TEMPERATURE DEPENDENCE OF
CRACK WAKE BRIDGING STRESSES IN
SILICON CARBIDE-REINFORCED ALUMINA COMPOSITES

By

G. Reza Sarrafi-Nour

A thesis submitted in conformity with the requirements for the degree of Doctor of Philosophy
Graduate Department of Metallurgy and Materials Science
University of Toronto

© Copyright by G. Reza Sarrafi-Nour (1999)
The author has granted a non-exclusive licence allowing the National Library of Canada to reproduce, loan, distribute or sell copies of this thesis in microform, paper or electronic formats.

The author retains ownership of the copyright in this thesis. Neither the thesis nor substantial extracts from it may be printed or otherwise reproduced without the author’s permission.
Temperature Dependence of Crack Wake Bridging Stresses in Silicon Carbide-Reinforced Alumina Composites

Ph.D., 1999
G. Reza Sarrafi-Nour
Department of Metallurgy and Materials Science
University of Toronto

ABSTRACT

R-curves for a SiC-platelet-reinforced alumina and a SiC-whisker-reinforced alumina were obtained between room temperature and 1400°C from chevron-notched flexure specimens. In response to evidence that R-curves alone provide inadequate characterization of the crack wake bridging processes due to their strong geometry dependence, a procedure based on a fracture mechanics weight function was developed to characterize the bridging stresses imposed on the crack surfaces by the bridging elements as a function of crack surface displacement. Finite element analysis was used to obtain the required weight function for crack surface tractions in specific chevron-notched flexure specimen geometries, and to find the crack opening displacement due to the applied load. An iterative scheme was devised to extract the bridging relation from the measured R-curves. The temperature dependence of the bridging stresses could then be studied through analysis of the R-curves measured for the composites.

Similar levels of bridging stresses were found in both of the composites. The bridging stresses in the SiC\textsubscript{pr}-Al\textsubscript{2}O\textsubscript{3} were found to be limited to a smaller crack surface displacement range, almost half of the value for the SiC\textsubscript{wr}-Al\textsubscript{2}O\textsubscript{3} material. These stresses were found to decay linearly with temperature in the whisker-reinforced material, while the limited non-linearity observed in the case of the platelet-reinforcement was attributed to the presence of an interfacial amorphous phase found in this composite. The unexpected similarity in the magnitude of the bridging stresses in the two composites is discussed in terms of the micromechanics of pullout bridging.

The crack tip toughness is found to increase with temperature in the whisker-reinforced material. In agreement with some previous studies of the composite, this toughening effect is associated with a microcrack zone formed ahead of the crack tip by the coalescence of diffusional cavities at the SiC\textsubscript{wr}/Al\textsubscript{2}O\textsubscript{3} interface. However, the results do not indicate any crack length dependence in this behavior or any influence from the microcrack zone on the bridging stresses.
# Table of Contents

<table>
<thead>
<tr>
<th>Chapter</th>
<th>Title</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Title Page</td>
<td>i</td>
</tr>
<tr>
<td></td>
<td>Abstract</td>
<td>ii</td>
</tr>
<tr>
<td></td>
<td>List of Figures</td>
<td>vi</td>
</tr>
<tr>
<td></td>
<td>List of Tables</td>
<td>x</td>
</tr>
<tr>
<td></td>
<td>Acknowledgements</td>
<td>xi</td>
</tr>
<tr>
<td>1.</td>
<td>Introduction</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td>1.0 Overview</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td>1.1 Objectives</td>
<td>3</td>
</tr>
<tr>
<td></td>
<td>1.2 Structure of the Thesis Document</td>
<td>3</td>
</tr>
<tr>
<td></td>
<td>References</td>
<td>4</td>
</tr>
<tr>
<td>2.</td>
<td>Background Overview</td>
<td>5</td>
</tr>
<tr>
<td></td>
<td>2.0 Introduction</td>
<td>5</td>
</tr>
<tr>
<td></td>
<td>2.1 R-curve Testing Methods</td>
<td>9</td>
</tr>
<tr>
<td></td>
<td>2.1.1 R-curve Measurement using Indentation Flaws</td>
<td>9</td>
</tr>
<tr>
<td></td>
<td>2.1.2 R-curve Measurement using Macro Flaws</td>
<td>12</td>
</tr>
<tr>
<td></td>
<td>2.1.2.1 Straight-Through Notched Specimens</td>
<td>12</td>
</tr>
<tr>
<td></td>
<td>2.1.2.2 Chevron-Notched Specimen</td>
<td>13</td>
</tr>
<tr>
<td></td>
<td>2.2 R-curve Behavior in Ceramic Systems: A Brief Review</td>
<td>16</td>
</tr>
<tr>
<td></td>
<td>2.2.1 Crack Face Bridging Mechanisms and Micromechanics</td>
<td>21</td>
</tr>
<tr>
<td></td>
<td>2.2.2 Temperature Dependence of R-curve Behavior in Ceramics</td>
<td>24</td>
</tr>
<tr>
<td></td>
<td>References</td>
<td>29</td>
</tr>
<tr>
<td>3.</td>
<td>Application of the Weight Function Method to the Analysis of the R-curve from a Chevron-Notched Specimen</td>
<td>35</td>
</tr>
<tr>
<td></td>
<td>3.0 Introduction</td>
<td>35</td>
</tr>
<tr>
<td></td>
<td>3.1 R-curve Response Due to Crack Wake Bridging Stresses</td>
<td>36</td>
</tr>
<tr>
<td></td>
<td>3.2 Analysis of the Stress Intensity Factor Due to Bridging Stresses for the Crack in a Chevron Notch</td>
<td>37</td>
</tr>
<tr>
<td></td>
<td>3.2.1 Application of the Weight Function for an Internal Crack in an Infinite Body</td>
<td>37</td>
</tr>
</tbody>
</table>
3.2.2 Slice Synthesis of the Crack Wake using the Weight Function for a
Through-Thickness Crack 39
3.2.3 Weight Function for Crack Surface Traction by Finite Element
Modeling 41
  3.2.3.1 Finite Element Modeling and Calculations 42
  3.2.3.2 Stress Intensity Factor and Weight Function Calculation 42
3.3 Calculation of the R-curve in a Chevron-Notched Specimen 46
  3.3.1 Evaluation of the R-curve Arising from Bridging Stresses 46
  3.3.2 Solution of the Integral Equation Arising from Crack Opening
    Displacements 47
  3.3.3 Examples of R-curves Due to an Exponential Bridging Relation 49
3.4 Determination of the Bridging Stress Distribution from the R-curve 52
  3.4.1 Determination of the Parameters from a Known Bridging Relation 52
  3.4.2 Determination of an Unknown Bridging Relation 52
3.5 Summary 54
3.6 Remarks on the Evaluation of the Toughness by Chevron-Notched Specimens
  in the Presence of R-curve Behavior 55
References 56

4. Experimental Procedure 59
4.0 Introduction 59
4.1 Materials, Processing and Fabrication 59
  4.1.1 SiC-Whisker-Reinforced Alumina 59
  4.1.2 SiC-Platelet-Reinforced Alumina 60
  4.1.3 Specimen Preparation 62
4.2 Fracture Tests 63
  4.2.1 Setup for High Temperature Fracture Test in Air 63
  4.2.2 Setup for High Temperature Fracture Test under Vacuum 64
  4.2.3 Measurement of Specimen Compliance during Fracture Test 64
  4.2.4 Testing Procedure 67
4.3 Remarks on Compliance Measurement at High Temperatures 69
References 70

5. Results 72
5.1 Calculation of the R-curves 72
5.2 Elevated Temperature Elastic Modulus 75
5.3 Deconvolution of Ko and Bridging Relation from the R-curve 76
LIST OF FIGURES

Fig. 1.1: Schematic representation of the crack wake bridging process and the associated bridging stresses in the crack wake region. 1

Fig. 2.1: Crack instability criterion in an ideal brittle behavior. 6

Fig. 2.2: Crack instability condition in a brittle solid with crack length dependence of the fracture toughness. 7

Fig. 2.3: Schematic of a penny/lateral crack system produced from a Vicker's indentation: cross-sectional view (left) and top view (right). 8

Fig. 2.4: Schematic of a fracture plane in a chevron-notched specimen. 14

Fig. 3.1: Comparison of bridging stress intensity factor arising from a constant traction on the wake of the crack in a chevron-notched specimen calculated using different methods. 39

Fig. 3.2: Schematic of the crack plane in a chevron-notched specimen and the slice representation of the crack wake. The shaded area on the crack plane represents the bridging interaction for one slice and the variable ζ defines the local position of the notch depth for each individual slice in the model. 40

Fig. 3.3: Finite element mesh used for the analysis of the chevron-notched flexure specimen in this work. 42

Fig. 3.4: Typical distributions of the weight function along the crack front in the chevron-notched specimen as a function of position, z, for a0/W=0.42 and a/W=0.7. (Reprinted from Engineering Fracture Mechanics, Vol. 59, G. R. Sarrafi-Nour, T. W. Coyle and T. Fett, "A Weight Function for the Crack Surface Tractions in Chevron-Notched Specimens," pp. 439-445, 1998, with permission from Elsevier Science.) 44

Fig. 3.5: Weight function for the crack surface tractions in chevron-notched specimen with a0/W=0.42. Symbols are finite element results and solid lines are the plots of Eq. 3.12 for the same a/W. Dotted line is the weight function for an edge crack in a finite plate at a/W=0.9. (Reprinted from Engineering Fracture Mechanics, Vol. 59, G. R. Sarrafi-Nour, T. W. Coyle and T. Fett, "A Weight Function for the Crack Surface Tractions in Chevron-Notched Specimens," pp. 439-445, 1998, with permission from Elsevier Science.) 45

Fig. 3.6: Calculated R-curves resulting from an exponential bridging relation in the chevron-notched flexure bar; characteristic bridging stress and displacement values are shown on the plot. 49

Fig. 3.7: Examples of the distribution of the bridging stresses resulting from an exponential bridging relation, characteristic values σp=20 MPa and δp=0.25 μm, over the crack wake in a chevron-notched flexure specimen. 50

Fig. 3.8: Examples of different profiles for a chevron notch crack obtained during R-curve calculations; σp=20 MPa and δp=0.5 μm. 50
Fig. 3.9: Comparison of the R-curves evolving from the exponential bridging law in the chevron-notched flexure specimen (lower family of curves) and in the same specimen containing a straight-through crack (upper family of curves); $\sigma_c=40$ MPa.

Fig. 3.10: Representation of the tangency condition of the applied stress intensity factor (dotted lines) to the R-curve (solid line) for a material susceptible to crack wake bridging. The R-curve is calculated for the exponential bridging relation with $\sigma_c=20$ MPa, $\delta=0.75$ $\mu$m and $K_c=2.5$ MPa$\sqrt{m}$.

Fig. 4.1: Typical microstructure of the materials used in this work; a) SiC-whisker-reinforced alumina, and b) SiC-platelet-reinforced alumina. The relief appearance of the microstructure of the platelet-reinforced composite is due to selective polishing of the alumina matrix during final polishing stage by colloidal silica (Bar=20 $\mu$m).

Fig. 4.2: Schematic representation of the indirect compliance measurement method.

Fig. 4.3: Finite element verification of the indirect compliance measurement method for the chevron-notched specimen using Eq. 4.4. The chevron-notch geometry modeled has $W/B=1.25$, $L/W=8$, $a/W=0.32$, $a/W=1$ and is considered to be under four-point flexure load such that $S/W=8$ and $S_2/S_7=2$.

Fig. 4.4: Typical load-displacement curves obtained during the fracture tests on the 20vol.% SiC-platelet-reinforced alumina between room temperature and 1400°C.

Fig. 4.5: Typical load-displacement curves obtained during the fracture tests on the 33vol.% SiC-whisker-reinforced alumina between room temperature and 1400°C.

Fig. 5.1: Examples of the R-curves from the SiC-platelet-reinforced alumina tested at various temperatures in air.

Fig. 5.2: Examples of the R-curves from the SiC-whisker-reinforced alumina; a) typical R-curves at various temperatures, and b) at room temperature.

Fig. 5.3: Variation of the elastic modulus as a function of temperature in the SiC-platelet- and SiC-whisker-reinforced alumina. (Note that the results from the whisker-reinforced composite are $E''=E(1-\nu^2)$ and the error bar represents one standard deviation calculated based on the values from three specimens.)

Fig. 5.4: A typical fit to an experimental room temperature R-curve from the platelet-reinforced composite by using the procedure described in section 3.4.2.

Fig. 5.5: a) Distribution of the bridging stresses in the crack wake, and b) calculated crack opening profile due to the applied load without the effect of the bridging stresses (dashed line) and under the influence of the bridging stresses. The data in both of the plots relate to the fit results of Fig.5.4.

Fig. 5.6: The constitutive bridging relation obtained by combining the results shown in Fig. 5.5a and Fig. 5.5b. Each symbol represents the data from one of the crack length domains shown in Fig. 5.5b.

Fig. 5.7: Variation of the maximum value of bridging stress deconvoluted from the R-curves of the SiC-platelet-reinforced alumina at various temperatures. The error bar on the data from high temperature tests under vacuum shows the scatter of the results from different specimens about the average value.
Fig. 5.8: Variation of the maximum value of bridging stress deconvoluted from the R-curves of the SiC-whisker-reinforced alumina at various temperatures. The error bar on the data shows the scatter of the results from different specimens about the average value.

Fig. 5.9: Typical constitutive bridging relations obtained from the analysis of the R-curves of the SiC-platelet-reinforced alumina.

Fig. 5.10: Typical constitutive bridging relations obtained from the analysis of the R-curves of the SiC-whisker-reinforced alumina.

Fig. 5.11: Variation of the $K_v$ deconvoluted from the R-curves of the SiC-platelet-reinforced alumina at various temperatures. The error bar on the data from high temperature tests under vacuum shows the scatter of the results from different specimens about the average value.

Fig. 5.12: Variation of the $K_v$ deconvoluted from the R-curves of the SiC-whisker-reinforced alumina at various temperatures. The error bar on the data shows the scatter of the results from different specimens about the average value.

Fig. 6.1: R-curves measured on silicon nitride specimens at elevated temperatures and polymethyl metacrylate, PMMA, at room temperature for experimental verification of the compliance measurement. The crack length at which the minimum value of the geometric function occurs for the chevron-notched specimen is indicated by the straight dashed-double-dotted line, $Y_{max}$ on the plot.

Fig. 6.2: An example of the notch tip of the SENB specimen used for the determination of the fracture toughness of the SiC-platelet reinforced composite. (Bar=50 µm)

Fig. 6.3: Comparison of an experimentally measured R-curve for the SiC-whisker-reinforced alumina at room temperature under the four-point bending condition prior to crack instability with the calculated R-curves based on a maximum bridging stress of 25 MPa and the assumption of an exponential bridging relation with characteristic displacements of 0.125 and 0.25 µm.

Fig. 6.4: TEM observation of the amorphous phase associated with the presence of the SiC-platelets: left) bright field image and the selected area diffraction pattern centered on the phase in the triple junction between the SiC$_p$ and Al$_2$O$_3$ grains showing a diffuse central spot, and right) dark field image of the amorphous phase running through the matrix grain boundary adjacent to the silicon carbide platelet. The image was taken with the aperture placed to avoid any diffraction coming from the alumina grains.

Fig. 6.5: Examples of the SiC-platelet facets on the fracture surface of the composite; a) room temperature; at higher temperatures indications of tracks of the amorphous phase (Fig. 6.4) could be found on some of the platelets as shown in micrograph b) at 800°C, c) at 1000°C and d) at 1200°C. At 1400°C, micrograph (e), such features could not be found on the facets of the platelets. (Bar=5 µm)

Fig. 6.6: Examples of the fracture surface of the SiC-platelet-reinforced alumina from various test temperatures under vacuum; a) room temperature, b) 800°C, c) 1200°C and d) 1400°C. (Bar=100 µm)

Fig. 6.7: Arrhenius plot of the velocity of the longitudinal sound waves as a function of temperature in the SiC-platelet-reinforced composite.
Fig. 6.8: Roughness profilometry results from the fracture surface of the SiC-platelet-
reinforced alumina specimens broken at various temperatures. The zero value on
the abscissa corresponds to a fictitious plane describing the average of the
distribution of the point elevations measured.

Fig. 6.9: Examples of the whisker-pullout length in the SiC-whisker-reinforced alumina
observed on the surface of the chevron notch: a) room temperature, and b) at
1200°C. The specimen from 1200°C test was etched in dilute HF acid solution to
remove the oxide glass layer from the surface. (Bar=1 μm)

Fig. 6.10: Examples of the microcracks formed within the damage zone in the vicinity of the
fracture surface in the SiC-whisker-reinforced alumina. Micrographs a-c and d-f
are from specimens broken at 1200°C and 1300°C, respectively. Marked area in
micrograph (b) is magnified in micrograph (c) and shows the coalescence of
cavities initiated at the SiC-Al₂O₃ interface to form the microcrack. Note that the
microcracks/cavities are always associated with the SiC-Al₂O₃ interface. SiC-
whiskers appear as the bright phase, arrows indicate the microcracks and FS
designates the fracture surface.

Fig. 6.11: Comparison between high temperature toughness results from Han et al [28] with
the $K_\text{c}$ values obtained from the analysis of the R-curves in this work. The error
bars on the data from this work represent the scatter of the results from different
specimens, while those of the results from [28] are one standard deviation quoted
by the authors in Table 2 of Ref. [28].

Fig. 6.12: Healing of a crack branch by the oxidation products in the SiC-whisker-reinforced
composite broken at 1200°C in air. The area defined by the square in micrograph
(a) is magnified in micrograph (b). Arrows are used to mark the crack trace for
better visibility. The images were obtained using field emission electron
microscope at an accelerating voltage of 1 kV. (Bar=5 μm)

Fig. 6.13: Comparison of the maximum bridging stress in the SiC-platelet-reinforced
alumina and the SiC-whisker-reinforced alumina as a function of temperature. The
error bars on the data points represent the scatter of the results from different
specimen about the average value. The data from the platelet-reinforced material
is the average of the results from tests both under vacuum and in air. The
maximum bridging stress value from the whisker-reinforced material at room
temperature (solid circle) is an estimated value based on the linear extrapolation of
high temperature results and the analysis of section 6.1.2.2.

Fig. 6.14: Variation of the normalized maximum bridging stress with temperature for the
SiC-reinforced alumina composites from this work and for monolithic aluminas
from Hay and White [17].

Fig. 6.15: Comparison of the initial value of the fracture toughness of the SiC-platelet-
reinforced alumina and SiC-whisker-reinforced alumina as a function of
temperature. The error bar on the data represents the scatter of the results from
different specimen about the average value. The data from the platelet-reinforced
material is the average of the results from tests both under the vacuum and in air.
LIST OF TABLES

Table 4.1: Properties of the SiC₆₆-reinforced alumina .......................... Page 60
Table 4.2a: Properties of the alumina powder ........................................ 61
Table 4.2b: Properties of SiC platelet powder .......................................... 61
ACKNOWLEDGEMENTS

I would like to first acknowledge Prof. Thomas W. Coyle for his contributions to and his special interest in my work. I had the opportunity to enjoy his trust during the course of my Ph.D. work and to develop myself both personally and intellectually. I also extend my sincere appreciation to Dr. Theo Fett, Institute for Material Research II (IMF-II), Research Center Karlsruhe/Germany, for his mentorship and supervision during my stay at IMF-II and for countless stimulating discussions, encouragement and attentive comments within the past two years and before.

I owe my most sincere thanks to many individuals who helped me with my work on various occasions and would like to mention them all. To those whom I may have forgotten momentarily while writing this acknowledgements section: I am also thankful to each and everyone of you!

I thank Connie Barry from Ceramics Processing Lab at McMaster University for hot-pressing and assistance with processing of the platelet-reinforced composites; she is best described for her challenging job in Rosarau Hamsu's thesis: "Connie Barry somehow managed to keep everything in the ceramics lab working, even the hot press". Arnold Hansma and Mike A. Mackay, AHCS Ltd, Burlington, Ontario, I would like to thank for giving me the privilege to use their surface grinding equipment on many occasions for machining flexure specimens.

I am grateful to Fred Neub and Sal Bocca for their assistance with electron microscopy throughout the course of my residency at the Department of Metallurgy and Materials Science, University of Toronto.

Eric Close, Keith Porter and John Ford from the machine shop in the Chemical Engineering Department: I thank them all for making various parts that I needed in my work, especially for their excellent job in making the high temperature mechanical testing module, and for their friendly technical assistance on various occasions.

The experimental results of this work would have not been completed without my unconstrained access to the Instron machine at the Center for Biomaterials, University of Toronto. I would like to thank Prof. Bob Pilliar and Chris Pereira for giving me the privilege to use their Instron.

The influence of my short stay at the Institute for Material Research II, Research Center Karlsruhe/Germany is apparent in many locations throughout this thesis document. I am grateful to Prof. Dietrich Munz for providing the guest researcher position at his institute and allowing me the privilege of using the equipment and facilities there and to the German Academic Exchange Service, DAAD, for supporting my visit. I would also like to thank the following fellow researchers at IMF II for their help and hospitality during my stay: Herbert Schneider for providing lab space and assistance with fracture experiments; Stefan Müller for the high temperature elastic modulus measurement of the SiC-platelet-reinforced composite; Gabrielle Rizzi for her help in using the ABAQUS finite element package, and Holger Kiewel, Rehiner Weiss and Robert Kühner for numerous useful discussions.
I am grateful to Dr. Choll K. Jun, Vice President, Greenleaf Corp., Saegertown PA, for generously donating the SiC-whisker-reinforced alumina and silicon nitride flexure specimens used in my research. In the same vein, I would like to thank Kevin Mawn, Materials Engineer at Greenleaf Corp., for his friendly treatment on numerous occasions and for the excellent quality of the large number of the specimens provided to me.

My friends at the University of Toronto were always a valuable source of encouragement and assistance. I would like to thank Vahid Safavi-Ardebili for his help, time and advice in the design and construction of various parts used in this research; Sasan Raghibzadeh for his useful comments on the optimization procedures and Masoud Shams for always being there eagerly listening to my descriptions of my work and giving useful comments. My fellow group members, Colin Ryan, Audrey Yakimov and Zhido Zheng, I thank them all for bearing with me and for their help throughout all these years.

I am thankful to my brothers and sisters and their families for their invaluable support and encouragement throughout my education.

To my wife Mitra, words can be only a poor gesture of my gratitude for her true love, incredible patience and absolute support throughout every moment of my Ph.D. work, which would have never been completed without her commitment.

I would like to dedicate this work to my wife Mitra and to the memories of my mother, whose unwavering support and encouragement in 24 years of my life paved the path to this conclusion and made this degree possible.
Chapter 1

Introduction

1.0 Overview

The importance of crack-wake-zone processes in toughening of ceramic materials has been known for almost two decades. When present, such processes influence the stress intensity factor at the tip of the crack by shielding the crack tip from the remotely applied stress field and, thus, toughening is imparted. Such crack tip shielding contributions usually vary with the crack length, leading to the rising crack-growth-resistance or R-curve behavior observed in some ceramic materials. The R-curves are important in demonstrating the presence of crack wake toughening mechanisms and establishing the crack instability criterion. Their utility, however, is limited to the specific crack, specimen, and loading geometry for which they were measured.

One of the toughening mechanisms with such characteristics pertinent to many ceramic materials is the crack wake bridging mechanism. When ligaments consisting of large grains or elongated second phase particles remain intact in the wake of an advancing crack, bridging from one crack wall to the other, they can exert closure tractions, \( \sigma_{\text{c}} \), on the crack walls which shield the crack tip from the applied stress intensity factor. This is schematically shown in Fig. 1.1. Dependent on their size and type, such bridges can survive crack opening displacements of several micrometers or more in ceramic composites reinforced with a discontinuous second phase or in monolithic ceramics containing large grains.

![Diagram of the crack wake bridging process](image)

Fig. 1.1: Schematic representation of the crack wake bridging process and the associated bridging stresses in the crack wake region.
Although a large amount of scientific endeavor has been devoted in the past to the characterization of the bridging toughening mechanism through the measurement of the resulting R-curve behavior, such efforts did not lead to satisfactory results. This is predominantly because the magnitude of the toughening and its variation with the crack length were found to be strongly dependent on geometrical parameters associated with the crack/specimen geometry used. A prominent example of this is the long-crack and short-crack R-curve debate; the magnitude of toughening obtained in long-crack R-curve studies was never found in the short crack domain.

A more useful characterization of the bridging toughening mechanism can be accomplished by the characterization of the bridging stresses themselves. The bridging stress varies with the crack opening profile, $\delta$, and, therefore, characterization of the bridging stress as a function of crack opening displacement, $\sigma_{br} = f(\delta)$, is desirable. In fact, the crack opening displacement is one of the hidden variables in an R-curve. The characterization of this bridging relation is expected to yield a description of the bridging toughening mechanism that is characteristic of the material, independent of crack, specimen, and loading type. The magnitude of the toughening for an arbitrary crack due to the bridging mechanism could then be determined when the toughness at the tip of the crack (a material characteristic value) and a fracture mechanics description of the cracked body are available.

The characterization of the bridging stress vs. crack opening displacement in a material may be carried out either directly or indirectly. The direct method implies that tensile tests be carried out on broken specimens held together only by the bridging ligaments on the crack surface [1]. The indirect method implies that the distribution of the bridging stresses be deconvoluted from measurement of a different behavior, e.g., deconvolution of the bridging stress from the crack opening profile of a crack containing bridges [2,3] or from the R-curve measurement results [4]. Each of these methods has been utilized on ceramics recently, although only in a few cases [1-7].

The general interest in ceramics for structural applications is mostly focussed on load bearing applications involving elevated temperatures. Therefore, the characterization of the crack wake bridging process at such temperatures would be of interest in the fields of both material design and development, and reliability of ceramic components. Although fracture toughness results on structural ceramics at high temperatures are now available in the literature, information on the crack wake bridging stresses at elevated temperature are very scarce in the literature. This reflects the difficulties associated with such characterizations and calls for contributions to this area in the field of fracture mechanics of ceramics.

Various discontinuous reinforcements including particulates, whiskers, and platelets, have been utilized to produce ceramic composites for structural applications. Amongst these, whiskers have been found the most promising type of reinforcement; however, materials containing whiskers are at the same time hard to process. Usually both toughness and strength improvements can be achieved by introducing whiskers into the matrix. In this line, many practices have been based on using SiC-whiskers due to their
unique high strength and high modulus. A commercial example for such composites is the well-known and established SiC-whisker-reinforced alumina cutting tools used for machining nickel-based super alloys.

An alternative reinforcement to SiC-whiskers with similar toughening effects could be SiC-platelets. In the case of platelet-reinforcement, part of the strength seems to be sacrificed for the toughness as the larger-size platelets tend to reduce the fracture strength of the composite. However, the ease of processing combined with toughening effects similar to whiskers may make platelets an alternative for less demanding structural applications or a partial substitute for whiskers. This would in turn necessitate further characterization of toughening contributions due to these types of reinforcements.

1.1 Objectives

This work intends to pursue two main objectives. The first objective is to extend the fracture mechanics weight function methodology to the analysis of the R-curves from chevron-notched specimens in order to utilize such R-curves to obtain the bridging constitutive relation through an iterative scheme. This will then furnish the basis for the second objective of this work: to study the bridging stresses and their temperature dependence in a SiC-platelet- and a SiC-whisker-reinforced alumina composite.

1.2 Structure of the Thesis Document

The thesis is organized as follows:

- Chapter 2, Background Overview, introduces the concept of R-curves in brittle materials and common methods of their measurement, and provides a brief review of R-curve behavior and the crack wake bridging toughening mechanism in ceramics.

- Chapter 3, Application of the Weight Function Method to the Analysis of the R-curve from a Chevron-Notched Specimen, focuses on the analysis of the stress intensity factor due to crack wake tractions in a chevron-notched specimen. Previous efforts in analyzing the stress intensity factor arising from the crack wake tractions in this geometry, based on the use of the weight function for a crack in an infinite body, are compared with two new analyses, a finite element analysis and a slice synthesis approach. This leads to the numerical derivation of the first weight function for crack surface tractions in a chevron-notched specimen using finite element analysis. This weight function is then used to examine the R-curve arising in this specimen geometry from crack wake bridging stresses. An iterative analysis scheme is sketched-out to allow deconvolution of the bridging stresses from an experimentally determined R-curve using the chevron-notched specimen modeled.

- Chapter 4, Experimental Procedure, describes the material processing, specimen preparation and testing procedures employed in this work. This includes processing of the SiC-platelet-reinforced alumina, preparation of the chevron-notched flexure specimens from the SiC-platelet-reinforced alumina and SiC-whisker-reinforced alumina composites, controlled fracture tests at room as well as elevated temperature, and the application of the indirect compliance measurement method and its finite
element verification in the case of the chevron-notched specimens. The culmination of this chapter will be the load-displacement curves obtained during controlled fracture tests at various temperatures up to 1400°C on the chevron-notched flexure specimens of the composites.

Chapter 5, *Results*, begins with the calculation of the R-curves from the load-displacement curves obtained in Chapter 4 via the compliance method, through a compliance-crack length relation obtained for the chevron-notched flexure specimen by finite element analysis. The R-curves are then submitted to the iterative analysis procedure described in Chapter 3. This results in the deconvolution of the constitutive bridging relations and the initial value of the fracture toughness at different temperatures, based on the implicit assumption of pullout bridging.

Chapter 6, *Discussion*, first examines the quality of the results obtained from the analysis of the R-curves in Chapter 5 by conducting independent experiments as well as by comparison of some of the results with values available in the literature. The variation of the crack tip toughness and bridging stresses as a function of temperature in each composite are then discussed separately based on microscopic examinations of the materials and the results from supplementary experiments. Finally, the temperature dependence of the bridging stresses and crack tip toughness are compared between the two composites.

Chapter 7, *Summary and Conclusions*, provides a brief review of Chapters 3 through 6 and presents the conclusions drawn from this work.

**References**

Chapter 2

Back Ground Overview

2.0 Introduction

For an ideal brittle material, the application of linear elastic fracture mechanics, LEFM, allows simple characterization of the stress field in the vicinity of the crack tip through the stress intensity factor, \( K \). The loading condition for crack advance in such a medium can be described as one which results in a stress intensity factor equal or greater than a material characteristic value, the so-called critical stress intensity factor, \( K_c \), or fracture toughness. Since crack propagation under mode I (opening mode) loading condition is of prime interest for ceramics, this critical value usually refers to fracture toughness under this mode or \( K_c \). (Our discussions in this work shall be limited to the mode I for the stress intensity factor, and therefore, for convenience the index “I” will be omitted hereafter.) The necessary condition for the crack advance in an ideal brittle material can thus be written, following the fracture mechanics formalism, as:

\[
K_{\text{appl}}(a) = \sigma_o Y \sqrt{a} = K_c \quad (2.1)
\]

where \( \sigma_o \) is a characteristic stress value describing the remotely applied load, \( Y \) is a geometric function describing the effect of the load/crack geometry on the stress intensity factor at the tip of a crack with the length equal to \( a \). Although Eq. 2.1 describes a necessary condition for crack propagation, it is insufficient to describe if the propagation would be catastrophic (unstable, critical or, equivalently, lead immediately to failure) or controlled. The stability requirement may be expressed by the derivative of the applied stress intensity factor relative to crack length [1]:

\[
\frac{dK}{da} > \frac{dK_c}{da} \Rightarrow \text{unstable crack propagation} \\
\frac{dK}{da} \leq \frac{dK_c}{da} \Rightarrow \text{stable crack propagation} \quad (2.2)
\]

Obviously, for an ideal brittle material with \( K_c = \sigma_f(a) \) the right side of the inequalities in Eq. 2.2 will be zero. This condition is shown schematically in Fig. 2.1. In this figure, a brittle material containing a preexisting crack of length \( a_o \) is considered to be under an applied load. The stress intensity factor for the crack/loading configuration, \( K_{\text{appl}} \), is assumed to follow the curves shown for different magnitudes of the applied load such that \( \sigma_f > \sigma_2 > \sigma_3 > \sigma_i \). In this crack system, catastrophic crack propagation would occur
when the applied stress intensity factor intersects $K_{IC}$, presented as a straight bold solid line in the figure, at crack length $a_o$.

![Stress Intensity Factor vs Crack Length](image)

Fig. 2.1: Crack instability condition in an ideal brittle behavior.

Many ceramic materials, both monolithic and composite, e.g., coarse-grained alumina, toughened ZrO$_2$, $\beta$-Si$_3$N$_4$, SiC$_w$-Alumina, SiC$_w$-Si$_3$N$_4$, fiber-reinforced ceramics, and similar materials are susceptible to some deviation from the ideal brittle behavior. This deviation is manifested as an apparent increase in the fracture toughness as a function of crack advance due to some operative toughening mechanisms. A careful examination of such behavior using fracture mechanics reveals that the fracture resistance of the material at the crack tip is still single-valued; however, the stress intensity factor at the crack tip is influenced by an additional component that appears on the right hand side of Eq. 2.1. This new interesting stress intensity factor component involves some complexities: it depends both on the microstructure of the material (of interest to material scientists) and on the geometry of the cracked body (of interest to fracture mechanics specialists). Superposability of stress intensity factors acting under the same mode allows us to rewrite Eq. 2.1 in a more general form to include the new stress intensity factor term and describe the necessary condition for crack advance:

$$K_{IC} = K_{tip} = K_{appl} + K_w$$  \hspace{1cm} (2.3a)

where $K_w$ reflects some interactions between the crack and the microstructure (or a specific toughening mechanism) of the material within the crack wake region. The $K_w$ term usually has a negative value,
implying closure action, and thus reduces the total stress intensity factor at the crack tip, $K_{ip}$. If $K_w$ were included in the left-hand side of Eq. 2.3a, i.e.,

$$K_{appl} = K_R = K_{ip} + K_w$$

(2.3b)

and the same crack configuration as shown in Fig. 2.1 were considered, we would arrive at an interesting situation. This is shown schematically in Fig. 2.2.

![Fig. 2.2: Crack instability condition in a brittle solid with crack length dependence of the fracture toughness.](image)

Right at the onset of crack propagation $K_w$ in Eq. 2.3b is equal to zero and $K_{ip}$ reaches the fracture resistance of the material ahead of the crack tip, $K_w$ i.e., $K_{appl} = K_R(a_o) = K_o$. Here, $K_R$ represents the fracture resistance of the material. It can be readily seen that the same crack configuration as was considered earlier would now require some stable crack propagation before reaching instability (which occurs at the tangency point between the applied stress intensity and fracture resistance curve). The stable crack growth is a direct consequence of the presence of $K_w$. Within the crack length regime bounded by $a_o$ and $a_f$ the brittle material presented in Fig. 2.2 is referred to as flaw-tolerant. It was the observation of evidence of such behavior in some ceramic systems that motivated many researchers to conduct detailed studies of this behavior between the late 80's and early 90's. A major idea behind most of these works was to tackle the problem of strength scatter in ceramics by tailoring flaw-tolerant microstructures.
The term $K_w$ can be considered to have caused crack tip shielding, because the stress intensity factor at the crack tip is smaller than the remotely applied value, $K_{appl} = K_R$, by the amount $K_w$. While for an external observer the stress intensity factor to cause further crack extension will be increasing, the observer at the crack tip will simultaneously measure only $K_w = K_{IC}$, the toughness of the material in the absence of any contributions from the crack wake.

Based on its global effect, the behavior described is known as Rising Crack Growth Resistance or, in short, R-curve behavior. Although here the letter "R" refers to the material fracture resistance energy from the energy approach to fracture mechanics, most of literature dealing with R-curve behavior in ceramics, and also the current work, employ the stress intensity factor concept. This is primarily due to the convenience that stress intensity factors of the same mode arising from different loading sources can be summed linearly. This rule was already employed in writing Eq. 2.3. However, to do the same summation in terms of energy, cross terms must be introduced [1]; the displacement resulting from each loading source contributes to the work done by the other loading sources.

In ceramic materials with R-curve behavior, $K_R$ is governed by the contribution of the crack wake to the stress intensity factor at the crack tip. Our current knowledge in the field of fracture mechanics of ceramics indicates that this contribution results from closure forces in the crack wake region. Two different sources have so far been identified to give rise to closure forces in the crack wake region. In one class of materials closure tractions are formed as a result of incomplete separation of the crack faces in the crack wake region due to the presence of unbroken ligaments such as large grains, fibers, or elongated second phase particles which are usually referred to as bridges [2,3]. The associated traction in the wake region is, therefore, referred to as a bridging stress. In the other class of materials the closure force in the crack wake is formed as a result of a dilation zone, which engulfs both the crack tip and crack wake, like transformation/microcracking in zirconia [4] or ferroelectric-domain switching [5] in piezo- and ferroelectric ceramics. Among the two major categories, the former is relevant to a wider range of ceramic materials, the so-called non-transformation ceramics, which include both monolithic and reinforced ceramic composites. It is the latter class of materials that will be focused on in this work. Due to the essential role of the crack wake and the microstructure in the R-curve behavior, the index of $K_R$ has sometimes been replaced by "w" or "µ" to reflect the wake or microstructural component of the stress intensity factor, respectively. A more appropriate index for this term, based on the objectives of this work, may be "br" to explicitly reflect the source of this stress intensity factor term; in this case the bridging interactions.

The source of the deviation from ideal brittle fracture behavior, as shown schematically in Fig. 2.2, for materials with bridging effects is now more obvious. One of the basic assumptions in LEFM is that the wake of the crack is traction free; the remotely applied stress would then be enough to characterize the stress intensity factor at the crack tip. Although the presence of bridges in the crack wake violates LEFM
assumptions, LEFM may still be utilized by considering the bridges as another crack loading system, and taking advantage of the superposition principle for the stress intensity factors.

The R-curve behavior has a positive influence on many mechanical properties of ceramic materials. For any mechanical property related to the response of the ceramic material to crack propagation, improvements can be expected if R-curve behavior is present. Some of the mechanical properties of ceramics that have been shown to improve as a result of the R-curve behavior include:

- strength of the material containing natural flaws
- thermal shock resistance
- cyclic fatigue
- failure caused by subcritical crack growth
- creep crack growth resistance

### 2.1 R-curve Testing Methods

The R-curve measurement on a material can be accomplished based on Eq. 2.1. By monitoring the crack length and the magnitude of the applied load during the course of crack propagation the stress intensity factor at each crack length can be obtained, assuming that a stress intensity solution for the crack and loading geometry is known. A stable crack growth condition is a prerequisite for such tests. Due to combined effects of low toughness and high modulus, a stable/control crack growth condition may not be easily fulfilled in a ceramic material. In addition, the requirement to measure the crack length may be difficult as a result of the crack size, the crack shape, or the testing environment.

R-curve behavior of ceramic materials has been characterized using two major types of techniques, with the essential difference between the two originating from the crack size domain used for the measurements. These are:

- R-curve measurement using macro flaws (cracks)
- R-curve measurement using micro (indentation or natural) flaws

#### 2.1.1 R-curve Measurement using Indentation Flaws

Like any other crack configuration, cracks formed around a hardness indentation, usually Vicker's or Knoop, in brittle materials may be used for toughness measurement and to study the R-curve behavior. As a consequence, of the localized elastic/plastic deformation under a sharp contact, a localized residual stress field is produced around the indentation [6]. Within this field a system of (half) penny-like cracks evolves to an equilibrium length, \( c_e \), along the impression diagonals (and lateral cracks develop on a plane near the impression base), as shown schematically in Fig. 2.3.
This arrest crack length is defined by the counterbalancing of the stress intensity factor arising from the residual contact, $K_{res}$, and the toughness of the material, $K_R$ (No specific reference is being made about the shape of the R-curve, e.g., flat or rising. $K_R$ can be simply considered as the toughness of the material for a crack length $c_o$) [7]:

$$K_{res} = \frac{\chi P}{c_o^{3/2}} = K_R$$

where $P$ is the indentation load and $\chi$ is the residual stress parameter describing the elastic/plastic response of the material upon deformation by the indenter. $\chi$ is usually expressed in terms of elastic modulus, $E$, and hardness of the material, $H$, and a proportionality constant, $\xi$, [8]:

$$\chi = \xi f(E/H)$$

Lack of a reliable physical model has resulted in the estimation of $\xi$ or $\chi$ using toughness values obtained for the material from other techniques, e.g., using long-crack toughness measurements [7]. Eq. 2.4 could be used to measure the R-curve by varying the indentation load and measuring the arrested crack length.

The experimental simplicity of strength measurements on specimens indented using various indentation loads has made the toughness and/or R-curve evaluation based on such measurements very attractive for a number of years. When an indented specimen is loaded to stress $\sigma$, under bending conditions for instance, the superposition of the applied and the residual stress intensity factors results in the total stress intensity factor, $K_{tot}$ [9]:
where $\psi$ is the geometric function for the crack system under study. At the critical condition, i.e., when failure occurs at $\sigma = \sigma_m$, the total stress intensity factor would be equal to the material fracture toughness, $K_{tot} = K_R$. Under such circumstance the decreasing residual and increasing applied stress intensity factors result in a certain amount of stable crack growth preceding failure, which occurs under the applied stress $\sigma_m$ at the crack instability length $c_m$. The magnitude of $K_R$ at the instability point can be written as [9,10]:

$$K_{R|c_m} = 4\left(\frac{\psi \chi^{1/3}}{3}\right)^{3/4}[\sigma_m P^{1/3}]^{3/4}$$ (2.7)

As can be seen from Eq. 2.7, a simple plot of the bending strength vs. indentation load on a logarithmic scale must yield a straight line with a slope equal to $-1/3$, if $K_R$ is constant. A deviation toward shallower slopes is taken as an indication of $R$-curve behavior. The $R$-curve can then be constructed from the strength data, assuming constant $\chi$ and $\psi$, by an appropriate derivation of $\sigma_m=f(P)$ using Eq. 2.6 and Eq. 2.7 and following an iterative scheme to compare the calculated indentation strength with the measured values [11] or measuring the crack instability length on dummy indents [12]. In another method of construction of the $R$-curve from the indentation strength data [13], a family of the total stress intensity factor versus crack length at each indentation load and the corresponding strength are plotted first. An envelope of tangency points is then graphically fitted to these curves to yield the $R$-curve. Hsueh and Becher [14] recently developed an analytic version of the tangency-envelope method. All such methods, however, still strongly rely on constant values for the residual stress parameter and crack shape factor. It must be realized that analyzing the indentation strength data to deconvolute the $R$-curve can lead to misleading results if $\chi$ (or $\xi$) depends on the indentation load, and the crack profile, and thus $\psi$, varies with crack extension. Although the as-indented radial crack possesses a half-penny (semi-circular) shape, this changes toward the semi-elliptical geometry as the crack extends upon loading [15]. Consequently, post-fracture determination of the crack shape at failure becomes vital in order to correctly determine $\psi$ in Eq. 2.7.

The stable crack extension regime, $c_o \leq c \leq c_m$, during loading the indented specimens also provides the possibility for in situ measurement of the crack length and thus determination of the $R$-curve. This can be accomplished by rearranging Eq. 2.6 [10]:

$$\frac{\sigma c}{P} = \frac{K_R}{P \psi} c^{3/2} - \frac{\chi}{\psi}$$ (2.8)
The calculation of $K_R$ from Eq. 2.8 becomes straightforward if $\chi$ and $\psi$ are constant. If $\psi$ varies during crack extension, then $\psi(c)$ has to be independently determined from crack contour development for a given indentation load $P$ to allow for the R-curve calculation. However, if $\chi$, $\psi$ and $K_R$ are all varying with the indentation load and crack length, the scheme required for the R-curve evaluation will be more difficult. In such a case, the crack extension curves from different indentation loads have to be considered as well.

A study on coarse-grained alumina by Bleise and Steinbrech [16] showed recently that a flat R-curve could be calculated from a set of indentation bending strength results combined with measurements of the crack length at instability from dummy indents even though the slope of the strength vs. indentation load data indicated an obvious deviation from $-1/3$. Similar results were also obtained in a study using the indentation strength method on zirconia [17]. In addition, some study has indicated that both $\chi$ and $\psi$ could be influenced by the environment [18]. Such results unequivocally demonstrate the need to determine the parameters $\chi$ and $\psi$ for each individual crack extension experiment. To overcome the difficulties and uncertainties associated with the residual stress parameter $\chi$, removal of the residual stress field by polishing the surface layer [19] or annealing the indented specimen [20] have been practiced in the literature. A method has also been suggested recently by Stech and Rödel [21] to calculate the R-curve for the indentation cracks without using any calibration parameters.

In general, although the indentation method is easy to employ, a reliable deconvolution of the R-curve from the experimental data strongly depends on correct determination of $\chi$ and $\psi$ values at each indentation load.

### 2.1.2 R-curve Measurement using Macro Flaws

#### 2.1.2.1 Straight-Through Notched Specimens

In methods using a macro flaw, the crack is grown from a notch and the stress intensity factor is measured during stable propagation of the crack. The crack extension length, over which the R-curve is monitored, spans between a few hundred micrometers to several millimeters. The R-curves obtained using such flaws are usually referred to as long-crack R-curves to distinguish them from the R-curves obtained from the short indentation or natural cracks. The notch is usually cut into the specimen such that it covers the thickness of the specimen to a certain depth, resulting in a through-thickness or straight-through crack geometry. Typical examples for test geometries with straight through-thickness cracks are Double-Cantilever Beam (DCB) specimen, Double-Torsion (DT) specimen, Compact Tension (CT) specimen and Single-Edge Notch Beam/Bend (SENB) specimen. The detailed description of such specimens and their geometric functions under different loading conditions can be found in any stress intensity factor handbook, e.g., Tada's handbook [22]. The choice of the specimen for the R-curve study is usually made
based upon the test objectives and testing environment, e.g., room temperature vs. high temperature. DCB, DT and CT specimens are mostly used for room temperature tests, with the DCB geometry being the most popular one due to the ease of achieving stable crack growth in this geometry. Under room temperature testing conditions the crack length during stable crack growth may conveniently be measured from traces of the crack front on the surface of the specimen in situ using a traveling microscope or be recorded by a video camera for later analysis. Although such crack length measurements can also be carried out at elevated temperature [23], the required facilities may not be accessible in many research laboratories. Therefore, especially in tests involving high temperatures, the crack length is frequently calculated from the compliance of the specimen (compliance, $C$, is defined as the displacement of the loading point divided by the load: multiplying the compliance by specimen thickness, $B$, and Young's Modulus results in the normalized compliance, $\lambda$) using the appropriate elastic compliance versus crack length solutions [22]. For this purpose, both the load and the load-point displacement, or crack mouth opening displacement, must be monitored during the fracture test to allow for the compliance measurement. For high temperature tests on ceramics, a flexure loading condition is easier to devise and, therefore, SENB specimens are popular for such test conditions. However, stable crack growth in this geometry requires a very narrow initial notch (notch width $\leq 50 \mu m$) and a very high stiffness load train, which usually can only be achieved through using a crack-stabilizing fixture [24]. Another major difficulty with testing ceramics is producing a sharp crack from the notch before the maximum load is achieved in the fracture test. This can be tackled by precracking the specimen using fatigue loading, e.g., following the method in [25], or loading the notched or indented specimens in a pre-cracking fixture [26]: however, the success rate in such experiments strongly depends on the user's experience with the material under study.

2.1.2.2 Chevron-Notched Specimen

An easier solution to the problem of forming a sharp crack prior to maximum load and obtaining crack growth stability is to use chevron-notched specimens [27]. In this type of specimen a triangular notch is cut into the thickness of the specimen such that the base of the triangle is formed either by the end of the specimen in the direction of the crack growth or by a fictive line passing through the thickness of the specimen. The fracture plane and the geometrical parameters describing the notch configuration in a chevron-notched specimen are shown in Fig. 2.4.

The high stress concentration at the tip of the chevron notch can result in crack nucleation at fairly low loads, removing the requirement for precracking. In addition, crack stability is improved in a chevron-notched specimen compared to a through-thickness crack geometry due to increasing crack front width with increasing crack length. A comprehensive review of chevron-notched specimens has been provided by Newman [28].
Fig. 2.4: Schematic of a fracture plane in a chevron-notched specimen.

Chevron-notched specimens have gained popularity in fracture toughness testing of ceramic materials since the late 80's. For tests aiming at characterization of the toughness of ceramics, the presence of the stable crack growth region assures the validity of the LEFM assumption of a sharp crack and allows the calculation of the critical stress intensity factor from the maximum load. There are, however, disadvantages in using chevron-notched specimens. This includes the lack of analytical stress intensity factor and compliance solutions for this crack geometry and the impossibility of performing in situ crack length measurements.

Bluhm [29] developed a method to analyze the three-dimensional crack configuration in a chevron-notched specimen using an approximate two-dimensional model, known as Bluhm's Slice Model. The chevron-notched specimen was treated as an assembly of length-wise slices along the width of the specimen. The crack in each slice was considered as a through-thickness crack. The compliance of the specimen could then be calculated from the compliance of the individual slices and a "shear correction" parameter, \( k \), to account for the inter-slice shear effects as [29,30]:

\[
\frac{1}{\lambda_{CN}} = \frac{\alpha - \alpha_o}{\alpha_i - \alpha_o} \frac{1}{\lambda(\alpha)} + \frac{k}{\alpha_i - \alpha_o} \int_{a_o}^{\alpha_i} \frac{1}{\lambda(\xi)} d\xi
\] (2.9)

where \( \lambda_{CN} \) and \( \lambda \) are the normalized compliance of the chevron-notched and straight-through specimen, respectively, \( \alpha \) is the normalized crack length, \( a/W \), and \( \alpha_o \) and \( \alpha_i \) are defined in Fig. 2.4. The shear correction factor, \( k \), essential in using this model, has to be obtained by comparison of experimental or numerical results (finite element or boundary element) with the results predicted from Eq. 2.9. Munz et al [30,31] employed Bluhm's model to calculate the geometric function, \( Y \), of various chevron-notched bar and short rod specimens from the compliance using:
\[ Y(\alpha) = \frac{1}{2} \frac{\alpha - \alpha_0}{\alpha - \alpha_0} d\alpha \]

In Bluhm's model the shear correction factor was assumed to possess a constant value greater than unity. It has been shown that this parameter depends on the crack length and thus has to be included in the integrand of the integral term on the right-hand side of Eq. 2.9 [32].

The geometric function of a chevron-notched specimen, as defined by Eq. 2.10, passes through a minimum that occurs at a specific crack length, \( \alpha_m \). This implies that once nucleated, increasing load will be required to grow the crack to the length \( \alpha_m \). For an ideal brittle material, or flat R-curve behavior, the amount of the stable crack extension preceding the maximum load, \( P_{\text{max}} \), is solely a property of the specimen geometry and \( P_{\text{max}} \) in the fracture test is achieved when the crack reaches this characteristic length. Thus, the toughness of the material can be unequivocally calculated from \( P_{\text{max}} \) and the minimum value \( Y_{\text{min}} \), without any requirement for crack length measurement. This is certainly a unique feature of chevron-notched specimens when they are used for toughness measurement on ceramics with a flat R-curve. The minimum value of the geometric function has been calculated in the literature for various chevron-notched specimens using different methods, e.g., by calibration against a known material [33], straight-through crack approximation [31], and using finite or boundary element methods [34,35].

When chevron-notched specimens are used to measure the toughness of ceramics exhibiting R-curve behavior the stable extension of the crack prior to the maximum load results in the accumulation of a finite amount of crack wake contribution to the stress intensity factor at the crack tip. In the presence of the R-curve behavior the relation between \( P_{\text{max}} \) and \( Y_{\text{min}} \) is lost completely, as \( P_{\text{max}} \) then corresponds to the tangency point of the applied stress intensity factor with the R-curve at a crack length that is larger than \( \alpha_m \). The apparent toughness calculated from \( P_{\text{max}} \) and \( Y_{\text{min}} \) in the presence of R-curve behavior, \( K_{\text{Q}} \), is not only larger than the real toughness value, but also has no physical meaning (discussed in more detail in 3.6).

Due to the ease of obtaining stable crack growth in this geometry, chevron-notched specimens have also become very popular for R-curve measurement on ceramics, especially at elevated temperature [36-43]. Typically the specimen is fractured under displacement control conditions, and the load and the displacement of the center-point on the specimen are recorded during the test. The dimensionless compliance is calculated from the test data and is corrected, by subtraction, for the compliance arising from the load train in the test setup (also referred to as the machine compliance). The compliance versus crack length, and thus the geometric function, is calculated using either Bluhm's slice model or finite element modeling. The measured compliance is then compared with the calculated compliance versus crack length curve to obtain the crack length and thus the geometric function. Finally, the stress intensity factor at each crack length is calculated from:
\[ K(\alpha_i) = \frac{P_i}{B\sqrt{W}}Y(\alpha_i) \]  

(2.11)

where \( P_i \) and \( \alpha_i \) are instantaneous load and instantaneous crack length at each point along the load-displacement curve, \( B \) is the specimen thickness and \( W \) is the specimen height. It has also been recognized that in the presence of R-curve behavior, the actual crack length may be shorter than the value calculated from the elastic compliance solution [23,44]. However, some experimental results [39] seem to indicate that the difference between the actual and calculated elastic crack length is not significant in the case of the chevron-notched specimens.

Deconvolution of the R-curve from the load-displacement curve of a chevron-notched specimen as described above is the approach used in this work. However, some modifications are employed, discussed later in chapter 4, to accommodate accurate compliance measurements.

### 2.2 R-curve Behavior in Ceramic Systems: A Brief Review

R-curve behavior in a ceramic material was first observed in monolithic alumina by Buresh and Pabst [45] about 25 years ago and was suggested to indicate the existence of a cumulative toughening mechanism in the wake of the crack. The first insight into the fracture process during stable propagation of cracks in a brittle material was made by Hübner and Jillek [46], who observed that fracture resistance of coarse-grained alumina improved by 70% during the stable crack extension experiment. However, it was found in the same study that samples notched to different depths yielded essentially the same fracture toughness when loaded to failure without any stable crack extension. From these observations, it was concluded that crack path roughness, intergranular fracture and crack branching during stable crack growth might be responsible for the improved fracture resistance.

It was not until the work of Knehans and Steinbrech [47] that the effect of the crack wake on the fracture resistance in a ceramic material was proven. By employing a simple yet clever experiment, this work revealed that the fracture resistance of coarse-grained alumina samples dropped to a value characteristic of the initial condition upon removal of the wake of a sharp crack. This clearly showed that the source of toughening was located in the crack wake and that the damage or microcrack zone near the crack tip in a coarse-grained alumina was far less effective than what initially had been believed. It was then Swanson [48] et al who showed the most direct evidence of the crack wake bridging process in the coarse-grained alumina by providing in situ micrographs. Later, an increasing number of published works became available on the observation of unbroken microstructural ligaments, or bridges, present in the crack wake of ceramic materials [49-51].
One of the issues realized in the early studies was that the R-curve of a material appeared to be different if a different crack geometry or a different notch depth were used [52,53]. Such evidence of geometry-dependence in the R-curve behavior was contrary to the traditional notion that, in contrast to metals, the R-curve of a brittle material was a material property and geometry independent [54].

All of the pioneering work on the R-curve behavior in ceramics was carried out in the long crack regime, i.e., cracks that initiated at the tip of a notch and propagated up to several millimeters. It was often in doubt if the R-curves from long cracks could be used to establish a reliable crack instability criterion in ceramics because failure in such systems occurred from flaws typically of the size of the microstructural elements. It was soon shown that the levels of R-curve behavior measured using long cracks did not exist for natural flaws [55]. This was indeed a serious problem considering that one of the primary focuses in ceramic R-curve research was to tackle problems that were related to the strength of the materials, e.g., strength variability, flaw tolerance, reliability, and life time prediction. These issues hinge on the crack instability criterion for natural flaws. If the R-curve were not a unique property of the material, then the curves obtained from long cracks would be completely irrelevant in such cases.

In the absence of a well understood correlation between the so-called long crack and short crack R-curves, efforts were focused on using indentation flaws to study and model R-curve behavior in ceramics, assuming that such flaws would properly represent strength-controlling flaws in ceramics. The contributions of Lawn and co-workers [6,7,9,11,13] to the development of indentation fracture mechanics and the modeling of R-curve behavior based on results from indentation flaws undoubtedly had a significant impact on the spread of indentation methods for R-curve characterization and increased understanding of the grain bridging process in ceramics. Although an implicit assumption in the R-curve studies using indentation flaws was that these R-curves were unique for the material and that the curves obtained from indentation flaws would be adequate to characterize the R-curve behavior, indications of the presence of geometry-dependent elements even within the short crack R-curves were seen [56]. A thorough understanding of the R-curve behavior in ceramics called for a clear vision of the geometry dependent element(s) of the R-curve behavior.

Initial efforts to model the R-curve behavior in a brittle material based on geometry-dependent entities were made by Foote et al [57] and Cotterell and Mai [58] in fiber-reinforced cementitious composites. The source of the R-curve behavior was considered to be the closure tractions from the bridging elements in the crack wake that, in turn, influenced the stress intensity factor at the crack tip. The bridging stress was assumed to follow the empirical tail-dominated relation:

\[
\sigma_{br} = \sigma_m (1-\frac{\delta}{\delta_f})^n
\]  

(2.12)
where \( \sigma_{br} \) is the bridging stress at the crack opening displacement \( \delta \) and \( \sigma_{m}, \delta_f \) and \( n \) are characteristic values describing the maximum bridging stress, the end of the bridging zone and the strain-softening behavior, respectively. By the calculation of the stress intensity factor arising from the bridging relation, the R-curve behavior in DCB and SENB specimens of fiber-reinforced cement composites was successfully modeled in these works. In [57] both exact and approximate solutions were obtained for the stress intensity factor arising from the bridging relation, Eq. 2.12, and used to determine the R-curve. The term "exact" was used to highlight taking into account the influence of the bridging stress on the crack opening profile, i.e., considering the effect of the closure tractions on the equilibrium crack shape, during the calculation of stress intensity factors. However, in the approximate solution method [57,58] this was neglected and the crack profile was assumed to be linear to simplify the stress intensity factor calculations.

Majumdar et al [59] modeled some aspects of the geometry dependence of the R-curve for long cracks in ceramics using a modified Dugdale approach [60], and considered the effect of finite specimen size with respect to crack length. Their results, however, indicated that the R-curves should be independent of the initial notch depth in a flexure specimen, which clearly contradicted the experimental results from Steinbrech on alumina [52]. Using an approach similar to works by Cotterell and Mai [58], Steinbrech et al [61] modeled the geometry dependence of the R-curves of coarse-grained alumina in SENB and DCB specimens based on crack opening displacement arguments. In this work the crack wake bridging contribution to the R-curve behavior was calculated through the J-integral method using the bridging stress relation presented in Eq. 2.12. One interesting result in [61] was that the length of the bridging zone formed in the crack wake was found to first increase and then decrease with increasing crack length. A generally well-accepted notion in the R-curve behavior of ceramics was the existence of a saturation length for the bridging zone, which in turn resulted in a plateau in the R-curve.

The modeling works on the effect of the crack geometry on the R-curve behavior due to the bridging process may be found to share at least one of the following assumptions:

- crack opening profile of specimen possesses a linear shape
- bridging stresses do not influence the crack profile shape
- a saturation length exists for the bridging zone

The first assumption originated from the studies of long cracks in cementitious materials [62] while the second assumption was made to simplify the calculation of the stress intensity factor related to the bridging tractions. It has to be noted that both theoretical [63,64] and experimental results [65,66] have indicated that the bridging stresses alter the shape of the crack profile. For an accurate calculation and modeling of the R-curve behavior the equilibrium crack profile under the influence of the bridging stresses has to be first calculated (iteratively) [63,64]. The complexities involved with such calculations seem to have been strongly promoting the above assumptions. These assumptions, in turn, facilitated transformation of the bridging relations of Eq. 2.12 from the crack opening displacement into position in
the crack coordinates and thus straightforward calculation of the resulting stress intensity factor. A major consequence of this transformation was the birth of the notion of a "characteristic bridging zone length" that could be traced in many R-curve research works.

Fett and Munz [63] discussed the R-curve behavior and its geometric elements in ceramics with crack wake bridging effects using the fracture mechanics weight function method. They showed that since the crack opening displacement and fracture mechanics weight functions were correlated and the bridging stresses evolved with the crack opening profile, each crack geometry would give rise to a different R-curve owing to the governing weight function. In addition, they showed that amongst other geometrical parameters R-curves were dependent upon the type of loading, i.e., tension versus bending, due to its effect on the crack opening profile.

It can be concluded at this stage that the geometric-element of the R-curve behavior due to bridging effects lies in the crack profile evolution for different crack geometries, which in turn results in different R-curve shapes under different conditions. At the same time, the characteristic material elements of the R-curve behavior would be such parameters as the fracture toughness of the material at the crack tip, $K_{IC}$ or $K_c$, and some characteristic parameters in the bridging relation*, e.g., $\sigma_0$, $\delta$, and $\alpha$ in Eq. 2.12, but not the bridging zone length. It may also be concluded that, if $K_c$, $\sigma_0$ and $\delta$ were considered material properties, R-curve behavior would become more of a fracture mechanics problem rather than a material issue. The trend in R-curve research after the early 90s seems to be indicative of such realization and the shift of the interest towards characterization of the bridging relation instead of the R-curve.

The first characterization of bridging stresses in a ceramic material goes back to an early work from Hsueh and Becher [68] on SiC$_w$-reinforced alumina. By assuming a fixed bridging zone length and neglecting the effect of the bridging stress on the crack opening displacement, Becher and Hsueh calculated from measured R-curves the distribution of the bridging stress as a function of crack face separation. Fett and Munz [69] obtained the bridging parameters by analyzing the R-curve results of Steinbrech et al on alumina [52]. Fett and Munz's method relied on an iterative analysis scheme based on the fracture mechanics weight function method and the assumption of an exponential bridging relation derived from the grain bridging model proposed by Mai and Lawn [70]. The exponential bridging relation:

$$\sigma = \sigma_0 \exp\left(-\frac{\delta}{\delta_0}\right)$$  \hspace{1cm} (2.13)

* It may be noted that even the bridging stress have been recently challenged as a material characteristic value by Fett [67]. It has been proposed that these stresses can be influenced by the T-stress term: the stress term, which describes a constant stress that is formed by the crack parallel to the crack direction.
was derived by considering a distribution function for the $\delta_f$ in Eq. 2.12, representing the distribution of the grain-size, and $n=1$ to describe pure friction. $\sigma_c$ and $\delta_i$ in Eq. 2.13 represent characteristic values of the bridging stress and the crack opening displacement, respectively. After obtaining $\sigma_c$, $\delta_i$, and $K_c$ from analyzing one of the R-curves, Fett and Munz used these values and successfully calculated the two other R-curves that had been obtained by Steinbrech et al on the specimens with two different notch depths. An important feature of this analysis was that no assumption was made regarding the shape of the equilibrium crack profile, thus allowing accurate calculation of the stress intensity factor due to bridging stresses. In further works Fett and co-workers used their fracture mechanics weight function methodology to obtain the bridging relation by analyzing crack opening profile measurement results [66,71,72] and the distribution of the bending strength in specimens containing natural flaws [73].

Hay and White [74,75] studied the crack bridging behavior of spinel and alumina experimentally by devising a post-fracture tensile testing technique. In this method, tensile tests were carried out on the specimens that were prepared from different positions within the wake of the crack from a DCB specimen. These samples were subsequently tested under tension to give a family of load-displacement curves. The load results were converted to the stress by dividing over the fraction of fracture surface area covered by intergranular fracture. Having undergone different amount of crack opening displacement prior to post-fracture tensile test, the stress-displacement from such specimens represented the remaining portion of the bridging stress relation. Thus, the envelope of the stress-displacement curves from the specimens obtained from various locations along the crack wake defined the variation of the bridging stresses as a function of opening displacement. The results indicated that the maximum bridging stress in the spinel varied from $-3.75$ MPa to $-13$ MPa with varying average grain size from $35 \mu m$ to $180 \mu m$ while the value for the coarse-grained alumina (average grain size $16 \mu m$) corresponded to $-46$ MPa.

Yu and Kobayashi [65] obtained the variation of bridging stresses as a function of crack opening displacement in a SiC$_m$-alumina composite by analyzing the crack opening profile data, obtained using the very precise Moire interferometry technique, through a combined finite element and iteration analysis. The crack profiles obtained in this work from stable crack growth in a wedge-loaded DCB and a SENB specimen resulted in a slightly different maximum bridging stress values in the analysis: $-23.5$ MPa for the SENB and $-21$ MPa for WL-DCB specimens, respectively. However, the resulting constitutive relation turned out to show the same trend in both of the specimens; the bridging stress fell down linearly from the maximum value to $-6$ MPa within the first $-3.5 \mu m$ and then smoothly decayed linearly to zero over the next some $18 \mu m$ of displacement. The slight difference between the bridging relations obtained from the two different types of specimens in this analysis was attributed to a different contribution of intergranular fracture between the two specimens. Following this work, Fett et al [72] analyzed the crack opening profile results from the SENB specimen in Yu and Kobayashi’s work using the weight function approach and the constitutive bridging relation was decovoluted from the crack opening profile. This analysis indicated a constitutive bridging relation somewhat different from the ones obtained previously.
The bridging stress rose to 25 MPa within the first \(-0.5\ \mu\text{m}\) of displacement, fell smoothly to \(-5\ \text{MPa}\) over the next \(3\ \mu\text{m}\) of displacement and was almost constant within the rest of the displacement range shown. Mori and Imai [76] devised a direct measurement method to characterize the bridging constitutive relation in ceramics based on using a double-edge-notched specimen mounted onto a stiff crack stabilizing fixture. The bridging stress results measured as a function of crack opening displacement for an alumina specimen was found to be satisfactorily expressed using the exponential relation, Eq. 2.13, with characteristic bridging stress and COD values of 59 MPa and 1.66 \(\mu\text{m}\), respectively.

Hypothetically, the knowledge of the crack tip toughness, bridging relation and appropriate fracture mechanics function describing the evolution of the stress intensity factor at the tip of the crack in a geometry should allow the determination of the R-curve in any crack configuration. This hypothesis was proven valid recently in a work by Fett et al [66] who calculated the short- and long crack R-curves for both coarse-grained and fine-grained alumina specimens by using the bridging relations that were obtained by analyzing crack profile measurement results on the materials and the appropriate weight function. The crack tip toughness was also estimated from the crack tip opening profile at the onset of the stable-crack-growth regime. The calculated R-curves were then compared with the experimentally measured R-curves and shown to be in good agreement. This undoubtedly provided solid evidence that the knowledge of the bridging constitutive relation and proper fracture mechanics formalism can mark an end to the short crack and long crack R-curve debates in ceramic materials.

### 2.2.1 Crack Face Bridging Mechanisms and Micromechanics

Our attention in the last parts of the previous section was redirected to the importance of the constitutive relation between the bridging stress and crack opening displacement, \(\sigma_{br} = f(\delta)\). The crack face bridging mechanism has long been studied and modeled in fiber-reinforced composites, e.g., [2, 77-80]. The existing bridging models in ceramics have also been based upon the models developed for fiber-reinforced cementitious materials or fiber-reinforced ceramics.

The general form of the constitutive bridging relation can be described by three distinct regions [79]: a linear rise from zero at \(\delta = 0\) to some stress value, further non-linear extension to a maximum stress and tail-off drop to zero at a characteristic rupture displacement. The linear segment of the relation represents the elastic stretch of the bridging ligament while the interface of the ligament/matrix is intact, i.e., the bridge is acting as a linear spring. When such an elastic bridging ligament fails, the bridging stress drops to zero instantaneously. If debonding occurs at the matrix/bridge interface prior to the rupture of the ligament, frictional contact at the interface results in a nonlinear stress-displacement region preceding the maximum bridging stress. If such a bridge fails at the crack plane, the bridging stress shall fall to zero instantaneously similar to the case for the elastic bridge. However, if the bridge fails within the debonded length, there will be a sudden drop from the maximum stress and the frictional pullout of the bridging
ligament will result in tail-off behavior to zero stress. Marshall and Evans [77] have adopted the rising portion of the constitutive relation as dominating the bridging behavior in fiber-reinforced ceramics. In this case the abrupt rupture of the bridging fibers results in the cut-off of the monotonically rising frictional bridging stress that is formed in the fiber due to frictional shear tractions at the interface of debonded fiber and matrix. Huseh and Becher [79,80] considered the entire constitutive relation to discuss the R-curve behavior in fiber and whisker-reinforce ceramics. Their calculated R-curves [80] for DCB specimens of SiC-whisker-reinforced alumina showed a significant rise from the matrix fracture toughness within the first 50-100 μm of crack length, which could not be observed in their experimental results. The sharp initial rise of the calculated R-curve is a consequence of the assumption of very large bridging stresses, arising from frictional bridges, close to the crack tip at short crack lengths.

Mai and Lawn [70] adopted the empirical relation presented by Eq. 2.12, which had been accepted for fiber-reinforced cement materials, for grain-bridging in ceramics following their experimental observations on alumina [48]. As was seen previously, this constitutive relation has also been adopted in numerous works on the analysis of bridging stresses and R-curve behavior in ceramic materials. The exponent n (or m as used by Mai and Lawn) in the bridging relation, Eq. 2.12, has been assumed to describe the nature of the bridging process. For n=0 this relation yields a uniformly distributed bridging stress; n=1 results in a linearly decaying bridging stress with crack opening displacement and has been accepted to describe a pure frictional pull-out process; n=2 is merely a fit value adopted empirically for fiber-reinforced concrete [81]. The value n=0 seems to be contradicting the mechanics of the bridging stresses in ceramics: the bridging tractions are formed as a consequence of crack opening displacement, and thus, must vary as a function of this parameter. In fact even Mai and Lawn [70] were reluctant to consider n=0 as a true physical value despite the good fit they obtained to the R-curve results from Swain on alumina using this value. It is to be remembered that the bridging relation presented in Eq. 2.12 describes the bridging event for a single bridging element as it is being encompassed by the crack. In a real microstructure the size of the bridging elements, e.g., large grains, and thus the critical opening displacement in Eq. 2.12 are statistically distributed. Therefore, the influence of the statistical nature of the size distribution of the bridging elements, if any, has to be included in the bridging stress function, as was done by Fett and Munz [69] and Sohn et al [82]. Nevertheless, Eq. 2.12 has been frequently used in the literature to obtain a fit to the R-curves in ceramics without any such statistical considerations, resulting in a priori assumption that all the bridges had a unique size.

We may note that our descriptions of the bridging problem so far has been based on a continuum approach, as such is Eq. 2.12. Although decisive in establishment of a basis to discuss and understand the global effects arising from the bridging process in the material, such an approach, due its nature, makes no attempt to elaborate the microstructural elements that control the bridging process. Such details are discussed in the micromechanical consideration of the bridging problem and have been dealt with extensively in the literature related to fiber-reinforced ceramics, e.g., the works by Evans and Marshall.
Bennison and Lawn [83] developed a micromechanical model for the grain bridging process based on the shear lag approach used by Marshall and Evans [77] to model the frictional bridging in fiber-reinforced ceramics. Bennison and Lawn's model considered an array of bridging grains of size $l$ with an effective spacing $d$ under normal residual compression $\sigma_R$ at the potential bridging grain/matrix interface. The constitutive bridging relations were derived for the frictional bridging process, assuming that the residual compression on the bridging grain initially helped open the crack, as:

$$\sigma_{br,fr}(\delta) = \left( \frac{2\mu \sigma_R E}{l^2} \right)^{1/2} \delta^{1/2} - \sigma_R$$

and for the pull-out bridging process as:

$$\sigma_{br,po}(\delta) = \frac{\mu \sigma_R \lambda \delta_f}{d^2} \left( \frac{2d^2}{l^2} - 1 \right) \left( 1 - \frac{\delta}{\delta_f} \right)$$

In Eqs. 2.14 and 2.15, $\sigma_{br}$ is the bridging stress and the indices "fr" and "po" refer to the frictional and pull-out processes, respectively, $\delta$ is the crack opening displacement, $\mu$ is the friction coefficient, $E$ is Young's modulus, $\lambda$ is the circumferential distance around the debonding grain, assumed as equal to $4l$, and $\delta_f$ is the critical crack opening displacement at which the grain disengages from the matrix. The cross-over of Eq. 2.14 and Eq. 2.15, the transition from frictional to pull-out bridging regime, could then be determined as:

$$\delta_+ / \delta_f = \left( \frac{\sigma_D}{\sigma_M} \right)^2 \left[ 1 + \frac{4 \sigma_M (\sigma_M + \sigma_R)}{\sigma_D^2} \right]^{1/2} - 1$$

where $\delta_+$ marks the end of frictional bridging zone, $\sigma_D$ is the shear-lag stress in Eq. 2.14 evaluated at $\delta=\delta_f$ in the absence of residual stress $\sigma_R$, and $\sigma_M$ is the sliding friction stress in Eq. 2.15 evaluated at $\delta=0$. The square-root dependence of the bridging stress on the displacement in Eq. 2.14 is a consequence of the shear-lag model. It can be readily seen that a strain-softening coefficient, $n$ in Eq. 2.12, equal to one has also been adopted in the pullout bridging relation described by Eq. 2.15. Comparison of the pullout bridging stress relations, Eq. 2.15 and Eq. 2.12, would also reveal the variables in the microstructure that control the magnitude the bridging stress, e.g., the friction coefficient and the residual stress at the sliding
interface or the size of the bridge. (Obviously, the maximum bridging stress in Eq. 2.12 and Eq. 2.15 are also correlated through a statistical function that should describe the size distribution of the bridging grains.) Amongst these variables, the friction coefficient and the residual stress at the sliding interface seems to be the most important controlling parameters, especially at elevated temperatures. A significant feature of the bridging model from Bennison and Lawn can be seen in Eq. 2.13: for \( \delta = 0 \) the bridging stress would be equal to \(-\sigma_R\), indicative of an opening stress at the beginning of the crack surface separation during debonding of the matrix from the potential bridging grain. This may explain Steinbrech and Slemenkel's observation [55] of a lower \( K_c \) for the natural flaws than the corresponding value for long cracks in alumina. Following fracture mechanics formalism, Bennison and Lawn [83] then used this bridging model to characterize the indentation strength behavior of alumina ceramics with different grain sizes. The R-curve deconvoluted from the indentation strength results using this model had a characteristic region that showed an initial fall from \( K_c \) for a crack size smaller than the grain size with the onset of the rise, from a \( K < K_c \) at a crack length equal to the grain size. A similar approach was later employed by Padture et al [84] to model the R-curve behavior due to bridging in two-phase ceramics.

For the whisker-reinforced ceramics Becher et al [80] have adopted constitutive relations similar to those developed for the fiber-reinforced materials [79]. The bridging relations for frictional bridging and pullout bridging in a whisker-reinforced ceramic were described as [80]:

\[
\sigma_{\text{br}}(\delta) = \frac{2\pi\delta E_w E_c}{d_w (1 - f) E_m} [1/2]
\]

\[
\sigma_{\text{pop}}(\delta) = 4\tau \frac{(l_p - 2\delta)}{d_w}
\]

where \( \tau \) is the frictional shear resistance of the debonded whisker/matrix interface, \( d_w \) is the diameter of whiskers, \( f \) is volume fraction of the whiskers and \( E \) is the elastic modulus with indices "w", "c" and "m" referring to whisker, composite and matrix, respectively. The frictional shear stress, \( \tau \) at the whisker/matrix interface was related to the radial (compressive) residual stress, \( \sigma_R \), and friction coefficient, \( \mu \), at the interface:

\[
\tau = \mu \sigma_R
\]

The significance of the interfacial properties and the role of residual stresses at the (reinforcement) bridge/matrix in the development of the bridging stresses can be directly seen in Eq. 2.13-Eq. 2.18. Such parameters can be used to tailor the toughening (and R-curve) response of ceramics.
2.2.2 Temperature Dependence of R-curve Behavior in Ceramics

Although a considerable number of studies have been carried out on the R-curve behavior and the effect of bridging stresses at room temperature, only a limited number of works can be found in the open literature to address the behavior at elevated temperatures. The first of such studies was carried out by Bornhauser et al [23] who studied the R-curve behavior of two different alumina materials, one pure and another containing 3% glass phase, between room temperature and 1000°C. Though the major objective of this work was to discuss fracture mechanics concerns regarding the applicability of LEFM to ceramic materials containing glass phase at high temperatures, some of the results obtained could be of interest to both material scientists and fracture mechanists. The R-curve of the materials were measured by first precracking of three-point flexure SENB specimens at room temperature and performing fast fracture tests under load-control condition at the desired temperature. The results of this study indicated that the pure, fine-grained alumina showed almost a flat R-curve through 1000°C while enhanced R-curve behavior could be observed in the material containing glass phase within the same temperature range. The observed R-curve behavior of the alumina samples with glass phase was suggested to result from an adhesive zone formed behind the crack tip by the viscous glass phase at elevated temperatures. Jakus et al [85] have shown that the tractions arising from a viscous bridging ligament were dependent both on temperature and displacement rate; the bridging tractions from viscous ligament increase with increasing displacement rate and decrease with increasing temperature. It may be noted that the alumina material with glass phase used in the work by Bornhauser et al [23] was quoted to have an averaged grain size of 11 μm: at this grain size the grain bridging process can also contribute to the R-curve behavior. In a subsequent research Wieninger et al [86] performed high temperature annealing on the precracked specimens of the same alumina with glass phase material as was used by Bornhauser et al and measured the fracture toughness of such specimens at room temperature. The results indicated that the annealing process, above 1000°C for 10 min, could significantly improve the room temperature R-curve behavior of such specimens. Increasing annealing time and temperature were also found to improve the room temperature R-curve behavior of such specimens. The fracture surface of the specimens annealed properly showed the presence of the bands of a second (glassy) phase along the edges of the grains. However, no results were presented related to the possible effect of the annealing process on the high temperature R-curve behavior.

Grimes et al [38] adopted the renotching technique to characterize and isolate the crack wake bridging contributions to the R-curve behavior of high purity silica-glass-phase sintered alumina at elevated temperatures using chevron-notched specimens. Their results indicated that the slope of the R-curves obtained at 950°C and 1200°C were significantly higher than those obtained at room temperature; this could be interpreted as increased contribution of the crack wake to the R-curve at those temperatures. Above 1200°C, however, the slope of the R-curve was found to dramatically decrease and appeared to be almost flat. In addition, the R-curves of the alumina material were found to initiate at decreasing stress
intensity factors with increasing temperature. The results from renotching experiments (almost 0.2 mm of the crack wake was left after the renotching operation) indicated a return of the stress intensity factor to the value close to that obtained from the straight-through notched specimen at all temperatures. However, the results of a renotching experiment at 1300°C indicated a critical stress intensity factor almost 20% lower than the value obtained on the straight-through notched specimen. This was considered by Grimes et al [38] to be an indication of a shift in the toughening mechanism with the onset of viscous behavior of the intergranular glass phase. Below 1200°C the mechanism responsible for the R-curve behavior was considered to have a high component of intergranular friction, while above this temperature the lubricated sliding due to increased softening of the silica-based intergranular glass phase deteriorated the R-curve behavior. In a similar work, White and Guazzzone [40] studied the R-curve behavior in a SiC\textsubscript{w}-reinforced alumina at room temperature, 1200°C and 1400°C using chevron-notched specimens. The results presented in their work indicated that the R-curve of the composite at room temperature fell between the R-curves at 1200°C and 1400°C, with the initiation level of the R-curves at 1200°C significantly higher than the corresponding value from room temperature. The authors then compared the fracture toughness values obtained from the chevron-notch specimens with those measured using straight-through notched specimens (notched specimens were prepared following the previous work by Grimes et al [38]). This comparison was expected to reflect the contribution of the crack wake; unstable crack propagation in a straight-through notched specimen precludes formation of the crack wake while stable crack growth prior to maximum load in the chevron-notched specimen results in some finite crack wake contribution on the measured toughness. The results indicated that while the toughness values measured using the straight-through notched specimens were almost insensitive to or even somewhat decreasing with the temperature through 1200°C, the values from the chevron-notched specimens were observed to pass through a maximum at 1200°C. As a result, it was concluded that the crack wake zone contribution in this composite improved with temperature through 1200°C and diminished at higher temperatures due to the softening of an intergranular glass phase. An increased whisker pullout length with increasing test temperature found in microscopic examination of the fracture surfaces of the composite was used as supporting evidence to the hypothesis of an increasing bridging contribution in the crack wake with increasing temperature. Based on these results the authors concluded that the toughening mechanism active in this material changed from a predominantly frontal zone at low temperature to one operative only in the following wake zone at 1200°C. In a following work, the same authors investigated SiC\textsubscript{w}-alumina composites that were prepared to result into two different whisker/matrix interfaces [41]. One type of the composite with designation "N" was prepared from as-received whiskers that had carbon residues on their surface and the other type, designated "Z", was prepared from the same whiskers after removing the carbon residues. The composite Z was the material that had been used in the previous study by the authors. The results indicated that, although tougher, composite N showed a much less pronounced R-curve behavior at room temperature. The high temperature toughness of the composite N was also
found dramatically lower than composite Z despite the fractographic results that indicated similar fracture surfaces for both composites Z and N. To support the hypothesis of two different dominant toughening mechanisms at room and elevated temperature in the composite, i.e., frontal microcracking [87,88] and crack wake bridging, the authors also presented some scanning acoustic microscopy results that identified microcracking around the whiskers within a subsurface layer adjacent to the fracture surface of composite Z at room temperature. The R-curve of the composite at room temperature shown by the authors (compare the first and second data point on the room temperature R-curve in Fig. 2 of Ref [40]), however, indicates run-arrest behavior characteristic of SiC-whisker-reinforced alumina. The arrest point following the unstable run on the presented data shows a reduction of the stress intensity factor relative the value prior to unstable crack run. This indicates that the toughening mechanism operative in the wake is sensitive to the state of crack propagation, i.e., stable or unstable. We may note from the classical studies on transformation toughening of ZrO₂ that the transformation or microcrack zones could be measured on the fracture surface of the specimens from the strength tests: a totally catastrophic crack propagation condition. Bridges, however, are found dominantly within the regions of the fracture surface produced by slow/stable crack propagation. During a fracture test condition where the crack growth proceeds in multiple stable/unstable propagation steps, each crack jump can result in a complete or partial destruction of the bridges formed in the crack wake. This may be imagined as a “natural” renotching process during which some part of the crack wake zone is removed and results in the well-known step like shape of the R-curves. Such an appearance can be observed in many long crack R-curve studies on SiC-whisker-reinforced alumina system, e.g., in [80,89,90].

Ohji et al [39] studied the R-curve behavior in a SiC-whisker-reinforced silicon nitride composite using chevron-notched flexure specimens between room temperature and 1300°C as a function of strain (displacement)-rate and found a strong dependence of the R-curves on the strain-rate at temperatures beyond 1000°C. Specimens broken at 1200°C showed more pronounced R-curve behavior with decreasing displacement rate during the fracture test: however, in contrast to the results from 1200°C, an inverse trend with the displacement rate was observed for the specimens tested at 1300°C. The R-curves at 1300°C were found almost parallel, having the same ΔK/Δa, but the curves initiated at a lower stress intensity factor with decreasing strain rate. One explanation for the observed rate sensitivity at 1300°C was given by the authors as being related to crack blunting and stress relaxation process due to grain boundary sliding in the vicinity of the crack tip, similar to what had been observed within the creep regime in SiC-whisker-reinforced alumina [91]. It has to be noted that a slower displacement-rate condition should be more similar to a creep condition. If creep relaxation at the crack tip were responsible for blunting (decreasing the crack tip curvature) of the crack tip, then one would observe an increase in the initial value of the toughness with decreasing strain rate. However, the observed trend at 1300°C is in the opposite direction of what could be accounted for by the creep effects. The observed trend for the R-curves of the material at 1300°C seems to closely resemble the effect of subcritical crack growth on the R-curve
behavior of a coarse-grained alumina at elevated temperatures reported by Webb et al [92]. Under such conditions, the crack propagation can be initiated at a stress intensity factor lower than the critical value, due to environmental effects at the crack tip. The subcritical crack growth process has a strong rate-dependent characteristic and is promoted below a threshold strain rate. Ohji et al also assessed the magnitude of the bridging tractions in the crack wake at 1300°C by analyzing the R-curve iteratively using an equation developed by Sakai and co-workers [93,94] to describe the stress intensity factor at the tip of a chevron-notch crack resulting from crack wake tractions. Following the assumption of a cubic stress distribution for the bridging tractions as a function of position behind the crack tip, the analysis of the R-curve resulted in a bridging stress distribution at 1300°C that had a maximum value of about 200 MPa close to the crack tip. From these results, the end of the bridging zone was also estimated to be at a COD of ~7μm.

White and Hay [95] reported the first experimental results on the measurement of the bridging stress in a coarse-grained high purity alumina between room temperature and 600°C using the post-fracture tensile test method. Their results indicated that the bridging stresses as a function of crack surface displacement decreased with increasing temperature. This trend included both the magnitude and the displacement range of the bridging stresses. In addition, it was observed that the ratio of intergranular/transgranular fracture for grains above a threshold size increased at 800°C relative to room temperature. This behavior was attributed to the lower pullout stresses associated with the diminishing residual stresses.

Fett et al [96] analyzed the elevated temperature R-curve results from a high purity coarse-grained alumina that had been obtained by Mundry [97] using SENB specimens between RT and 1000°C. Using the fracture mechanics weight function method, the R-curves were analyzed assuming that the material possessed an exponential bridging constitutive relation, Eq. 2.13. Following the analysis of the R-curve from room temperature, the value of the characteristic displacement (δc in Eq. 2.13) obtained was assumed to be temperature independent and was used in the analysis of the R-curves from 700 and 1000°C. The characteristic bridging stress (σm in Eq. 2.13) obtained from the R-curves found to decrease almost linearly with increasing temperature, indicating that there was a contribution to this parameter from temperature-dependent internal residual stresses caused by the thermal expansion anisotropy of aluminum oxide. However, the assumption of the same characteristic displacement in the bridging relation for all temperatures may be arguable. This parameter is related to the crack surface displacement at which the bridging grain disengages from the matrix and the statistical size distribution of (large) bridging grains. The micromechanics of the grain bridging process and the experimental data from White and Hay [95] described earlier suggest that this parameter is temperature sensitive.

Xu et al [20] studied the R-curve behavior of alumina between room temperature and 1300°C using an indentation strength technique. In order improve the reliability of the R-curve results, the residual contact field from the indentation process was annealed out at 1350°C, the crack length was obtained by
measuring surface traces of the cracks on surviving dummy indentations and the ellipticity of the cracks were measured using SEM observation on the fracture surface of the specimens. The R-curves were calculated from the indentation-strength data at different temperatures and were further analyzed iteratively based on the bridging model developed by Bennison and Lawn [83] to obtain the initial toughness and bridging parameters. The results indicated that while the thermal residual stress showed a linear decaying trend with increasing temperature, the bridging stress was relatively constant, ~70 MPa, below 700°C and moderately decreased with increasing temperature above this temperature. The results for the friction coefficient at the interface of the bridging grains with the matrix showed that this parameter was monotonically increasing with increasing temperature. The temperature dependence for the friction coefficient was related to the absence of a glass phase in the high-purity hot-pressed alumina material used for the study. Xu et al attributed the behavior of the bridging stress below 700°C to the counterbalance between the residual stress and friction coefficient; beyond this temperature the increase in the friction coefficient was inadequate to offset the effect of the residual stress.

It may be concluded that at elevated temperatures the R-curve and toughening behavior due to bridging mechanism is most influenced by the nature of the bridge matrix interface and the state of the residual stresses at this interface. The composite response of these two parameters to the temperature may result in a complex temperature dependency with multiple temperature ranges dominated by each of these parameters. It has to be remembered that in addition to the bridging parameters, the crack tip toughness can also influence the R-curve through its influence on the crack opening profile.

References


35. A. R. Ingraffea R. Perucchio, T. Y. Han, W. H. Gerstle and Y. P. Huang, "Three-Dimensional Finite and Boundary Element Calibration of the Short-Rod Specimen"; pp. 49-68 of Ref. [27].


Chapter 3

Application of the Weight Function Method to the Analysis of the R-curve from a Chevron-Notched Specimen

3.0 Introduction

The geometry dependence of the R-curve behavior in ceramics leads to some complexities in characterizing fracture resistance and the associated toughening mechanisms present in the material. It also results in major obstacles to establishing a crack propagation criterion applicable to crack geometries other than the specific geometry for which the R-curve was determined. Therefore, it is necessary to obtain the geometry-independent, material characteristic elements, which give rise to an R-curve. Such elements are, in fact, characteristic of the toughening mechanism(s) present in the material/microstructure under study. The importance of such characteristic information is dual; we may use such information to understand and tailor the specific toughening mechanisms of the material, or predict the conditions necessary for crack propagation in different crack geometries through a proper fracture mechanics formalism.

In the presence of bridging toughening mechanisms, such characteristic information represents the constitutive relation between the bridging stress, \( \sigma_b \), and the crack opening displacement (COD), \( \delta \), i.e., the description of how bridging stresses develop as a function of crack opening displacement [1]. The knowledge of such a relationship would enable us, through a proper fracture mechanics methodology, to correlate and compare the R-curves from different crack geometries [2-4].

The focus in this chapter is the establishment of a methodology to obtain such characteristic information from an R-curve obtained from a chevron-notched flexure specimen [5,6]. Although such information may be obtained more directly by performing post-fracture tensile tests [7,8] or by analyzing crack opening profile measurements [9,10], the requirement of elaborate instrumentation in these methods makes an analysis based on the R-curve measurements an attractive alternative especially for high-temperature studies beyond 1200°C.

Chevron-notched specimens have been used in many crack growth studies on ceramic materials [11-17] and were also adopted for the current work. This type of specimen is in fact very popular for toughness measurements on ceramics due to the ease of crack nucleation from the tip of the triangular notch and the well-known characteristic region of stable crack growth preceding the maximum load in a fracture test. The presence of the stable crack growth region assures existence of a sharp crack and allows the calculation of the critical stress intensity factor from the maximum load. Compared to a through-the-thickness crack geometry, further stable crack growth beyond the maximum load can also be obtained using a chevron-notched specimen with much less stringent requirements on the compliance of the testing machine and the
load-train of the testing setup. This relates to the elastic energy stored in the test setup/specimen system [18] and, thus, makes the chevron-notched specimens very interesting.

The major disadvantages of the chevron-notched specimens, however, are the lack of analytical solutions for the stress intensity factor arising from different loading conditions in this geometry and the infeasibility of performing in-situ crack length measurements.

A new fracture mechanics weight function-based approach to the analysis of the R-curve obtained from a chevron-notched flexure specimen is presented in the following. The development of the weight function for crack surface tractions in a chevron-notched flexure specimen required for this approach is described. This methodology will become the major tool in Chapter 5 to analyze and understand the R-curve results from the fracture test that will be presented in Chapter 4.

### 3.1 R-curve Response Due to Crack Wake Bridging Stresses

The analysis of an observed R-curve is based on the relationship among the stress intensity factor acting at the crack tip, \( K_{sp} \), the stress intensity factor arising from the remotely applied load, \( K_{app} \), and the stress intensity factor resulting from the presence of shielding mechanisms such as bridging ligaments in the crack wake, \( K_{br} \):

\[
K_{tip} = K_{app} + K_{br}
\]  
(3.1a)

At the critical condition, \( K_{sp} \) will be equal to a characteristic value, \( K_o \). The value of \( K_o \) represents the fracture resistance of the material in the absence of any crack wake contributions giving rise to R-curve behavior. It will be more convenient if Eq. 3.1a is rearranged such that the terms representing the material response are all brought on one side of the equation:

\[
K_{app} = K_o - K_{br}
\]  
(3.1b)

With \( K_o \) a characteristic material value, the analysis of the R-curve behavior will be reduced to understanding the term \( K_{br} \). It is this term that defines the shape and magnitude of an observed R-curve. Being a stress intensity factor, \( K_{br} \) can be considered to be composed of a stress intensity factor solution and a stress distribution. The stress distribution in this case represents the bridging stress that is acting over the crack wake, \( \sigma_{br}(x,a) \), with \( x \) and \( a \) representing a position and crack length in the crack coordinate system, respectively. The required stress intensity factor solution in this case is unknown and is to be sought. Since the bridging stress evolves as a function of crack opening displacement, \( \sigma_{br}=f(\delta) \), the resultant stress in the crack coordinate system can indeed be very complicated. This results in the requirement that a large number of different stress intensity factor solutions be available for different
bridging stress distributions and crack lengths within the same specimen geometry in order to handle the term \( K_b \). The three-dimensional nature of the crack problem in a chevron-notched specimen calls for such stress intensity factor solutions to be obtained by employing numerical methods, e.g., finite element or boundary element modeling. Such an approach would certainly not be practical due to the amount of calculation required.

3.2 Analysis of the Stress Intensity Factor Due to Bridging Stresses for the Crack in a Chevron Notch

A powerful method to analyze the stress intensity factor arising from any arbitrary loading is the weight function method [19-21]. Fett, [22], Fett and Munz [3,23,24], Fett et al [4,25,26] and Foote et al [1] have used this method to analyze the R-curve behavior and the bridging stress contributions in a variety of cases. In simple words, the weight function is the stress intensity factor arising from a pair of symmetric loads acting on the crack surfaces; the Green’s function [27] of the stress intensity factor problem. The \( K_{br} \) term can be expressed by the integral of the product of the weight function, \( h \), and the bridging stress, \( \sigma_{br}(x) \), over the section of the crack with bridging interactions:

\[
K_{br} = K(a) = \int_0^a h(x,a) \sigma_{br}(x) dx
\]  

(3.2)

If the weight function, \( h \), for the crack surface tractions in a chevron-notched specimen were available, the problem of the R-curve and the effect of the bridging stress in this specimen geometry could be analyzed based on the Eq. 3.2. Such a weight function has not appeared in the open literature for a chevron-notched geometry.

3.2.1 Application of the Weight Function for an Internal Crack in an Infinite Body

Previously, Sakai and Inagaki [28] and Sakai and Bradt [29] used the weight function for an internal crack in an infinite body to compare the R-curves obtained from the crack growing in a chevron-notched specimen and the same specimen with a straight-through crack in the presence of a constant bridging traction. The weight function for an internal crack in an infinite body under symmetrical load [30] is given by:

\[
h(x,a) = \frac{1}{\sqrt{\pi a}} \left[ \left( \frac{a+x}{a-x} \right)^{1/2} + \left( \frac{a-x}{a+x} \right)^{1/2} \right] = \frac{2}{\sqrt{\pi a}} \frac{a}{\sqrt{a^2-x^2}}
\]  

(3.3)

The analysis by Sakai and co-workers [28,29] resulted in the following expression for the stress intensity factor arising from a constant bridging stress, \( \sigma_b \):
\[
\Delta K = 2\sqrt{\frac{a}{\pi b(a)}} \int_{a}^{\infty} \frac{b(x)}{\sqrt{a^2 - x^2}} dx
\]  
(3.4)

where \(b(a)\), the width of the crack front, is equal to the specimen width, \(B\), and to \(B(a-a_c)/(a_1-a_c)\) for specimens with a straight-through notch and with a chevron notch (shown in Fig. 3.2), respectively. In the case of a constant traction acting in the wake of the crack growing in a chevron notch also a closed form solution was derived for Eq. 3.4 [29]. Comparing Eq. 3.4 with Eq. 3.2, it can be readily seen that the bridging tractions were assumed to be proportional with the ratio of \(b(x)/b(a)\); \(\sigma(x) = \sigma_a b(x)/b(a)\), where \(b(x)\) is the local width of the crack wake. Ohji et al [12,17] have used the function given by Eq. 3.4 to deconvolute the distribution of bridging stresses along the crack wake, \(\sigma_w(x)\), from R-curves obtained from chevron-notched flexure specimens of SiC-whisker-reinforced Si\(_3\)N\(_4\) and laminated SiC-whisker-reinforced Si\(_3\)N\(_4/\)Si\(_3\)N\(_4\) composites.

Although it seems rather obvious that the application of the weight function of an internal crack in an infinite body to the crack problem in the chevron-notched specimen will not lead to satisfactory results, the accuracy of the results obtained by using Eq. 3.4 was examined by comparison with results obtained from finite element modeling. The details of the finite element model and stress intensity factor calculations will be presented later in this chapter. For this analysis a constant stress, \(\sigma_a\), was placed over the triangular wake of the crack in chevron-notched flexure specimen and the stress intensity factor at each crack length was obtained by calculating the average value of the stress intensity factor distribution along the crack front. The results from the finite element modeling are compared with the results obtained by employing Eq. 3.4 in Fig. 3.1.

The results presented in Fig. 3.1 indicate that Eq. 3.4 does not adequately describe the shape and the magnitude of the stress intensity factor arising from the wake loading of the crack in the chevron-notched specimen. The major inadequacy of Eq. 3.4 is, as pointed out, in employing an improper weight function. All weight functions follow the form:

\[
h = \sqrt{\frac{2}{\pi a}} \left( \frac{1}{\sqrt{1 - \rho}} + \sum_{n=1}^{\infty} B_n (1 - \rho)^{(2n-1)/2} \right), \quad \rho = x/a
\]  
(3.5)

where the singular term dominates the near-tip region and the further coefficients, \(B_n\), are functions of the crack length \(a\) and depend on the special specimen type. As can be seen by comparing Eq. 3.3 with Eq. 3.5, the weight function for an internal crack in an infinite body contains only the near tip term, \((a^2-x^2)^{1/2}\), i.e., the stress intensity factor at the tip of such a crack configuration is not influenced by the boundaries of the body, which are located in an infinite distance from the crack tip. Clearly, this cannot be the case for the
Fig. 3.1: Comparison of bridging stress intensity factor arising from a constant traction on the wake of the crack in a chevron-notched specimen calculated using different methods.

crack in a chevron-notched specimen or any other finite body. As a crack grows into a finite body, the contribution of the terms involving the $B_n$ coefficients in Eq. 3.5 to the stress intensity factor at the crack tip becomes increasingly dominant. Following the above description we can also see from Fig. 3.1 that the deviation of the results obtained by using Eq. 3.4 from the actual value becomes more severe as the crack extends deeper into the chevron notch.

3.2.2 Slice Synthesis of the Crack Wake using the Weight Function for a Through-Thickness Crack

As a better alternative to Eq. 3.4, an averaged stress intensity factor may be obtained for the crack surface tractions in the chevron-notched specimens by considering the wake of the crack in this geometry to be composed of an infinite number of slices with through-thickness cracks, each of a different initial notch depth. This is shown schematically in Fig. 3.2. We may then treat each slice in the model as a straight-through crack and attempt to calculate an average stress intensity factor from the stress intensity factors produced by loading the wake of each of these slices. This can be treated as an analogy to the approach used in the well-known Bluhm's Slice Model [32], which was developed to calculate the compliance of the chevron-notched specimen from the sum of the compliance of many slices, each considered as a specimen with a straight-through crack under the same loading condition.

First, consider again the stress intensity factor resulting from constant surface tractions, $\sigma_o$, which can be obtained by averaging the stress intensity factors arising from $n$ slices:
where $K_{w,CN}$ refers to the stress intensity factor due to loading of the triangular crack wake in a chevron-notched specimen and $K_i$ is the stress intensity factor arising from the wake loading of one slice in the model.

\begin{equation}
K_{w,CN} = \frac{\sum_{i=1}^{n} (K_i)_i}{n}
\end{equation}

![Diagram](image)

**Fig. 3.2:** Schematic of the crack plane in a chevron-notched specimen and the slice representation of the crack wake. The shaded area on the crack plane represents the bridging interaction for one slice and the variable $\zeta$ defines the local position of the notch depth for each individual slice in the model.

To calculate the $K_i$'s in Eq. 3.6, Eq. 3.4 can be used along with an appropriate weight function describing the same specimen with a through-thickness crack, e.g., in the case of a flexure bar specimen the weight function for an edge crack in a rectangular plate [31]:

\begin{equation}
h(x,a) = \sqrt{\frac{2}{\pi a}} \frac{1}{\sqrt{1-x/a}} \left[1 + \sum_{\nu,\mu} A_{\nu,\mu} \alpha^\mu (1 - \frac{x}{a})^{\nu+1}\right]
\end{equation}

where $x$ is the distance along the crack of length $a$, $\alpha=a/W$, $W$ is the thickness of the plate in the direction of crack growth and the coefficients $A_{\nu,\mu}$ are constants for the weight function given in Appendix I.a. Rewriting Eq. 3.6 using integral instead of summation notation gives, after changing the variables and the direction of the integration:
where $K_{w,ST}$, the stress intensity factor arising from crack surface loading of a straight-through crack of initial notch depth $a_o$ by the constant traction $\sigma_o$, is given, following Eq. 3.2, by:

$$K_{w,ST} = \sigma_o \int_{a_o}^{a} h(x,a) dx$$

(3.9)

The results predicted by Eq. 3.8 are shown in Fig. 3.1 by the dashed line. We can see from the results in Fig. 3.1 that the slice approach describes the trend in the stress intensity factor arising from loading of the crack wake surfaces in a chevron-notched specimen well, although the magnitude is overestimated.

To refine the slice approach further, a correction factor may be introduced before the integral in Eq. 3.8 to obtain a better estimation of the magnitude of the stress intensity factor. Such a correction factor can be obtained by comparing the finite element results with the analytical ones. This method was further explored by finite element modeling of two additional distributions of the tractions over the crack wake: linearly increasing and linearly decreasing towards the crack tip. The results of this analysis indicated that the correction factors required were dependent not only on the crack length but also on the shape of the traction distribution. Such a dependency makes this approach unsuitable for quantitative analysis of the $R$-curve, although it may be sufficiently accurate for many purposes and would be preferred to the use of Eq. 3.4.

### 3.2.3 Weight Function for Crack Surface Tractions by Finite Element Modeling

In the previous section we attempted to obtain the $K_{sr}$ term for a chevron-notched specimen by implementing the weight function of a straight-through crack in a slice approach and observed that the accuracy of the results was dependent on obtaining a correction factor. If we had the weight function for the crack surface tractions in a chevron-notch specimen, the analysis of the $K_{sr}$ would be simplified to a large extent. Unfortunately, such a weight has not appeared in the literature, nor could we employ the Bluhm's Slice Model to obtain a weight function from an available weight function of a straight-through geometry.

In the following section, a numerical solution is presented for a weight function for crack wake tractions in a chevron-notched specimen. The weight function is obtained by three-dimensional finite element analysis of chevron-notched flexure specimens of specific geometries. The analysis will consider the stress intensity factor arising from a line load acting in the wake of the crack at different positions between the notch apex and the crack tip.
3.2.3.1 Finite Element Modeling and Calculations

Due to the symmetry of the problem, only one quarter of the specimen was modeled. The specimens considered were flexure bars with W/B=1.25, L/W=8, a/W=1, and a0/W=0.22, 0.32, 0.42. The parameters describing the notch geometry are shown in Fig. 3.2; \( L \) is the specimen length. The Poisson's ratio was taken as 0.28. These geometries were modeled in ABAQUS\(^4\) using 9368 nodes, 2012 elements and about 28100 degrees of freedom. Combinations of quadratic isoparametric and quarter-point elements [33,34] were used to model the specimen. To simulate the stress field singularity at the crack tip eight quarter-point elements, in the shape of collapsed hexahedrons, were used at the crack tip. The length of the crack front was divided into five such segments. The rest of the model was built up using quadratic isoparametric, hexahedron and pentahedron, elements. An example of the finite element mesh is shown in Fig. 3.3.

![Finite Element Mesh](image.png)

Fig. 3.3: Finite element mesh used for the analysis of the chevron-notched flexure specimen in this work.

3.2.3.2 Stress Intensity Factor and Weight Function Calculation

In a one-dimensional problem, the weight function, \( h(x,a) \), is the stress intensity factor at the tip of a crack of length "a" resulting from a point load at a position \( 0 < x < a \). For the current problem, where the crack geometry must be described in two dimensions, the concept of the point load was replaced by the line load. The line load, \( P \), was applied in such a way that the ratio of \( P \) over \( b(x) \), the local width of the crack wake at distance \( x \) from the notch mouth, was kept constant throughout the entire analysis. The stress intensity factor, \( K \), was also assumed to be constant along the crack front and to have a value equal to the average over those calculated at each position along the crack front. The calculated results, to be shown

\(^4\) ABAQUS/STANDARD Ver. 5.4. Hibitt, Karlsson & Sorensen, Inc., Pawtucket, RI 02860-4847
later, indicate that this is a fair assumption for the current problem, as the calculated values for $K$ vary only slightly over $>70\%$ of the crack front length, from mid plane towards the corner of the notch, although the values do increase rapidly approaching the intersection of the crack front and the notch. This is in agreement with previous finite element results by Raju and Newman [35] on short bar and short rod chevron-notched specimens. Also, in the work by Raju and Newman the stress intensity factor results calculated from the compliance of the specimen are in good agreement with the average of the stress intensity factor distribution along the crack front. This points toward the fact that the average of the stress intensity factor along the crack front in a chevron-notched specimen adequately describes the stress intensity factor at the crack tip.

The value of the weight function for the crack surface tractions at any arbitrary position, $x$, can be found by dividing the average stress intensity factor along the crack front, resulting from the application of the line load $P$ at the position $x$, by the load per unit length:

$$h(x,a) = \frac{K(a)}{P / b(x)}$$

where $b(x)$, the local width of the crack wake, is equal to $B(x-a_o)/(d_0-a_o)$.

There are several methods which allow one to estimate the magnitude of the stress intensity factor at the crack tip using finite element analysis, e.g., crack opening displacement [36], and nodal force [37]. Here a $J$-integral based method [38] was used to estimate the stress intensity factor at the crack tip. This method of stress intensity evaluation is a robust technique as it is less sensitive to the accuracy of the displacement solution in the near tip region and allows accurate evaluation of the stress intensity factor at the crack tip even using a relatively coarse mesh [39]. Three $J$-integral contours are calculated at each nodal position along the crack front by employing the $J$-integral routine in the finite element code and the stress intensity at that position is calculated from the average value of the three $J$-contours. The condition of plane strain is assumed and the $J$-results are converted to stress intensity factor, $K$, using:

$$K = \sqrt{JE^*}$$

where $E^*$ is the plane strain elastic modulus, $E/(1-\nu^2)$.

Some typical results for the distribution of $K$ along the crack front are shown in Fig. 3.4. As pointed out earlier, it can be seen that the value of the $K$ is almost constant along the major portion of the crack front and tends to grow rapidly when approaching the intersection of the crack front and the notch. This is in agreement with the earlier studies by Raju and Newman [40] and Ingraffea et al [41] who observed the same type of stress intensity distribution. However, as discussed by Raju and Newman [40], finite element analysis cannot adequately evaluate the stress intensity factor at locations where the crack front intersects
another boundary. In fact, the results of this study also indicated a higher scatter in the values of the J-integral between different contours passing through the nodes, which are located at this intersection.

As no analytical solution is available for the stress intensity factor in the chevron-notched specimen, the calculated values are checked for self-consistency. The J-contours should be path independent for an elastic case. In fact, as indicated in [38], due to inaccuracy in the numerical integration, the values for different contours around the same position would never be identical. The degree of path dependency of the J-contours, however, may be taken as an indication of the quality of the finite element mesh used. Such inspections indicate that the maximum deviation from the average value for the three J-contours at each position along the crack front is about 3%, except for the contours passing through the nodes located at the intersection of the crack front and the notch. In order to assess the overall quality of the finite element model the constraint conditions at the crack front were modified such that a straight-through crack was simulated. Using a procedure similar to that described above, the value of the weight function for a straight through crack was calculated at the x=0 plane of the model for a/W=0.5 at x/a=0 and x/a=0.92, and for a/W=0.80 at x/a=0 and x/a=0.95. The results were then compared with the boundary collocation results for the edge crack in a rectangular plate [31] and found to be in agreement within better than 2.2%.
The numerical results, given in Appendix I.b, for the weight functions obtained through this analysis can be expressed in the crack length range \( \alpha_c \leq \alpha \leq 0.9 \) by the following fit relation (compare with the general form given in Eq. 3.5):

\[
h(x,a) = \sqrt{\frac{2}{\pi(a-a_0)}} \frac{\xi}{\sqrt{1-\xi}} \left[ 1 + \sum_{\nu,\mu=0} A_{\nu\mu} \frac{\alpha^\mu}{(1-\alpha)^{3/2}} (1-\xi)^{\nu+1} \right]
\]  \hspace{1cm} (3.12)

where \( \alpha = a/W \), \( \xi = (x-a_c)/(a-a_c) \) and the coefficients \( A_{\nu\mu} \) are given in Appendix I.c. (It can easily be shown that the dimensionless parameter \( \xi \) results in \( 1/N \) singularity as \( x \) approaches \( a \).) The values calculated by finite element and Eq. 3.12 are plotted in Fig. 3.5 for \( a/W=0.42 \) together with the weight function of the edge crack in a rectangular plate for \( a/W=0.9 \).

---

**Fig. 3.5:** Weight function for the crack surface tractions in chevron-notched specimen with \( a/W=0.42 \). Symbols are finite element results and solid lines are the plots of Eq. 3.12 for the same \( a/W \). Dotted line is the weight function for an edge crack in a finite plate at \( a/W=0.9 \). (Reprinted from Engineering Fracture Mechanics, Vol. 59, G. R. Sarrafi-Nour, T. W. Coyle and T. Fett, "A Weight Function for the Crack Surface Tractions in Chevron-Notched Specimens," pp. 439-445, 1998, with permission from Elsevier Science.)
It is useful to keep the following important points in mind when employing the weight functions obtained:

- The weight functions obtained are strictly valid for the notch geometries modeled, i.e. specific $W/B$, $a/W$ and $a_1/W$.
- The coefficients of the weight functions for any other $a/W$, with $W/B=1.25$ and $a_1/W=1$, within the range $0.22 \leq a/W \leq 0.42$ may be obtained by employing interpolation, e.g. using cubic-spline method, on the coefficients $A_{lw}$ of Eq. 3.12 as a function of $a/W$.
- As long as $L/W \geq 2$, we can expect no effect from specimen length on the value of the weight function. This can be inferred from the Boundary Collocation results obtained by Fett and Munz [31] on the weight function for an edge crack in a rectangular plate.

3.3 Calculation of the R-curve in a Chevron-Notched Specimen

With an appropriate weight function available, the R-curve problem can now be analyzed in the chevron-notched specimen geometries modeled. To furnish some basis for the next section, where a methodology based on the weight function will be developed to extract the characteristic bridging information from a measured R-curve, the evolution of some R-curves for cracks growing in one of the chevron notch geometries considered will be examined. The following specimen geometry is taken as an example: $W/B=1.25$, $a/W=0.32$ and $L/W=8$. This is also specimen geometry used for the experimental R-curve tests on the materials chosen for this study.

When analyzing the effect of the crack wake bridging tractions on the stress intensity factor, it is the shape and magnitude of the R-curve that is of prime interest: how the $K_r$ term in Eq. 3.1 evolves as a result of the operation of a known bridging constitutive relation, $\sigma_0=f(\delta)$. In most circumstances, however, the R-curve is available and the shape and magnitude of the crack wake bridging tractions are to be found. The latter case is more related to the material discipline where microstructural design and characterization of material behavior is of special interest. It should be noted that the basis of the analysis is the same in either case, i.e. starting from Eq. 3.1b. The procedure leading to deconvolution of the bridging tractions from an experimental R-curve obtained using the chevron-notched flexure specimen will be the focus of section 3.3.2.

3.3.1 Evaluation of the R-curve Arising from Bridging Stresses

Consider a four-point flexure specimen containing a chevron notch of the given geometry and assume that the material has grain-bridging behavior. The objective is to predict the R-curve that would arise as a result of the bridging interactions in such specimen. For the case of frictional grain bridging Mai and Lawn [32] have proposed a model which relates the bridging stress in a single bridging element to the crack opening displacement through a power law relation:
\[ \sigma_{br} = \sigma_o (1 - \delta / \delta_c)^m, \quad m = 0, 1, 2, \ldots; \quad \delta / \delta_c \leq 1 \quad (3.13) \]

where \( \sigma_o \) is a characteristic bridging stress, \( \delta_c \) is the critical displacement beyond which the bridging stress vanishes and \( m \) is the strain-softening exponent, most frequently taken as equal to 1 to represent pure friction. The grain size, and thus the critical displacement, are statistically distributed. After proper mathematical treatment of the distribution function, Eq. 3.13 yields, for \( m = 1 \) [22]:

\[ \sigma_{br} (\delta) = \sigma_o \exp\left(-\frac{\delta}{\delta_o}\right) \quad (3.14) \]

where \( \sigma_o \) and \( \delta_o \) are characteristic values describing the magnitude and the distribution range of the bridging tractions. Such dependence has been shown to be a good description of the frictional grain bridging in a wide range of coarse-grained alumina materials [22,25,43]. In the presence of crack wake (bridging) tractions, the total stress intensity factor at the crack tip is written following Eqs. 3.1b and 3.2 as:

\[ K_{appl}(a) = K_o + \int_{a_0}^a h(x,a) \sigma_{br}(\delta(x,a)) dx \quad (3.15) \]

and the total crack opening displacement, \( \delta(x,a) \), will be:

\[ \delta_{total}(x,a) = \delta_{appl}(x,a) + \delta_{br}(x,a) \quad (3.16) \]

Since the total crack surface displacement that appears on the left-hand side of Eq. 3.16 is also embedded in the right-hand side of Eq. 3.16, Eq. 3.16 represents an integral equation*.

### 3.3.2 Solution of the Integral Equation Arising from Crack Opening Displacements

The evolution of the R-curve can be predicted by solving the integral equation, Eq. 3.16, which results in the distribution of the bridging stress in the wake of the crack. The contribution of the bridging stress to the COD, \( \delta_{br} \), can be obtained using the relation proposed by Rice [44]:

\* In the presence of the bridging stress, the total stress acting on the crack surfaces is the sum of applied and the bridging stress, \( \sigma_{appl}(x) + \sigma_{br}(x) \). The total crack opening displacement can be written using the weight function \( h(x,a) \) and following Rice [44]:

\[ \delta(x,a) = \frac{1}{E} \int_x^a h(x,a') \left( \int_0^a h(x',a') (\sigma_{appl}(x') + \sigma_{br}(\delta) dx') da' \right) \]
\[ \delta_{br}(x,a) = \frac{1}{EF} \int_{x}^{a} h(x,a') K_{br}(a') da \] (3.17)

which, after inserting \( K_{br} \) from Eq. 3.2 gives:

\[ \delta_{br}(x,a) = \frac{1}{EF} \int_{x}^{a} h(x,a')[ \int_{x}^{a} h(x',a') \sigma_{br}(x') dx'] da' \] (3.18)

For the opening displacement component arising from the applied stress intensity factor, \( \delta_{\text{appl}} \), we use a finite element solution obtained for the chevron-notched bar under the four-point bending condition by employing the finite element model used in section 3.2.3.1. As a result of the variation of the stress intensity factor along the crack front, the COD field will also vary along the crack tip. Consistent with the assumptions made during the weight function calculations, the crack opening displacement is taken as the average of the opening displacements at each position \( x \) over the width of the crack. The crack opening displacement results from the finite element analysis, converted into crack opening compliance, can be expressed by the following fit relation:

\[ \frac{BE}{P} \delta(x,a) = \sum_{n=0}^{d} C_n(a) \left(1 - \frac{x}{a}ight)^{n+1/2}, \quad 0.33 \leq x/W \leq a/W \] (3.19)

with the fit coefficients \( C_n(a) \) given in Appendix I.d for \( 0.4 \leq \alpha \leq 0.9 \).

To obtain a numerical solution for Eq. 3.16 several strategies have been employed, with the successive approximation method being the most popular one [45]. For the current analysis, a solution method based on least-squares procedure was adopted and is described briefly in the following.

In the first step, a polynomial expansion is assumed for the bridging stress as a function of the crack coordinates, \( x \) and \( a \), e.g.:

\[ \sigma_{br}(x) = \sigma_0 \sum_{n=0}^{\infty} A_n (a-x)^n \] (3.20)

where \( \sigma_0 \) is the characteristic bridging stress and \( A_n \) are the coefficients of the bridging stress polynomial. Inversion of the bridging relation, Eq. 3.14, gives:

\[ \delta(x) = -\delta_0 \ln\left( \frac{\sigma_{br}(x)}{\sigma_0} \right) \] (3.21)
Replacing $\delta(x,a)$, $\delta_{br}(x,a)$ in Eq. 3.16 using Eq's 3.18-3.21 and truncating the series in Eq. 3.20 to $N$ terms gives, after rearrangement:

$$-\delta_0 \ln \sum_{m=0}^{N} A_n (a-x)^{m/2} = \delta_{app}(x) + \sigma_0 \sum_{m=0}^{N} A_n \chi_n(x)$$  \hspace{1cm} (3.22)

with

$$\chi_n = \int_{x}^{a} h(x,a') \int_{a_0}^{x'} h(x',a')(a'-x')^{m/2} \, dx' \, da'$$

The coefficients, $A_n$, of the bridging stress polynomial can be obtained by using a least-squares procedure such that both sides of Eq. 3.22 are the same within a prescribed error. The bridging stress intensity factor, and thus the R-curve, can be obtained by inserting the bridging stress distribution, Eq. 3.20, into Eq. 3.2.

### 3.3.3 Examples of R-curves Due to an Exponential Bridging Relation

The R-curves shown in Fig. 3.6 and Fig. 3.9 are calculated using the scheme described in the previous section, with the series in Eq. 3.22 truncated to 10 terms ($N=9$), for a model material with the following properties: $E'=400$ GPa, $K_0=2.5$ MPa.$\sqrt{m}$, $\sigma_0=20, 40$ MPa, $\delta_0=0.25, 0.50, 0.75 \mu m$.

![R-curves](image)

**Fig. 3.6:** Calculated R-curves resulting from an exponential bridging relation in the chevron-notched flexure bar; characteristic bridging stress and displacement values are shown on the plot. (Reprinted from Ceram. Eng. Sci. Proc., Vol. 19, Iss. 4, G. R. Sarrafi-Nour, T. Fett and T. W. Coyle “Finite Element Analysis of Crack Wake Tractions in Chevron-Notched Bend Bar,” pp. 193-201, 1998, with permission from The American Ceramic Society.)
Example crack profiles and bridging stress distributions in terms of the crack coordinates calculated during the solution procedure are shown in Fig. 3.7 and Fig. 3.8, respectively.

![Graph showing bridging stresses distribution](image)

**Fig. 3.7:** Examples of the distribution of the bridging stresses resulting from an exponential bridging relation, characteristic values $\sigma_c=20\text{MPa}$ and $\delta_c=0.25\text{um}$, over the crack wake in a chevron-notched flexure specimen. (Reprinted from Ceram. Eng. Sci. Proc., Vol. 19, Iss. 4, G. R. Sarrafi-Nour, T. Fett and T. W. Coyle “Finite Element Analysis of Crack Wake Traction in Chevron-Notched Bend Bar,” pp. 193-201, 1998, with permission from The American Ceramic Society.)

![Graph showing opening displacement](image)

**Fig. 3.8:** Examples of different profiles for a chevron notch crack obtained during R-curve calculations; $\sigma_c=20\text{MPa}$ and $\delta_c=0.5\text{um}$. (Reprinted from Ceram. Eng. Sci. Proc., Vol. 19, Iss. 4, G. R. Sarrafi-Nour, T. Fett and T. W. Coyle “Finite Element Analysis of Crack Wake Traction in Chevron-Notched Bend Bar,” pp. 193-201, 1998, with permission from The American Ceramic Society.)
It is useful at this point to look at the effect of the crack geometry on the shape and magnitude of the R-curve by comparing the R-curves arising from the same constitutive bridging relation in a straight-through notched flexure specimen. The straight-through notched specimen is considered to have the same geometry and notch depth and be under the same loading condition as considered for the chevron-notched specimen.

The R-curves for the flexure specimen with a straight-through crack are calculated by interpolating the bridging stress intensity factor solutions presented by Fett and Munz [45]. The solutions in [45] were obtained using the same methodology and the weight function for an edge crack in a rectangular plate (Eq. 3.7). These R-curves are compared with those calculated for the chevron-notched specimen in Fig. 3.9.

Fig. 3.9: Comparison of the R-curves evolving from the exponential bridging law in the chevron-notched flexure specimen (lower family of curves) and in the same specimen containing a straight-through crack (upper family of curves); \( \sigma_c = 40 \) MPa. (Reprinted from Ceram. Eng. Sci. Proc., Vol. 19, Iss. 4, G. R. Sarrafi-Nour, T. Fett and T. W. Coyle "Finite Element Analysis of Crack Wake Tractions in Chevron-Notched Bend Bar," pp. 193-201, 1998, with permission from The American Ceramic Society.)

These results illustrate that the same constitutive bridging relation and material properties give two essentially different R-curves, both in shape and magnitude, for the two crack geometries. R-curves from a chevron-notched specimen show a moderate rise during initial crack growth compared to the rise, proportional to \( \sqrt{\Delta a} \), within the same crack growth region for a straight-through crack.
3.4 Determination of the Bridging Stress Distribution from the R-curve

In the previous section, the weight function method was employed to estimate the shape and magnitude of the R-curve in the chevron-notched specimen under the assumption of a known constitutive bridging relation. In this section, we will focus our efforts on a reverse scheme to obtain the distribution of the bridging tractions as a function of crack profile from the R-curve results. Such method has been developed and successfully applied by Fett [22] and Fett et al [25] to the R-curve results obtained from flexure specimens of different aluminum oxide materials containing a through-thickness crack under the assumption of a known constitutive bridging relation. The fundamentals of this approach are based on the relations governing the R-curve behavior, i.e., Eq. 3.1b, Eq. 3.15 and Eq. 3.16.

3.4.1 Determination of the Parameters from a Known Bridging Relation

When the R-curve and the constitutive bridging relation are both known, the parameters of the bridging relation can be obtained by fitting Eq. 3.15 to the measured R-curve through different iterative strategies, e.g., least-squares. This may be accomplished by moving backward through a scheme similar to the one presented by Eq. 3.22-Eq. 3.14. An easier approach, as described by Fett et al [25], could be based upon using tabular solutions obtained for Eq. 3.16 under the specific constitutive bridging relation. In this case, the iterative scheme needs only to search the solution tables for a set of parameters of the constitutive relation that results in the best fit of Eq. 3.15 to the measured R-curve.

3.4.2 Determination of an Unknown Bridging Relation

In many circumstances, as in this work, the bridging relation is unknown and the objective is to characterize the constitutive relation of the material through R-curve measurements. When only the R-curve is available from the experiment, the $K_{appl}$ from the left-hand side of the Eq. 3.15 is known and we can still fit the right-hand side of this equation to the measured R-curve. Our iterative scheme, however, cannot directly proceed by solving Eq. 3.16. This is because a numerical solution for Eq. 3.16 may be feasible only when the constitutive bridging relation is known or a certain form has been assumed for such a relation. To tackle this problem, Eq. 3.16 is temporarily ignored and a polynomial distribution for the unknown bridging stress as a function of crack coordinates is assumed. The polynomial expansion of the shielding stresses introduced in [46] may be found suitable for this purpose:

$$
\sigma_{br}(x,a) = \sum_{i,j=0} C_{ij} (a - x)^i a^j
$$

There are two main advantages in adopting such a form for the bridging stress. The bridging stress distribution function presented in Eq. 3.23 makes no explicit reference to a specific constitutive bridging relation, nor does it make any specific assumption regarding the shape of the crack profile with respect to
the position along the crack wake as is often done, e.g., the well known linear profile assumption [2]. It is important to appreciate that, when going backward from the R-curve towards the distribution of the bridging stress, the goal is to find out about a distribution from a set of final integral products and that a large number of possible distributions could give rise to the same integral value. In order to reduce this mathematical complexity, bounds can be placed on the bridging stress polynomial function, Eq. 3.23, based on knowledge of the mechanics of the specific bridging behavior, e.g., in the case of this work the frictional pullout bridging. These constraints can be formulated as follows:

- \( \sigma_{br} \geq 0 \) for all \( x \), and \( a_o \leq x \leq a \): \( K_o = K_{appl} - K_{br} \) and, therefore, positive sign for \( \sigma_{br} \).

- \( \frac{\partial \sigma_{br}}{\partial x} \geq 0 \) for all \( x \), \( a_o \leq x \leq a \): the pullout bridging stress vanishes in the crack wake.

- \( \sigma_{br}(x=a, a) = \text{constant} \) for all \( a \)

Although implementing such constraints into an iterative scheme may lead to some complexity, they are useful in avoiding convergence into solutions that are not physically meaningful. Using Eq. 3.23, Eq. 3.15 can now be re-written as:

\[
K_{appl}(a) = K_o + \sum_{i,j=0}^{n} C_{ij} \cdot a^j \cdot \int_{a_o}^{a} h(x,a) \cdot (a-x)^i \, dx
\]  

(3.24)

where the coefficients \( C_{ij} \) are unknown. The value of \( K_o \) is either known from an independent test or can be taken as unknown along with the coefficients \( C_{ij} \). With respect to ceramics reinforced with a discontinuous second phase, the matrix toughness has been used in the past to represent the value of \( K_o \), e.g., in work by Becher et al on SiC-reinforced alumina [47]. It has to be noted that in reinforced ceramics, \( K_o \) can also be influenced by non-shielding toughening mechanisms operative at the tip of the crack, e.g., crack deflection due to the second phase or crack branching. Therefore, assigning the matrix toughness to \( K_o \) can lead to overestimation of the crack wake contribution by the amount of the non-shielding component. In some studies conducted using a chevron-notched specimen, the value of \( K_o \) has also been estimated by extrapolation of the R-curve to \( a_o \), the initial notch depth [12,17]. As can be seen from the weight function and from the R-curves calculated in the preceding section, in the presence of a finite bridging stress at very small COD's close to the crack tip \( K_{br} \) vs. crack extension rises in a square-root shape as \( a \) approaches \( a_o \), thus making accurate estimation of \( K_o \) in a test involving stable crack growth impossible. However, a more critical source of error, with respect to a chevron-notched specimen, in such an estimation may arise from the difficulty in separating the onset of non-linearity, and therefore the onset of crack growth, from the linear regime of the load-displacement curve. Since the geometric function for the crack in a chevron-
notched specimen (as defined by Eq. 2.10) also has a square-root singularity at \( a = a_\circ \), underestimation of the crack length in this region can lead to erroneously large values for \( K_{\text{app}} \) in the very initial steps of crack growth. (To the author's best of knowledge and experience, the portion of the R-curve corresponding to the crack length region close to the onset of non-linear behavior in the load-displacement curve may not be calculated correctly by using the customary approach based on the compliance solution. Additional reasons for this could be following. First, the actual notch tip geometry deviates from the ideal case, where the apex is considered as sharply pointed and this leads to a non-singular behavior. Second, the shape of the crack front from the crack that initially bursts into the apex of the notch may deviate from the equilibrium straight-line shape. Third, the actual geometric function in the short crack length region may be sensitive to the notch-width, similar to the case of through-thickness notched specimens [48].) Therefore, if not available from an accurate independent test, the best approach would appear to be to take the value of \( K_c \) as an unknown parameter along with the coefficients of the bridging stress polynomial.

The best set of coefficients \( C_i \) and \( K_c \) can be determined by using a least-squares iterative scheme based on Eq. 3.24 and the constraints described above, such that:

\[
\sum (K_{\text{app}, \text{calculated}} - K_{\text{app}, \text{measured}})^2 = \min \tag{3.25}
\]

This procedure will yield the distribution of the bridging stress as a function of position along the crack wake for each crack length within the range of the measured R-curve.

The crack surface displacements arising from the bridging stress distributions, \( \delta_{\text{cr}} \), can be obtained by inserting the bridging stress polynomial, Eq. 3.23, in Eq. 3.18. Combining these data with the crack opening displacement arising from the applied load, \( \delta_{\text{app}} \), from the finite element solution, Appendix I.d, will result in the total crack opening displacements, \( \delta_{\text{total}}(x,a) \), described by Eq. 3.16. Finally, the bridging relation, \( \sigma_B = f(\delta) \), can be obtained by correlating the bridging stress results from Eq. 3.23 with the crack opening displacement results, \( \delta_{\text{total}}(x,a) \), through the crack coordinates \( x \) and \( a \).

This numerical scheme constitutes the major tool used to analyze the experimental R-curve results that will be presented in chapter 5.

3.5 Summary

The problem of the R-curve arising from the crack wake bridging interactions during propagation of the crack in a chevron-notched specimen was analyzed by employing fracture mechanics weight function method. In the absence of the exact weight function for crack surface tractions in a specific chevron notch geometry a slice approach was shown to be adequate for qualitative analysis of the stress intensity factor arising from bridging stresses. To accomplish the higher accuracy required for a quantitative assessment of
the R-curve problem, finite element analysis was next employed to obtain numerical solutions for this weight function in the chevron-notched flexure specimen geometries of interest.

The utility of the numerically derived weight functions was demonstrated by calculating theoretical R-curves arising from a specified constitutive bridging relation and the geometry dependence of the R-curve behavior due to bridging interactions was revisited by comparing some of the calculated R-curves with those calculated for the same constitutive bridging relation acting in the same specimen but containing a through-thickness crack.

Finally, a methodology based on the fracture mechanics weight function method was devised to iteratively extract the constitutive bridging relation from an experimentally measured R-curve using one of the chevron-notch geometries analyzed. This methodology will be utilized in the proceeding chapters to analyze and study the temperature dependence of bridging stresses in two different SiC-reinforced alumina ceramic composites.

3.6 Remarks on the Evaluation of the Toughness by Chevron-Notched Specimens in the Presence of R-curve Behavior

Another implication of the R-curve analysis presented in section 3.3.3 relates to the accuracy of the toughness calculation from the maximum load, \( P_{\text{max}} \), and the minimum value of the geometry correction factor, \( Y_{\text{min}} \), for the chevron-notched specimen when the material shows R-curve behavior due to bridging effects. This has been discussed qualitatively by Munz [49]. For the chevron-notched bar considered in this study \( Y_{\text{min}} \), calculated based on the finite element compliance solution and using Eq. 2.10, occurs at a relative crack length of about 0.48. The maximum load in a fracture test using chevron-notched specimen corresponds to the tangency point between the R-curve and the applied stress intensity factor curve. This is quantitatively shown in Fig. 3.10 for one of the calculated R-curves of Fig. 3.6. When the R-curve is flat, the tangency occurs at \( Y_{\text{min}} \) and, therefore, fracture toughness can be calculated unambiguously from the maximum load. In the presence of the R-curve, however, the tangency occurs at a crack length beyond the one corresponding to \( Y_{\text{min}} \). For the R-curves shown in Fig. 3.6 and Fig. 3.9 this tangency occurs \( 0.5 < \alpha < 0.65 \); for the R-curve shown in Fig. 3.10, the tangency occurs at \( \alpha W=0.53 \). It can also be seen that the magnitude of the \( K_{\text{br}}/K_c \) at such crack lengths is not negligible in any of the data sets presented in Fig. 3.6 and Fig. 3.9.
Fig. 3.10: Representation of the tangency condition of the applied stress intensity factor (dotted lines) in a chevron-notched flexure specimens to the R-curve (solid line) calculated based on the exponential bridging relation with $\sigma_p=20$ MPa, $\delta_0=0.75$ µm and $K_e=2.5$ MPa$\sqrt{m}$.

References


Chapter 4

Experimental Procedure

4.0 Introduction

Improvement in the fracture resistance of ceramic materials has often been achieved by introducing high strength ceramic whiskers [1-3]. Simultaneous improvements in both toughness and strength obtained by incorporating whiskers in a number of ceramic matrices have made this type of reinforcement a very unique and attractive choice. Another type of reinforcement of some potential interest are plate-like particles or platelets [4-11], which seem to give toughness improvements comparable to that observed in their whisker-reinforced counterpart. However, the strength performance of the composites containing platelets seems to have been compromised, as all such composites show strength values lower than the parent unreinforced matrix [4,8,10,11]. This behavior can be related to an increased flaw size in the microstructure due to the presence of larger platelets. Another aspect of the mechanical properties of platelet-reinforced materials that has attracted some interest is the R-curve behavior. Some studies have indicated that in such systems crack wake bridging by the platelets can significantly contribute to the crack tip shielding and thus toughening and R-curve behavior in the material [11-13]. Although, as discussed in preceding chapters, an R-curve is not a material property due to its dependence on the crack/loading geometry, the unique underlying bridging stress that evolves as a function of the crack opening displacement is characteristic of the specific microstructure and reinforcement/matrix interface. In order to compare the bridging response at room and elevated temperatures resulting from whisker and platelet reinforcements, two aluminum oxide based composites were chosen for this study and are described in the following.

4.1 Materials, Processing and Fabrication

4.1.1 SiC-Whisker-Reinforced Alumina

The composite chosen for this purpose was a commercially available cutting tool material (Grade WG-300, manufactured by Greenleaf Corporation, Saegertown, Pa), which contains 33 vol.% SiC whiskers in an alumina matrix. This composite is produced in the form of plates by uniaxial hot-pressing of a powder blend, prepared through a proprietary process, containing both alumina and SiC whiskers. The basic properties of this material are given in Table 4.1 based on the information given by Han et al [14] and by the manufacturer. The hot-pressed, high purity composite is almost theoretically dense and exhibits very clean boundaries with very little or no glassy phase at both matrix grain boundaries and whisker/matrix interfaces [14,15]. A typical microstructure of this composite is shown in Fig. 4.1a. Regions
of reinforcement clustering are evident in the micrograph resulting in whisker rich and whisker poor regions in the microstructure.

Table 4.1: Properties of the SiC-reinforced alumina

<table>
<thead>
<tr>
<th>Property</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Composition</td>
<td>Al₂O₃ + 33 vol.% SiC whiskers</td>
</tr>
<tr>
<td>Impurities</td>
<td>Trace amounts of: Ca, Fe, K, Mo, S and Si</td>
</tr>
<tr>
<td>Average matrix grain size</td>
<td>~1.5 μm</td>
</tr>
<tr>
<td>Whisker size</td>
<td>Diameter: 0.1-1.0 μm, aspect ratio: 10-100</td>
</tr>
<tr>
<td>Color</td>
<td>Green/Grey</td>
</tr>
<tr>
<td>Density</td>
<td>3.74 g/cm³</td>
</tr>
<tr>
<td>Porosity</td>
<td>Gas-tight</td>
</tr>
<tr>
<td>Young’s Modulus</td>
<td>393 GPa</td>
</tr>
<tr>
<td>Shear Modulus</td>
<td>158 GPa</td>
</tr>
<tr>
<td>Poisson’s ratio</td>
<td>0.23</td>
</tr>
<tr>
<td>Strength (4-point bend)</td>
<td>690±41 MPa</td>
</tr>
<tr>
<td>Weibull parameter</td>
<td>16</td>
</tr>
</tbody>
</table>

The samples for this study were received in two batches of machined flexure specimens with nominal dimensions \( W=5.00 \text{ mm}, B=4.00 \text{ mm}, L=47\text{mm} \) and \( W \) oriented parallel to the hot-pressing axis. This resulted in both the crack plane and growth direction aligned parallel to the hot-pressing axis. It is known that hot-pressed whisker-reinforced materials show preferred whisker orientation on the plane perpendicular to the pressing axis, thus resulting in lateral isotropic microstructure [16,17]. The preferred orientation affects the toughness of the composite in different directions [18].

4.1.2 SiC-Platelet-Reinforced Alumina

The system chosen for this purpose was a composite of alumina containing 20 vol.% SiC platelets. The choice of this system was made based upon a previous study [19], which examined the R-curve behavior of this material in the short-crack regime by performing in-situ crack growth observation and indentation strength tests. The composite material was prepared from a commercially available alumina oxide powder (APA 0.5, Ceralox Corporation, Tucson, AZ) and a SiC-platelet powder (~400 mesh, Third Millennium Products, Knoxville, TN) through a hot-pressing process. The properties of the raw materials are shown in Table 4.2a and Table 4.2b based on the information from the suppliers.

The composite material was prepared by mechanical mixing of alumina and SiC-platelet powders in an attrition mill using zirconia milling media and ethanol as vehicle. Following a two-hour deagglomeration of the alumina powder in the attrition mill, the SiC-platelets were added into the mill and mixed with the alumina for 15 minutes at low speeds to avoid milling of the platelets.
Table 4.2a: Properties of the alumina powder

<table>
<thead>
<tr>
<th>Property</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Composition</td>
<td>99.97% α-Al₂O₃ + 500 ppm MgO</td>
</tr>
<tr>
<td>Impurities (trace)</td>
<td>Ca, Ga, Na, Fe, Mn, Si</td>
</tr>
<tr>
<td>Specific surface area</td>
<td>8.5 m²/g</td>
</tr>
<tr>
<td>Median particle size</td>
<td>0.4 μm</td>
</tr>
<tr>
<td>Sintered density</td>
<td>3.96 g/cm³ (2hrs at 1510°C)*</td>
</tr>
<tr>
<td>Linear shrinkage</td>
<td>17%</td>
</tr>
</tbody>
</table>

*measured on a 10g pellet, pressed at 34.5 MPa in a 2.54 cm diameter die.

Table 4.2b: Properties of the SiC-platelet powder

<table>
<thead>
<tr>
<th>Property</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Composition</td>
<td>99.9% α-SiC</td>
</tr>
<tr>
<td>Platelet diameter</td>
<td>3-30 μm, average=10 μm</td>
</tr>
<tr>
<td>Platelet thickness</td>
<td>0.5-3 μm</td>
</tr>
</tbody>
</table>

After the mixing stage, the zirconia balls were separated from the mixture and the slurry was transferred into a beaker. The vehicle was then evaporated at 80-90°C while stirring the mixture vigorously to minimize settling of the SiC-platelets. The dried powder batch was finally passed through a 150 mesh screen prior to being loaded into the graphite die for hot-pressing.

Two different batches were produced following the route described. The first batch was hot-pressed at 1600°C for 2hrs in vacuum and under 50 MPa of pressure in a 50.8 mm diameter graphite die. The density of the composite, measured by water displacement technique, was greater than 99% of the theoretical density (based on a theoretical density of 3.83 g/cm³ calculated for the composite from the linear rule of mixtures). The average matrix grain size was determined to be about 7.5 μm based on intercept measurements. A large number of matrix grains in this composite were found to show irregular or non-equiaxed shapes. In order to suppress the growth of the alumina matrix grains during hot-pressing, and thus reducing the potential contribution of the matrix grains to the R-curve behavior, a second version of the composite was prepared following the same route with the addition of Y₂O₃ as dopant. The mixed dopant formed by Y₂O₃ and the MgO, present in the as-received alumina powder, has been used in SiCₐ-alumina systems as grain-growth inhibitor [20]. The addition of Y₂O₃ was achieved by using an aqueous solution of Y(NO₃)₃·6H₂O which was added during the milling stage. The amount of yttrium nitrate in the solution was adjusted such that the concentration of each dopant was 200 ppm in the final batch. The composite material produced from this batch under the same hot-pressing condition used for the undoped composition had a relative density of 95%, indicating that the dopant had effectively slowed down the diffusional processes. The results of the hot-pressing study on this composition indicated that in order to
achieve a composite with a relative density of 99%, the doped composition had to be hot-pressed for 1 hr at 1650°C in vacuum and under 50 MPa of pressure. The composite produced using this hot-pressing condition had an average matrix grain-size of about 5.0 μm and the shape of the matrix grains was generally equiaxed. To produce test specimens, the composite was hot pressed in a 76 mm diameter graphite die under the conditions described. Image analysis on cross-sections cut parallel to the hot-pressing axis showed some degree of preferred orientation of the platelet C-axis parallel to the hot-pressing axis and, therefore, this direction was chosen to be parallel to both crack plane and growth direction in the test specimens. A typical microstructure of the platelet-reinforced composite is shown in Fig. 4.1b. The elastic modulus, $E$, and shear modulus, $G$, of the composite were measured ultrasonically at room temperature in the direction parallel to the hot-pressing axis to be 414 GPa and 171 GPa, respectively. From these values a Poisson's ratio of 0.21 was calculated for this composite. No significant variation in the wave velocity was found in the direction perpendicular to the hot-pressing axis.

![Typical microstructure of the materials used in this work: a) SiC-whisker-reinforced alumina, and b) SiC-platelet-reinforced alumina. The relief appearance of the microstructure of the platelet-reinforced composite is due to selective polishing of the alumina matrix during final polishing stage by colloidal silica (Bar=20 μm)](image)

**4.1.3 Specimen Preparation**

Following hot pressing, the disks were sliced and cut into bars with rectangular cross-section. The bars were then machined using a 400 grit diamond wheel on a surface grinding machine to produce beam samples of $W=5.00$ mm, $B=4.00$ mm and $L=45.00$ mm, with $W$ chosen to be parallel to the hot-pressing axis. Some specimens had to be prepared with $W=4.00$ mm, $B=3.20$ mm, and $L=45.00$ mm, resulting in the same $W/B$ ratio but a higher span-to-width, $S/W$, ratio. This was due to technical difficulties with the hot-pressing equipment, which allowed only two batches of this composite to be produced successfully. The samples for testing in air were therefore prepared from cut-off pieces or out-of-tolerance specimens
already made from these two batches. To maintain the same chevron notch geometry amongst all of the specimens a different \( W \) and \( B \) had to be adopted for this set of specimens such that \( W/B = 1.25 \). Proper adjustments of the compliance, geometric function and crack opening displacement from the applied load were made for this group of specimens during the calculations.

The chevron notch was prepared using a fixture mounted on the Y-axis of an X-Y linear stage with a nominal displacement resolution of 0.01 mm mounted on a laboratory precision cut-off machine (Accutom-2, Streuers Ltd., USA) allowing precise positioning of the sample relative to the blade of the machine, as shown in Appendix IIIa. The initial notch depth, \( a_n/W \), of the chevron-notched specimen chosen in this study was equal to 0.32, resulting in a notch apex angle, \( \theta \), of -61.0 degrees. The fixture held the sample during the cutting stage at \( \theta/2 \) relative to the tip of the diamond blade of the cut-off machine. With the angle of the notch apex fixed, the size of \( a_n/W \) (or equivalently \( a_n/W \)) was set by adjusting the Y-position of the specimen relative to the top of the blade. After adjusting the desired \( a_n/W \), notching of the specimen was performed by moving the fixture towards the blade of the machine at a controlled feed rate in the range of 1-2 mm/min. One side of the triangular notch was produced during each pass.

Post-fracture measurements of the initial notch depth, \( a_n \), on a large number of specimens, both glass dummies and actual specimens, showed that the scatter in the \( a_n/W \) was in the order of ±0.01. A typical fracture plane from a chevron-notched specimen produced is shown in Appendix IIIa. Both 300 \( \mu \)m and 200 \( \mu \)m diamond-impregnated blades were used to cut the chevron notch into the specimens. The majority of the samples were notched, however, using the 300 \( \mu \)m thick blade at 2600-3000 rpm. The notch widths produced from these blades were usually 50-80 \( \mu \)m above the nominal blade thickness.

4.2 Fracture Tests

Controlled fracture tests were conducted both at room and elevated temperatures under flexure loading mode. High temperature tests on the platelet-reinforced composite were performed both in air and under vacuum, while the whisker-reinforced composite was tested only in the ambient atmosphere. All of the tests were performed on servo hydraulic load frames. The flexure-testing fixture was made from SiC and conformed to ASTM C1211-9 [21] with outer and inner spans of 40 mm and 20 mm in the four-point and 40 mm span under the three-point flexure conditions. The details of each testing setup are described in the following.

4.2.1 Setup for High Temperature Fracture Test in Air

Testing at high temperatures in air was achieved by designing and building a high temperature testing unit which included the load transfer parts, displacement measuring elements and a clam-shell box furnace with molybdenum disilicide heating elements. This setup was used on the frame of a servo-hydraulic testing machine (Instron 8501, Instron Ltd., Burlington, ON) and is shown in Appendix IIIb. The temperature was controlled and monitored during the test by using a closed-loop feed back control unit
with type S (Pt/10%Rh-Pt) thermocouple which was placed adjacent to one face of the specimen. The loading of the specimen on the fixture was achieved by using an alumina push rod and tube. The load was measured using a 3000lb (13,361 kN) tension/compression load cell. The displacement measurement is discussed separately in section 4.2.3.

4.2.2 Setup for High Temperature Fracture Test under Vacuum

This set up was used during a five-month visit at the Institute for Material Research II, Research Center Karlsruhe, Karlsruhe/Germany. The setup consisted of a load frame (Instron 1196, Instron, Germany), onto which a servo-hydraulic unit (Instron 8500, Instron, Germany) was mounted. The load was measured through a tension/compression load cell with a 5kN capacity. The high temperature environment was provided on this setup by using a resistance furnace with molybdenum semi-cylindrical heating elements. Loading of the specimen inside the furnace was achieved by using solid tungsten rods attached to both the machine actuator and the load cell. The temperature of the furnace was controlled and measured through a closed-loop control unit using a type S thermocouple with its tip embedded into the SiC-fixture close to the position of the sample using an aluminum oxide adhesive. The embedded thermocouple was checked against a standard thermocouple using a thermocouple calibration unit and found to agree with the standard thermocouple within at most 5°C between room temperature and 1000°C. High vacuum, better than 6x10⁻⁵ torr at testing temperatures ≤ 1400°C, was achieved on this setup through a combination of rotary and turbomolecular pumps.

4.2.3 Measurement of Specimen Compliance during Fracture Test

Due to the hidden nature of the notch surfaces in a chevron-notched specimen, crack length cannot be measured in-situ from the traces of the crack front on the notch surfaces during a fracture test. It is therefore very common to estimate the crack length in such specimens through compliance measurement [22-24]. (Some work has also been carried out based on post-loading measurements of the crack length and successive loading/unloading of the specimen [25].)

The loading point compliance is conventionally measured by using a sensor, e.g., linear variable displacement transducer (LVDT), which is mounted on the base or on the actuator of the testing machine and measures the relative displacement at the center-point of the tensile face of the flexure specimen during the test. This displacement is then converted to the specimen compliance by subtracting the so-called machine compliance, usually measured separately by using a known sample under the same loading condition, from the measured compliance. For high temperature experiments, performing such calibration tests accurately can be tedious. Additionally, uncertainties in such measurements would be unavoidable due to settling of the testing apparatus and flattening of the rollers of the loading fixture at their contact points due to creep effects at high temperatures. To perform accurate compliance measurements White et al [26] used a laser interferometry technique and measured the crack mouth compliance on chevron-notched flexure specimen. The crack length was then obtained by using a calibration curve calculated from the
crack mouth compliance by finite element analysis. Structural ceramics possess a high elastic modulus and, therefore, crack mouth opening displacement of the small specimen sizes employed in such tests is very small, usually in the order of a few micrometers. As a result, such displacements may only be measured accurately by employing measurement techniques of high resolutions, e.g., optical interferometry as was used by White et al [26]. This would, in turn, require facilitating a testing environment that satisfies the stringent requirements of an optical measurement, e.g., using a vibration free environment or air table; these may not be readily available in mechanical testing lab.

An easier, yet precise, approach to the compliance measurement is based upon indirect measurement of the loading-point compliance [27-29]. In this technique a relative displacement is measured on the tensile face of the flexure specimen between the midpoint and two other points symmetrically located at some distance on either side of the midpoint on the specimen. This is shown schematically in Fig. 4.2.

Fig. 4.2: Schematic representation of indirect compliance measurement method.

The measured differential displacement, \( \delta_1 \), leads to a fictitious compliance, \( C_f = \delta_1/P \), which may be converted to the true load-point compliance of the specimen, \( C = \delta/P \), through analytical relations. For a specimen without any crack, such an analytical relation can be easily obtained from the fundamentals of elastic beam theory [30]. For a specimen with a through-thickness crack, Hilmsoit [27] derived an analytical relation between the apparent compliance, \( C_f \), and the true compliance, \( C \), by superposition of the two limit cases of pure bending and pure hinge behavior:

\[
C = \kappa C_1 + \lambda C_o
\]  

(4.1)

where \( C_o \) is the compliance of the beam without a crack from bending theory:

\[
C_o = \left( \frac{S_1 - S_2}{W} \right)^2 \frac{1}{BE} \left[ \frac{S_1 + 2S_2}{4W} + \frac{W(1 + \nu)}{2(S_1 + S_2)} \right]
\]  

(4.2)

and the coefficients \( \kappa \) and \( \lambda \) depend only on the testing geometry and can be obtained through the following relations [27,28]:

\[
\text{Fig. 4.2: Schematic representation of indirect compliance measurement method.}
\]
with the geometrical parameters $S_1$, $S_2$, $\beta$ and $c$ defined in Fig. 4.2. The compliance relation described by Eq. 4.1 has been verified both numerically and experimentally for a straight-through notched specimen under the four-point flexure condition [28]. However, the validity of this relation for the chevron-notched flexure specimen was under question and had to be verified. This uncertainty is related to the definition of the $C_0$ term in Eq. 4.1. Obviously, this term could no longer be replaced by the compliance of the solid beam. The first guess about the magnitude of $C_0$, by way of comparison with the specimen containing a straight-through crack, would be the initial compliance of the chevron-notched specimen. In order to verify this hypothesis, finite element analysis was employed and showed that the hypothesis was wrong. In a series of finite element analysis, it was found that for a chevron-notched specimen $C_0$ in Eq. 4.1 had to be replaced by the compliance of the flexure specimen with a straight-through crack of a depth equal to the initial notch depth of the chevron-notched specimen, i.e., $C_{n\text{-through}(a/W)}$:

\[ C = \kappa S_1 + \lambda C_{n\text{-through}(a/W)} \]

The results from finite element verification of Eq. 4.4 are shown in Fig. 4.3 by plotting the deviation of the compliance calculated based on Eq. 4.4, by using $C_f$ results from the finite element analysis, relative to the compliance obtained from the finite element modeling. The ratios $\beta/S_1$ shown on the plot designate the relative position of the point at which the displacement $\delta_f$ was obtained from the finite element model to calculate the fictitious compliance $C_f$. It can be seen that the predictions made by Eq. 4.4 are in very good agreement with finite element results and, therefore, this method could reliably be adopted for the compliance measurements on chevron-notched specimens.

In the actual experimental setup, shown in Appendix IIIb, a single LVDT (DFG-1 Gauging Transducer, ±1 mm measuring range, Solartron Metrology, UK) was used to collect the differential displacement, $\delta_f$. For this purpose, the LVDT was placed at the center of a small seesaw mechanism [31]. The measured displacements from two locations each 15 mm away from the center-point of the specimen (parameter $\beta$ in Fig. 4.2 equal to 5 mm) were transferred by two alumina rods to the two sides of the seesaw and caused the displacement of the body of the LVDT, while the core of the LVDT sensed the displacements of the center-point on the specimen.
Fig. 4.3: Finite element verification of the indirect compliance measurement method for the chevron-notched specimen using Eq. 4.4. The chevron-notch geometry modeled has $W/B=1.25$, $L/W=8$, $a_d/W=0.32$, $a_s/W=1$ and is considered to be under four-point flexure load such that $S'/W=8$ and $S_s/S_t=2$.

4.2.4 Testing Procedure

All of the fracture experiments were carried out under displacement control condition using an actuator speed of 0.050 mm/min. The heating and cooling rates employed for the high temperature tests were 15°C/min and samples were soaked at the testing temperature for 30 minutes prior to initiating the test. A small amount of pre-load, less than 4 N, was used during the heat-up ramp to secure the position of the specimen on the fixture during this stage. A minimum of three successful tests was performed at each condition except for the tests performed on the platelet-reinforced composite in air. Due to lack of a sufficient number of specimens in this case only two specimens were tested at each temperature, except at 1000°C, where three tests were carried out. The load and displacement data were collected during the test by using A/D data-acquisition boards. A 16-bit and a 12-bit data acquisition board were used to collect load and displacement data in the test setups for testing under vacuum and in air, respectively: a 20-40 Hz data acquisition rate was used during all of the experiments. Stable crack growth was achieved for the platelet-reinforced composite specimens under the 4-point flexure condition. However, no stable crack growth could be achieved beyond, or far enough beyond, the maximum load under the four-point flexure condition for the whisker-reinforced material. This can be related to an insufficient stiffness of the load-train of the
Fig. 4.4: Typical load-displacement curves obtained during the fracture tests on the 20vol.%-SiC-platelet-reinforced alumina between room temperature and 1400°C in air. (Specimen size: $W=4.00$ mm, $B=3.2$ mm.)

Fig. 4.5: Typical load-displacement curves obtained during the fracture tests on the 33vol.%-SiC-whisker-reinforced alumina between room temperature and 1400°C in air.
setup used for testing in air and the characteristics of the crack growth process in this composite that will be discussed later. As a result fracture tests on the whisker-reinforced composite were conducted under three-point flexure condition with the span width equal to 40 mm. Typical load-displacement curves obtained during the fracture tests for each composite are shown in Fig. 4.4 and Fig. 4.5.

4.3 Remarks on Compliance Measurement at High Temperatures

An issue of significant influence on the accuracy of the displacements measured using the floating seesaw mechanism (Appendix III.b) at elevated temperatures is the temperature stability of the furnace during the time frame of the controlled fracture test. This becomes especially influential when the three alumina-rods transferring the displacements from the specimen to the seesaw mechanism housing the LVDT (See Appendix III.b) are not exposed identically to the temperature fluctuations. For example, the center alumina rod transferring the displacement from the midpoint-point of the specimen to the core of the LVDT may be passed through the tube supporting the base of the SiC bending-fixture and the two other rods transferring the displacements from the points close to the lower-span rollers run on the outside of the support tube into the furnace. As a result the outside rods can be exposed to direct radiation from the heating elements while the one at the center will be masked form this direct radiation by the tube wall. In an ideal case, the test is performed after a stable equilibrium condition has been established in the furnace. For furnaces controlled by closed-loop PID (Proportional Integral Derivative) controllers, maintaining such a stable equilibrium strongly depends on the tuning parameters adopted for the controller, which may well vary with temperature and furnace load/working condition. This may result in the temperature fluctuating around the set point by a few degrees over a time period of several minutes. With the alumina rods in different thermal environments, there would be a response-lag between the expansion/contraction of the center alumina rod with the ones located outside if such thermal fluctuations occur. Depending on the length of the rods extended into the hot-zone of the furnace and the amplitude of the temperature fluctuations around the set point, this can give rise to a false displacement in the order of a few tenths- to a few micrometers superimposed in either direction (contraction or expansion) on the displacement data. The superimposed displacement, in turn, affects the calculated compliance, crack length and thus the stress intensity factor. In order to maintain accuracy of the compliance measurements using the apparatus described in section 4.2.3 at high temperatures and to avoid distortions of the displacement data, the following recommendations are made:

- The alumina rods should be placed in the same thermal environment; they should ideally be placed inside the tube supporting the loading fixture.

*Theoretical analysis based on energy balance in the machine + specimen system indicated that for the current chevron-notched geometry the ratio of machine compliance to that from the specimen has to be at least < 0.8 in order to maintain the stable crack growth during the test beyond the maximum load.*
The length of the alumina rods extended into the hot-zone should be minimized; using the lowest possible position in the hot-zone is recommended. Alternatively, materials with smaller thermal expansion coefficients, such as SiC or silica-glass rods (softening temperature should be considered, as the rods will be exposed to few MPa of compressive stress due to the load imposed by the springs) may be used for this purpose.

Thermal fluctuations inside the furnace, within the time frame of the test, are to be minimized; ideally kept zero or less than \( \pm 1^\circ C \).

Note that with just 10 cm length of the alumina rods extended into the furnace, a \( \pm 1^\circ C \) temperature oscillation between the rods (\( \alpha_{T=1000^\circ C}=8.4\times10^{-6}/^\circ C \)) can cause the LVDT to read displacement fluctuations in the order 1\( \mu \)m! This amount of the displacement is comparable with the total displacements within the elastic region, usually in the order of a few micrometers. Clearly, larger fluctuations in combination with a larger exposed length of the displacement pickups could also affect the displacement measurement within the inelastic region, too.

References


Chapter 5

Results

5.1 Calculation of the R-curves

R-curves were calculated from the load-displacement information obtained from the fracture experiments by using a compliance solution based on finite element analysis of the chevron-notched specimen. The compliance solution was obtained using the same finite element model as described in chapter 3 under a four-point flexure loading condition with \( S_t = 40 \text{ mm} \) and \( S_x = 20 \text{ mm} \). The compliance results (Appendix I.e) from the finite element analysis can be expressed using the following fit relation:

\[
\lambda(\alpha) = \lambda_0 + (\frac{\alpha}{1-\alpha})^2 \sum_{i=0}^{5} A_i \alpha^i
\]

(5.1)

where \( \lambda(\alpha) \) is the dimensionless compliance (\( \lambda = \frac{BE' \cdot LPD}{P} \); \( B \) the specimen width, \( E' \) the plane strain elastic modulus, \( LPD \) the loading-point displacement and \( P \) is the load), \( \lambda_0 = \lambda(\alpha_0) \) is the compliance of the chevron-notched specimen without any crack and \( A_i \) are the fit coefficients, given in Appendix I.f. Using the compliance relation, Eq. 5.1, the geometric function for the chevron-notched specimen, \( Y(\alpha) \), was calculated from [1]:

\[
Y(\alpha) = \sqrt{\frac{1}{2} \frac{\alpha_1 - \alpha_0}{\alpha - \alpha_0} \frac{d\lambda}{d\alpha}}
\]

(5.2)

The compliance and geometric functions for the three-point bending condition and for the four-point bending condition with \( (S_t - S_x)/W = 5 \) were derived from the geometric function calculated from the finite element compliance solution (Appendix I.d and Appendix I.e) by considering the effect of the span-to-width ratio and were verified using Bluhm’s Slice Model [2] as described in Appendix II.a.

The crack length at any position on the non-linear segment of the load-displacement curve was estimated by calculating the compliance at that position using Eq. 4.4 and comparing this value with Eq. 5.1. It is acknowledged that in the presence of bridging tractions, such method of determination of the crack length may underestimate the real crack length. This is because the same bridging stresses that influence the stress intensity at the crack tip affect the displacement of the crack walls and thus the loading points [4]. However, the R-curve calculated from this so-called “elastic-equivalent crack length”, and hence the bridging tractions deconvoluted therefrom, can be treated as a lower bound estimate.
The applied stress intensity factor at each crack length, $a$, was then obtained by using [1]:

$$K(a) = \frac{P_i}{B \sqrt{W}} Y(a)$$

(5.3)

where $P_i$ is the instantaneous load and $W$ is the thickness of the specimen in the direction of crack growth. Typical $R$-curve results obtained at different temperatures for the SiC-platelet-reinforced alumina and for the SiC-whisker-reinforced alumina are shown in Fig. 5.1 and Fig. 5.2, respectively. The room temperature $R$-curve results for the whisker-reinforced composite are shown separately in Fig. 5.2b. The run/arrest nature of the crack growth phenomena in the whisker-reinforced composite, also visible as saw-tooth features on the load-displacement curves, have been reported previously [4]. They could be observed most profoundly at room temperature (Fig. 5.2b) and diminished as the test temperature was raised. It can be seen from Fig. 5.2b that after each unstable crack run the stable crack growth reinitiates at a lower stress intensity factor indicating that most of the bridges formed in the crack wake have been destroyed. As a result, a direct evaluation of the bridging stresses from the $R$-curves of the composite at room temperature could not be accomplished; however, the lowest stress intensity factor observed at longer crack lengths on these $R$-curves was adopted as $K_0$. (An estimation of the magnitude of the bridging stress at room temperature will be provided at the beginning of Chapter 6 by calculating some $R$-curves and comparing those curves with the initial region of stable crack growth on a measured $R$-curve.)

![Fig. 5.1: Examples of the R-curves from the SiC-platelet-reinforced alumina tested at various temperatures in air.](image-url)
Fig. 5.2: Examples of the R-curves from the SiC-whisker-reinforced alumina; a) typical R-curves at various temperatures, and b) at room temperature.
5.2 Elevated Temperature Elastic Modulus

Calculation of the COD fields, an important element of the analysis methodology presented in chapter 3, required the knowledge of the elastic modulus of the materials used within the temperature range of this study. For the platelet reinforced composite the elastic modulus was measured between room temperature and 1300°C ultrasonically at 15 MHz [5]. For this purpose, the velocity of longitudinal and lateral ultrasonic waves were first measured at room temperature which resulted in the elastic modulus, $E$, shear modulus, $G$, and thus the Poisson's ratio, $\nu$, of the composite. The velocity of the longitudinal waves was then measured as a function of temperature by using a glass composition as coupling medium between the ultrasonic probe and the specimen at high temperature [5]. Under the assumption that Poisson’s ratio was independent of temperature, the measured longitudinal wave velocities were converted to elastic modulus using [6]:

$$\nu = \frac{0.5(C_l/C_t)^2 - 1}{(C_l/C_t)^2 - 1}$$

(5.4)

$$E = 2(1 + \nu)\rho C_l^2$$

(5.5)

where $C_l$ and $C_t$ are the velocity of the longitudinal and lateral ultrasonic waves and $\rho$ is the density of the material. For the whisker-reinforced alumina composite, the elastic modulus was indirectly deduced by comparing the slope of the linear portion of the load-displacement curves with the initial compliance of the specimen from the finite element solution. Since the displacement measurements were carried out directly on the specimens, the initial slope of the load-displacement curve combined with the finite element results ought to provide a reasonably accurate estimate of the plane strain elastic modulus, $E’$. The modulus results from both of the composites are shown in Fig. 5.3.

![Graph showing variation of elastic modulus as a function of temperature](image)

Fig. 5.3: Variation of the elastic modulus as a function of temperature in the SiC-platelet- and SiC-whisker-reinforced alumina. (The results from the whisker-reinforced composite are $E’=E(1-\nu^2)$ and the error bar represents one standard deviation calculated based on the values from three specimens.)
The adoption of $E'$ instead of $E$ for the modulus values determined from the measured compliance within the linear region of the load-displacement curves follows a series of finite element analyses indicating variation of the compliance in such region with variation of $v$ and that the modulus obtained from such determination had a value closer to $E'$ than $E$.

5.3 Deconvolution of $K_c$ and Bridging Relation from the R-curve

Based on the methodology described in section 3.4.2 of this work, each individual R-curve was analyzed to obtain $K_c$ and the distribution of the bridging stress as a function of crack opening profile. The numerical routines required for this analysis were developed using a commercial mathematical software (Matlab 5/Matlab 5 Optimization Toolbox, The MathWorks, Inc., Natick, MA) and Eq. 3.23 was used to obtain a least-squares fit to the R-curve results (the least-squares routine was developed based on the constrained optimization tool available in the Optimization Toolbox of Matlab 5). The fit procedure yielded the unknown parameters $K_c$ and the coefficients of the bridging stress polynomial in Eq. 3.22. An example of a fit curve result and the corresponding distribution of the bridging stress on the crack surfaces are shown in Fig. 5.4 and Fig. 5.5a, respectively.

In the next step, the CODs from the bridging stress distribution and the applied load were calculated using Eq. 3.17 and Eq. 3.18 and the total COD was obtained by summing these two displacements based on Eq. 3.15. Some example crack opening profiles calculated for the R-curve results shown in Fig. 5.4 are shown in Fig. 5.5b. Correlating the bridging stress data from Fig. 5.5a with the COD data from Fig. 5.5b through the crack length and position co-ordinates along the crack path, the distribution of the bridging stress as a function of COD could be obtained and is shown in Fig. 5.6. (It is reminded that the COD on the abscissa of Fig. 5.6 and on the ordinate of Fig. 5.5b correspond to the displacement of one crack wall relative to the crack plane. The actual crack opening displacement is thus obtained by multiplying these values by two.)

One of the difficulties encountered while applying this procedure to some of the R-curves was the convergence of the fitting routine to a set of coefficients for the bridging stress polynomial, which resulted in a dependence of constitutive bridging relation on the crack length, i.e., a non-unique $\sigma_b=f(\delta)$ for different crack lengths. Since such results were of no physical meaning, a more rigorous procedure was employed to obtain a fit to the measured R-curve if such solutions were encountered. In this case, the distribution of bridging stress as a function of COD was calculated for a series of crack lengths within the range of the experimental results during each iteration step by the fit algorithm, i.e., $\sigma_b=f(\delta)$a. In the next step, an average bridging relation, $<\sigma_b>=f(\delta)$, was calculated at overlapping COD points between these dummy crack lengths. The routine was then set to fit Eq. 3.23 to the R-curve results from the experiment such that the deviation of the resulting $\sigma_b=f(\delta)$ at each of these crack lengths from the calculated average distribution was minimized simultaneously with Eq. 3.25, i.e., the bridging stress distribution had to satisfy the additional condition:
Fig. 5.4: A typical fit to an experimental room temperature R-curve from the platelet-reinforced composite by using the procedure described in section 3.4.2. (Reprinted from Ceram. Eng. Sci. Proc., Vol. 19, Iss. 4, G. R. Sarrafi-Nour, T. W. Coyle “Analysis of the Bridging Zone Contribution to the R-curve Behavior of SiC-Platelet-Reinforced Alumina using the Chevron-Notched Bend Bar Specimen,” pp. 203-211, 1998, with permission from The American Ceramic Society.)

Fig. 5.5: a) Distribution of the bridging stresses in the crack wake, and b) calculated crack opening profile due to the applied load without the effect of the bridging stresses (dashed line) and under the influence of the bridging stresses. The data in both of the plots relate to the fit results of Fig. 5.4. (Reprinted from Ceram. Eng. Sci. Proc., Vol. 19, Iss. 4, G. R. Sarrafi-Nour, T. W. Coyle “Analysis of the Bridging Zone Contribution to the R-curve Behavior of SiC-Platelet-Reinforced Alumina using the Chevron-Notched Bend Bar Specimen,” pp. 203-211, 1998, with permission from The American Ceramic Society.)
Although the numerical implementation of this procedure may be somewhat difficult to elaborate, its effectiveness in converging to solutions with self-consistent constitutive bridging relations is certainly attractive.

\[
\sum (\sigma_{br}(\delta)_{ij} - \langle \sigma_{br}(\delta) \rangle)^2 = \min
\]  

(5.6)

By analyzing all of the R-curves, the variation of \( K_o \) and the distribution of bridging stresses as a function of COD were obtained at different temperatures. The results of this analysis are presented in Fig. 5.7 through Fig. 5.12. The maximum value of the bridging stress, \( \sigma_{br,max} \), is shown as a function of temperature in Fig. 5.7 and Fig. 5.8 for the platelet-reinforced and whisker-reinforced composite, respectively. For comparison, some literature results \([7,8]\) for the maximum bridging stress found in a 30vol.% SiC-whisker-reinforced alumina at room temperature are also shown in Fig. 5.8. Some typical constitutive bridging relations deconvoluted from the R-curves of each composite are shown in Fig. 5.9 and Fig. 5.10 at different temperatures to demonstrate the variations in the interaction range of the bridging stress with temperature in each material. The variation of \( K_o \) with temperature for each composite is shown in Fig. 5.11 and Fig. 5.12.
Fig. 5.7: Variation of the maximum value of bridging stress deconvoluted from the R-curves of the SiC-platelet-reinforced alumina at various temperatures. The error bar on the data from high temperature tests under vacuum shows the scatter of the results from different specimens about the average value.

Fig. 5.8: Variation of the maximum value of bridging stress deconvoluted from the R-curves of the SiC-whisker-reinforced alumina at various temperatures. The error bar on the data shows the scatter of the results from different specimens about the average value.
Fig. 5.9: Typical constitutive bridging relations obtained from the analysis of the R-curves of the SiC-platelet-reinforced alumina.

Fig. 5.10: Typical constitutive bridging relations obtained from the analysis of the R-curves of the SiC-whisker-reinforced alumina.
Fig. 5.11: Variation of the $K_o$ deconvoluted from the R-curves of the SiC-platelet-reinforced alumina at various temperatures. The error bar on the data from high temperature tests under vacuum shows the scatter of the results from different specimens about the average value.

Fig. 5.12: Variation of the $K_o$ deconvoluted from the R-curves of the SiC-whisker-reinforced alumina at various temperatures. The error bar on the data shows the scatter of the results from different specimens about the average value.
**References**


Chapter 6

Discussion

6.0 Introduction

The main objective of the current chapter is to provide explanation for the results obtained in Chapter 5 and possibly correlate them with the microstructure of the material. This will be accomplished by providing additional supporting results from complementary experiments, referring to observations made in the literature and suggesting possible relations between the microstructure and the behavior of material.

Following a brief section on the assessment of the quality of the results obtained in Chapter 5 from the analysis of the R-curves based on the weight function methodology described in Chapter 3, the results and behavior pertinent to each of the composite materials and the effect of temperature will be discussed in a separate section. Finally, the toughening trend and the bridging behavior will be compared between the two composite materials.

In order to avoid redundancy, figures from other parts of this work will not be reproduced here, unless combination of the data from different figures is found necessary.

6.1 Evaluation of the Quality of the Results

Before entering into the discussions of the material behavior based on the results obtained from the experimental measurements and the analysis presented in chapters 4 and 5, it is important to assess the quality of the experimental results as well as the values obtained from the analysis of those results. This will help to establish confidence in the reliability of the procedures and results.

6.1.1 Load-displacement Curves and R-curves

The key elements in extracting the R-curve from the load-displacement curve obtained during the fracture test on a chevron-notched specimen are the correct measurement of the loading-point displacement and availability of a reliable compliance/geometric function solution.

The quality of the finite element mesh and some of the finite element results were verified in Chapter 3 by calculating values for the weight function of the through-thickness crack using the finite element model and comparing the values with those from the literature.

The indirect compliance measurement method used was shown in Chapter 4 to be of high accuracy based on the finite element results shown in Fig. 4.3. Additional verification was sought for the accuracy of the load-displacement curves indirectly by conducting fracture tests on chevron-notched specimens of various materials, including glass, monolithic alumina, monolithic silicon carbide and various grades of
silicon nitride, and calculating the (plane-strain) elastic modulus of these specimens at room temperature from the slope of the elastic region of the load-displacement curves. In all of such cases, except for the SiC-whisker-reinforced alumina composite (WG-300) used in this work, an agreement within better than 4% was found between the elastic modulus determined from the compliance measurement and the values determined independently (e.g., by an ultrasonic method) or reported in the literature. Considering that minor geometrical variability was inevitable amongst various specimens, this is a good agreement between the modulus results. As for the SiC-whisker-reinforced alumina, the elastic modulus and Poisson’s ratio quoted by the manufacturer (Table 4.1) were 393 GPa and 0.23, respectively, and have been determined by the ultrasonic resonance method. These values lead to a plane-stain elastic modulus of 415 GPa which appeared to be significantly different from the average value of 472 GPa determined from the compliance measurement during fracture tests. This could be correlated to the influence of the preferred orientation of the SiC-whiskers within the plane perpendicular to the hot-pressing direction (section 4.1.1). Similar dependence of the modulus of elasticity on the direction was also observed by Zeng et al [1] who used the Herzian indentation for elastic modulus determination in various ceramics, including a SiC-whisker-reinforced alumina. As described in section 4.1.1, the specimens in this work were intentionally prepared from the hot-pressed blank such that both crack plane and crack growth direction were parallel to the hot-pressing axis. This led to the preferred orientation of the SiC-whiskers relative to the notch/crack plane normal (in this regard, the orientation of the specimen used for the ultrasonic determination of the elastic properties of the composite, Table 4.1, is not known). Under such circumstance, the linear rule of mixture:

\[ A_c = \sum_{i=1}^{n} V_i A_i \]  

where \( A \) is the property, \( V \) is the volume fraction, index “e” refers to the composite and index “i” refers to each constituent, could be used to provide an estimate for the elastic properties of the composite. Such estimation has also been adopted by Becher et al [2] in the case of similar SiC-whisker-reinforced alumina composites and under similar testing conditions. To estimate the elastic properties of the whisker-reinforced composite, the elastic properties of SiC-whisker and alumina from Becher et al [2] were employed: alumina: \( E=400 \) GPa and \( v=0.24 \); SiC-whisker: \( E=550 \) GPa and \( v=0.24 \). Calculations based on using these values in Eq. 6.1 gave \( E=449.5 \) GPa, \( v=0.223 \), and thus \( E'=473 \) GPa, for a 33 vol.% SiC-whisker-reinforced alumina. It can be seen that the calculated value for (plane strain) modulus, \( E' \), is in good agreement with the average value determined from the compliance measurement. Thus, the modulus values determined from the compliance of the whisker-reinforced composite specimens reflect the effect of the preferred whisker orientation relative to the loading direction during the fracture tests.

Further to the verification of the compliance measurements and R-curve calculations from the measured load-displacement curves, fracture tests were also carried out on test specimens made of
polymethyl methacrylate, PMMA, and monolithic silicon nitride\(^*\), which had essentially flat R-curve behavior. Some typical R-curves calculated from the load-displacement curves of these PMMA and silicon nitride specimens are shown in Fig. 6.1 (The silicon nitride material was found to show subcritical crack growth effect under the test conditions employed at 1200°C. This is observed as a falling trend in the otherwise flat R-curve of the material).

![R-curves](image)

**Fig. 6.1:** R-curves measured on silicon nitride specimens at elevated temperatures and polymethyl metacrylate, PMMA, at room temperature for experimental verification of the compliance measurement. The crack length at which the minimum value of the geometric function occurs for the chevron-notched specimen is indicated by the straight dashed-double-dotted line, \(y_{min}\), on the plot.

### 6.1.2 Toughening Parameters Deconvoluted from the R-curve

**6.1.2.1 Crack Tip Toughness, \(K_t\)**

To assess the values obtained for \(K_t\) as a fit parameter, independent fracture tests were conducted at room temperature on three four-point-bend SENB specimens, 3.6 mm x 4.5 mm x 45 mm, from the platelet-reinforced alumina composite with a notch depth \(a/W=0.57\). The notch was cut into the specimens using a 50\(\mu\)m-thick diamond impregnated disk, resulting in a notch-tip radius of \(~30\ \mu\)m. The notch tip of one such specimen is shown in Fig. 6.2. These specimens were then fractured under a cross-head speed of  

\* Grade GSN, Greenleaf Corporation, Saegertown PA
fracture microscopic observations. The fracture toughness of these specimens was calculated from the maximum load and the geometric function for the straight-through crack given by Srawley and Gross [3].

Fig. 6.2: An example of the notch tip of the SENB specimen used for the determination of the fracture toughness of the SiC-platelet reinforced composite. (Bar=50 μm)

The average fracture toughness value obtained from these tests was 4.64± 0.10 MPa√m. The analysis of the room temperature R-curves from six specimens of the composite prepared from two different batches resulted in $K_0=4.17$ and 4.22 MPa√m for two specimens and $K_0=4.55-4.70$ MPa√m for the other four specimens. This indicates a satisfactory agreement between the measured and the fit value for the initial toughness in the composite. These values are also in good agreement with the results presented by Belmonte et al [4], who studied the R-curve behavior of alumina reinforced with various amounts of SiC-platelets using SENB specimens. The room temperature R-curve presented by Belmonte et al for the composite containing 20 vol.% fine SiC-platelets started at ~4.62 MPa√m.

6.1.2.2 Bridging Stresses

As the bridging stresses are experimentally difficult to measure and data for ceramics reinforced with a discontinuous phase especially scarce in the literature, a direct quantitative assessment of the accuracy of the bridging stress values obtained from the analysis of the R-curves, as was done for $K_0$, was not possible. However, attempts were made to provide justification for the bridging stress results based on the available literature values for SiC-whisker-reinforced alumina. The major obstacle encountered in this case was the difficulty in obtaining a long-enough continuous R-curve for the whisker-reinforced composite at room temperature such that the curve could be analyzed reliably. Numerous attempts in this direction failed to provide an R-curve suitable for the deconvolution of the bridging stresses. As a result, only a qualitative verification could be established by comparing the maximum bridging stress estimated from a linear extrapolation of the high temperature results to room temperature with the results from Yu and Kobayashi [5] and Fett et al [6] for a 30 vol.% SiC-whisker-reinforced alumina, as was shown in Fig. 5.8. It can be
[5] and Fett et al [6] for a 30 vol.% SiC-whisker-reinforced alumina, as was shown in Fig. 5.8. It can be seen in Fig. 5.8 that the extrapolation of high temperature results to room temperature yields a value in a very good agreement with those reported in [5,6]. In the next step the R-curve for the composite was calculated, based on the procedure outlined in Chapter 3 assuming this value for the maximum bridging stress and that the exponential relation, Eq. 2.12, could approximate the bridging relation. The exponential form was assumed based on the resemblance between this bridging constitutive relation at extended CODs and the bridging relations deconvoluted from the R-curves in this work. The solutions for the bridging stress intensity factors were then sought for $K_e$ ranging 5.5-6.3 MPa.$\sqrt{m}$ and a characteristic displacement of 0.125-0.25 μm. The characteristic displacement range was chosen based on SEM observations of the crack trace on the surface of the notch of the specimens broken at room temperature indicating a whisker pullout length ranging from 0.3-0.8 μm with an average value of about ~0.5 μm. Some of these calculated R-curves are compared with one room temperature experimental R-curve in Fig. 6.3. The abrupt variations in the stress intensity factor on the experimental R-curve are due to the crack-arrest behavior of the composite described previously. The experimental R-curve shown in Fig. 6.3 appears to be in good agreement with the R-curves calculated for $K_e$ values of 5.8-5.9 MPa.$\sqrt{m}$; however, the effect of the characteristic displacement values chosen appear to be minor within the crack length range for which the R-curve was available.

Fig. 6.3: Comparison of an experimentally measured R-curve for the SiC-whisker-reinforced alumina at room temperature under the four-point bending condition prior to crack instability with the calculated R-curves based on a maximum bridging stress of 25 MPa and the assumption of an exponential bridging relation with characteristic displacements of 0.125 and 0.25 μm.
6.2 Temperature Dependence of the Toughening Behavior in SiC-Platelet-Reinforced Alumina

6.2.1 Crack Tip Toughness

The initial values of the fracture toughness, $K_c$, obtained from the analysis of the R-curves of the SiC-platelet-reinforced composite at different temperatures, are shown in Fig. 5.11. The major distracting feature of these values is the scatter in the $K_c$ results from the R-curves obtained under vacuum. As described in section 4.3, the processing defects caused by degraded hot-pressing conditions did not allow the production of further specimens of this composite. It may be speculated that such defects were also present in the two batches of the composite used to produce fracture specimens and could be responsible for the large scatter in $K_c$. In the course of this work, defective materials were obtained from several hot-pressing trials which seemed to strongly indicate a large heat-loss through the lower ram of the hot-press when using the 70 mm internal-diameter graphite die (this die was used to produce specimens for the fracture tests). This was demonstrated by the presence of a central region of a lighter color and lower density on the side of the hot-pressed blank facing the lower ram of the hot-press. In the most severe cases, when a large mass (280-320g) of powder had been loaded into the die, large radial cracks could be observed on the same face of the hot-pressed blank. Such defects could not be observed in the materials processed in a smaller die, 12.5 mm in diameter, used for the optimization of hot-pressing schedule. The heat-loss seemed to result in differential sintering between the lower and the upper faces as well as between the edges and center of the bottom face of the hot-pressed blank, thus promoting crack formation in the blank. Such cracks could be identified during visual inspection of some of the hot-pressed blanks after removal of the surface layer and slicing the blank. In some cases these cracks could be identified only under SEM. Obviously, the presence of defects of this nature could severely deteriorate the fracture resistance of the material at the tip of the growing cracks. Keeping this in mind and combining the results from tests in air and under vacuum, the average trend of the results presented in Fig. 5.11 seems to suggest a smoother drop in $K_c$ at temperatures below 1000°C.

The rapid deterioration of the crack tip toughness above 1000°C may be correlated with the softening of the amorphous phase found at some of the (grain) boundaries associated with SiC-platelets in TEM observations, as shown in Fig. 6.4. This amorphous phase was found to be present mostly as isolated pockets at the triple junctions formed by the SiC-platelet and the alumina matrix grains (Fig. 6.4, left image). In some less frequent occasions, the amorphous phase was observed to have penetrated into the matrix grain boundaries adjacent to the SiC-platelets (Fig. 6.4, right image) but not between the grains within the matrix. Traces of this amorphous phase could also be found during SEM fractographic examinations carried out on the fracture surface of the specimens broken under vacuum, as shown in Fig. 6.5. While the facets of the platelets on the fracture surface from room temperature tests were found essentially clean (Fig. 6.5a), those from higher temperatures contained the traces of the amorphous second phase. At 800°C (Fig. 6.5b) this could be observed as a band-structure on the platelet facets that resembled
a replica of the matrix grain/platelet interfaces where the glass phase had been residing. At 1000°C (Fig. 6.5c) the previous structure was found to have been replaced by the isolated (droplet-like) islands of the amorphous phase; this indicates that the amorphous phase could undergo viscous deformation at this temperature. Such islands of a second phase were also observed in the SiC-platelet-reinforced alumina composites studied by Belmonte et al at 1200°C [4].

![Image](https://via.placeholder.com/150)

**Fig. 6.4:** TEM observation of the amorphous phase associated with the presence of the SiC-platelets: left) bright field image and the selected area diffraction pattern centered on the phase in the triple junction between the SiC$_{pl}$ and Al$_2$O$_3$ grains showing a diffuse central spot, and right) dark field image of the amorphous phase running through the matrix grain boundary adjacent to the silicon carbide platelet. The image was taken with the aperture placed to avoid any diffraction coming from the alumina grains.

If the amorphous phase at the boundary could be considered as one very rich in silica, the viscosity of such a phase at 1000°C would be near the annealing point ($\eta \approx 10^{12}$ Pa.s) [7] while at 1200°C such a composition would yield a viscosity well within the annealing range ($\eta \approx 10^9$ Pa.s) [7]. However, it is noted that even a low bulk impurity concentration may result in a considerable accumulation of the impurities at the grain boundary interfaces [8,9] due to limited solubility of the solute in the bulk of material which could strongly influence the viscosity of the grain boundary phase.

No indication of the amorphous phase could be found on the facets of the SiC-platelets observed on the fracture surface of the composite at 1400°C (Fig. 6.5e). This may indicate of a redistribution of the amorphous phase at this temperature into the matrix grain boundaries. If the liquid phase formed by further softening of the amorphous phase at such temperatures could completely wet the grain boundaries between the alumina matrix grains, it would penetrate and preferentially reside in such regions.
Fig. 6.5: Examples of the SiC-platelet facets on the fracture surface of the composite; a) room temperature; at higher temperatures indications of tracks of the amorphous phase (Fig. 6.4) could be found on some of the platelets as shown in micrograph b) at 800°C, c) at 1000°C and d) at 1200°C. At 1400°C, micrograph (e), such features could not be found on the facets of the platelets. (Bar=5 μm)
Fig. 6.6: Examples of the fracture surface of the SiC-platelet-reinforced alumina from various test temperatures under vacuum; a) room temperature, b) 800°C, c) 1200°C and d) 1400°C. (Bar=100 μm)
6.2.2 Bridging Stress

The variation of the maximum bridging stress and examples of typical bridging relations calculated from the R-curves at different temperatures are shown in Fig. 5.7 and Fig. 5.9, respectively. In general, the maximum bridging stress can be observed to decrease relative to the value at room temperature with increasing temperature. This is an expected trend based on the role of the residual radial stresses at the interface between matrix grains and the reinforcement. The residual stresses are a consequence of thermal expansion mismatch between different phases or different crystallographic orientations, and processing of the material at elevated temperatures. The exact nature and magnitude of these stresses in a composite depends on the thermal expansion mismatch, elastic properties, volume fraction of the second phase and the geometrical nature of the second phase [10-12]. Upon cooling from the sintering temperature, such stresses may be relaxed by creep or viscoelastic processes within the first few hundred degrees below the processing temperature; however, in the absence of such processes at lower temperatures the stresses are built up and stored in the microstructure. Due to the larger thermal expansion coefficient of the alumina matrix (8.6 x 10^{-6}°C^{-1} [10]) than the SiC reinforcement (~4 x 10^{-6}°C^{-1} for the cubic phase [9] and 3.2-4.5 x 10^{-6} °C^{-1} for the hexagonal phases [10]), the radial component of the residual stress at the matrix/reinforcement interface is compressive [10-14].

As can be seen from Eq. 2.17 and Eq. 2.18, the radial residual stress gives rise to the bridging stress during the reinforcement pullout process through the shear tractions that act along the debonded interface between the matrix and the bridging reinforcement. A detailed analysis of the temperature dependency of the pullout bridging stress is complex due to the simultaneous effect of temperature on multiple parameters influencing the micromechanics of bridging. However, a simplified picture of temperature dependency of the pullout bridging stress may be drawn by considering Eq. 2.15, Eq. 2.17 and Eq. 2.18. Here, the variable temperature enters most directly through the dependence of shear tractions at the interface, \( \tau \), on the normal compressive residual stress, \( \sigma_r \), at the bridge/matrix interface. The residual stress is in turn proportional to the thermal expansion coefficient mismatch between the two phases (here the matrix and reinforcement), \( \Delta \alpha \), the difference between the processing or a characteristic temperature and working temperature, \( \Delta T \), and a constant, \( A \), describing elastic properties and volume fractions of each phase. This leads to:

\[
\sigma_{br,po} \propto \tau(l_{po} - 2\delta) \propto \mu \sigma_r(l_{po} - 2\delta)
\]

\[
\sigma_{br,po} \propto A\mu\Delta \alpha \Delta T(l_{po} - 2\delta)
\]  

(6.2)

where \( \mu \) is the friction coefficient at the interface and \( l_{po} \) and \( \delta \) are the pullout length of the bridge and displacement of one crack wall relative to the crack plane, respectively. It may be assumed that \( \mu \) would not vary significantly with increasing temperature, if the interface is free of a (amorphous) second phase, i.e. the interface is clean. Assuming a temperature range where no drastic change would occur in the elastic properties of the material, the temperature response of the pullout bridging stress will be described by
$\Delta \alpha \Delta T$ and $l_{po}=f(T)$. It has been generally observed that the reinforcement pullout length increases with increasing temperature [15-17]. It will be shown later that this behavior leads to bridging interactions being maintained over a wider crack opening displacement range. Under such circumstances, the residual stresses, and thus $\Delta \alpha \Delta T$ term, can be considered to dominantly describe the temperature dependence of the bridging stresses. Although in $\Delta \alpha \Delta T$ term the thermal expansion coefficient of each phase would also depend on temperature, the variation of the latter with temperature is overshadowed in the residual stress problem by the magnitude of temperature variation itself. This would, therefore, result in a linear dependence of the residual stresses on temperature, provided that there would be no discontinuity in the behavior of the material, due to a phase transition or microcracking. Indeed, studies of residual strains and stresses in SiC-whisker-reinforced alumina composites between room temperature and 1000°C by Majumdar et al [18] using neutron diffraction analysis and by Ballard et al [19,20] using x-ray diffraction provide experimental evidence for such a behavior. These studies indicate that both residual strains and residual stresses in the composite decline linearly with increasing temperature.

The bridging stress results presented in Fig. 5.7 can be seen to generally indicate a linear trend, except between 800°C and 1000°C. However, if the characteristics of the interface, e.g., friction coefficient or viscoelastic behavior due to an amorphous phase, vary significantly with temperature the composite effect would no longer lead to a linear dependence on temperature as described in Eq. 6.2. Such non-linear effects can also be clearly seen in the maximum pullout bridging stress results provided by White and Hay [17] for a high purity alumina and an alumina containing glass phase at the grain boundaries (these results will be shown later in Fig. 6.14). In this case, the temperature dependence of the maximum pullout stress is entirely non-linear for the latter, while that from the former indicates a linear trend. Having this in mind, the variation of the maximum bridging stress with increasing temperature in the composite near 1000°C is suggestive of such effects. The anomalous trend between 800°C and 1000°C in the data shown in Fig. 5.7 is believed to be associated with the viscoelastic deformation of the amorphous phase shown to be present in this composite and the shear-rate dependence associated with such a deformation mechanism. In the presence of a viscoelastic second phase at the matrix/bridge interface, the shear deformation is also dependent on the rate of slip at the interface [21], i.e., the magnitude of the bridging stress also would depend on the crack opening displacement rate. Such rate dependency has been previously observed in R-curve studies on SiC-whisker-reinforced silicon nitride at 1200°C [22]. In that case a material with an essentially flat R-curve (corresponding to no bridging tractions) under a fast displacement rate condition was shown to demonstrate a significant R-curve behavior (corresponding to a finite amount of bridging tractions) upon reducing the displacement rate during the fracture test.

The onset temperature for the viscoelastic behavior of the amorphous interfacial phase may also be seen in the velocity of the longitudinal ultrasonic waves used for the determination of elastic modulus at high temperatures (Fig. 5.3). In order to accentuate the temperature effects on the velocity data, the velocity vs. temperature data are plotted in Fig. 6.7 using an Arrhenian presentation (the value of velocity at 1400°C was obtained by the extrapolation of a polynomial fit to data measured between 660°C and 1350°C). This
plot also clearly shows the presence of two temperature domains: a region with a smooth variation limited to \( T < -950^\circ C \) and a region of sharp variation above approximately 1050\(^\circ C\). If lines were fit to the data within each temperature domain, \( T \leq 800^\circ C \) and \( T > 950^\circ C \), the intersection of the two linear trends would occur at \(-930^\circ C\). (Here, it is noted that the temperature dependence of elastic modulus in various monolithic ceramics [23] indicates a linear drop with increasing temperature in materials with no amorphous grain boundary phase. Therefore, the Arrhenian presentation of such a linear trend should appear as a non-linear behavior, as can also be seen for the data in the lower temperature region of Fig. 6.7. However, to help determine a transition temperature into the region speculated to be influenced by a thermally activated process, the lower temperature region of Fig. 6.7 is also approximated by a linear behavior.) This could be a reasonable estimate of the temperature for the onset of the viscoelastic behavior of the amorphous phase at the SiC-platelet/alumina interface and is in agreement with the temperature dependence of the bridging stress results shown in Fig. 5.7.

![Arrhenius plot](image)

Fig. 6.7: Arrhenius plot of the velocity of the longitudinal sound waves as a function of temperature in the SiC-platelet-reinforced composite.

The exact determination of the influence of the viscous flow behavior due to the amorphous phase on the magnitude and the distribution of the bridging stresses requires further experiments at significantly different displacement rates to identify the type of the viscous behavior, e.g., shear-thinning or shear-thickening. Such experiments have been conducted by White and Hay [17] on a set of post-tensile fracture test specimens of alumina with a glass phase at the grain boundary. This analysis indicated that as the displacement rate at 1170\(^\circ C\) was raised (corresponding to an increase in the shear rate imposed on the viscoelastic grain boundary layer) from 0.25 \( \mu m/min \) to 2.54 \( \mu m/min \), the maximum pullout bridging stress
appeared to increase by ~10%. The increase of the pullout bridging stress with increasing shear rate indicates of a shear-thickening behavior from the viscous glass phase present at the grain boundaries.

Examples of the shape of the constitutive bridging relations obtained from the R-curves are shown in Fig. 5.9. In general, in addition to the decreasing stresses with increasing temperature, it can be seen that the bridging relation tends to tail-off at larger (crack) opening displacements. This is in agreement with observations made on the fracture surface indicative of larger platelet pullout length with increasing test temperatures in this work, Fig. 6.6, and with the previous observations in whisker-reinforced alumina [15,16]. Due to the tortuosity of the fracture surface and the three-dimensional nature of the platelet shape compared to whiskers, the pullout length was hard to characterize by microscopic methods. Instead, the roughness of the fracture surface of test specimens broken under vacuum at different temperatures was characterized as an indicator of the pullout length using an optical roughness profilometer*. For this purpose a total area of ~0.52 mm² was analyzed for one representative sample per test temperature. This was accomplished by performing the analysis on several smaller areas (~0.04 mm²) of the fracture surface and combining the data from these tests to produce the global results for each test specimen. During each individual measurement, the data were corrected for the underlying macroscopic curvature and tilt of the fracture surface using the mathematical filters provided in the analyzing instrument software. The results of this analysis are shown in Fig. 6.8 by plotting the cumulative distribution of point-height on the fracture surface at different temperatures.

![Roughness profilometry results](image)

**Fig. 6.8:** Roughness profilometry results from the fracture surface of the SiC-platelet-reinforced alumina specimens broken at various temperatures. The zero value on the abscissa corresponds to a fictitious plane describing the average of the distribution of the point elevations measured.

---

* WYCO Profilometer NT2000, WYCO Ltd., Tucson, AZ
It may be noted that microscopic examination of the fracture surfaces, e.g. micrographs shown in Fig. 6.6, did not indicate any significant variation in the fracture mode of the matrix grains, which was found to be predominantly intergranular. Therefore, the results from the analysis of fracture surface roughness should adequately reflect contributions from the reinforcing platelets. The distributions shown in Fig. 6.8 also indicate clearly a broadening towards larger values as the temperature is increased and are in agreement with the extension of the tail-off behavior of the constitutive bridging relations calculated from the R-curves (See Fig. 5.9 for instance).

Noteworthy, the cumulative distributions in Fig. 6.8 seem also to degenerate into two visible clusters; one cluster describes the temperature range $RT \leq T \leq 1000^\circ C$ and the other covers the temperatures $1200^\circ C$ and $1400^\circ C$. A significant softening of the intergranular amorphous phase appears to have strongly promoted (lubricating effect) the platelet pullout at $1200^\circ C$, thus resulting in a significant difference between the fracture surface roughness at $1200^\circ C$ relative to $1000^\circ C$. However, further temperature increase to $1400^\circ C$ does not seem to have yielded a similar drastic change in the fracture surface roughness profile. Interestingly, it was shown in the micrographs of Fig. 6.5 that the facets of the SiC-platelets at this temperature did not show any indication of the presence of an amorphous phase.

Speculation on the potential contribution of matrix grain bridging to the observed R-curve behavior of the composite was the incentive to reduce the matrix grain size in the composite, as a previous study by Chou and Green [24] on this composite system had reported a flat R-curve in the short crack regime. As described in section 4.1.2 the composites prepared at the earlier stages of this work as well as in a previous study [25] had an average matrix grain size of ~7.5 μm and most of the matrix grains showed a non-equiaxed shape. One room temperature R-curve from this material was also analyzed for comparison and resulted in a maximum bridging stress of 26 MPa and a $K_c$ value of 4.17 MPa√m. These values are within the range of the values obtained for the composite with a finer matrix grain size. However, the constitutive relation could not be extracted from this R-curve, as a solution for the COD due to the applied load was not available for the particular specimen geometry used in these R-curve measurements. Therefore, it is concluded that within the matrix grain size range described for the two materials the contribution of the matrix grains to the crack wake bridging behavior of the composite is negligible and the bridging process is predominantly driven by the contributions from the SiC-reinforcement.

### 6.3 Temperature Dependence of the Toughening Behavior in SiC-Whisker-Reinforced Alumina

#### 6.3.1 Crack Tip Toughness, Bridging Stress, and Pullout Length

The R-curve results from the whisker-reinforced alumina composite were shown in Fig. 5.2 as a function of temperature. As described earlier, the crack growth behavior of the composite at room and lower temperatures had a significant tendency towards the run-arrest behavior. In most of such cases, see for instance the R-curves shown in Fig. 5.2.b, following the crack arrest point the crack first propagated
The onset of the stable propagation was at a lower stress intensity factor than the value of the R-curve prior to the crack arrest point. This process usually occurred in multiple steps until the crack had grown completely into the notch, and could be observed with the same severity up to 600°C. As described previously, each unstable jump after the crack arrest could lead to partial destruction of the bridging elements in the crack wake, thus reducing the contribution of the crack wake to the stress intensity factor. It was observed in some cases that successive arrest points resulted in a characteristic stress intensity factor suggesting that the bridges formed in the crack wake were completely destroyed during unstable crack propagation. The crack arrest/growth behavior was observed to be the most pronounced at room temperature and to diminish with increasing temperature and could not be observed at and beyond 1000°C.

Due to the lack of an adequately wide and continuous region on the R-curves from room temperature tests on the composite, these R-curves could not be analyzed to obtain the distribution of the bridging stresses. Therefore, the bridging at room temperature was estimated by the linear extrapolation of high temperature results and the procedure described in section 6.1.2.2. Nevertheless, the minimum stress intensity factors following the unstable crack propagation steps on the R-curves at room temperature, especially if they occurred at more than one location and at relatively large crack lengths, were adopted as an acceptable estimate of $K_c$ for the particular specimen. Acceptance of such values as an estimate of $K_c$ followed the shape of the weight functions for cracks in finite bodies. For short crack lengths the tractions near the crack tip contribute most significantly to the stress intensity factor, while for longer crack lengths the tractions that are acting far away from the crack tip are more important.

It can be clearly seen in Fig. 5.2 that compared to the R-curves at room temperature the R-curves at 1000°C and 1200°C appear at higher stress intensity factors. This is similar to the behavior observed by White and Guazzone [15] for the SiC-whisker-reinforced alumina at 1200°C relative to room temperature. This apparent improvement was discussed by the authors to be due to an increasing contribution of the bridging mechanism in the R-curve behavior of the composite at elevated temperatures based upon the observation of an increasing whisker-pullout length with increasing temperature and comparison of the toughness values from chevron-notched and SENB specimens. In agreement with the microscopic observations from the previous works [15,16], the whisker-pullout length in the composite studied here was also observed to increase with test temperature. Examples of the whisker-pullout length at room temperature and 1200°C are shown in the micrographs of Fig. 6.9. However, analysis of the R-curves clearly shows that the maximum value of the bridging stress in the composite is declining with increasing temperature, as presented in Fig. 5.8. The trend of these results with temperature appears to agree with expectations based on the Eq. 6.2.

Detailed microscopic examinations [26,27] of this SiC-whisker-reinforced alumina have indicated that the composite is essentially free of a glass phase and an accumulation of an amorphous phase at the triple junctions or whisker/matrix interface could be observed very rarely. Nevertheless, close examinations of the interface between the SiC-whisker and alumina matrix grains have revealed a 1-nm thick (few atomic
layers) "nano-crystalline" phase at such boundaries [27]. It may be, therefore, anticipated that the temperature dependence of the bridging tractions would closely resemble a linear dependence on temperature owing to the contributions from the residual stresses. The linear fit of the maximum bridging stress data shown in Fig. 5.8 by the solid line seems to satisfactorily describe the trend of the data presented.

Fig. 6.9: Examples of the whisker-pullout length in the SiC-whisker-reinforced alumina observed on the surface of the chevron notch: a) room temperature, and b) at 1200°C. The specimen from 1200°C test was etched in dilute HF acid solution to remove the oxide glass layer from the surface. (Bar=1 μm)

As was described and discussed in the previous section for the bridging stress results from the platelet-reinforced composite, the common observation of an increased pullout length of the reinforcement with increasing temperature in such composite systems results in a larger critical crack opening displacement in the constitutive bridging relation, e.g., a larger $\delta_c$ in Eq. 3.12 (or $\delta_o$ in Eq. 3.13). Similar to the constitutive bridging relations obtained from the R-curves of the platelet-reinforced composite (Fig. 5.9), those obtained for the whisker-reinforced composite (Fig. 5.10) also indicate the extension of the bridging stress distribution towards larger crack opening displacements with increasing temperature. Based on the bridging results presented in Fig 5.8 and Fig. 5.10, it is concluded that the contribution of the bridging stresses to the toughening behavior of this SiC-whisker-reinforced alumina diminishes with increasing temperature. However, a more precise statement on the toughening due to the bridging mechanism ought to include the variation with temperature of the integral of the constitutive bridging relation, i.e. the area under the bridging stress-displacement curve. The variation of both the maximum bridging stress and the critical displacement with temperature may bring about the condition that the area under the bridging stress-displacement curves at higher temperature would be larger than those at lower temperature, if the entire curve is considered. Clearly, in such a case the statement regarding variation with temperature of the contribution of the bridging process to the toughness ought to refer to the COD range.
The question now arises as to what is giving rise to the "improvement" of the R-curve behavior. The \( K_0 \) values obtained from the R-curves, as shown in Fig. 5.12, imply that the initial toughness of the material improves with increasing temperature and passes through a maximum at 1200°C. The improvement of the toughness of the composite with increasing temperature is in general agreement with the results from Han et al [28] and Hanson et al [29,30], who studied the high temperature toughness in this whisker-reinforced alumina by using precracked bend specimens. Although the authors described this whisker-reinforced composite as one showing little R-curve behavior, the R-curves and bridging stress results from this study clearly indicate otherwise. In fact, one of the reasons for the difference in the magnitude of toughness improvement with temperature reported in [29,30] with the results obtained in this study could be due to neglecting the effect of the crack wake bridging and the R-curve on the toughness measured with the precracked specimens.

The studies by Han and Suresh [26] and Han et al [28] on the toughness and crack growth behavior of this whisker-reinforced composite have indicated that a damage zone containing microcracks is formed ahead of the crack tip and leads to crack tip shielding at elevated temperatures. To verify the presence of such a damage zone in the specimens broken at elevated temperatures, the region adjacent to the fracture surface was examined on selected specimens using field emission SEM at an accelerating voltage of 1 kV. For this purpose, the specimens were cut perpendicular to the fracture surface along the direction of crack growth. The as-cut plane perpendicular to the fracture surface was then carefully polished to a mirror finish (more than 100 \( \mu \)m in-depth was removed from the as-cut plane) using various diamond polishing media and low loads to minimize the potential damage induced during specimen preparation. The SEM micrographs in Fig. 6.10 show examples of the damage observed adjacent to the fracture surface at 1200°C and 1300°C. To verify that such damage was neither induced by the sample preparation procedure nor existed at lower temperatures, a sample was also prepared under the same condition from a specimen broken at ambient temperature. No microcracks or damage could be found in this sample.

The principal mechanisms responsible for the formation of the damage zone ahead of the crack tip in this composite at high temperature have been described to be distributed diffusional cavitation and microcracking [26,28]. Cavitation is promoted in the whisker-reinforced composite at high temperatures (typically above 1200°C [26,31]) by formation of amorphous silica and alumino-silicate glasses at the SiC-Al₂O₃ interface because of an oxidation reaction. The stress-assisted viscous flow of the glass phase at high temperatures promotes extensive cavitation along the Al₂O₃-SiC interfaces and at the grain triple junctions in the region ahead of the crack tip. The oxidation of the SiC leading to the formation of the amorphous phase, and cavitation in turn, has been shown to strongly depend on the loading rate, temperature and stress intensity factor [26,28,32,33]. Generally, higher temperatures, slower loading rates or higher applied loads give rise to the formation of a larger amount of glass phase at the Al₂O₃-SiC interface [28]. The coalescence of the interfacial/intergranular cavities leads to the formation of microcracks within the damage zone. No attempt was made to observe and characterize the interfacial oxide layer in this work.
Fig. 6.10: Examples of the microcracks formed within the damage zone in the vicinity of the fracture surface in the SiC-whisker-reinforced alumina. Micrographs a-c and d-f are from specimens broken at 1200°C and 1300°C, respectively. Marked area in micrograph (b) is magnified in micrograph (c) and shows the coalescence of cavities initiated at the SiC–Al2O3 interface to form the microcrack. Note that the microcracks/cavities are always associated with the SiC–Al2O3 interface. SiC-whiskers appear as the bright phase, arrows indicate the microcracks and FS designates the fracture surface.
However, under similar testing conditions the interfacial layer has been found to be essentially a silica glass* [28].

The damage/microcrack zone produced by the process described above seems to possess some similarities with the microcrack zone formed in cement-based composites [34]. The microcrack damage zone in the latter is also formed at the crack tip as a consequence of the high local stress field in this region, although in the former thermally activated processes (diffusion and viscous flow) also play a leading role. The conventional microcracking process observed in ceramics such as ZrO₂-toughened alumina [35] or TiB₂-reinforced silicon carbide [36] relies on the presence of tensile residual/transformation stress at the grain boundaries. Upon imposition of a critical level of stress, e.g. the one associated with the crack tip, normal to such boundaries the microcrack site is activated and the microcrack extends over the grain boundary. The microcrack zone formed includes two shielding components [37,38]: the modulus shielding due to a lower effective elastic modulus for the material within the damage zone, and the dilatational shielding due to the permanent strain associated with the opening of the microcracks. In contrast, the microcrack damage zone formed in the whisker-reinforced composite at elevated temperature appears to initiate from the boundaries that are under residual compression at lower temperatures and has time-dependent characteristics due to contribution of the diffusion process.

To provide experimental evidence for the toughening effects associated with the microcrack zone ahead of the crack tip, Han et al [28] conducted two types of fracture experiments using four-point-bend SENB specimens containing a fatigue-precrack (precrack length ≤ 200μm) produced by compression-compression fatigue loading (over 10⁶ cycles). It is to be noted that crack wake bridges may not survive such a fatigue loading condition and, therefore, the wake of such a precrack can be considered traction-free. Hence, in the absence of any further stable crack growth, the fracture toughness characteristics of the material at the tip of such a pre-crack can be calculated from the maximum fracture load and the total crack length (notch length + precrack length). In one set of tests, the precracked specimens were loaded to failure at various temperatures (T ≤ 1500°C) under load control condition (dK/dt = 0.026 MPa√m.s⁻¹) and the fracture toughness was calculated based on using the precrack length. The authors indicated that they did not observe any clear indication of stable crack growth up to about 1200°C and the fracture toughness could be unambiguously obtained from the fracture load. However, at and beyond 1300°C indications of stable crack growth were reported. The results of these tests are compared with the Kc results from this work in Fig. 6.11. In the second set of experiments the authors induced the damage ahead of the crack tip in the precracked specimens by loading the specimens at 1300°C and 1400°C to a stress intensity factor close to the critical value from the uninterrupted tests. The damage introduced at high temperatures in the vicinity of the precrack tip was then frozen in by removing the load and holding the specimen at temperature to anneal the residual stresses due to the deformations at the crack tip. The specimens were then cooled slowly

* Although internal oxidation leading to formation of glass phase is usually limited to creep test conditions, the presence of the notch with the through-thickness precrack in the fracture mechanics experiments provides for the transport of the oxidizing atmosphere between the fracture surfaces of the crack to the highly stressed region at the crack tip, thus promoting extensive oxidation within such regions.
to room temperature. The fracture toughness of the pre-damaged specimens at room temperature was shown by the authors to be significantly higher than that of the virgin material, indicating toughening contributions from the damage zone ahead of the crack tip.

![Graph showing comparison between high temperature toughness results and Kc values.](image)

**Fig. 6.11:** Comparison between high temperature toughness results from Han et al [28] with the Kc values obtained from the analysis of the R-curves in this work. The error bars on the data from this work represent the scatter of the results from different specimens, while those of the results from [28] are one standard deviation quoted by the authors in Table 2 of Ref. [28].

Although no pre-damaging experiment was carried out at 1500°C by Han et al, the authors concluded by comparing the fracture toughness at this temperature with the value from room temperature that at 1500°C, though larger in size, the damage zone degraded the fracture toughness of the material. The toughening effect from the microcrack damage zone was further analyzed semi-quantitatively by the authors based on microcracking models describing the modulus-softening contribution of a microcrack zone to toughening.

### 6.3.2 Bridging in the Presence of a Microcrack Zone

Before comparing the toughness results obtained in this work with those from Han et al, a question arises regarding the bridging stresses deconvoluted from the high temperature R-curves of the composite. The question may be developed as following:

If the microcrack zone formed ahead of the crack tip at high temperature is giving rise to toughening, will this zone also possess R-curve characteristics? If it does, how could the bridging results from high
temperature R-curves be substantiated considering the potential R-curve contributions due to the microcrack damage zone?

As soon as characteristic components of a toughening mechanism change with the crack length, R-curve behavior should be expected. For the cases of transformation and microcracking mechanisms, the variation of the zone size and zone shape with crack length leads to the R-curve behavior. It may be first debated if the size and shape of the microcrack zone varied with the crack length, and hence gave rise to an increasing R-curve behavior [39]. Initially the microcrack zone would form at the crack tip during the loading stage, giving rise to energy dissipation and, at the same time, the cracked body would become more compliant. Although the crack length has not changed physically yet, this would be interpreted as crack advance by a finite amount in a compliance determination of the crack length; a similar postulate has been made regarding phase-transformation shielding of a crack in a finite body and its effect on the R-curve measured using a compliance-based determination of the crack length [40]. Eventually, actual crack propagation would ensue when the microcrack process zone reaches a critical size. From this point on, the length of the microcrack zone would vary continuously by the amount of crack advance, Δa. It follows then that the critical stress intensity factor at the onset of the actual crack propagation contains the contribution of the microcrack zone formed at the crack tip. Further energy dissipation by the process zone thus remains proportional to Δa and, therefore, the stress intensity factor required to continue advancing the crack remains constant unless the crack approaches the boundaries of the specimen, at which point the stress intensity factor would vary considerably.

According to the above description optical measurement of the crack length in the presence of the microcrack process zone would yield a flat R-curve initiated at \( K_c \), yet the compliance determination of the crack length in this case would yield an R-curve that would initiate at \( K_c' \) and rise to the actual \( K_c \) within a short interval of apparent crack advance. This is schematically shown in Appendix II.b.

In agreement with the above arguments, some microcrack modeling work [41] has indicated that microcracks in the crack wake have little or no effect on the toughness. However, finite element simulation [42] has shown that crack tip shielding could be influenced by microcracks in the crack wake and, thus, R-curve behavior would be expected. The interaction between multiple toughening (R-curve) mechanisms has been discussed frequently in the ceramics literature in the past. Understanding of this complex problem is still incomplete due to multiple possible interactions between the mechanisms involved. In the case of R-curves arising from the combination of microcrack and bridging mechanisms, however, a simplified analysis has been presented by Horii [34] for such a circumstance in a cementious material. The calculated R-curves for the operation of the bridging zone in the absence and in the presence of a microcrack zone based on the characteristic parameters of the toughening mechanisms adopted from the experimental observations in concrete in Horii’s work (Fig. 8 in Ref [34]) indicate dominance of the R-curve behavior by the bridging stresses.

The potential contribution of the damage zone to the observed R-curve behavior in the whisker-reinforced composite at elevated temperatures may be further investigated through the bridging stress
results obtained from the analysis of the R-curves of the composite. The $K_e$ results presented in Fig. 6.11 seem to indicate that microcrack formation is activated at temperatures above 1000°C and that its contribution grows to a maximum value at 1200°C. It may be noted that the R-curve results in this work mostly cover a crack length between ~400-600μm to ~3500μm. If the damage zone contributed significantly to the R-curve behavior of the composite in this crack length regime, an anomalous trend in the deconvoluted bridging stress would be seen within the temperature range where microcrack formation occurred. This would happen because the deconvolution procedure blindly considers any variation in the R-curve to be due to bridging tractions in the crack wake region. No such anomaly is visible in the bridging stress results of Fig. 5.8. (It was argued above that if a microcrack zone developed at the crack tip, a compliance technique would interpret this as an increase in crack length, resulting in an apparent R-curve associated with the development of the microcrack zone. However, such an “apparent” R-curve behavior is not seen in the present results. It is believed that this is because the “apparent” R-curve behavior occurred at “apparent” crack lengths smaller than the crack lengths for which the stress intensity factor could be reliably determined.)

It is noteworthy that up to 1200°C there is good agreement between the results from this work and those from Han et al (Fig. 6.11), although the two studies employ different crack geometries. Had the R-curve effects from the microcrack zone been significant, the $K_e$ results from this study would have been different from those of [28]. Note again that the toughness results from Han et al [28] at T<1200°C were obtained without any indication of stable crack growth and those here were determined by the fit procedure which extrapolates the R-curves to the notch tip while considering the variation of the bridging stress intensity factor with the crack length as understood from the rest of the R-curve. It may be concluded from this comparison that the microcrack zone formed at the crack tip at high temperature results in toughening but no R-curve effect.

### 6.3.3 Crack Healing at Elevated Temperatures

Further comparison of the results shown in Fig. 6.11 indicates that the results from the two studies appear to be considerably different at T>1200°C. Although Han et al reported that they observed indications of stable crack growth at T>1200°C, the significant difference between the toughness values from the two studies seen in Fig. 6.11 cannot be accounted for by the small amount of bridging stress that exists at such temperatures. Based on the observations made on the material, two possible effects may be considered to have led to the existing difference in $K_e$ results (assuming that the damage zone ahead of the crack tip results in no R-curve behavior, otherwise the R-curve effect would be another possibility.). These are loading rate dependency of the damage zone formation, and healing of the crack in the precracked specimens due to oxidation. Formation of cavities and their coalescence, and thus the formation and size of the damage zone, was indicated to be dependent on the loading rate [26,28,32,33]: slower loading rates promote more cavitation. Examination of the load-displacement curves from this study indicates that the average loading rate to the maximum load was approximately 1-1.5 N/s during the tests at 1200°C-1400°C.
compared to a loading rate of \(-3.5\) N/s used in [28]. This would be expected to result in more cavitation at the tip of the crack (notch) in this work, and so a higher toughness. However, the difference in the loading rate does not seem to have significantly influenced the results obtained below 1200°C, as both test conditions seem to have yielded virtually the same toughness results. Nevertheless, since the mechanism of damage formation ahead of the crack tip is both time-dependent and thermally activated, the similarity of the results at lower temperature may not necessarily imply that the same trend would be maintained as the temperature is raised.

Alternatively, healing of the crack in the precracked specimens used in [28] may have contributed to the observed difference. Both the observations of this work and those by Han et al indicate the presence of a thick glassy oxide layer on the fracture surface of the specimens tested at \(T>1200°C\). The formation of the glass phase can lead to the healing of the precrack by joining the crack surfaces together. In fact, such a process was observed in this work in a crack branching off the main crack during microscopic observations of the microcracks in the specimen tested at 1200°C, as shown in Fig. 6.12. (We may note here that fresh crack surfaces are produced when crack grows into the chevron notch at high temperatures and that the samples from this work were not soaked at the test temperature after finishing the test. Hence, the feature shown in Fig. 6.12 has been exposed to \(T>1000°C\), only during the cooling stage, for \(-15\) minutes. Thus such effect can be more significant when the specimens containing a precrack is heated up to and soaked at the test temperature.) A similar effect has been speculated to be responsible for some erroneously high toughness values measured on silicon nitride precracked specimens tested at 1200°C in air [43]; such anomalous fracture toughness results were not obtained for the same specimens if tested in a nitrogen atmosphere.

![Fig. 6.12](image.png)

**Fig. 6.12:** Healing of a crack branch by the oxidation products in the SiC-whisker-reinforced composite broken at 1200°C in air. The area defined by the square in micrograph (a) is magnified in micrograph (b). Arrows are used to mark the crack trace for better visibility. The images were obtained using field emission electron microscope at an accelerating voltage of 1 kV. (Bar=5 \(\mu m\))
In addition, it is also noted that the magnitude of crack branching shown to exist in this material at elevated temperatures in [26,28] was not observed during microscopic examinations of this work. This may also contribute to the observed difference compared to the previous research.

6.4 **Comparison between the Platelet- and Whisker-reinforced Alumina**

The effect of reinforcement geometry on the toughening and bridging stresses can be established by comparing the toughening parameters obtained for these materials. However, due to different volume fractions of the reinforcement in the two materials and the potential influence of processing-induced defects in the platelet-reinforced composite, such a comparison may not yield a clear judgement of the relative effectiveness of the two reinforcements.

The temperature dependence of the maximum bridging stress and the initial value of the fracture toughness are compared in Fig. 6.13 and Fig. 6.15, respectively. The comparison of the temperature dependence of bridging stresses in the two composites indicates that almost the same magnitude of bridging stresses exists in the two composites. The temperature dependence of the bridging stresses, although generally similar, appears to be different for the SiC-platelet-reinforced alumina in the vicinity of 1000°C. This behavior was discussed based on the viscoelastic effects due to the presence of an amorphous phase at some of the SiC-platelet/alumina interfaces and the associated rate dependency of the viscoelastic deformation of an amorphous phase at elevated temperatures. In general, the bridging stresses are found to decline with increasing temperature in both of the composite materials due to the effect of the normal compressive residual stress at the interface between the alumina grains and SiC-reinforcements on the bridging stresses formed during the pullout process.

It may be found somewhat interesting that the magnitude of the bridging stresses are almost the same in the two composites, although the whisker-reinforced material contains a larger volume fraction of the reinforcement and thus a larger number density of potential bridging sites on the crack plane, a higher aspect ratio, and a finer size for whiskers (0.1-1.0 μm in diameter and aspect ratio of 10-100) relative to the platelets (3-30μm in diameter and aspect ratio of ~10). In addition, the three-dimensional geometry of platelets may be expected to be more restrictive in the pullout process and platelets would be more susceptible to failure due to bending near the crack surfaces. In fact, the bridging relations deconvoluted from the R-curves, Fig. 5.14 and Fig. 5.15, clearly show that the pullout performance of the platelets is restricted to a crack opening displacement that is smaller than that of the whisker-reinforced composite by almost a factor of two.

The bridging stress relation given by Eq. 2.17 describes the evolution of the pullout bridging stress in a single bridging whisker. A similar relation may be derived for a bridging platelet assuming a square-plate-like geometry for the platelet (see Appendix III.b for the derivation):
where \( d_{pl} \) is the thickness of the platelet.

\[
\sigma_{po,pl} = 2\tau \frac{l_{po} - 2\delta}{d_{pl}}
\]  

(6.2)

Fig. 6.13: Comparison of the maximum bridging stress in the SiC-platelet-reinforced alumina and the SiC-whisker-reinforced alumina as a function of temperature. The error bars on the data points represent the scatter of the results from different specimen about the average value. The data from the platelet-reinforced material is the average of the results from tests both under vacuum and in air. The maximum bridging stress value from the whisker-reinforced material at room temperature (solid circle) is an estimated value based on the linear extrapolation of high temperature results and the analysis of section 6.1.2.2.

In a continuum approximation the bridging stress that exists in individual bridging reinforcements is related to the globally observed value through the (area) fraction of the active bridging elements on the crack plane; this fraction usually possess statistical characteristics. Hence, a thorough comparison of the pullout bridging stress between the whisker- and platelet-reinforced materials requires quantitative information on the size distribution of the reinforcements and their pullout length. In the absence of such information, a qualitative comparison may be made using Eq. 2.17 and Eq. 6.2. As can be seen from the bridging relations extracted from the R-curves of the composites (Fig. 5.9 and Fig. 5.10), the bridging stresses from the platelet-reinforced composite vanish over a smaller COD range than from the whisker-reinforced composite. However, for a first order estimate we may consider that pullout length is the same
for both of the reinforcements. Based on the microscopic observations made on the materials, this seems to be a fair assumption at least for the room temperature condition. Although microscopic examinations indicate that the thickness of the platelets is usually a factor of 5 larger than the diameter of whisker, we consider the thickness of the platelets and diameter of the whiskers to be the same to simplify arriving at an upper bound value for the frictional pullout bridging stresses. The friction coefficient at the interface between alumina and SiC-platelets and alumina and SiC-whiskers are also assumed to be the same, at least in the lower temperature range where this interface in the SiC-platelet/alumina case is still not affected by the viscoelastic behavior of the amorphous phase found at some of the interfaces. Under such conditions, the magnitude of the pullout bridging stresses may be traced back into the state and the magnitude of the residual stresses at the interface between the alumina and SiC-reinforcement. Analysis of the residual stresses in the SiC-alumina composite system indicates that the interface between alumina and SiC grains is under residual compression and that the magnitude this stress component depends on the volume fraction, shape, and aspect ratio of the inclusion [11-13]. Typically for 30vol.% SiC-whiskers (cubic phase) with an aspect ratio of 10 or larger, the radial component of the residual stress at the interface is around $-1.0 \text{ GPa}$. [11-13]. For the $\alpha$-SiC-platelets (hexagonal phase) in alumina, Chou and Green [44] analyzed the residual stresses as a function of volume fraction and aspect ratio of the inclusion by considering the platelet as an ellipsoidal disk. This analysis indicated that the radial component of the residual stresses at the interface was dependent on the position along the interface; however, the dependence was found to be very weak and the stress quickly reached a plateau value. For a 20vol.% concentration of the $\alpha$-SiC platelets with an aspect ratio of 10 the plateau value for the radial component of the residual stress at the interface was calculated to be about $-0.3 \text{ GPa}$ [44], roughly one-third of the same residual stress component in the whisker-reinforced material. Becher et al [2] assumed a friction coefficient of $\sim 0.1$ at the interface of SiC-whisker/alumina, which leads to a shear strength of 100 MPa for a debonded interface (assuming a radial residual compression of stress of $-1 \text{ GPa}$). Considering the same value for the friction coefficient, the shear strength of the debonded interface in the case of the SiC-platelets would be 30 MPa ($0.1 \times 0.3 \text{ GPa}$). The magnitude of the frictional pullout bridging stress in the platelet-reinforced material thus would be on the order of less than 30% of the value in the whisker-reinforced composite.

It seems as if all of the parameters explicitly affecting the magnitude of the pullout bridging stresses in the composite are in favor of a considerably lower bridging stress in the platelet-reinforced material. Two arguments may potentially justify the observed magnitude of the bridging stress in the material reinforced with the platelets. The first argument concerns the state of the residual stresses at the interface between the platelet and the matrix. In the analysis of the residual stresses due to thermal expansion mismatch in composite materials, shear stresses are assumed a priori equal to zero following the notion that the thermal strains are dilational in nature [11]. In addition, the shape of the second particles is considered to be non-faceted, e.g., spherical, cylindrical or ellipsoidal disk. It may be noted that, in contrast to such smooth (zero-faceted) geometries, for a faceted inclusion both normal and shear residual stresses can exist at the interface between the inclusion and the matrix [13]. The actual geometry of the SiC-platelets also
possesses, to some extent, faceted characteristics; the majority of the platelets have multiple edges and corners thus forming facets at the perimeter of the platelet. The residual shear may interact constructively or destructively with the shear tractions that are produced during pullout of the reinforcement [45] and, if present, should be considered in the micromechanical derivation of the bridging stresses [46,47]. If the interaction between the residual shear stress and pullout shear stress were constructive, the shear stress term in Eq. 6.3 would be larger than the product of the radial residual stress and the friction coefficient of the debonded interface, leading to improvement of the pullout stress. The order of magnitude of the residual shear stresses calculated for the faceted inclusions in [13] seems to be on the order of the shear stresses estimated in the previous paragraph.

The second argument may be constituted around the contributions to the pullout bridging stress from sources other than thermal residual stresses, e.g., asperity loading and rotation of the bridging elements in their sockets due to their geometry/size [48] (such mechanisms, however, are more frequently observed in the case of grain bridging materials). Although bridges are most commonly observed in materials with thermal residual stresses, the existence of R-curve behavior and crack wake zone tractions in materials like spinel [48,49], which has a cubic crystalline structure, indicates that such stresses can exist even in the absence of thermal residual stresses. In this case, grain size, grain morphology and topography and size distribution control the evolution of bridging stresses during the grain-pullout process. For example, the study of bridging tractions in spinel by White and Hay [48] indicated that the magnitude of the bridging stress in spinels of various grain size ranged between 3.5 to 13 MPa.

There are indications that pullout bridging for whiskers is not effective in the absence of residual compressive stresses. In this regard, the author has examined various silicon nitride materials for R-curve behavior and bridging stresses [50]. All of these materials (one hot-pressed, one sintered silicon nitride and one sintered and HIP heat-treated silicon nitride) possessed similar microstructure composed of ~90% high-aspect-ratio β-Si₃N₄ whiskers dispersed in an α-Si₃N₄ matrix. From the three materials examined, however, only one showed indication of R-curve behavior and bridging stresses in spite of their similar microstructures. Although pullout features could also be observed on the fracture surface of the two other silicon nitride materials, the R-curves from these materials were essentially flat. It was concluded that in the case of the material with R-curve behavior the composition of the grain boundary phase would be such that the β-Si₃N₄ whiskers could be under residual compression and give rise to frictional forces during pullout.

Comparison of the results presented in Fig. 6.12 indicates that while the bridging stress is almost nil at 1400°C in the whisker-reinforced material, the material reinforced by the platelets shows a finite value of about 5.6 MPa at this temperature. If we consider that both of the composites are hot-pressed at temperatures above 1600°C and the creep relaxation during cooling from the hot-pressing temperature in both of these materials is limited to 1300°C-1400°C, the bridging stress driven by the thermal residual stresses can be expected to vanish at about the same temperature in both of the composites. If the bridging stress in the platelet-reinforced composite at 1400°C (in the absence of thermal residual stresses) were due
to the contributions from the geometry of the reinforcement, it could be assumed independent of or insensitive to the temperature. Therefore, subtracting the amount of the bridging stress at 1400°C from the bridging stresses should yield that component of the bridging stresses that is due to the thermal residual stresses. The variation of the latter with temperature may then be expected to be the same for the two composites. To examine this hypothesis the data from Fig. 6.12 are re-plotted in Fig. 6.13 based on the description given after normalizing the maximum bridging stress at each temperature to the value at room temperature. The new graph gives us the opportunity to compare the results from this work on the reinforced aluminas with the results from White and Hay on two different monolithic alumina materials [17].

![Graph](image)

**Fig. 6.14:** Variation of the normalized maximum bridging stress with temperature for the SiC-reinforced alumina composites from this work and for monolithic aluminas from Hay and White [17].

It can be seen that after the subtraction of the value of the bridging stress at 1400°C from the platelet-reinforced composite data set the difference between the temperature dependence of the maximum bridging stress in the whisker- and in the platelet-reinforced materials is now almost invisible, if the behavior of the platelet-reinforced composite about 1000°C is neglected. Upon applying this offset subtraction to the bridging stresses of the platelet-reinforced material, both of the composites appear to rely on the same source of bridging stresses. Note that the line representing the trend of the data from the reinforced materials in Fig. 6.14 is the fit to the data from the whisker-reinforced material only.

Comparison between the temperature trend for the initial value of the fracture toughness from the two composites, Fig. 6.15, indicates two essentially different temperature dependencies at temperatures above...
800°C. Below this temperature, the $K_c$ from both of the composites may be considered insensitive to temperature. (It is assumed that the scatter in the data from the platelet-reinforced composite is an artifact caused by hot-pressing-induced defects).

Fig. 6.15: Comparison of the initial value of the fracture toughness of the SiC-platelet-reinforced alumina and SiC-whisker-reinforced alumina as a function of temperature. The error bar on the data represents the scatter of the results from different specimen about the average value. The data from the platelet-reinforced material is the average of the results from tests both under the vacuum and in air.

Above 1000°C, the crack tip toughness of the platelet-reinforced composite shows a more rapid deterioration with increasing temperature, which was related to softening of the residual glass phase at the interface between the matrix and the reinforcement. For the whisker-reinforced material the trend with temperature was found to be different above 1000°C due to the toughening effect exerted by a microcrack damage zone ahead of the crack tip, which in turn was promoted by the diffusion-induced cavitation along the whisker/matrix boundaries. It is interesting to note that a similar behavior cannot be observed in the platelet-reinforced composite, although an amorphous phase already exists at some of boundaries associated with the SiC-platelets (Fig. 6.5) and this, following the discussions on the whisker-reinforced material, should promote cavitation. This may reflect the effect of chemistry/viscosity of the glass phase at the specific temperature and the magnitude of the damage induced ahead of the crack tip by cavitation.

As discussed in the microcrack shielding model [38] and also noted by Han et al [28] the growth and coalescence of microcracks lead to deterioration of the intrinsic toughness of the material at the crack tip while the formation of the microcrack zone itself shields the crack tip from the far field stress intensity
factor. The two mutually competing processes, crack tip shielding and deterioration of the crack tip resistance due to microcrack coalescence, result in a net effect, which may be toughness enhancement or deterioration (anti-shielding or amplification). Whether the net result from this dualism leads to toughening or toughness deterioration depends strongly on the size, density and the position of the microcracks.

The toughness trend with increasing temperature in the platelet-reinforced composite seem to indicate that the damage zone, if also formed in this material, must have fulfilled the local crack tip condition in such a way that the toughness deterioration has been dominant. It may be considered that a larger size of the platelet, and thus a larger local interface with the matrix grains, may lead to the formation of a larger crack by the coalescence of the interfacial cavities and hence a larger microcrack size in the damage zone. This would in turn promote deterioration of the toughness, as was observed at higher temperatures in the whisker-reinforced material.

References


44. Y. -S. Chou and D. J. Green, "Residual Stress-Induced Spontaneous Microcracking in a-SiC Platelet Al2O3 Composites," J. Mater. Sci., 29, 5725-5731 (1994).


Chapter 7

Summary and Conclusions

7.0 Summary

The contribution of the crack wake bridging tractions to the stress intensity factor at the tip of the crack propagating in a chevron-notched flexure specimen was analyzed by employing the fracture mechanics weight function method. Since three degrees of freedom: depth of the apex tip, \(a_o\), depth of the notch, \(a_t\), and specimen width-to-breadth ratio, \(W/B\), describe the geometry of the triangular crack wake, each notch geometry within a specific type of the specimen requires its own weight function (or ideally a weight function that includes all these variables) to accomplish such an analysis. In addition, the variation of the crack width during propagation in a chevron notch limits the applicable analytical techniques to the numerical methods capable of handling three-dimensional problems, e.g., finite element analysis. However, a two-dimensional slice synthesis of the triangular crack wake contributions to the stress intensity factor at the crack tip, using an appropriate weight function from a through-thickness crack geometry, was shown to provide a viable alternative for qualitative discussion of the crack wake effects when the true weight function for the chevron-notched specimen under study was unavailable.

To allow quantitative analysis of the contribution of the bridging stress, \(\sigma_b(x,a)\), to the stress intensity factor at the crack tip numerical solutions were achieved for the crack surface traction weight function in a chevron-notched flexure specimen for three different \(a_t/W\) values from a series of finite element analyses of the specimen geometry. The magnitude of the weight function was obtained by calculating the average stress intensity factor at the crack tip arising from a unit line-load acting over the width of the crack wake in various positions between the tip of the apex and tip of the crack. Although the magnitude of the stress intensity factor, and hence the weight function, was found to vary along the crack front, it was almost constant over a large fraction of the crack front. The values of the weight function as a function of the position of the line-load and crack length were then used to obtain the coefficients of a polynomial fit, satisfying the general shape of the weight functions, in order to simplify further numerical application of the weight function.

The weight function method was used to calculate the R-curves which would arise from specific bridging relations, \(\sigma_b=f(\delta)\), in one of the chevron-notched specimen geometries modeled. The crack opening profile is influenced by both the applied and the bridging stresses in the crack wake. Therefore, correct estimation of the distribution of the bridging stresses and thus the resulting stress intensity factor at the crack tip required solving an integral equation involving the crack opening displacement, \(\delta\), to obtain the equilibrium crack profile. The exact distribution of the bridging stresses in the crack wake, and thus the
stress intensity factor at the crack tip arising from this stress distribution, \( K \), could be obtained by inserting the equilibrium crack profile into the bridging relation. Some examples of the calculated R-curves were compared with the R-curves which would arise from the same \( \sigma_0 = f(\delta) \) in the same specimen with a straight through-thickness crack to illustrate the geometry dependence of the R-curve behavior due to crack wake bridging interactions and to stress the significance of the bridging relation and the form of the weight function in the R-curve problem.

The main goal of this work was to estimate the bridging relation from the experimentally determined R-curves using chevron-notched specimens. A procedure was devised to iteratively deconvolute \( \sigma_0(x,a) \), and thus \( \sigma_0 = f(\delta) \), from the R-curve through a least-squares fit scheme under the implicit assumption that the underlying bridging process was dominated by the frictional pullout.

Two different aluminum oxide ceramic composites each containing a different SiC-reinforcement were subjected to controlled fracture tests under flexure loading conditions between room temperature and 1400°C using the chevron-notch specimen geometry analyzed. One material, a commercially available ceramic cutting tool, contained 33 vol.% of SiC-whiskers as reinforcement. The other material was fabricated by incorporating 20 vol.% SiC-platelets into an alumina matrix and hot-pressing the mixture at 1600°C-1650°C.

A large number of fracture tests were conducted on each of the materials to collect at least three load-displacement curves at each testing temperature: ambient, 800°C, 1000°C, 1200°C and 1400°C. The R-curves were calculated from the load-displacement curves by using the compliance vs. crack length solution obtained by finite element analysis of the specimen. To avoid any influence from the compliance of the load train of the testing setup, the displacement was measured as a differential displacement directly on the tensile face of the specimens between the center-point and two other points symmetrically located about this point. The true compliance of the specimen was calculated from this apparent compliance by employing a closed form relation verified by finite element analysis.

The R-curves from the whisker-reinforced material showed strong run/arrest behavior at room temperature leading to partial destruction of the crack wake during multiple unstable crack jumps following the crack arrest points. As a result of this behavior, the room temperature R-curves from the whisker-reinforced material could not be analyzed to obtain the distribution of the bridging stresses. This crack growth behavior was found to diminish with increasing temperature. The rest of the R-curves determined for test specimens of the two composites were analyzed using the weight function method and the iterative scheme developed to obtain self-consistent \( \sigma_0 = f(\delta) \) and the initial value of the fracture toughness, \( K \), characterizing the toughness of the material in the absence of the crack wake bridging contributions. The \( K \) values from the analyses of the R-curves from the platelet-reinforced composite were verified against the values determined by independent toughness tests using SENB specimens with a notch tip radius of ~30 μm. The maximum bridging stress value from the whisker-reinforced material was compared to literature values for a similar whisker-reinforced alumina composite.
In many circumstances the iterative scheme developed to analyze the R-curves converged to $\sigma_{br}(x,a)$ solutions that resulted in self-consistent (within a small scatter or with minor deviations at large crack opening displacements) $\sigma_{br}=f(\delta)$. However, in some other cases the resulting $\sigma_{br}(x,a)$ from the analysis led to non-unique $\sigma_{br}=f(\delta)$ relations. For these cases, a more rigorous analysis scheme was utilized to assure convergence to self-consistent bridging stress distributions. In this procedure, the distribution of bridging stress as a function of $\delta(x,a)$ was calculated for a series of crack lengths within the range of the experimental results during each iteration step by the fit algorithm. An average $\sigma_{br}=f(\delta)$ was then calculated from this set of bridging relations. The routine was then set to perform the curve fit analysis to the R-curve data from the experiment such that the deviation of the resulting $\sigma_{br}=f(\delta)$'s, calculated from the distribution of the bridging stress $\sigma_{br}(x,a)$ at each of these crack lengths, from their average distribution was simultaneously minimized.

The maximum bridging stress at room temperature was found to be $-23$ MPa in the SiC-platelet-reinforced material and was estimated to be $-25$ MPa in the SiC-whisker-reinforced material based on extrapolation of the trend of these values at high temperatures to room temperature. In both of the composites the maximum bridging stress was found to diminish with increasing temperature while the crack opening displacement range for which bridging interactions exist appeared to extend with increasing temperature. Both of these effects reflected the contribution of the radial residual compressive thermal stresses on the bridging reinforcement to the evolution of the bridging stresses. Both residual stresses and bridging stresses would be, therefore, expected to decrease with increasing temperature. However, the reduction of the radial compressive residual stress simultaneously improves the pullout length of the reinforcement by increasing the debond-length at the reinforcement/matrix interface, increasing both the frictional area and survival chance of the bridges at larger crack opening displacement. This in turn would be manifested in improvement of the R-curve at larger crack length extensions due to the significance of the tractions remote from the crack tip in finite bodies.

In the whisker-reinforced material the maximum bridging stress was found to decrease linearly with increasing temperature while for the platelet-reinforced composite some non-linear effects could be observed near 1000°C. In addition, while no bridging stress could be observed at 1400°C in the whisker reinforced composite, i.e., the R-curve was flat, a finite amount of bridging stress could still be found in the platelet-reinforced material under the same condition. The non-linear behavior of the bridging stresses with temperature around 1000°C in the platelet-reinforced composite was related to the viscoelastic effects from an interfacial amorphous phase found to exist at some of the boundaries associated with the SiC-platelets. Most of the amorphous phase was found in triple junctions formed by the matrix grains and SiC-platelet facets. At higher temperatures, however, no significant non-linear influence could be observed. Presumably the low viscosity of the amorphous phase at such temperatures together with its limited concentration and possible redistribution reduced the influence of this phase under these conditions. The absence of such non-
linearity in the bridging behavior from the whisker-reinforced composite reflected the clean interface between the whiskers and matrix grains in this material. The bridging relations obtained from the R-curves also indicated that the distribution of the bridging stresses in the platelet-reinforced composite was limited to a crack opening displacement almost a factor of two smaller than that from the whisker-reinforced material.

The crack tip toughness of the platelet-reinforced composite was found to diminish with increasing temperature, more drastically above 1000°C, as could be expected by virtue of the presence of the interfacial amorphous phase. For the whisker-reinforced material this value was found to be insensitive to temperature up to 800°C and to increase with increasing temperature to a maximum value at 1200°C followed by reduction toward 1400°C. At all these temperatures, however, the crack tip toughness showed a value above that at room temperature. In agreement with a previous study of this material, a microcrack damage zone which was observed to form near the crack tip was found to be responsible for the crack tip toughening at elevated temperatures. This microcrack zone was produced ahead of the crack tip by coalescence of cavities formed at the interface of the SiC/Al₂O₃ within the region of the material associated with the crack tip stress field. The presence of the microcrack damage zone and cavitation, and their association with the SiC/Al₂O₃ interface could be verified through microscopic examination of the regions of the material adjacent to the fracture surface. No indication of an R-curve behavior associated with the microcrack zone of this kind could be observed, nor did the bridging stress results appear to be influenced by the presence of this microcrack zone.

Micromechanics models of frictional pullout bridging indicate that the magnitude of the bridging stresses in platelet-reinforced material should be considerably lower than in whisker reinforced material. This was not consistent with the experimental results. Two hypotheses were suggested to explain this inconsistency. One hypothesis involved the effect of the residual stresses; the interface between the platelet and matrix might be under residual shear, which could influence the frictional pullout stresses. This hypothesis was developed based on some recent work on the residual stresses in ceramics indicating that the interface of a faceted inclusion with the matrix may experience residual shear and the significance of interfacial shear stresses to the evolution of the bridging stresses. The second hypothesis was that there were contributions from bridging stress interactions other than those due to residual stress induced friction. This followed the observation of a finite bridging stress in the material at 1400°C, where the residual stresses had to be completely relaxed. Processes like grain rotation and asperity loading during pullout were considered to be responsible for this behavior. Such bridging processes were also considered in the past to be responsible for the bridging stresses observed in monolithic cubic ceramics like spinel, and could be expected to be temperature insensitive. Removing the temperature independent component of the bridging stress from the data for the platelet-reinforced material and expressing the bridging results from the two composites in a normalized form based on their room temperature value, the maximum bridging stress results from the two materials appeared to share the same linear trend vs. temperature.
7.1 Conclusions

7.1.1 Theoretical Analysis

- The contribution of the crack wake tractions to the stress intensity factor at the tip of the crack, and thus the R-curve, in the chevron-notched flexure specimens were successfully analyzed by employing the fracture mechanics weight function method. This was accomplished by obtaining numerical solutions for the crack surface traction weight function and for the crack opening profile due to the applied load by using finite element analysis of this crack geometry.

- The theoretically calculated R-curves for an exponential bridging relation could give the geometry dependence of the R-curves by comparing the R-curves arising from crack propagation in the chevron-notched specimen with those from the through-thickness crack geometry. These R-curves also showed a much smoother initial rise in the chevron-notched specimen while those from the through-thickness crack showed a rise proportional to $\sqrt{\Delta a}$ within the same crack propagation range.

- The iterative analysis scheme developed based on the weight function method could be used successfully to deconvolute initial value of the fracture toughness and the distribution of the bridging stresses from the R-curves measured in the experiments. However, yielding a self-consistent bridging stress as a function of crack opening profile from the analysis sometimes required a more rigorous analysis scheme involving calculation of the equilibrium crack profile, and thus the distribution of the bridging stresses as a function of crack opening displacement, at each iteration step.

7.1.2 Experimental Analysis

- The maximum bridging stress at room temperature in a 33 vol.% SiC-whisker- and a 20 vol.% SiC-platelet-reinforced alumina were estimated to be ~25 and ~23 MPa, respectively. These stresses were found to decrease with increasing temperature; for the whisker-reinforced material the bridging stress vanished at $T>1300^\circ C$ while for the material reinforced with the platelets a maximum bridging stress of ~5.5 MPa could still be found at 1400$^\circ C$.

- A linear dependence on temperature was found for the bridging stresses in the whisker-reinforced material. This could be explained based on the role of the residual stresses on the evolution of the bridging stresses and the presence of an interface free of an amorphous phase.
between SiC-whiskers and alumina matrix grains in this composite. However, the bridging stress vs. temperature from the platelet-reinforced material showed some anomalous non-linear behavior about 1000°C, which was related to the viscoelastic behavior of an interfacial amorphous phase found at some of the boundaries associated with the SiC-platelets in this composite. However, further analysis indicated that except for the non-linear behavior about 1000°C in the platelet reinforced material, the temperature dependence of the bridging stresses in both of the composites could be considered as essentially following the same linear trend with increasing temperature.

- An additional temperature effect on the bridging stresses in both of the composites was found to be an increase of the crack opening displacement with bridging interaction with increasing temperature. In general, the bridging relations decovoluted from the R-curves of the composites showed a tendency towards larger crack opening displacements as the temperature was raised. This was found to be in accordance with the microscopic observations of increased pullout length of the reinforcement.

- A microcrack damage zone formed by cavitation at the SiCw/Al2O3 interface within the high local stress field at the crack tip was found to exist in the composite at elevated temperatures. This microcrack zone was also found to give rise to toughening effects, since at all temperatures between 1000°C and 1400°C the crack tip toughness of the material turned out to possess a value larger than that from room temperature.

- The results obtained for the whisker-reinforced material indicated no visible R-curve effect or influence on the bridging stresses due to the presence of the microcrack zone at such temperatures.
Appendix I

Fit Coefficients and Numerical Results

I.a: Coefficients of Eq. 3.6

The coefficients, $A_{n\mu}$, of the weight function of an edge crack in a finite plate [1]:

$$h(x,a) = \frac{2}{\pi a} \sqrt{\frac{1}{1-x/a}} \left[ 1 + \sum_{n=1}^{\infty} \frac{A_{n\mu} a^{\mu}}{(1-a^{2})(1-x/a)^{\nu+1}} \right]$$

<table>
<thead>
<tr>
<th>$\nu$</th>
<th>$A_{n\mu}$</th>
<th>$A_{1\mu}$</th>
<th>$A_{2\mu}$</th>
<th>$A_{3\mu}$</th>
<th>$A_{4\mu}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0.4980</td>
<td>2.4463</td>
<td>0.0700</td>
<td>1.3187</td>
<td>-3.067</td>
</tr>
<tr>
<td>1</td>
<td>0.54165</td>
<td>-5.0806</td>
<td>24.3447</td>
<td>-32.7208</td>
<td>18.1214</td>
</tr>
<tr>
<td>2</td>
<td>-0.19277</td>
<td>2.55863</td>
<td>-12.6415</td>
<td>19.763</td>
<td>-10.9860</td>
</tr>
</tbody>
</table>

I.b: Finite element results for the crack surface traction weight functions.

$h(\xi,a)$, in the chevron-notched bend bar specimens $W/B=1.25$, $L/W=8$, $a/W=1$ and $a/W=0.22, 0.32$ and 0.42.

<table>
<thead>
<tr>
<th>$a/W$</th>
<th></th>
<th>$a/W$</th>
<th></th>
<th>$a/W$</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.4</td>
<td>0.222</td>
<td>0.283</td>
<td>0.250</td>
<td>0.200</td>
</tr>
<tr>
<td>0.5</td>
<td>0.149</td>
<td>0.604</td>
<td>0.789</td>
<td>1.959</td>
</tr>
<tr>
<td>0.6</td>
<td>0.083</td>
<td>0.762</td>
<td>0.583</td>
<td>3.879</td>
</tr>
<tr>
<td>0.8</td>
<td>0.069</td>
<td>1.444</td>
<td>0.414</td>
<td>6.084</td>
</tr>
<tr>
<td>0.9</td>
<td>0.059</td>
<td>4.197</td>
<td>0.235</td>
<td>13.923</td>
</tr>
</tbody>
</table>

Table 1.1: $h(x,a)$ for $a/W=0.22$

<table>
<thead>
<tr>
<th>$a/W$</th>
<th></th>
<th>$a/W$</th>
<th></th>
<th>$a/W$</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.4</td>
<td>0.750</td>
<td>0.298</td>
<td>0.450</td>
<td>0.450</td>
</tr>
<tr>
<td>0.5</td>
<td>0.166</td>
<td>0.253</td>
<td>0.333</td>
<td>0.656</td>
</tr>
<tr>
<td>0.6</td>
<td>0.107</td>
<td>0.304</td>
<td>0.257</td>
<td>0.707</td>
</tr>
<tr>
<td>0.8</td>
<td>0.079</td>
<td>0.584</td>
<td>0.242</td>
<td>0.813</td>
</tr>
<tr>
<td>0.9</td>
<td>0.062</td>
<td>1.014</td>
<td>0.233</td>
<td>0.833</td>
</tr>
</tbody>
</table>

Table 1.2: $h(x,a)$ for $a/W=0.32$

<table>
<thead>
<tr>
<th>$a/W$</th>
<th></th>
<th>$a/W$</th>
<th></th>
<th>$a/W$</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.4</td>
<td>0.375</td>
<td>0.298</td>
<td>0.450</td>
<td>0.450</td>
</tr>
<tr>
<td>0.5</td>
<td>0.166</td>
<td>0.253</td>
<td>0.333</td>
<td>0.656</td>
</tr>
<tr>
<td>0.6</td>
<td>0.107</td>
<td>0.304</td>
<td>0.257</td>
<td>0.707</td>
</tr>
<tr>
<td>0.8</td>
<td>0.079</td>
<td>0.584</td>
<td>0.242</td>
<td>0.813</td>
</tr>
<tr>
<td>0.9</td>
<td>0.062</td>
<td>1.014</td>
<td>0.233</td>
<td>0.833</td>
</tr>
</tbody>
</table>

Table 1.3: $h(x,a)$ for $a/W=0.42$

<table>
<thead>
<tr>
<th>$a/W$</th>
<th></th>
<th>$a/W$</th>
<th></th>
<th>$a/W$</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.4</td>
<td>0.375</td>
<td>0.339</td>
<td>0.450</td>
<td>0.450</td>
</tr>
<tr>
<td>0.5</td>
<td>0.166</td>
<td>0.321</td>
<td>0.333</td>
<td>0.656</td>
</tr>
<tr>
<td>0.6</td>
<td>0.107</td>
<td>0.304</td>
<td>0.257</td>
<td>0.707</td>
</tr>
<tr>
<td>0.8</td>
<td>0.079</td>
<td>0.584</td>
<td>0.242</td>
<td>0.813</td>
</tr>
<tr>
<td>0.9</td>
<td>0.062</td>
<td>1.014</td>
<td>0.233</td>
<td>0.833</td>
</tr>
</tbody>
</table>
l.c. Coefficients of Eq. 3.11

The coefficients, \( A_{\nu_0} \), of the fit relation

\[
h(x, a) = \sqrt{\frac{2}{\pi}} \frac{\xi}{(a - a_0) \sqrt{1 - \xi}} \left[ 1 + \sum_{\nu, \mu=0}^\infty \frac{A_{\nu_0}}{(1 - \alpha)^{3/2}} (1 - \xi)^{\nu+1} \right]; \quad \xi = \frac{x - a_0}{a - a_0}
\]

to the numerical results of Appendix I.b:

\[
A_{\nu_0}; \ a_0/W=0.22
\]

<table>
<thead>
<tr>
<th>( \nu=0 )</th>
<th>( \mu=0 )</th>
<th>1</th>
<th>2</th>
<th>3</th>
</tr>
</thead>
<tbody>
<tr>
<td>v=0</td>
<td>-2.0785</td>
<td>2.7446</td>
<td>7.8175</td>
<td>-7.6139</td>
</tr>
<tr>
<td>1/2</td>
<td>0.9784</td>
<td>4.9004</td>
<td>-17.9156</td>
<td>15.9411</td>
</tr>
<tr>
<td>v=0</td>
<td>-0.0054</td>
<td>-6.3133</td>
<td>16.1245</td>
<td>-11.1495</td>
</tr>
</tbody>
</table>

\[
A_{\nu_0}; \ a_0/W=0.32
\]

<table>
<thead>
<tr>
<th>( \nu=0 )</th>
<th>( \mu=0 )</th>
<th>1</th>
<th>2</th>
<th>3</th>
</tr>
</thead>
<tbody>
<tr>
<td>v=0</td>
<td>-3.5928</td>
<td>8.6026</td>
<td>-1.3268</td>
<td>-2.8235</td>
</tr>
<tr>
<td>1/2</td>
<td>7.5188</td>
<td>-25.8126</td>
<td>27.4251</td>
<td>-6.4034</td>
</tr>
<tr>
<td>v=0</td>
<td>-6.6844</td>
<td>24.8966</td>
<td>-29.8404</td>
<td>11.1440</td>
</tr>
</tbody>
</table>

\[
A_{\nu_0}; \ a_0/W=0.42
\]

<table>
<thead>
<tr>
<th>( \nu=0 )</th>
<th>( \mu=0 )</th>
<th>1</th>
<th>2</th>
<th>3</th>
</tr>
</thead>
<tbody>
<tr>
<td>v=0</td>
<td>-5.6694</td>
<td>12.3795</td>
<td>-2.9021</td>
<td>-3.2004</td>
</tr>
<tr>
<td>1/2</td>
<td>13.5020</td>
<td>-38.0586</td>
<td>30.7401</td>
<td>-3.8265</td>
</tr>
<tr>
<td>v=0</td>
<td>-11.3203</td>
<td>33.8084</td>
<td>-31.7464</td>
<td>8.5356</td>
</tr>
</tbody>
</table>

l.d. Coefficients of Eq. 3.19

The crack opening displacement solution \( \delta(x,a) \) obtained by finite element analysis for the crack in chevron-notched specimen, \( W/B=1.25, a_o/W=0.32 \) under four-point bending load with \( S_1/W=8 \) and \( S_1/S_2=1 \) can be expressed by the best-fit polynomial relation (Eq.3.19):

\[
BE \frac{\delta(x,a) P}{P} = \sum_0^4 C_n(a) \left(1 - \frac{x}{a}\right)^{n+1/2}, \quad 0.33 \leq x/W \leq a/W
\]

to the finite element solution for the average crack opening displacement \( \delta \) at each position \( x \) along the crack length \( a \) of the chevron-notched specimen: \( W/B=1.25, a_o/W=0.32, a_o/W=1 \) under four-point being load \( P \) with the span ratio of \( S_1/S_2=2 \) and \( S_1/W=8 \).
**C\(_\alpha\)(\(\alpha\))**:

<table>
<thead>
<tr>
<th>(\alpha=\alpha/W)</th>
<th>0.4</th>
<th>0.5</th>
<th>0.6</th>
<th>0.7</th>
<th>0.8</th>
<th>0.9</th>
</tr>
</thead>
<tbody>
<tr>
<td>(C_0)</td>
<td>11.9650</td>
<td>12.7387</td>
<td>15.9083</td>
<td>22.6629</td>
<td>38.1221</td>
<td>106.2460</td>
</tr>
<tr>
<td>(C_1)</td>
<td>-42.6968</td>
<td>-18.8005</td>
<td>-9.0129</td>
<td>7.6707</td>
<td>106.904</td>
<td>529.7930</td>
</tr>
<tr>
<td>(C_2)</td>
<td>575.7240</td>
<td>177.8930</td>
<td>120.7170</td>
<td>101.7520</td>
<td>232.5890</td>
<td>541.6720</td>
</tr>
<tr>
<td>(C_3)</td>
<td>-3854.3600</td>
<td>-637.4520</td>
<td>-332.4530</td>
<td>-256.3440</td>
<td>536.7330</td>
<td>458.0810</td>
</tr>
<tr>
<td>(C_4)</td>
<td>9843.9700</td>
<td>823.1050</td>
<td>318.0680</td>
<td>209.3460</td>
<td>-449.1680</td>
<td>-165.0010</td>
</tr>
</tbody>
</table>

**I.e:**  Finite element results for the compliance vs. crack length of the chevron-notched bend bar specimen

Normalized compliance as a function of crack length of the chevron-notched bend bar specimen, \(W/B=1.25, \alpha/W=0.32\) under four point bending condition \(S_p/W=8\) and \(S_p/S_Z=2\), calculated by finite element modeling:

<table>
<thead>
<tr>
<th>(\alpha=\alpha/W)</th>
<th>0.4</th>
<th>0.5</th>
<th>0.6</th>
<th>0.7</th>
<th>0.8</th>
<th>0.9</th>
</tr>
</thead>
<tbody>
<tr>
<td>(\lambda(\alpha))</td>
<td>122.091</td>
<td>141.936</td>
<td>180.673</td>
<td>264.455</td>
<td>499.027</td>
<td>1731.0</td>
</tr>
</tbody>
</table>

**I.f:**  Coefficients of Eq.5.1

The numerical values of Appendix I.e can be expressed by the best-fit polynomial relation:

\[
\lambda(\alpha) = \lambda_\alpha + (\frac{\alpha}{1-\alpha})^2 \sum_{i=0}^{5} A_i \alpha^i \quad \alpha=\alpha/W, \quad 0.32 \leq \alpha \leq 0.9
\]

with \(\lambda_\alpha=112.655\) also obtained by finite element analysis and the fit coefficients given in the following table:

<table>
<thead>
<tr>
<th>i</th>
<th>0</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
</tr>
</thead>
<tbody>
<tr>
<td>(A_i)</td>
<td>-614.8050</td>
<td>4638.0788</td>
<td>-13416.2585</td>
<td>19553.3486</td>
<td>-14340.3355</td>
<td>4203.2797</td>
</tr>
</tbody>
</table>

**References:**

Appendix II

Supplementary Calculations and Models

II.a: Derivation of the Geometric Function and Crack Opening Displacement Solutions as a Function of Span-to-Width Ratio. \((S_1-S_2)/W\) for the Chevron Notch Geometry \(W/B=1.25, a/W=0.32\) and \(a/W=1\):

In order to reduce the time required for the finite element calculations, the geometric function and crack opening displacement solutions required for the other specimen size/loading configurations used in this work were derived from the original finite element solution, Appendix I.d and I.e and, through the following procedure.

It is assumed that similar to the case for the through-thickness crack under flexure [1] the geometric function, \(Y(\alpha)\), for the specific chevron-notch geometry is linearly proportional to the span-to-width ratio:

\[
Y(\alpha) = \frac{3S_1-S_2}{2W} \frac{\sqrt{\alpha F(\alpha)}}{1-\alpha^{3/2}} \quad (\text{II.1})
\]

To verify if this relation (Eq. II.1) also holds for the chevron-notched specimens, we may use the Bluhm's slice model Eq.2.9. This will in turn require the knowledge of the shear correction factor, \(k\), which also varies with the span-to-width ratio, loading configuration as well as with the crack length. It can be easily seen, for instance by comparing the compliance solution of Appendix I.d with the results from the Bluhm's model, that neglecting the latter would cause only an error of \(-3\%\) in the compliance vs. crack length, \(\lambda(\alpha)\), and \(Y(\alpha)\) functions. The initial value of the shear correction factor can be obtained by a simple finite element modeling. In this case the finite element model is a simple three-dimensional quarter-specimen model composed of 500-700 linear elements. This gives \(k=1.600\) and \(k=1.536\) for the three-point bending load configuration and for the \((S_1-S_2)/W=10\) used in this work, respectively.

Using the above values of \(k\) the \(\lambda(\alpha)\) and hence \(Y(\alpha)\), can be calculated for each of these configurations. For this purpose the compliance of the specimen with the through-thickness crack, required in the Bluhm's model, under the four-point bending condition was calculated numerically from the geometric function solution from Gross and Srawly [1] by using the relation:

\[
\lambda(\alpha) = \lambda_0 + 2 \int_0^\alpha Y^2(\alpha) d\alpha \quad (\text{II.2})
\]
where $\lambda_o$ is the normalized compliance of the solid beam without a crack. For the three-point bending condition, the compliance crack length relation for the specimen with a through-thickness crack was obtained from the compliance solutions presented by Fett [2] for various $S/W$ ratios.

The $F(\alpha)$ from the right-hand side of Eq. II.1 calculated for each of the configurations in question and from the geometric function calculated from the finite element compliance solution of Appendix I.e are shown in Fig. II.1.

![Graph](image)

**Fig. II.1:** Verification of the Eq. II.1 for the chevron-notch specimen geometry used.

The minor deviation visible in the region $\alpha_o < \alpha < 0.4$ is due to the sensitivity of the compliance solution from the Bluhm's slice model to variation of the shear correction factor with the crack length.

Similarly, a linear dependence on the span-to-width ratio will also hold for the crack opening displacement solution, Appendix I.f. However, for the compliance solution it will hold, following Eq. II.2, that:

$$
\Delta \lambda(\alpha) = \lambda(\alpha) - \lambda(\alpha_o) = \left(\frac{S_1 - S_2}{W}\right)^2 f(\alpha)
$$

(II.3)

where $\lambda(\alpha_o)$ will be 299.697 and 201.495 for the three-point bending condition with $S/W=8$ and for the four-point bending condition with $(S_1-S_2)/W=10$, respectively. The $\lambda(\alpha)$ functions calculated based on the above relation are compared with those obtained through Bluhm's slice model in Fig. II.2. It must be
remembered that the span-to-width ratio is also implicit in the finite element compliance solution, Appendix I.d.

\[ \text{Fig. II.2: Verification of Eq. II.3.} \]

**II.b: Toughening and R-curve Due to a Microcrack Zone ahead of the Crack Tip**

The effect of the microcrack zone ahead of the crack tip on the toughening and R-curve behavior may be described through the following consideration [3].

During loading of a specimen containing a sharp crack, a microcrack process zone is formed ahead of the crack tip without any crack growth. The size of the process zone is proportional to square of the applied stress, \( \sigma^2 \). As a result of formation of microcracks at the grain boundaries of a proper orientation some energy will be consumed. This will in turn increase the specimen compliance; the increase of the specimen compliance would be interpreted as an indication of crack advance, if the compliance method is used for crack length determination, although the physical length of the crack is still the initial length, \( a_0 \). This is schematically shown in Fig. II.3b. When the process zone reaches a critical length, the crack and the zone size then increase by \( \Delta a \).

From this visualization it follows then
The critical stress intensity factor at the onset of the actual crack extension contains the influence of the initial process zone at the crack tip.

Further energy consumption by the process zone is proportional to \( \Delta a \) and, therefore, the stress intensity factor required for further crack extension remains constant, i.e., the R-curve would be flat. Only when the zone approaches the specimen boundaries should a clear change be observed in the stress intensity factor.

**Fig. II.3:** Schematic representation of crack/microcrack zone interaction after [3]: a) initial crack configuration, b) formation of the initial process zone, c) process zone at the verge of crack extension and d) crack extension.

The R-curves obtained from the true crack measurement and compliance estimation of the crack due to the process visualized in Fig. II.3 is shown in Fig. II.4.

**Fig. II.4:** Schematic of R-curve due to crack propagation in the presence of the microcrack zone of Fig. II.3.
II.c: \textbf{Frictional pullout stress from the plate like particle}

The frictional stresses exerted parallel to side $l_i$ and perpendicular to side $l_2$ of the plate-like particle shown in the schematic follows:

\[
\sigma_{po} = \frac{2\tau(l_2 + d)(l_{po} - 2\delta)}{l_2d}
\]  

where $\tau$ is the shear traction acting on the frictional interfaces between the plate-like particle and the matrix. If it is assumed that $l_1=l_2=l$ and $R=\nu d$ this will give:

\[
\sigma_{po} = \frac{2\tau.R.d(1+\frac{1}{R})(l_{po} - 2\delta)}{Rd^2}
\]  

Considering that for $R > 10$ the term $(1+1/R)=1$, this gives:

\[
\sigma_{po} = \frac{2\tau(l_{po} - 2\delta)}{d}
\]  

\textbf{References}


Appendix III

Experimental Apparatus

III.a: Setup for the Preparation of the Chevron Notch

The setup used for cutting a chevron notch into the flexure specimens, as described in section 4.1.3, is shown in Fig. III.1a. The details of the cutting fixture, specimen and diamond blade configuration are shown in Fig. III.1b. Each side of the triangular notch is produced in one run as the notching fixture approaches and passes the rotating diamond blade (Fig. III.1b). The fracture plane from a specimen produced by using this setup is shown in Fig. III.1c. The specimen shown has W=5.00 mm, B=3.99 mm and a_n=1.65 mm (as was measured on the fracture plane).

Fig. III.1: Chevron notch cutting setup: a) apparatus, b) fixture details and c) a typical notch produced.
III. b: Test Setup for Controlled Fracture Test at High Temperatures in Air

The complete test setup used for fracture tests at room and elevated temperature in air is shown in Fig. III.2. A closer view of the displacement-measuring unit of the high-temperature testing module is shown in Fig. III.3. As described in section 4.2.3, the LVDT is housed in the sea-saw mechanism underneath the water-cooled base of the setup (Fig. III.3) and measures the differential displacement on the tensile face of the specimen between the center point and two other points located symmetrically about the center point, close to the lower-rollers, through the three alumina rods. As such, the measured differential displacement remains undisturbed by the compliance of the load-train of the test setup.

A close view of the bend fixture holding a specimen under the four-point bending condition is presented in Fig. III.4 and clearly shows the arrangement of the alumina rods. Note that in this picture the right half of the specimen is located behind the guide block on the right. The chevron notch on the specimen is located right at the top the center alumina rod and is visible in this image.

Fig. III.2: The test setup used for the fracture tests at room and elevated temperatures in air.
Fig. III.3: Close view of the displacement measuring mechanism of the high-temperature fracture test setup.

Fig. III.4: Close view of the SiC bending-fixture holding a chevron-notched specimen under four-point loading condition.