Stress-Induced Heat Generation and Strain Localization in Polycrystalline and Nanocrystalline Nickel

by

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A thesis submitted in conformity with the requirements for the degree of Master of Applied Science
Graduate Department of Materials Science and Engineering
University of Toronto

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Master of Applied Science
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Abstract
Commercially available polycrystalline Ni (Ni200; grain size: 32 μm) and electrodeposited nanocrystalline Ni (grain size: 57 nm), Ni-2.6%Fe (grain size: 25 nm) and Ni-8.5%Fe (grain size: 20 nm) were analyzed for the phenomena of stress-induced heat generation and strain localization during plastic deformation at room temperature (i.e. 25°C). Tensile specimens according to ASTM E8 standard dimensions were tested at strain rates of 10⁻²/s and 10⁻¹/s, respectively, to record the amount of heat dissipated and the change of localized strain using a high resolution infrared (IR) detector and digital image correlation (DIC) camera, respectively. Results have shown that the maximum temperatures that were recorded in nanocrystalline Ni and Ni-Fe alloys were at least 30°C lower than the onset temperatures for subgrain coalescence previously measured through differential scanning calorimetry. It can be concluded that thermally activated grain growth during tensile testing of nanocrystalline Ni and Ni-Fe alloys is not likely to occur.
Acknowledgments

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<th>Symbol</th>
<th>Definition</th>
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<tbody>
<tr>
<td>d</td>
<td>Average grain size</td>
</tr>
<tr>
<td>$D_{gb}$</td>
<td>Grain boundary diffusion coefficient</td>
</tr>
<tr>
<td>$D_v$</td>
<td>Lattice diffusion coefficient</td>
</tr>
<tr>
<td>k</td>
<td>Material constant</td>
</tr>
<tr>
<td>$k_b$</td>
<td>Boltzmann’s constant</td>
</tr>
<tr>
<td>$T$</td>
<td>Temperature</td>
</tr>
<tr>
<td>$\dot{T}$</td>
<td>Rate of temperature increase</td>
</tr>
<tr>
<td>$f_{IN}$</td>
<td>Intercrystalline volume fraction</td>
</tr>
<tr>
<td>$f_{GB}$</td>
<td>Grain boundary volume fraction</td>
</tr>
<tr>
<td>$f_{TJ}$</td>
<td>Triple junction volume fraction</td>
</tr>
<tr>
<td>H</td>
<td>Hardness</td>
</tr>
<tr>
<td>$H_0$</td>
<td>Material constant</td>
</tr>
<tr>
<td>b</td>
<td>Burger’s vector</td>
</tr>
<tr>
<td>$L_j$</td>
<td>Length of individual grain boundary</td>
</tr>
<tr>
<td>c</td>
<td>Heat capacity</td>
</tr>
<tr>
<td>p</td>
<td>Driving force</td>
</tr>
<tr>
<td>$k$</td>
<td>Heat conductance</td>
</tr>
<tr>
<td>$\dot{W}_p$</td>
<td>Rate of plastic work</td>
</tr>
<tr>
<td>n</td>
<td>Strain hardening exponent</td>
</tr>
</tbody>
</table>
Greek Symbols

$\Delta$  Grain boundary width

$\dot{\varepsilon}$  Strain rate

$\sigma$  Stress

$\rho$  Material density

$\sigma_0$  Material constant

$\sigma_y$  Yield strength

$\sigma_{UTS}$  Ultimate tensile strength

$\tau$  Torque

$\theta_j$  Grain boundary misorientation angle

$\gamma_j$  Individual grain boundary energy

$\beta$  Taylor-Quinney coefficient

$\varepsilon^e$  Elastic strain

$\varepsilon^p$  Plastic strain

$\alpha$  Thermal expansion coefficient

$\lambda$  Lame constants

$\mu$  Lame constants

$\dot{\varepsilon}^e_{kk}$  Elastic strain rate

$\dot{\varepsilon}^p_{ij}$  Plastic strain rate

$\sigma_{ij}$  Flow stress

$\varepsilon_{22}$  Width strain

$\varepsilon_{33}$  Thickness strain
<table>
<thead>
<tr>
<th>Acronyms</th>
<th>Definition</th>
</tr>
</thead>
<tbody>
<tr>
<td>%E.L.</td>
<td>Percentage elongation</td>
</tr>
<tr>
<td>SEM</td>
<td>Scanning electron microscopy</td>
</tr>
<tr>
<td>TEM</td>
<td>Transmission electron microscopy</td>
</tr>
<tr>
<td>EDS</td>
<td>Energy dispersive X-ray spectroscopy</td>
</tr>
<tr>
<td>MDSC</td>
<td>Modulated differential scanning calorimetry</td>
</tr>
<tr>
<td>NBED</td>
<td>Nanobeam electron diffraction</td>
</tr>
<tr>
<td>SAD</td>
<td>Selected area diffraction</td>
</tr>
<tr>
<td>DIC</td>
<td>Digital image correlation</td>
</tr>
<tr>
<td>IR</td>
<td>Infra red</td>
</tr>
<tr>
<td>UTS</td>
<td>Ultimate tensile strength</td>
</tr>
<tr>
<td>FCC</td>
<td>Face centre cubic</td>
</tr>
<tr>
<td>VHN</td>
<td>Vickers hardness number</td>
</tr>
<tr>
<td>FEM</td>
<td>Finite element modeling</td>
</tr>
<tr>
<td>MD</td>
<td>Molecular dynamics</td>
</tr>
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</table>
1. Introduction

Over the past several years, considerable progress has been made regarding the understanding of the mechanical properties of nanocrystalline materials, in particular nanocrystalline metals [e.g. Weertman, 2007, Koch, 2007, Whang, 2011]. For the case of nanometals produced by the electrodeposition method, significant increases in yield strength and hardness can be attributed to the Hall-Petch grain size hardening mechanism [e.g. Erb et al., 2007]. The effects of grain size on other mechanical properties such as the Young’s modulus, wear resistance and coefficient of friction of nanocrystalline electrodeposits are also well documented [e.g. Erb et al., 2007; Erb, 2010]. However, some of the details related to issues such as deformation mechanisms, intrinsic ductility or strain hardening capacity of these materials are still not well understood.

One particular concern that needs to be addressed is the possibility of localized heating of specimens during tensile testing which could have a considerable effect on the microstructural evolution and the resulting tensile properties. Previous studies have reported grain growth effects during tensile testing in nanocrystalline Ni [Dalla Torre et al., 2002], Ni-Fe [Fan et al., 2006] and Co-P [Fan et al., 2006], all produced by the electrodeposition method. For example, nanocrystalline Ni with a grain size of 20 nm was tested in tension at strain rates between $10^{-5}$/s and $10^{-1}$/s. Shear bands were observed at fracture angles of $55 – 65^\circ$ with respect to the tensile axis [Dalla Torre et al., 2002]. Transmission electron microscopy in one of the shear band regions at the higher strain rates revealed considerable grain growth. It was speculated that this grain growth was due to the kinetic energy during high speed deformation which could result in local hot spots within the shear band reaching temperatures on the order of $300^\circ$C. For the case of nanocrystalline Ni-18 wt% Fe electrodeposits, substantial grain growth was also observed at a strain rate of $10^{-3}$/s [Fan et al., 2006]. In this case, the grain size increased from 23 nm in the as-plated state to 60 nm in the deformed materials. Some abnormal grain growth was also observed, leading to sizes of some of the grains in excess of 200 nm.

With respect to the thermal stability of nanocrystalline Ni, it has been previously shown that annealing above $100^\circ$C can already result in some sub-boundary coalescence [Klement et al., 1995]. Major grain growth occurred at temperatures between $260^\circ$C and $290^\circ$C depending on
starting grain size and impurity content [Erb et al., 2007]. The major grain growth temperature for Ni-20% Fe alloy was 380°C.

In recent years, infrared imaging technology in conjunction with mechanical property testing has developed rapidly [Rogalski, 2002]. For example, high resolution infrared imaging technology is of large interest in the analysis of thermoelastic stress development [Backman and Greene, 2008] which effects cold expansion of holes [Backman et al., 2010] in aerospace components. The infrared technique has also been applied successfully to study heat generation due to plastic deformation in tensile testing. Heat generation during plastic deformation at strain rates up to 10/s with the infrared technique has been analyzed for TRIP steels [Rusinek and Klepaczko, 2009]. The results showed a maximum temperature increase of 35K within the strain hardening regime; 58K near the necking regime and 90K just before sample fracture. Temperature increases during deformation could lead to thermal softening, which in turn could affect the failure process. It is well known [Bever et al., 1973; Taylor and Quinney, 1934] that most of the energy of plastic deformation is dissipated as heat and the remaining balance stored in the material as strain energy (e.g. lattice distortions, defect formation). This subject is of considerable interest in the study of stored energy and its effects on the process of recrystallization during deformation.

Another issue that needs to be addressed is the phenomenon of strain localization during deformation of nanocrystalline electrodeposits, which could have a considerable effect on the overall mechanical properties. In recent years, digital image correlation (DIC) strain measurements [Chu et al., 1985] during tensile testing have been widely used to study such strain localizations. Examples include studies on the effect of cold expansion of holes using thermoelasticity and digital correlation [Backman et al., 2008, Backman et al., 2010] and the analysis of stress distributions on turbine blades [Backman and Greene, 2008]. The effects of strain localization on mechanical properties have been documented for several materials. One particular study on strain localization using DIC acquired full-field necking behaviour of mild steel deformed at a strain rate of 10^-3/s [Coppietenrs et al., 2011]. Localized strain gradually developed and built up during the deformation process. The results showed that the highest localized strain was located at the necking zone beyond ultimate tensile strength and the position of highest localized strain corresponded to the area of fracture. In another study, the
rapid evolution of shear banding close to the necking region was captured with the DIC method on fully dense nanocrystalline Ni at a strain rate of $10^{-4}$/s [Zhu et al., 2010].

The purpose of this study is to correlate the phenomena of stress-induced heat dissipation and strain localization for both polycrystalline and nanocrystalline Ni and to analyze the coupling effect to the thermal-mechanical properties during plastic deformation. A specific objective of this work is to quantify the amount of heat dissipated during plastic deformation for both polycrystalline and nanocrystalline Ni and to evaluate the possibility of thermally activated grain growth in nanocrystalline Ni due to stress-induced heat generation during deformation.

This thesis is organized as follows: Chapter 2 is divided into four sections presenting a literature review on the current knowledge of the synthesis and the mechanical properties of electrodeposited nanocrystalline materials, the Hall-Petch strengthening mechanism, as well as the deformation mechanisms in nanocrystalline materials. Previous studies dealing with the effects of strain localization and heat generation during deformation will also be reviewed. Chapter 3 presents the experimental details regarding experimental design, the various experimental techniques used in the characterization of the samples and subsequent property measurements. Chapter 4 comprises results and discussion of the current study. An analysis of the strain localization and heat generation due to plastic deformation as a function of grain size and strain rates will be presented. Chapter 5 contains the conclusions from this work and Chapter 6 gives recommendations for future work on this subject.
2. Literature Review

The effects of grain size on the mechanical properties are well documented for nanocrystalline materials. However, the possibility of localized strain and heating of specimens during tensile testing could have an effect on the microstructure, as well as the overall mechanical properties of these materials. Section 2.1 of the literature review covers the fundamentals of nanocrystalline materials, the electrodeposition synthesis method and mechanical properties in terms of the Hall-Petch mechanism. Section 2.2 addresses different types of deformation mechanisms for these materials. Sections 2.3 and 2.4 review the effects of strain localization and heat generation during plastic deformation, respectively.

2.1 Electrodeposited Nanocrystalline Materials

2.1.1 Nanocrystalline Materials Overview

The relationship between mechanical properties and grain size has been a subject of numerous investigation since the early work of Hall [Hall 1951] and Petch [Petch 1953]. Reduced grain sizes in crystalline materials contribute to higher yield strength and hardness. This is due to the fact that grain boundaries are very effective barriers for dislocation slip (section 2.1.3). One particular way to express the effect of grain size on the intercrystalline defect density (grain boundaries, GB, triple junctions, TJ) is to present the volume fractions of atoms associated with these defects for varying grain sizes. Palumbo et al. [1990] proposed a regular 14-sided tetrakaidecahedron as a grain shape model (Fig. 2.1) to estimate the volume fractions of atoms located at all interfacial defects ($f_{IN}$), grain boundaries ($f_{GB}$) and triple junctions ($f_{TJ}$) as a function of grain size $d$. With a grain boundary thickness ($\Delta$), the volume fractions of the different components were estimated according to Eqs. (1–3).

\[
f_{IN} = 1 - \left(1 - \frac{\Delta}{d}\right)^3 \tag{1}
\]

\[
f_{GB} = \frac{3\Delta}{d}\left(1 - \frac{\Delta}{d}\right)^2 \tag{2}
\]

\[
f_{TJ} = f_{IN} - f_{GB} \tag{3}
\]
Figure 2.1: (a) The 14-sided tetrakaidecahedron, and (b) The arrangement of several 14-sided tetrakaidecahedra to form a polycrystalline structure.

GB: grain boundary; TJ: Triple junction [Palumbo et al. 1990]

Figure 2.2 shows the corresponding plot of the Eqs. (1 – 3) for grain sizes smaller than 1 μm and assuming a reasonable grain boundary thickness of Δ = 1 nm. At grain sizes above 100 nm, the intercrystalline volume fraction is relatively small (less than 3%). However, for grain sizes less than 10 nm, the intercrystalline volume fraction increases rapidly to about 50% at a grain size of 5 nm. In other words, there is a substantial increase in the volume fraction of atoms that reside at grain boundaries and triple junctions [Palumbo et al. 1990] in nanocrystalline materials. Other studies (e.g. Weertman 2007, Koch, 2007) also pointed out that the volume fraction of the intercrystalline component increases greatly when the grain size is reduced to the nano-regime (i.e. less than 100 nm).

With the increase of intercrystalline volume fraction, nanocrystalline materials below 100 nm grain size exhibit many outstanding properties, such as: ultra-high yield and ultimate tensile strengths, excellent wear resistance, high solute solubility and diffusivity, superior magnetic properties and electrical resistance, as well as appealing superplastic formability at low temperature in some materials [Gleiter, 1989].
Figure 2.2: Volume fractions of all atoms associated with the interfaces in fully dense nanocrystalline materials as a function of grain size, assuming a regular 14-sided tetrakaidecahedron as the grain shape and a grain boundary thickness $\Delta$ of 1 nm [Palumbo et al. 1990]

2.1.2 Synthesis of Electrodeposited Nanocrystalline Materials

Researchers in the past tried numerous synthesis techniques to fabricate nanocrystalline materials with grain sizes less than 100 nm, such as inert gas condensation [e.g. Gleiter 1989, Sanders et al. 1997], ball milling [e.g. Fecht 2002, Morris and Morris 1997] and severe plastic deformation [e.g. Valiev et al. 1993, Popov et al. 1997]. In the attempt to control the volume fractions of grain boundaries and triple junctions in the material, Erb et al. [2007] applied the process of electrochemical synthesis to electrodeposit nanocrystalline metals to achieve outstanding physical, chemical and mechanical properties. Electrodeposition is a process that requires an external power supply (direct current or pulsed current) to connect a cathode and anode immersed in an electrolyte to drive the deposition. Metal ions dissolved at the anode deposit onto the cathode with both electrodes immersed into a solution which contains a salt of
the metal to be plated. The cations of the salt solution carry a positive charge and are attracted to the negatively charged cathode. When cations reach the cathode, they are reduced to the metallic form. The result is a layer of metal deposited on the cathode. Figure 2.3 shows a simple direct current electrodeposition setup.

![Diagram of a direct current electrodeposition setup](image)

*Figure 2. 3: Schematic diagram of a direct current electrodeposition setup*

By changing the operating parameters such as the plating bath composition, temperature, pH, current density, current waveform and bath agitation during the electrodeposition process, the quality and the microstructure of the final product can be significantly influenced [El-Sherik and Erb 1995]. Nanocrystalline materials can be produced by electrodeposition at high overpotential and at conditions of low atom surface diffusion rates for which the nucleation of new grains is favored over the growth of existing grains. If growth conditions are maintained to favor massive nucleation, electrodeposition has the ability to produce fully-dense bulk nanocrystalline metals and alloys with very small grain size (i.e. less than 100 nm).
2.1.3 Mechanical Properties of Nanocrystalline Materials

The strength dependence of polycrystalline materials on grain size has been described by the classical Hall-Petch relationship [Hall 1951, Petch 1953]. Hall-Petch strengthening is interpreted on the basis of the dislocation pile-up theory during plastic deformation in polycrystalline material. Dislocations pile up against grain boundaries, where grain boundaries act as barriers to further dislocation motion [e.g. Reed-Hill et al. 1994]. Materials show an increase in yield strength ($\sigma_y$) and hardness ($H$) with decreasing grain size from the micrometer to the nanometer range. The Hall-Petch relationship is expressed as Eqs. (4 – 5)

$$\sigma_y = \sigma_0 + k d^{-1/2}$$  \hspace{1cm} (4)

$$H = H_0 + k' d^{-1/2}$$  \hspace{1cm} (5)

where $\sigma_y$ and $H$ are the yield strength and hardness of the materials, $\sigma_0$ and $H_0$ represent the frictional stress required to move individual dislocations within individual grains, $k$ and $k'$ are material constants and $d$ is the average grain size.

Figure 2.4 clearly shows the Hall-Petch relationship for the case of flow stress $\sigma_y$ as per Eq. (4). Flow stress increases constantly with the reduction of grain sizes until ~ 100 nm. Once the grain size decreases below 100 nm, many nanocrystalline materials show a reduced slope with decreasing grain size. It is generally believed that for grain sizes within the range of 100 nm to 10 nm, deformation which, for large grain sizes is mainly due to dislocation activity, starts to involve other grain-boundary-assisted mechanisms such as grain boundary slip/sliding or diffusional creep [e.g. Chokshi et al. 1989, Van Swygenhoven and Derlet 2001, Kumar et al. 2003].

Further grain size reduction resulted in softening of many nanomaterials [Chokshi et al. 1989, Palumbo et al. 1990, Erb et al. 2007]. In these cases, the flow stress or hardness of nanocrystalline materials were observed to peak at typically 10 nm grain size (Fig. 2.4). The negative flow stress/hardness behaviour is the so-called inverse Hall-Petch effect. For example, the softening behaviour was reported by Palumbo et al. [1990] for a series of electroplated nanocrystalline Ni-P alloys (grain size range: 3-20 nm). Palumbo et al. showed that dislocation
activity apparently is no longer dominant and other grain boundary-assisted deformation mechanisms could control plastic deformation in this grain size range.

Figure 2.4: Hall-Petch relationship for the flow stress ($\sigma_y$) of polycrystalline materials as a function of grain sizes [Kumar et al. 2003]

When the grain size in Ni is reduced to the nano-regime (approximately less than 100 nm), the generation of dislocations is limited to grain boundaries and dislocation movement across the grains is still achievable. However, further decrease in grain size (to approximately 10 nm) would make dislocation bowing out of grain boundaries difficult for nanocrystalline Ni [Ebrahimi et al. 2004]. The large grain boundary areas could instead contribute to a sliding mechanism to accommodate plastic strain. This will be discussed in section 2.2, together with other possible deformation mechanisms in nanocrystalline metals.

2.2 Deformation Mechanisms of Nanocrystalline Materials

2.2.1 Grain Boundary Sliding
As the grain size in nanocrystalline material decreases, dislocation slip activity (Fig. 2.5a) is no longer the dominant deformation mechanism. Many other types of grain-boundary-assisted deformation mechanisms are activated instead. One mechanism is grain boundary sliding (Fig. 2.5b) which originally was proposed by Gifkins and Snowden [1966] for polycrystalline
materials. Grain boundary sliding is a deformation mechanism which accommodates plastic strain by the relative sliding of the adjacent grains. The sliding behaviour of grains is to maintain grain contiguity and to prevent the formation of internal voids and cracks from the grain separation, which is achieved by diffusional movement of atoms along the grain boundary during the shear process which occurs in the direction of the grain boundary [Dieter 1988].

![Figure 2.5: Types of deformation mechanisms in nanocrystalline materials](image)

(a) Dislocation slip; (b) Grain boundary sliding; (c) Diffusional creep;
(d) Grain rotation; (e) Mechanical twinning [Erb et al. 2004]

### 2.2.2 Diffusional Creep

In conventional polycrystalline materials, diffusional creep (Fig. 2.5c) occurs by the transport of vacancies/atoms along grain boundaries or the lattice at high temperatures and relatively low stresses. Diffusional flow can either be controlled by the Nabarro-Herring (lattice diffusion) or Coble-type (grain boundary diffusion) creep processes. The two types of diffusional creep mechanisms are expressed as follows [Dieter 1988]:
where $\dot{\varepsilon}$ is the strain rate, $\sigma$ is the applied stress, $b$ is the Burgers vector of the dislocation, $D_v$ and $D_{gb}$ are the lattice and grain boundary diffusion coefficients, respectively, $k_B$ is Boltzmann’s constant, $T$ is the temperature and $d$ is the grain size. At lower temperatures in the nanocrystalline grain size regime, Coble-type (Eq. 7) of grain-boundary diffusional creep dominates over Nabarro-Herring type (Eq. 6) creep. Coble [1963] observed that the reduction of grain size enhances the diffusional creep rate through grain boundary diffusion as a result of the increase of grain boundary density.

### 2.2.3 Grain Rotation

A recent investigation of grain growth revealed the possibility of grain rotation (Fig. 2.5d) induced grain coalescence during mechanical deformation [Moldovan et al. 2002]. Considering a grain in a two-dimensional polycrystals, the driving force for grain rotation can be expressed in the form of the cumulative torque ($\tau$)

$$
\tau = \sum_j L_j \frac{d\gamma_j}{d\theta_j}
$$

where $L_j$ represents the lengths of individual grain boundaries with energies $\gamma_j$; $\theta_j$ is the misorientation angle across the grain boundary between a grain and some adjacent grain $j$ [Moldovan et al. 2002]. This constitutive model (Eq. 8) shows that grain rotations are more favorable in nano-scaled grain sizes with generally high grain boundary energies and low misorientation angles.

Grain rotation resulting in grain coalescence is of importance during the nucleation stage of recrystallization or in grain growth processes during deformation. Li [1962] suggested the possibility of subgrain rotation during recrystallization, especially at small grain sizes where the kinetics of rotation is more rapid. Wang et al. [2008] studied the in-situ tensile deformation of nanocrystalline Ni (grain size: 20 nm) using nanobeam electron diffraction (NBED).
Nanocrystalline Ni showed evidence of deformation induced grain rotation in which small grains in the agglomerate rotated and adjusted their orientation to coalesce into larger grains of about 35 nm in size.

A series of molecular dynamics studies by Cahn and co-workers [Cahn et al. 2006] have also shown that stress-induced grain boundary migration may also result in grain rotation resulting in grain growth. In these studies, it was pointed out that such a phenomenon could be of particular importance in nanocrystalline materials.

### 2.2.4 Mechanical Twinning

Mechanical twinning (Fig. 2.5e) is an alternative mode of plastic deformation in nanocrystalline materials. Experimental and molecular dynamics modeling studies have shown that twinning is often observed in nanocrystalline materials of low stacking fault energies to accommodate deformation stress [Schiotz 2004, Yamakov et al. 2002, Karimpoor et al. 2003, Kumar et al. 2003]. Twinning appears in the form of partial dislocations nucleating at grain boundaries [Froseth et al. 2005].

An atomic simulation study on nanocrystalline Cu (grain size: 39 nm) by Schiotz [2004] showed that deformation twinning occurs after a total strain of 6.45% at high strain rates of 5 x 10^8/s. A significant number of partial dislocations were generated, separated by narrow stacking fault ribbons. Another simulation study by Yamakov et al. [2002] revealed the mechanism for twin formation in nanocrystalline Al (grain size: 45 nm). A variety of processes involving the interactions of dislocation with grain boundaries and the formation of deformation twins were observed after approximately 12% plastic strain. Simulations provided an insight into the possibility that twins can originate at both grain boundaries and in the grain interiors; the latter resulting from the interactions of stacking faults.

On the other hand, experimental results were also presented which support the importance of twinning for the mechanical properties of nanocrystalline metals. Mechanical twinning was suggested to occur in nanocrystalline Co (grain size: 12 nm) to accommodate plastic strain [Karimpoor et al. 2003]. In contrast to the response of polycrystalline Co in which deformation
is dominated by dislocation slip, the twinning in nanocrystalline Co was possible due to the much higher flow stress and tensile strength achievable in these materials. Similar twinning events were also reported during in-situ TEM tensile testing of electrodeposited nanocrystalline Ni (grain size: 50 nm) [Kumar et al. 2003].

2.3 Effect of Strain Localization during Deformation

2.3.1 Plastic Instability and Localized Plastic Strain

When the applied stress in a tensile test exceeds the yield strength ($\sigma_y$), plastic deformation will occur along the gauge length of the test sample. This local extension under tensile loading causes a simultaneous area constriction and the deformation usually is concentrated in this most highly stressed region [e.g. Hertzberg 1996]. The localized plastic deformation strain hardens the material, which tends to increase the load-carrying capacity of the specimen as deformation increases. The load-carrying capacity is opposed by the gradual decrease in the cross-sectional area of the specimen as it elongates. Necking or localized deformation begins at maximum load, where the increase in stress due to decrease in the cross-sectional area of the specimen becomes greater than the increase in the load-carrying ability of the metal due to strain hardening. This condition of instability leading to localized deformation is defined by a 2-step process (Fig. 2.6).

The first process is diffuse necking, a form of unstable flow in a sheet tensile specimen. Diffuse necking initiates and may terminate in fracture. However, it is often followed by a second instability process called localized necking. In this instability mode, the localized necking forms a narrow band, inclined at an angle to the specimen axis, across the width of the specimen [Dieter 1988]. Plastic deformation concentrates in the localized neck and fracture eventually initiates and propagates through it. An analysis based on plasticity theory predicts that the localized neck will most likely form at a certain angle. For isotropic materials, the relative values of the width strain ($\varepsilon_{22}$) and thickness strain ($\varepsilon_{33}$) are equal. The expression

$$\tan^2 \theta = \varepsilon_{22}/(\varepsilon_{22} + \varepsilon_{33})$$  \hspace{1cm} (9)
shows that the orientation of shear bands ($\theta$) is about $35^0$ or $55^0$ with respect to the tensile axis [Reid 1973]. Lee et al. [2011] examined the shear angles of annealed aluminum sheets under tensile deformation and confirmed that the angle was about $50 - 60^0$ from the tensile axis (Fig. 2.6).

![Image of localized necking in annealed aluminum sheet](left) [Lee et al. 2011]; and schematic diagram of diffuse necking and localized necking in a sheet tensile specimen (right) [Dieter 1988]

2.3.2 Methods of Measuring Localized Strain

In the past, plastic strains during deformation were mainly measured using strain gauges. With the advancement in new imaging technologies, researchers are now able to map localized strain patterns in the sample gauge with a non-contact and non-destructive method. The digital image correlation (DIC) system is capable to map two- or three-dimensional strain contour maps on sample surfaces. Figure 2.7 shows a typical optical image acquisition system for the two-dimensional DIC method. Specimens are aligned perpendicularly to the CCD camera along the loading system. White light sources are used to illuminate the specimen surface during the experiment. After recording the digital images of the specimen surface before and after deformation, the DIC computes the motion of each image point by comparing the displacement of each evenly spaced virtual grid on the digital images to obtain the full deformation [Pan et al. 2009] (Appendix A).
Coppieters et al. [2010] analyzed the post-necking behaviour of mild steel using the DIC technique. Figure 2.8 shows the computed strain fields at two different load steps (i.e. B and C). At load step B close to the onset of necking, strain fields are evenly distributed with the gradual increase of strain from both ends (i.e. blue color) to the center of the sample gauge (i.e. red color). At load step C within the necking region, the sample experienced both diffuse and localized necking. The highest strain was detected at the localized necking zone shown as the concentrated red spot at the center of the specimen.

Figure 2.7: Typical digital image correlation (DIC) system for the two-dimensional DIC method [Pan et al. 2009]

Figure 2.8: Force-Elongation curve of the central zone of mild steel samples [Coppieters et al. 2010]
Figure 2.9: Evolution of the longitudinal strain in the necking zone of mild steel with the increase of loading steps shown [Coppieters et al. 2010]

Figure 2.9 shows the evolution of the longitudinal strain in the necking area during the experiment. The recorded amount of plastic strain was low initially in the early stage of loading. However, increasing load yielded higher longitudinal plastic strain. The onset of necking is indicated at the highest plastic strain which later corresponded to the position of fracture.

Finite element (FE) modeling has been reported to simulate the localized stress state of specimens with different geometries during elongation. For example, Guo et al. [2010] assessed the effect of sample geometry on the ductility of nanocrystalline materials using FE modeling (Figure 2.10). Nanocrystalline material with grain size less than 100 nm exhibited different stress distributions before and after necking.

Under a constant tensile load, the 1 mm specimen gauge showed a relative homogeneous stress distribution as the strain level increased, and necking was not quite observable. On the other hand, the 2 mm specimen gauge showed a less homogeneous stress distribution within the gauge section and necking appeared to be initiated from the center. When the gauge length was increased even further, significant localized stress evolution and concentration were observed. As a result, necking started and became localized at the region of highest plastic strain.
2.3.3 Shear Band Evolution of Nanocrystalline Materials

Shear banding has been observed during deformation of both polycrystalline and nanocrystalline metals. The occurrence of intense shear banding is a prelude to failure in many sheet metals. Duan et al. 2005 conducted a combined experimental and numerical study on the intense shear banding observed for polycrystalline AA6111 Al alloy under uniaxial tension testing. Results showed that the FE model predicts the plastic strain distribution along the gauge section (Fig. 2.11a). Within the shear band at this deformation stage, the strain rate recorded by Duan et al. was 50 times higher than the average strain rate of $3 \times 10^{-2}/s$. The predicted shear band angle to the loading direction was $59^0$. The experimentally measured distribution by the DIC method was very close with a shear band angle of $62.9^0$ (Fig. 2.11b). Similar shear localization effects were observed in polycrystalline AA5754 Al alloy sheets [Kang et al. 2008] and ultrafine lamellar Ti-Fe eutectics (grain sizes: 20 – 30 μm) [Zhu et al. 2011].

Past studies associated the observation of shear bands during localized deformation of nanocrystalline metals with their limited ductility. Examples include nanocrystalline Fe (grain size: 20 nm) after microhardness testing [Malow and Koch 1998], nanocrystalline Fe (grain size: 268 nm) after compression testing [Wei et al. 2002] and nanocrystalline Ni (grain size: 20 nm) after high-rate tensile testing [Dalla Torre et al. 2002]. The results of these studies
suggested that shear localization is the primary mode for the onset of plastic deformation in nanocrystalline metals and develops early failure in these materials.

Figure 2.11: Plastic strain distribution of aluminum sheet alloys under uniaxial tension (a) FEM predicted distribution (b) Measured distribution by the DIC method [Duan et al. 2005]

Jia et al. 2003 evaluated the formation of shear bands in nanocrystalline Fe (grain size: 268 nm) as a function of plastic strain using quasi-static compression testing. The process of shear banding was observed in nanocrystalline Fe at 3.7% (Fig. 2.12a) and 7.8% (Fig. 2.12b) plastic strains. Three pre-existing shear bands denoted I, II and III grew much broader and propagated (i.e. band tip A) after 7.8% plastic strain. In addition, a new band IV was developed after 7.8% strain, indicating the formation of new shear bands at higher plastic strain levels.

Zhu et al. 2010 captured the propagation of shear bands in fully dense electrodeposited nanocrystalline Ni (grain size: 20 nm) during uniaxial tensile testing at a quasi-static strain rate of $10^{-4}$/s. During the tensile process, the evolution of surface strain on the sample at different strain levels was imaged by the DIC method. The stress-strain curve of the fully dense electrodeposited nanocrystalline Ni sample can be divided into three stages (Fig. 2.13).
Stage I corresponds to the elastic deformation region where there is no evidence of shear banding. Stage II represents the strain hardening region. Strain distribution was uniform both in longitudinal and transverse directions (Fig. 2.14a) and gradually the onset of shear banding was observed (Fig. 2.14b). In contrast, rapid evolution of shear banding was observed in stage III which corresponds to Figures 2.14 (c – f). With increasing engineering strain within the necking region, strain localization zones began to concentrate in the middle of the sample, forming band-like structures. Shear banded zones gradually intensified and formed a shear angle with respect to the loading direction. After the oriented band structure became dominant, the samples fractured in these regions.

During highly localized mechanical deformation of nanocrystalline metals, grain coalescence during the shear banding process was reported in several studies. Examples of past studies include experimental microtensile tests on nanocrystalline Al thin films (grain size: 40 nm) [Gianola et al. 2006] and numerical MD simulations for nanocrystalline Ni (grain size: 5 nm) [Farkas et al. 2006]. These studies showed that high applied stresses are required to trigger grain coalescence. From an atomic scale model in FCC nanocrystals [Wei and Gao 2008], it was speculated that the associated coalescence process during deformation is governed by thermally assisted diffusion and sliding along grain boundaries.
Figure 2.13: Engineering stress-strain curve of the fully dense electrodeposited nanocrystalline Ni. Three stages (I, II and III) are presented at different engineering strains [Zhu et al. 2010]

Stage I: Elastic deformation
Stage II: Strain hardening
Stage III: Necking

Figure 2.14: Strain maps of nanocrystalline Ni sample captured by DIC corresponding to consecutive points taken at different engineering strains in Figure 2.13 [Zhu et al. 2010]
Transmission electron microscopy in one of the shear band regions revealed considerable grain growth [Fan et al. 2006]. It was speculated that this grain growth was due to the kinetic energy during high speed deformation which could result in local hot spots within the shear band reaching a temperature on the order of 300°C. In contrast, other papers showed that the microstructure change was due to stress-driven grain growth. Xu et al. 2010 observed a transition from global grain growth to local grain coarsening in electrodeposited nanocrystalline Ni-Co alloys (grain sizes: 11 – 22 nm) during tensile deformation. With increasing Co content (i.e. 2.4% – 59.3%) , the suppressed stress-induced grain growth caused by sufficient solute pinning at grain boundaries delayed shear band plasticity instability and resulted in enhanced ductility. Sharon et al. 2011 conducted microtensile tests on nanocrystalline Pt thin films. The result of grain size increases from 20 nm in the initial state to 33 nm in the deformed state provided evidence for stress-induced grain growth from the applied tensile stress since the homologous temperature of 0.146 was likely too low to cause thermally activated grain growth.
2.4 Effect of Stress-Induced Heat Generation during Deformation

2.4.1 Laws of Thermodynamics and Models for Heat Dissipation

The transient heat equation [e.g. Rittel 1999] expresses the relationship between the heat generation rates and the spatial-temporal variation of the temperature as follows:

\[ k \nabla^2 T - \alpha (3\lambda + 2\mu) T_0 \dot{\varepsilon}_e + \beta \sigma_{ij} \dot{\varepsilon}_{ij}^p = \rho c \dot{T} \]  \hspace{1cm} (10)

where \( k \) is the heat conductance and \( \alpha \) the thermal expansion coefficient. \( \rho, c, \lambda \) and \( \mu \) stand for the material’s density, heat capacity and Lame constants, respectively. \( T \) and \( \dot{T} \) represent the temperature and rate of temperature increase. The strain rates \( \dot{\varepsilon} \) are divided into elastic \( (\dot{\varepsilon}_e) \) and plastic \( (\dot{\varepsilon}_p) \). \( \sigma_{ij} \) represents the flow stress during deformation [Rittel 1999].

If adiabatic conditions prevail \((k \nabla^2 T = 0)\) in the case of high strain rate loading and the elastic contribution is neglected as \( \alpha (3\lambda + 2\mu) T_0 \dot{\varepsilon}_e = 0 \), Eq. 10 is reduced to:

\[ \beta = \frac{\rho c \dot{T}}{\sigma_{ij} \dot{\varepsilon}_{ij}^p} = \frac{\rho c \dot{T}}{dW_p} \]  \hspace{1cm} (11)

The ratio of dissipated to plastic power, \( \beta \), corresponds to the Taylor-Quinney coefficient. Part of the mechanical energy expended during a plastic deformation process in metals is converted into heat, with the remainder as stored energy. The stored energy is an essential feature of the cold-worked state and represents the change in internal energy of the metal [Hodowany et al. 1999]. Since the early works of Farren and Taylor [1925] and Taylor and Quinney [1934], the fraction of the rate of plastic work dissipated as heat, \( \beta \), is often assumed to be a constant parameter of 0.9 for most metals during deformation. However, this ratio may exceed unity and may even reach values as high as 2, in the case of highly localized processes [Rittel 1999]. This means that stored energy in the system is possibly released and contributes to instantaneous dissipation. This is an important issue since the driving force for localized band propagation takes the form of an energy release rate [Rittel et al. 2006, Yang and Wang 2006].
Aravas et al. [1990] proposed the residual stress theory to determine $\beta$ for polycrystalline metals. Residual stress theory identifies the stored energy as residual stresses generated in the metal in the form of lattice imperfections after unloading. Results calculated from uniaxial stress-strain curves indicate that the stored energy increases with increasing hardening capacity of metals [Aravas et al. 1990].

The residual stress theory in Figure 2.15a [Hodowany 1997] shows a dependence of $\beta$ as a function of strain hardening exponent ($n$) and plastic strain ($\varepsilon^p/\varepsilon_0$). With higher strain hardening exponent and plastic strains, the predicted values $\beta$ is lower and decreases continuously. The results showed that ductile metals with high hardening capacity have a higher ratio of stored energy (i.e. lower $\beta$). The $\beta$ curve peaks in the beginning of plastic strain due to the onset of strain hardening during deformation [Aravas et al. 1990].

Figure 2.15: Fraction of plastic work dissipated as heat predicted by (A) residual stress theory, and (B) dislocation theory [Hodowany 1997]
A different type of model, proposed by Zehnder [1990], associated the stored energy with dislocations, dislocation interactions and residual strains caused by incompatible slips in grains of different orientation in metals, as well as taking into account of the concept of dislocation multiplication and density suggested by Nabarro [1987]. Figure 2.15b of the dislocation model shows that $\beta$ i) increases with increasing plastic strain and ii) decreases with higher hardening exponent, similar to the residual stress theory.

2.4.2 Methods of Measuring Heat Dissipation

Taylor and Quinney [1937] set up the first experiments to measure the heat evolution in mild steel at different plastic strains in torsion. A thermocouple was affixed onto the outer surface of the sample and temperature was recorded directly using a galvanometer. The second testing method was to remove the sample from the load fixture after deformation and dropping it immediately into a calorimeter. Both measurements showed an equivalent temperature increase of $64^0\text{C}$. Sachdev and Hunter [1982] used an infra-red detector to measure the temperature increase during tensile tests of dual phase steel. This steel showed a maximum temperature increase of $76^0\text{C}$ at a strain rate of $1.5 \times 10^{-2}/\text{s}$. Ayres [1985] measured thermal gradients due to deformation in tensile specimens of 1008 aluminum-killed (AK) steel with thermocouples. AK steel reached a maximum temperature increase of $90^0\text{C}$ at a strain rate of $10^{-1}/\text{s}$. Lin and Wagoner [1987] also used thermocouples to measure temperature changes during deformation on interstitial-free steels and stainless steels type 310. Both steels showed temperature increases by $75^0\text{C}$ and $118^0\text{C}$, respectively at a strain rate of $10^{-1}/\text{s}$.

With the advancement of infrared detector technologies for non-contact and non-destructive measurements, Mason et al. [1994] were able to detect the changes in temperature during the deformation of 2024 aluminum and Ti-6Al-4V titanium. 2024 aluminum showed a temperature increase of $100^0\text{C}$ for a strain rate of $3 \times 10^{3}/\text{s}$, whereas Ti-6Al-4V titanium showed the same temperature increase for a strain rate of $1.5 \times 10^{3}/\text{s}$.
Rusinek and Klepaczko [2009] analyzed the effect of heat generation on the mechanical properties of TRIP steels during plastic deformation at strain rates up to 10/s with the infrared technique. Their work showed that the increase of temperature was associated with the stress level during deformation (Fig. 2.16). In the strain hardening region A, the recorded temperature increase was 35K. The increase of temperature was more rapid around the necking region B to 58K. The final temperature increase at sample fracture was around 90K. Figure 2.17 shows the observable temperature changes acquired from infrared images of mild steel deformed at a strain rate of $10^{-2}$/s. The highest temperature was localized at the center of the sample gauge where most of the heat was dissipated. In the case of mild steel, the maximum temperature increase was 25K above room temperature. Table 2.1 summarizes the list of methods and the recorded temperature increases reported in different recent studies.

![Figure 2.16](image-url)

Figure 2. 16: Temperature increase and stress evolution of TRIP steels during plastic deformation at $\dot{\varepsilon} = 10$/s and $T_0 = 300K$ [Rusinek and Klepaczko 2009]
Figure 2. 17: Temperature increase of mild steel in quasi-static loading $\dot{\varepsilon} = 10^{-2}/s$ and $T_0 = 300K$ during deformation [Rusinek and Klepaczko 2009]

Table 2.1. List of methods and recorded temperature increases in different studies

<table>
<thead>
<tr>
<th>Reference</th>
<th>Method</th>
<th>Material</th>
<th>Strain rate ($\dot{\varepsilon}$)</th>
<th>Temp increase</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sachedev and Hunter [1982]</td>
<td>Infrared detector</td>
<td>Dual phase steel</td>
<td>$1.5 \times 10^{-2}/s$</td>
<td>76°C</td>
</tr>
<tr>
<td>Ayres [1985]</td>
<td>Thermocouples</td>
<td>AK steel</td>
<td>$10^{-1}/s$</td>
<td>90°C</td>
</tr>
<tr>
<td>Lin and Wagoner [1987]</td>
<td>Thermocouples</td>
<td>Stainless steel type 310</td>
<td>$10^{-1}/s$</td>
<td>118°C</td>
</tr>
<tr>
<td>Mason et al. [1994]</td>
<td>Infrared detector</td>
<td>2024 Aluminum</td>
<td>$3 \times 10^{-3}/s$</td>
<td>100°C</td>
</tr>
<tr>
<td>Mason et al. [1994]</td>
<td>Infrared detector</td>
<td>Ti-6Al-4V titanium</td>
<td>$1.5 \times 10^{-3}/s$</td>
<td>100°C</td>
</tr>
<tr>
<td>Rusinek and Klepaczko [2009]</td>
<td>Infrared detector</td>
<td>TRIP steel</td>
<td>10/s</td>
<td>90°C</td>
</tr>
<tr>
<td>Rusinek and Klepaczko [2009]</td>
<td>Infrared detector</td>
<td>Mild steel</td>
<td>$10^{-2}/s$</td>
<td>25°C</td>
</tr>
</tbody>
</table>
2.4.3 Localized Grain Growth of Nanocrystalline Materials

Miniature samples were designed in past studies to investigate material properties of electrodeposited nanocrystalline materials. Dalla Torre et al. [2002] tested electrodeposited Ni (grain size: 20 nm) with sample sizes of 3 to 20 mm at room temperature and at strain rates between $10^{-5}$ and $10^{3}$/$s$. In the low strain rate regime less than $10^{-2}$/$s$, Dalla Torre et al. observed very low plasticity with little evidence of necking in the fracture areas. The 20 mm-sized samples showed two different types of morphologies at their fracture surfaces. Figure 2.18 shows a combination of a very rough and irregular sample surface with some portions of columnar surface structures. On the other hand, samples with sizes of 3 mm showed only a single type of surface structure after fracture (Fig. 2.19).

![Figure 2.18: SEM micrographs of a fractured nanocrystalline Ni specimen (size: 20 mm) tested at a low strain rate [Dalla Torre et al. 2002]](image1)

![Figure 2.19: SEM micrograph of a fractured nanocrystalline Ni specimen (size: 3 mm) tested at a low strain rate [Dalla Torre et al. 2002]](image2)
Dalla Torre et al. also tested the material at a high strain rate of $10^3$/s to observe again the changes in the microstructures of nanocrystalline Ni samples. The 20 mm-sized sample showed a clear change in the fracture mode with a stronger reduction in the cross-sectional area due to necking. The fracture surface was examined by TEM and revealed a conjugated shear band region (Fig. 2.20).

![TEM images and selected area diffraction (SAD) patterns of a locally deformed area taken from the shear band of a 20 mm nanocrystalline Ni deformed at 1.7 x $10^3$/s. (a) The central region in the shear band, (b) region from which the SAD pattern (c) is taken, (d) magnified region from which the SAD pattern (e) is taken [Dalla Torre et al. 2002]

Fan et al. [2006] were able to test larger tensile samples according ASTM standard at strain rates up to 10/s. They observed that nanocrystalline Ni-Fe alloys (grain size: 23 nm) with 70 mm standard ASTM size tested at a strain rate of $10^3$/s showed dimples and evidence of post-necking deformation on the fracture surface at low (Fig. 2.21a) and high (Fig. 2.21b) magnifications. TEM images of the surface of the fractured nanocrystalline Ni-Fe alloy showed evidence of grain growth during tensile deformation (Fig. 2.22). The arrows in Figure 2.22b indicate the large grains that underwent abnormal grain growth during tensile elongation from $\varepsilon = 4.0\%$ to $\varepsilon = 6.4\%$. Grain size distribution statistics showed a change in average grain sizes from the as-deposited state (Fig. 2.23a) and the deformed state (Fig. 2.23b). The material with an average as-deposited grain size of 23 nm grew significantly to a broader distribution with
grains larger than 200 nm. The large fraction of large grains (d > 200 nm) suggests the occurrence of abnormal grain growth. The largest grain growth occurred within the strain rate range of $10^{-2}/s$ to $10^{-1}/s$ (Fig. 2.24a) and at larger tensile strains (Fig. 2.24b).

Figure 2.21: SEM images of the fractured nanocrystalline Ni-Fe alloy deformed at $10^{-3}/s$ (a) at low magnification, (b) at high magnification [Fan et al. 2006]

Figure 2.22: Bright-field TEM images of nanocrystalline Ni-Fe alloy after deformed at $10^{-3}/s$ to (a) $\varepsilon = 4.0\%$, and (b) $\varepsilon = 6.4\%$ [Fan et al. 2006]

It was suggested by Holm et al. [2003] that abnormal grain growth is due on non-uniform grain boundary mobility driven by thermal activation. When metals deform plastically, a portion of plastic work during deformation would be released in the form of heat. The excess heat enthalpy stored in the grain boundaries drives grain growth.
The driving force \( (p) \) for grain growth is related to grain size \( (d) \) as Eq. 12 [Haessner 1978]:

\[
p = \frac{3\gamma}{d}
\]  

(12)

With a constant interface enthalpy \( (\gamma) \), a reduction of grain sizes from 10 mm to 10 nm would lead to a significant enhancement of the driving force for grain growth by a factor of 1000.

Figure 2. 23: Grain size distribution statistics of nanocrystalline Ni-Fe alloy in the (A) as-deposited state, and (B) after deformation at \( 10^3/s \) to \( \varepsilon = 6.4\% \) [Fan et al. 2006]

Figure 2. 24: Average grain sizes of nanocrystalline Ni-Fe alloy at various (a) strain rates, (b) tensile elongation [Fan et al. 2006]
2.4.4 Thermally Induced Grain Growth in Nanocrystalline Electrodeposits

The thermal stability of electrodeposited nanocrystalline Ni was previously investigated by modulated differential scanning calorimetry (MDSC) [Klement et al. 1995, Turi 1997]. Turi analyzed the peak temperature for major grain growth for nanocrystalline Ni. Because of the large volume fraction of interfaces in nanocrystalline materials, the heat release during grain growth for nanocrystalline Ni can be measured accurately. For example, the major grain growth in nanocrystalline Ni of 30 nm grain sizes was around 266°C (Fig. 2.25a). However, at a temperature before the onset of major grain growth, nanocrystalline Ni already goes through a sequence of relaxation processes which results in lower energy release [Klement et al. 1995]. Klement et al. suggested that the beginning of subgrain coalescence occurred at 100°C. As for nanocrystalline Ni-10%Fe, higher temperatures at 260°C and around 396°C are required for either subgrain coalescence or grain growth, respectively (Fig. 2.25b), which could be due to the formation of an ordered Ni$_3$Fe phase [Turi 1997].

![MDSC heat flow curve for 30 nm nanocrystalline Ni](Turi 1997)
Figure 2. 25b: MDSC heat flow curve for 13 nm nanocrystalline Ni-10%Fe [Turi 1997]

\[ T_p = 396.31^\circ C \]

\[ \Delta H_{\text{total}} = 15.85 \text{ J/g} \]

\[ T_p = 396.31^\circ C \]

\[ \Delta H_{\text{total}} = 925.73 \text{ J/mol} \]
3. Experimental Procedures

3.1 Experimental Design

This study deals with the evaluation of the thermal-mechanical properties of bulk polycrystalline and nanocrystalline Ni and Ni-Fe alloys using samples prepared according to ASTM standard. Experiments were divided into two parts. The first part studied the effect of in-situ heat generation during deformation, while the second part studied the effect of strain localization on the mechanical properties. The mechanical property testing experiments were conducted at the Institute of Aerospace Research of the National Research Council Canada. All samples were loaded on a MTS servo-hydraulic machine for tensile testing at strain rates of \(10^{-2}/s\) and \(10^{-1}/s\). Temperature changes on one side and localized tensile strain on the other side of each sample were measured and captured simultaneously using both a high resolution infrared camera (Deltatherm 1410) and a high resolution Digital Image Correlation Camera (Allied Vision Technologies), respectively (Fig. 3.1). Temperature and strain were continuously recorded to final fracture. Samples after fracture were observed under the Scanning Electron Microscopy (SEM) to characterize their fracture topography.

![Experimental setup](image)

Figure 3.1: Experimental setup (A) MTS Servo-Hydraulic Tensile Machine (B) Infra-red Detector (C) Digital Image Correlation System
3.2 Materials

Tensile coupons of electrodeposited nanocrystalline Ni and Ni-Fe alloys were provided by Integran Technologies Inc., Toronto, Ontario. Polycrystalline Ni (commercially available Ni 200) and the electrodeposited nanocrystalline Ni and Ni-Fe samples were machined by the electrical discharge machining (EDM) method to produce dog bone shape tensile test samples according to the ASTM E8 standard [ASTM International, 2004] (Fig. 3.2a) with thicknesses varying from 0.9 to 1.0 mm (Table 3.1). Samples were labeled according to the different electrodeposition batch numbers provided by Integran. For example, samples C065-1 and C162-1 were samples 1 from batch C065 nickel and C162 nickel-iron batch, respectively. Figure 3.2b shows a picture of the tensile samples prepared by electrodeposition and EDM.

Figure 3. 2a: Geometry of the tensile samples used in the current study

<table>
<thead>
<tr>
<th>Sample Batches</th>
<th>Thickness (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>10-N200</td>
<td>1.00</td>
</tr>
<tr>
<td>07-PUN1-J024</td>
<td>1.00</td>
</tr>
<tr>
<td>06-N50F-C162</td>
<td>0.94</td>
</tr>
<tr>
<td>06-N50-C065</td>
<td>0.92</td>
</tr>
</tbody>
</table>
3.3 Microhardness Testing and Grain Size Measurements

Vickers microhardness tests were conducted on all samples. The testing load was set to 100 g for a loading time of 10 second. Each sample was tested at least 5 times to obtain an average hardness value. For the nanocrystalline materials, the hardness values were used to estimate grain sizes by comparison with previous hardness-grain size relationships conducted in our group on these types of materials. The grain size of the polycrystalline Ni 200 material was determined by optical microscopy.

3.4 MTS Servo-Hydraulic Machine

All tensile tests were performed according to the specifications of ASTM standard E8M-01 [ASTM Standard, 2004]. The MTS servo-hydraulic machine was calibrated according to standard procedures prior to experiments and aligned properly to the infrared detector and the digital image correlation camera. Samples were tested at room temperature at strain rates of $10^{-2}$/s and $10^{-1}$/s.
3.5 Digital Image Correlation (DIC) Technique

The changes of localized strain patterns during deformation were captured by a high resolution Digital Image Correlation Camera (Allied Vision Technologies) (Fig. 3.3). Samples prior to loading the samples were lightly abraded using 320 silicon carbide paper and the DIC system was calibrated. Samples with abraded-surfaces facing towards the CCD camera were illuminated by a source of white light during the entire tensile test. The images were then post-processed through a DIC system to calculate the maximum displacement and the localized strain distributions. Localized strains contours were mapped along the sample gauge over a length of 40 mm up to engineering UTS at the two different strain rates.

![Digital Image Correlation Technique](image)

Figure 3. 3: Digital Image Correlation technique (DIC) system (left); area of strain detection (right)

3.6 Infrared Imaging (IR)

A high resolution infrared camera (Deltatherm 1410) was used to measure the temperature changes in the samples during tensile deformation (Fig. 3.4). Prior to the experiment, the infrared camera was calibrated and the facing sides of the tensile samples were spray painted black to equalize emissivity for better infrared detection. Frame rates of the infrared detector were set to 15 frames per second to best capture the temperature profiles along sample gauges. Images captured during the experiment were then post-processed through a Thermoelastic Stress Analysis (TSA) system to locate the temperature increase of all samples along the gauge section during tensile testing. The maximum temperature along the sample gauges was recorded for all samples.
3.7 Scanning Electron Microscopy (SEM)

Two SEM’s (Hitachi S-570 and S-6600) at the University of Toronto were used to analyze the fracture surfaces of the tensile samples. Low and high magnification images of the fracture surfaces were taken which were used for characterization on the microstructure and fracture topography.

3.8 Energy Dispersive X-ray Spectroscopy (EDS)

The compositions of the different sample batches were analyzed using a Link Analytical LZ-5 EDS detector attached to the Hitachi S-570 SEM. The EDS system was calibrated using a standard cobalt sample prior to each experiment. Each sample was tested at least three times at low magnification at different areas to determine the average chemical composition. The characteristic X-rays emitted from the samples during bombardment with the incident electron beam were analyzed to determine their compositions.
4. Results and Discussion

4.1 Sample Compositions

The compositions of all samples as determined by EDS are listed in Table 4.1. Batch 06-N50-C065 has the highest percentage of 8.5 wt% Fe. The weight percentage of iron is lower in 06-N50F-C162, at 2.6 wt% Fe. Materials 07-PUN1-J024 and 10-N200 were pure nickel samples with no iron additions.

Table 4.1: Composition of all sample batches

<table>
<thead>
<tr>
<th>Sample Batches</th>
<th>wt % Fe [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>10-N200</td>
<td>0</td>
</tr>
<tr>
<td>07-PUN1-J024</td>
<td>0</td>
</tr>
<tr>
<td>06-N50F-C162</td>
<td>2.6</td>
</tr>
<tr>
<td>06-N50-C065</td>
<td>8.5</td>
</tr>
</tbody>
</table>

4.2 Microhardness Tests and Grain Sizes

The grain size of polycrystalline Ni was determined by standard metallography. Figure 4.1a shows an example of an optical micrograph for the Ni200 material used in this study. A grain size distribution histogram is shown in Figure 4.1b, based on a total grain count of 400 grains. The average grain size of this material was 32 μm.

The grain sizes of the nanocrystalline Ni and Ni-Fe alloys were determined by an indirect method. The microhardness of these materials was used to derive their grain sizes by comparison to previous microhardness-grain size relationships established by other members of our group which are reproduced here as Figures 4.2a and 4.2b, respectively.
The Vickers microhardness values and grain sizes for all materials tested in this study are summarized in Table 4.2.

Figure 4. 1a: Optical Image of 10-N200

Figure 4. 1b: Grain Size Distribution of 10-N200

Average: 32 μm
STDEV: 16.1 μm
Figure 4. 2a Vickers hardness as a function of grain sizes ($d^{-1/2}$) for as-prepared nanocrystalline Ni [El-Sherik et al. 1992]

Figure 4. 2b Vickers hardness as a function of grain sizes ($d^{-1/2}$) for as-prepared nanocrystalline Ni-Fe [Cheung et al. 1995]
Table 4.2: Microhardness and the grain sizes of all sample batches

<table>
<thead>
<tr>
<th>Sample Batches</th>
<th>wt % Fe [%]</th>
<th>Vickers Hardness (VHN)</th>
<th>Grain Size</th>
</tr>
</thead>
<tbody>
<tr>
<td>10-N200</td>
<td>0</td>
<td>137 ± 9</td>
<td>32 μm</td>
</tr>
<tr>
<td>07-PUN1-J024</td>
<td>0</td>
<td>415 ± 8</td>
<td>57 nm</td>
</tr>
<tr>
<td>06-N50F-C162</td>
<td>2.6</td>
<td>463 ± 10</td>
<td>25 nm</td>
</tr>
<tr>
<td>06-N50-C065</td>
<td>8.5</td>
<td>487 ± 6</td>
<td>20 nm</td>
</tr>
</tbody>
</table>

4.3 Tensile Properties

The tensile properties of all samples were tested at two different strain rates $10^{-2}/s$ and $10^{-1}/s$, respectively. When comparing samples tested at a constant strain rate (e.g. $10^{-2}/s$, Fig. 4.3a), nanocrystalline Ni-8.5%Fe with a grain size of 20 nm showed the highest values in yield strength and UTS at 1130 MPa and 1741 MPa, respectively. On the other hand, polycrystalline Ni at a grain size of 32 μm showed the lowest yield strength and UTS at 208 MPa and 442 MPa, respectively. The other two nanocrystalline materials had yield and tensile strengths between these two extremes. The increase of yield strength and UTS with decreasing grain size is due to the Hall Petch strengthening mechanism. With the refinement of grain sizes, there is also a tradeoff between the increase in yield strength and UTS and the reduction in ductility as can be clearly seen in Figure 4.1a. A similar trend in the tensile properties was found at a strain rate of $10^{-1}/s$ (Fig. 4.3b). Small changes in yield strength, UTS and elongation were observed as the applied strain rate increased (Table 4.3). Yield strength, as well as UTS, increased slightly in the nanocrystalline Ni and Ni-Fe samples. Polycrystalline Ni was not affected when the strain rate was changed. There are no significant changes in sample elongation in both nanocrystalline and polycrystalline Ni samples for the two strain rates.
Figure 4. 3a: Example of the stress-strain curve of all samples at a strain rate of $10^{-2}/s$

Figure 4. 3b: Example of the stress-strain curve of all samples at a strain rate of $10^{1}/s$
Table 4.3: Tensile properties of all samples at strain rates of $10^{-2}$/s and $10^{-1}$/s

<table>
<thead>
<tr>
<th>Samples</th>
<th>wt % Fe [%]</th>
<th>Grain Size</th>
<th>Strain Rate (/s)</th>
<th>Yield Strength (MPa)</th>
<th>UTS (MPa)</th>
<th>Elongation (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>10-N200-4</td>
<td>0</td>
<td>32 μm</td>
<td>$10^{-2}$</td>
<td>208</td>
<td>442</td>
<td>50.4</td>
</tr>
<tr>
<td>10N200-5</td>
<td>0</td>
<td>32 μm</td>
<td>$10^{-1}$</td>
<td>208</td>
<td>445</td>
<td>51.0</td>
</tr>
<tr>
<td>07-PUN1-J024-3</td>
<td>0</td>
<td>57 nm</td>
<td>$10^{-2}$</td>
<td>950</td>
<td>1504</td>
<td>8.2</td>
</tr>
<tr>
<td>07-PUN1-J024-2</td>
<td>0</td>
<td>57 nm</td>
<td>$10^{-1}$</td>
<td>970</td>
<td>1508</td>
<td>8.0</td>
</tr>
<tr>
<td>06-N50F-C162-1</td>
<td>2.6</td>
<td>25 nm</td>
<td>$10^{-2}$</td>
<td>1010</td>
<td>1579</td>
<td>7.6</td>
</tr>
<tr>
<td>06-N50F-C162-2</td>
<td>2.6</td>
<td>25 nm</td>
<td>$10^{-1}$</td>
<td>1100</td>
<td>1763</td>
<td>7.5</td>
</tr>
<tr>
<td>06-N50-C065-1</td>
<td>8.5</td>
<td>20 nm</td>
<td>$10^{-2}$</td>
<td>1130</td>
<td>1741</td>
<td>7.7</td>
</tr>
<tr>
<td>06-N50-C065-6</td>
<td>8.5</td>
<td>20 nm</td>
<td>$10^{-1}$</td>
<td>1150</td>
<td>1867</td>
<td>7.5</td>
</tr>
</tbody>
</table>
4.4 Stress-Induced Strain Localization

4.4.1 Localized Strain Contours

When metals deform plastically, they experience diffuse and localized necking as engineering strain increases (see Fig. 2.6). The localized plastic strain increases and the region of the highest strain indicates the position of sample fracture. The DIC system captured the evolution of localized strain at different levels of engineering strain and strain rate. In the following figures, the percent strain numbers listed at the bottom are the overall engineering strain values. The colours show localized strains throughout the gauge sections. The strain values increase from blue to green to yellow and red. Figure 4.4a shows the localized strain contours of polycrystalline Ni deformed at $10^{-2}$/s. From 15% to 45% strain, i.e. within the uniform deformation region, polycrystalline Ni shows an increase of localized tensile strain along the loading axis. The change of color from dark purple to warm blue, green and yellow indicates the increase of localized strain at different levels of overall engineering strain. Two highly localized strain regions located along the sample gauges show inhomogeneous flow beginning at about 25% engineering strain. One of the two localized strains with the highest strain values indicates the position of fracture (i.e. dashed line). At the higher strain rate at $10^{-1}$/s, the localized strain on polycrystalline Ni is more uniform (Fig. 4.4b) and only one region developed highly localized strain which later resulted in fracture.

With reduced grain size, the localized strain in nanocrystalline Ni developed in a somewhat different way. At the strain rate of $10^{-2}$/s, nanocrystalline Ni shows changes in localized strain as indicated in Figure 4.5a. From 2.5% to 5.5% strain, the localized strain increases rather slowly until engineering UTS. At around 8.0% engineering strain, the region of the highest localized strain indicates the position of fracture at an angle of $62^0$ (i.e. dashed line), which was measured on the actual fractured sample and the localized strain image. Diffusional necking on nanocrystalline Ni samples is less pronounced than for polycrystalline Ni samples. Similar to polycrystalline Ni, the strains on nanocrystalline Ni at the higher strain rate of $10^{-1}$/s are more evenly distributed (Fig. 4.5b). However, the angle of fracture still remained at $62^0$ in the region of the highest localized strain.
Figure 4. 4a: Localized strain contours on polycrystalline Ni deformed at $10^{-2}$/s. The dashed line indicates the position of fracture at the region of highest localized strain.

Figure 4. 4b: Localized strain contours on polycrystalline Ni deformed at $10^{-1}$/s. The dashed line indicates the position of fracture at the region of highest localized strain.
Figure 4. 5a: Localized strain contours on nanocrystalline Ni deformed at $10^{-2}$/s. The dashed line indicates the position of fracture at the region of highest localized strain.

Figure 4. 5b: Localized strain contours on nanocrystalline Ni deformed at $10^{-1}$/s. The dashed line indicates the position of fracture at the region of highest localized strain.
Figure 4. 6a: Localized strain contours on nanocrystalline Ni-2.6%Fe deformed at $10^{-2}$/s. The dashed line indicates the position of fracture at the region of highest localized strain.

Figure 4. 6b: Localized strain contours on nanocrystalline Ni-2.6%Fe deformed at $10^{-1}$/s. The dashed line indicates the position of fracture at the region of highest localized strain.
Figure 4. 7a: Localized strain contours on nanocrystalline Ni-8.5%Fe deformed at $10^{-2}$/s. The dashed line indicates the position of fracture at the region of highest localized strain.

Figure 4. 7b: Localized strain contours on nanocrystalline Ni-8.5%Fe deformed at $10^{-1}$/s. The dashed line indicates the position of fracture at the region of highest localized strain.
Nanocrystalline Ni-2.6%Fe (Figs. 4.6a and 4.6b) and Ni-8.5%Fe (Figs. 4.7a and 4.7b) showed a development of localized strain contours similar to nanocrystalline Ni at both strain rates of $10^{-2}$/s and $10^{1}$/s, respectively. In spite of the changes in strain rates, the orientation of the regions of highest localized strain corresponding to the position of fracture remained constant at $60^0$ and $62^0$ for nanocrystalline Ni-2.6%Fe and Ni-8.5%Fe, respectively.

4.4.2 Effects of Grain Sizes and Strain Rates on Strain Localization

Localized strain patterns were generated through the digital image correlation (DIC) system. Strain fields were analyzed up to ultimate engineering tensile strength (UTS) and were plotted as a function of distance over the 40 gauge length. Figure 4.8a shows the localized tensile strain contours in polycrystalline Ni at overall engineering strain levels of 15%, 25%, 35% and 45% at a strain rate of $10^{-2}$/s. Along the gauge section, localized tensile strain increases differently with increasing overall engineering strain. Beyond 35% overall engineering strain, the localized strain levels begin to exceed the overall applied strain in two regions at about 15 and 33 mm, respectively. Polycrystalline Ni undergoes a transition from homogeneous to inhomogeneous plastic flow. For example at 33 mm, the localized strain at 45% overall engineering strain had a value of 62%, which is about 1.4 times higher than the overall applied strain. Due to inhomogeneous plastic flow, two highly regionally localized strained regions were found along the gauge length. One of the two peaks with the localized strain at 55% within the necking zone, eventually corresponded to the position of fracture: the region at 17 mm (i.e. red dashed line).

In comparison with the strain rate at $10^{-2}$/s, localized tensile strain development in polycrystalline Ni at a strain rate of $10^{1}$/s is less pronounced with reduced local concentration (Fig. 4.8b). Polycrystalline Ni shows a more homogeneous deformation with evenly distributed strain fields. At 45% overall engineering strain, one of the strain fields located at 29 mm (i.e. red dashed line) along sample gauges corresponded to the position of fracture at a value of 33% within the necking zone.
Figure 4. 8a: Localized tensile strain contours (in %) in polycrystalline Ni at a strain rate of $10^{-2}$/s for different overall engineering strain levels. The red dashed line indicates the position of fracture.

Figure 4. 8b: Localized tensile strain contours (in %) in polycrystalline Ni at a strain rate of $10^{-1}$/s for different overall engineering strain levels. The red dashed line indicates the position of fracture.
Figure 4. 9a: Localized tensile strain contours (in %) in nanocrystalline Ni at a strain rate of $10^{-2}$/s for different overall engineering strain levels. The red dashed line indicates the position of fracture.

Figure 4. 9b: Localized tensile strain contours (in %) in nanocrystalline Ni at a strain rate of $10^{-1}$/s for different overall engineering strain levels. The red dashed line indicates the position of fracture.
Figure 4. 10a: Localized tensile strain contours (in %) in nanocrystalline Ni-2.6%Fe at a strain rate of $10^{-2}$/s for different overall engineering strain levels. The red dashed line indicates the position of fracture.

Figure 4. 10b: Localized tensile strain contours (in %) in nanocrystalline Ni-2.6%Fe at a strain rate of $10^{-1}$/s for different overall engineering strain levels. The red dashed line indicates the position of fracture.
Figure 4. 11a: Localized tensile strain contours (in %) in nanocrystalline Ni-8.5%Fe at a strain rate of $10^{-2}/s$ for different overall engineering strain levels. The red dashed line indicates the position of fracture

Figure 4. 11b: Localized tensile strain contours (in %) in nanocrystalline Ni-8.5%Fe at a strain rate of $10^{-1}/s$ for different overall engineering strain levels. The red dashed line indicates the position of fracture
In contrast, the localized strains on nanocrystalline Ni with 57 nm grain size are much lower than for polycrystalline Ni at both strain rates. Within the necking zone at engineering UTS (i.e. 5.5% overall engineering strain), nanocrystalline Ni deformed at $10^{-2}/s$ (Fig. 4.9a) indicates a localized strain of 4.5%, which later forms the position of fracture at 18 mm (i.e. red dashed line). As in the case of polycrystalline Ni, the localized strains at the higher strain rate of $10^{-1}/s$ are more evenly distributed (Fig. 4.9b). Localized strain of 5.6% within the necking zone later corresponds to the position of fracture at 21 mm along the gauge length (i.e. red dashed line).

With the further refinement of grain size, samples of nanocrystalline Ni-Fe behaved similarly to nanocrystalline Ni. Nanocrystalline Ni-2.6% Fe at 25 nm grain size deformed at strain rate of $10^{-2}/s$ (Fig. 4.10a) to 5.5% engineering strain exhibits a localized strain of 2.2%, which later forms the position of fracture at 24 mm (red dashed line). At the higher strain rate of $10^{-1}/s$ (Fig. 4.10b), the position of fracture is located at 20 mm with a localized strain at 5.1% within the necking zone. Similar strain patterns were found on nanocrystalline Ni-8.5% Fe at 20 nm grain size. Within the necking zone, localized strain at the strain rate of $10^{-2}/s$ reaches 4.2% at 22mm and forms the position of fracture (i.e. red dashed line) (Fig. 4.11a), whereas for the higher strain rate at $10^{-1}/s$ the position of fracture was located at 19 mm with a localized strain of 4.3% (i.e. red dashed line) (Fig. 4.11b).

Table 4.4 summarizes the localized strains recorded at the positions of fracture for polycrystalline and nanocrystalline Ni and Ni-Fe samples for the two strain rates. As expected from the stress-strain curves, the results show that the localized strain in nanocrystalline Ni and Ni-Fe is much lower than that in polycrystalline Ni. This can be explained by the differences in the strain hardening capacity and overall ductility.
Table 4.4 Summary of the highest overall and localized strains recorded in all samples

<table>
<thead>
<tr>
<th>Samples</th>
<th>Grain Size</th>
<th>Strain Rate (1/s)</th>
<th>Highest Overall Engineering Strain at Necking (%)</th>
<th>Highest Localized Strain at Position of fracture (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Poly Ni</td>
<td>32 μm</td>
<td>$10^{-2}$</td>
<td>45</td>
<td>55 (17 mm)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$10^{-1}$</td>
<td>45</td>
<td>33 (29 mm)</td>
</tr>
<tr>
<td>Nano Ni</td>
<td>57 nm</td>
<td>$10^{-2}$</td>
<td>5.5</td>
<td>4.5 (18 mm)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$10^{-1}$</td>
<td>5.5</td>
<td>5.6 (21 mm)</td>
</tr>
<tr>
<td>Nano Ni-2.6%Fe</td>
<td>25 nm</td>
<td>$10^{-2}$</td>
<td>5.5</td>
<td>2.2 (24 mm)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$10^{-1}$</td>
<td>5.5</td>
<td>5.1 (20 mm)</td>
</tr>
<tr>
<td>Nano Ni-8.5%Fe</td>
<td>20 nm</td>
<td>$10^{-2}$</td>
<td>5.5</td>
<td>4.2 (22 mm)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$10^{-1}$</td>
<td>5.5</td>
<td>4.3 (19 mm)</td>
</tr>
</tbody>
</table>
4.4.3 Fracture Surfaces

In order to gain further insight into the different plastic deformation mechanisms of polycrystalline and nanocrystalline Ni and Ni-Fe alloys, post-fracture analysis of the failed surfaces was performed. The fracture surfaces of all samples were examined in Hitachi SEM S570 and S6600 scanning electron microscopes. Photographs were taken at low magnification. Higher magnification micrographs were taken in selected areas. The SEM images showed numerous features indicating a high degree of ductility in all materials, such as: dimples, microvoids and vein-like structures, which were all quite similar to those typically found on ductile polycrystalline metals.

**Dimples**

Dimples were the most common feature observed on ductile materials. Dimples are formed by microvoid coalescence during deformation, by a process of internal and localized necking. Coarse dimples were easily shown on polycrystalline Ni at 32 μm grain size (Fig. 4.12). Much finer dimples under higher magnification were also found on nanocrystalline Ni and Ni-Fe alloys at 57 nm, 25 nm and 20 nm grain size, respectively (Figs. 4.13 to 4.15) indicating ductile fracture.

**Deep Microvoids**

Microvoids are usually much larger and deeper than the surrounding dimples. For most microvoids, the inner surface showed a contour pattern indicating that they progressively increased in size as a result of the growth and coalescence of microvoids formed during deformation. Polycrystalline Ni at both strain rates (Figs. 4.12a and 4.12b) showed substantial amounts of deep microvoids surrounded by coarse dimples, located at different areas of the samples (i.e. position A, B, C and D). The sizes of microvoids and dimples were observed to be smaller at the higher strain rate (Fig. 4.12b).
Step Structures

Step structures (Fig. 4.13a) for example located at position A had many vertical steps where no microvoid coalescence and dimples were observed in nanocrystalline Ni. Within the step structure, vein-like structures were often observed, sometimes referred to as the “river pattern” (e.g. Fig. 4.13b). Similar features were also found on nanocrystalline Ni-2.6%Fe and Ni-8.5%Fe alloys but were less noticeable under the same magnification (Figs. 4.14 and 4.15).

Cleavage-like Structures

Cleavage-like structures were found on Figure 4.13a at position B where both dimples and cleavage-like structures were observed on the sample fracture surface suggesting the possibility of a mixed fracture mode.

Table 4.5 Fracture features observed on all samples

<table>
<thead>
<tr>
<th>Sample</th>
<th>Grain Size</th>
<th>Strain Rate (/s)</th>
<th>Features</th>
</tr>
</thead>
<tbody>
<tr>
<td>Poly Ni</td>
<td>32 μm</td>
<td>10^2</td>
<td>Deep microvoids surrounded by coarse dimples</td>
</tr>
<tr>
<td></td>
<td></td>
<td>10^1</td>
<td>Deep microvoids surrounded by less coarse dimples</td>
</tr>
<tr>
<td>Nano Ni</td>
<td>57 nm</td>
<td>10^2</td>
<td>Mostly fine dimples, steps, vein-, cleavage-like</td>
</tr>
<tr>
<td></td>
<td></td>
<td>10^1</td>
<td>Mostly fine dimples, vein-like structures</td>
</tr>
<tr>
<td>Nano Ni-2.6%Fe</td>
<td>25 nm</td>
<td>10^2</td>
<td>Mostly fine dimples, steps, vein-like structures</td>
</tr>
<tr>
<td></td>
<td></td>
<td>10^1</td>
<td>Mostly fine dimples, vein-like structures</td>
</tr>
<tr>
<td>Nano Ni-8.5%Fe</td>
<td>20 nm</td>
<td>10^2</td>
<td>Mostly fine dimples, less noticeable vein-like</td>
</tr>
<tr>
<td></td>
<td></td>
<td>10^1</td>
<td>Mostly fine dimples, vein-like structures</td>
</tr>
</tbody>
</table>
Figure 4. SEM Micrographs of polycrystalline Ni at strain rate of $10^{-2}$/s at various positions
Figure 4. 12b SEM Micrographs of polycrystalline Ni at strain rate of $10^1$/s at various positions
Figure 4. 13a SEM Micrographs of nanocrystalline Ni at strain rate of $10^{-2}$/s at various positions
Figure 4. 13b SEM Micrographs of nanocrystalline Ni at strain rate of $10^{-1}$/s at various positions
Figure 4. 14a SEM Micrographs of nanocrystalline Ni-2.6%Fe at strain rate of $10^2$/s at various positions
Figure 4. 14b SEM Micrographs of nanocrystalline Ni-2.6%Fe at strain rate of $10^1$/s at various positions
Figure 4. 15a SEM Micrographs of nanocrystalline Ni-8.5%Fe at strain rate of $10^2$/s at various positions
Figure 4. 15b SEM Micrographs of nanocrystalline Ni-8.5%Fe
at strain rate of $10^{-1}$/s at various positions
4.5 Stress-Induced Heat Generation

4.5.1 In Situ Heat Generation

When metals are deformed, part of the plastic work is dissipated as heat during the deformation process. In this study, the amount of heat released was measured and recorded by the infrared (IR) detector at different engineering strain levels during the experiment. Figure 4.16a shows selected infrared images of polycrystalline Ni deformed at the strain rate of $10^{-2}$/s to the various overall strain levels indicated at the bottom of the individual snapshots. With the increase of engineering strain from 15% to 45%, the localized temperature along the sample gauge increases, indicated by the changes in color from light blue to yellow, orange and red. Heating is located at the center of the gauge length and the dashed line at 50% engineering strain indicates that the position of fracture is in the area of highest temperature (i.e. red color). At the higher strain rate of $10^{-1}$/s, the effect of heating on polycrystalline Ni is more pronounced (Fig. 4.16b). Heating is relatively uniform across the entire gauge length. The position of fracture is indicated within the area of highest temperature at 50% engineering strain (i.e. dashed line).

Nanocrystalline Ni deformed at the strain rate of $10^{-2}$/s generates a slight increase in temperature as engineering strain increases from 2.5% to 5.5% (Fig. 4.17a). Compared to polycrystalline Ni, heat is more concentrated at the center of the sample gauge, close to the area where final fracture will occur. Again, the dashed line corresponds to the position of fracture at an angle of $62^0$ along the loading axis. Similar to polycrystalline Ni, the effect of heating at higher strain rate is more noticeable. The area of highest temperature at 50% engineering strain indicated by the dashed line locates the position of fracture at an angle of $62^0$ (Fig. 4.17b).

Results were similar for nanocrystalline Ni-2.6%Fe and Ni-8.5%Fe. During the uniform deformation from 2.5% to 5.5% overall engineering strain, there are little changes in temperature for nanocrystalline Ni-2.6%Fe at the strain rate of $10^{-2}$/s (Fig. 4.18a). However, heat is more localized at the center of the sample gauge after necking and forms a fracture angle of $60^0$ at 7.5% overall engineering strain (i.e. dashed line).
Figure 4. 16a: Infrared images of polycrystalline Ni deformed at a strain rate of $10^{-2}/s$.

The dashed line at 50% engineering strain indicates the position of final fracture.

Figure 4. 16b: Infrared images of polycrystalline Ni deformed at a strain rate of $10^{-1}/s$.

The dashed line at 50% engineering strain indicates the position of final fracture.
Figure 4. 17a: Infrared images of nanocrystalline Ni deformed at a strain rate of $10^{-2}$/s. The dashed line at 8% engineering strain indicates the position of final fracture.

Figure 4. 17b: Infrared images of nanocrystalline Ni deformed at a strain rate of $10^{-1}$/s. The dashed line at 8% engineering strain indicates the position of final fracture.
Figure 4. 18a: Infrared images of nanocrystalline Ni-2.6%Fe deformed at a strain rate of $10^{-2}$/s. The dashed line at 7.5% engineering strain indicates the position of final fracture.

Figure 4. 18b: Infrared images of nanocrystalline Ni-2.6%Fe deformed at a strain rate of $10^{-1}$/s. The dashed line at 7.5% engineering strain indicates the position of final fracture.
Figure 4. 19a: Infrared images of nanocrystalline Ni-8.5%Fe deformed at a strain rate of $10^{-2}$/s. The dashed line at 7.5% engineering strain indicates the position of final fracture.

Figure 4. 19b: Infrared images of nanocrystalline Ni-8.5%Fe deformed at a strain rate of $10^{-1}$/s. The dashed line at 7.5% engineering strain indicates the position of fracture.
Nanocrystalline Ni-8.5\%Fe at the strain rate of $10^{-2}/s$ also gradually generates a temperature increase until about 5.5\% within the uniform deformation (Fig. 4.19a). The dashed line indicates the position of fracture at an angle of 62° after necking at the area of highest temperature. The heating patterns for both nanocrystalline Ni-2.6\%Fe (Fig. 4.18b) and Ni-8.5\%Fe (Fig. 4.19b) at $10^{-1}/s$ are relatively more distinct but the fracture angles remain at 60° and 62° respectively, within the areas of highest temperature.

4.5.2 Conversion of Plastic Work to Heat

Recalling from Eq. 11 regarding the heat conversion factor, the Taylor-Quinney coefficient $\beta$, during mechanical deformation, $\beta = \frac{\rho c_r}{\sigma_{ij} e_{ij}^p} = \frac{\rho c_r}{dW_p}$, shows that part of the work done ($dW_p$) on the material contributes to heat generation ($\Delta T$) while the rest remains as stored energy. Depending on the material properties (i.e. material density, heat capacity and flow stress) and the strain rate, the heat conversion factor (i.e. Taylor-Quinney coefficient) varies from material to material. Both the material density and heat capacity of polycrystalline and nanocrystalline Ni and Ni-Fe samples were previously determined as 8.908 g/cm$^3$ and ~ 0.48 J/gK, respectively [Turi and Erb 1995]. With different strain rates at $10^{-2}/s$ and $10^{-1}/s$, the acquired flow stress from the stress-strain curves (e.g. Appendix B) and the recorded rates of temperature increase, the value of $\beta$ for each material can therefore be determined.

Figure 4.20 shows the effect of strain rate on the Taylor-Quinney coefficient for polycrystalline Ni. At a strain rate of $10^{-2}/s$, the heat conversion factor in the beginning of the deformation process fluctuates around 0.5. As engineering plastic strain increases, the conversion factor decreases gradually to 0.2. Most of the plastic work done during deformation contributes to stored energy. In contrast, the heat conversion factor in polycrystalline Ni at the higher strain rate behaves differently. The heat conversion factor is initially at 0.8. However, the curve increases steadily with deformation and reaches a value of over 1.4 at the end of 40\% plastic strain. At a strain rate of $10^{-1}/s$, most of the energy from plastic work in polycrystalline Ni is converted to heat as plastic strain increases.
Figure 4.20: Effect of strain rate on the Taylor-Quinney coefficient for polycrystalline Ni

Figure 4.21: Effect of strain rate on the Taylor-Quinney coefficient for nanocrystalline Ni
Figure 4.22: Effect of strain rate on the Taylor-Quinney coefficient of nanocrystalline Ni-2.6%Fe

Figure 4.23: Effect of strain rate on the Taylor-Quinney coefficient of nanocrystalline Ni-8.5%Fe
The effect of strain rate on the heat conversion factor in nanocrystalline Ni was also analyzed. With the refinement of grain size, nanocrystalline Ni converts increasing more plastic work to heat during the deformation process (Fig. 4.21). At the strain rate of $10^{-2}$/s, the conversion factor initially is at 0.3 and increases to 0.7 by the end of 4% plastic strain. The heat conversion factor at a strain rate of $10^{-1}$/s follows similar trend and is slightly higher than for the lower strain rate.

The heat conversion factors in nanocrystalline Ni-Fe samples also increases with increasing strain rate and overall plastic strain (Figs. 4.22 and 4.23). Within the 4% plastic strain, the conversion factor of nanocrystalline Ni-2.6%Fe, initially at 0.6 for both strain rates, increases to 0.8 and 1.0 for the strain rates $10^{-2}$/s and $10^{-1}$/s, respectively (Fig. 4.22). Nanocrystalline Ni-8.5%Fe at $10^{-1}$/s converts heat from 0.55 to 0.9, whereas the conversion factor at $10^{-2}$/s only increases slightly to 0.6 (Fig. 4.23).

These results show that the heat conversion factors $\beta$ for polycrystalline and nanocrystalline Ni and Ni-Fe alloys change with strain rate and overall plastic strain. Polycrystalline Ni deformed at strain rate of $10^{-2}$/s follows the residual stress theory [Aravas et al 1990, see Fig. 2.15a], in which most of the mechanical energy is converted to stored energy as residual stresses (i.e. lattice imperfections) and contributes to strain hardening during the deformation processes. With increasing strain rate to $10^{-1}$/s, polycrystalline Ni follows more the dislocation theory [Zehnder 1990, see Fig. 2.15b]. Mechanical energy is partially stored due to dislocation interactions and multiplication during deformation. However, as mentioned by Rittel [1999] some of the stored energy in a material can be released and contributes to instantaneous dissipation during the deformation process. As a result, $\beta$ of polycrystalline Ni exceeds unity and reaches values in excess of 1.4 at 40% overall engineering strain.

With the reduction of grain size, deformation processes in nanocrystalline materials are more and more controlled by grain boundaries and triple junctions [e.g. Van Swygenhoven et al. 2001; Kumar et al. 2003]. The effects of lattice imperfections, dislocation interactions and multiplication are less pronounced as compared to polycrystalline materials. As a result, most of the mechanical energy during deformation in both nanocrystalline Ni and Ni-Fe is released as heat. The increase of strain rate favors a higher heat conversion factor in both nanocrystalline Ni and Ni-Fe.
4.5.3 Effects of Grain Sizes and Strain Rates on Heat Generation

The maximum temperatures within the sample gauges were recorded for tests conducted at room temperature (i.e. 25°C). The results were analyzed for the two different strain rates and correlated with the true stress-strain curves. Figure 4.24a shows the relationship between the true stress-strain curve and the maximum temperature increase of polycrystalline Ni deformed at a strain rate of 10^{-2}/s. Within the region of uniform deformation up to 38% true strain, the curve of temperature increase is relatively linear and recorded an increase of 170°C just before the onset of necking at ~ 650 MPa true stress and ~ 38% true strain. Beyond necking, a rapid increase in temperature was recorded and the maximum increase was 250°C at nearly 40% true strain. The maximum temperature of the sample was therefore 500°C at the point of sample fracture. Polycrystalline Ni converts more plastic work to heat energy during deformation at a higher strain rate of 10^{-1}/s (Fig. 4.20). The temperature increase is therefore more rapid (Fig. 4.24b) even though there are no major changes in the true stress-strain curves for the two strain rates. At the onset of necking, the maximum temperature increase was 450°C. A further rapid increase to nearly 700°C was recorded just before sample fracture. This means that the maximum temperature detected in the sample gauge was around 950°C in polycrystalline Ni at fracture, about twice the temperature recorded for the strain rate of 10^{-2}/s.

Nanocrystalline Ni shows a different behaviour in converting plastic work to heat. Figure 4.25a exhibits the true stress-strain curve and the maximum temperature increase in nanocrystalline Ni deformed at 10^{-2}/s. Close to the onset of necking at 1600 MPa true stress and 6.2% true strain, the temperature increase recorded was 150°C. As deformation continues to final fracture, the rise in temperature was 340°C, close to 8% true strain. In other words, the final temperature was around 590°C at fracture. Similar to polycrystalline Ni, nanocrystalline Ni showed no significant changes in the stress-strain curve at a strain rate of 10^{-1}/s (Fig. 4.25b). The temperature detected at the onset of necking also was ~ 150°C but the final increase was 440°C. In other words, the final temperature at fracture was 690°C, just 100°C higher than for the strain rate of 10^{-2}/s.
Figure 4. 24a: Relationship between the true stress-strain curve and the maximum temperature increase for polycrystalline Ni deformed at a strain rate of $10^{-2}$/s

Figure 4. 24b: Relationship between the true stress-strain curve and the maximum temperature increase for polycrystalline Ni deformed at a strain rate of $10^{1}$/s
Figure 4.25a: Relationship between the true stress-strain curve and the maximum temperature increase for nanocrystalline Ni deformed at a strain rate of $10^{-2}/s$.

Figure 4.25b: Relationship between the true stress-strain curve and the maximum temperature increase for nanocrystalline Ni deformed at a strain rate of $10^{-1}/s$. 

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Figure 4.26a: Relationship between the true stress-strain curve and the maximum temperature increase for nanocrystalline Ni-2.6%Fe deformed at a strain rate of $10^{-2}$/s.

Figure 4.26b: Relationship between the true stress-strain curve and the maximum temperature increase for nanocrystalline Ni-2.6%Fe deformed at a strain rate of $10^{-1}$/s.
Figure 4. 27a: Relationship between the true stress-strain curve and the maximum temperature increase for nanocrystalline Ni-8.5%Fe deformed at a strain rate of $10^{-2}$/s.

Figure 4. 27b: Relationship between the true stress-strain curve and the maximum temperature increase for nanocrystalline Ni-8.5%Fe deformed at a strain rate of $10^1$/s.
With further grain size refinement, nanocrystalline Ni-2.6%Fe at strain rates of $10^{-2}$/s (Fig. 4.26a) and $10^{-1}$/s (Fig. 4.26b) generated a temperature increase of about $16^0$C at nearly 6% plastic strain. As the samples went through necking, sharp increases in temperature to $57^0$C at 8% true strain and $66^0$C at 7% true strain were detected for $10^{-2}$/s and $10^{-1}$/s at final fracture, respectively. Nanocrystalline Ni-8.5%Fe showed the following results (Figs. 4.27a and 4.27b). Before the onset of necking, the temperature increases were about $15^0$C and $21^0$C for strain rates of $10^{-2}$/s and $10^{-1}$/s, respectively. As plastic strain increased, the maximum temperatures were $59^0$C for the strain rates of $10^{-2}$/s and $71^0$C for $10^{-1}$/s, respectively.

Table 4.6 summarizes all maximum temperatures of all materials at necking and final fracture for both strain rates. As mentioned in the previous section, nanocrystalline Ni and Ni-Fe exhibited relatively high heat conversion factors $\beta$ during deformation. However, nanocrystalline samples reach very high temperatures due to the limited ductility (i.e. engineering strain).

**Table 4.6 Summary of the maximum temperatures recorded at room temperature (i.e. 25$^0$C) for all samples**

<table>
<thead>
<tr>
<th>Samples</th>
<th>Grain Size</th>
<th>Strain Rate (/s)</th>
<th>At Necking ($^0$C)</th>
<th>At Fracture ($^0$C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Poly Ni</td>
<td>32 $\mu$m</td>
<td>$10^{-2}$ $10^{-1}$</td>
<td>42 70</td>
<td>50 95</td>
</tr>
<tr>
<td>Nano Ni</td>
<td>57 nm</td>
<td>$10^{-2}$ $10^{-1}$</td>
<td>40 40</td>
<td>59 69</td>
</tr>
<tr>
<td>Nano Ni-2.6%Fe</td>
<td>25 nm</td>
<td>$10^{-2}$ $10^{-1}$</td>
<td>41 42</td>
<td>57 66</td>
</tr>
<tr>
<td>Nano Ni-8.5%Fe</td>
<td>20 nm</td>
<td>$10^{-2}$ $10^{-1}$</td>
<td>40 46</td>
<td>59 71</td>
</tr>
</tbody>
</table>
4.6 Influence of Localized Heating on Microstructure

As discussed in the introduction, several previous studies have demonstrated considerable instability of the microstructure of nanocrystalline metals during plastic deformation. This instability was manifested in grain growth observed close to the fracture surfaces in materials after tensile testing. The two mechanisms suggested for grain growth were i) localized heating in narrow shear bands resulting in thermally activated grain growth [e.g. Dalla Torre et al. 2002] and ii) subgrain coalescence by a grain rotation mechanism [e.g. Moldovan et al. 2002; Wang et al. 2008]. There is also the possibility of stress-induced grain boundary migration as proposed from simulation studies by Cahn and co-workers[e.g. Cahn et al. 2006].

The main objective of the current study was to assess the likelihood of thermally activated grain growth by measuring the actual temperature profiles in the gauge sections of nanocrystalline Ni and Ni-Fe alloys during tensile testing. The results have shown that the maximum temperatures that were recorded in these materials was at least 30°C lower than the onset temperatures for subgrain coalescence previously measured for these materials through differential scanning calorimetry. On the basis of these results, it can be stated that thermally activated grain growth during tensile testing of nanocrystalline Ni and Ni-Fe alloys is not likely to occur.
5. Conclusions

Overall tensile properties, stress-induced heat generation and strain localization in polycrystalline and nanocrystalline Ni and Ni-Fe alloys during plastic deformation were reported in the current study. The observed tensile properties (i.e. yield strength, ultimate tensile strength and elongation) of polycrystalline and nanocrystalline alloys at strain rates of $10^2$/s and $10^1$/s were consistent with results of previous studies on similar materials.

The events of strain localization in polycrystalline and nanocrystalline Ni and Ni-Fe alloys during plastic deformation were captured with a digital image correlation (DIC) system and the evolution of localized strain at different levels of engineering strain and strain rate prior to sample fracture was recorded. Both polycrystalline and nanocrystalline Ni and Ni-Fe alloys showed increases of localized tensile strain with increasing overall engineering strain values. Localized strains in nanocrystalline Ni and Ni-Fe alloys were considerably lower than in polycrystalline Ni. This can be explained by the differences in the strain hardening capacity and overall ductility. For both strain rates, the positions of fracture within the necking zone were constant at ~ $60^\circ$ for nanocrystalline Ni and Ni-Fe alloys and $90^\circ$ for polycrystalline Ni.

SEM images of the fracture surfaces showed numerous features which support the ductility observed in the stress-strain curves for all materials, such as: dimples, microvoids and vein-like structures. Polycrystalline Ni with a much higher ductility indicated deep microvoids surrounded by coarse dimples. Evidence of fine dimples and cleavage-like structures on the sample fracture surfaces of nanocrystalline Ni alloys suggests the possibility of a mixed fracture mode. Similar features were also found on nanocrystalline Ni-Fe alloys but less noticeable at the same magnification. The sizes of microvoids and dimples were observed to be smaller at the higher strain rate.

This study also investigated the phenomenon of stress-induced heat generation in all materials. The amount of heat released was measured and recorded at room temperature (i.e. $25^\circ$C) by an infrared (IR) detector at various overall strain levels during tensile deformation. Heating in nanocrystalline Ni and Ni-Fe alloys was concentrated at the center of the sample gauge, close to the area where final fracture occurred. The positions of final fracture corresponded to the areas
of highest temperature, at an angle of ~ 60° with respect to the tensile axis. Polycrystalline Ni tested at the higher strain rate showed considerable heating over the entire gauge length.

The heat conversion factor (i.e. Taylor-Quinney coefficient \( \beta \)) for polycrystalline Ni deformed at strain rate of \( 10^{-2} \) /s follows the residual stress theory, in which most of the mechanical energy is converted to stored energy as residual stresses (i.e. lattice imperfections) and contributes to strain hardening during the deformation processes. However, polycrystalline Ni follows more the dislocation theory due to dislocation interactions and multiplications as the strain rate was increased to \( 10^{-1} \) /s. The result of \( \beta \) for polycrystalline Ni exceeding unity and reaching values in excess of 1.4 is likely due to instantaneous heat dissipation during the deformation process. With deformation processes in nanocrystalline Ni and Ni-Fe alloys being more controlled by grain boundaries and triple junctions, most of the mechanical energy during deformation is released as heat (i.e. high \( \beta \) value). In general, the increase of strain rate favors a higher \( \beta \) value in both nanocrystalline Ni and Ni-Fe alloys.

Maximum temperatures at various points along the true stress-strain curves were recorded for all materials at different strain rates. All samples showed a significant increase in temperature after the onset of necking. Since polycrystalline Ni has a relatively high heat conversion factor \( \beta \) at the strain rate of \( 10^{-1} \) /s, the temperature increased rapidly and reached ~ 95°C, about twice the temperature recorded for the strain rate of \( 10^{-2} \) /s. Nanocrystalline Ni and Ni-Fe reached average maximum temperatures up to ~ 59°C and ~ 70°C at the strain rates of \( 10^{-2} \) /s and \( 10^{-1} \) /s, respectively.

According to earlier differential scanning calorimetry studies, the beginning of subgrain coalescence in nanocrystalline Ni was around 100°C. The results of the current study have shown that the maximum temperatures that were reached in these materials was at least 30°C lower than the onset temperatures for subgrain coalescence. Therefore, thermally activated grain growth during tensile testing of nanocrystalline Ni and Ni-Fe alloys is not likely to occur. However, the possibilities of grain growth due to a grain rotation mechanism or stress-induced grain boundary migration in nanocrystalline metals cannot be ruled out on the basis of this particular study.
6. Recommendations for Future Work

Recommendations for future work include:

1. Conduct microstructural analysis at the localized hot spots near the fracture areas of the nanocrystalline Ni and Ni-Fe samples using Transmission Electron Microscopy (TEM) to check for grain size gradients. There have been suggestions in the literature that grain growth may accompany shear band formation.

2. Measure microhardness or nanoindentation hardness profiles along the gauge lengths on the fractured samples. Such hardness profiles could potentially reveal any structural changes within the gauge section as a result of the tensile deformation.

3. Conduct quasi-static strain rate experiments (e.g. at $10^{-5}$/s) on similar samples to cover a broader range of strain rates. Lower strain rates are expected to generate reduced heating during sample deformation.

4. Examine localized strains within the elastic region of polycrystalline and nanocrystalline Ni and Ni-Fe alloys in much more depth using the DIC method. Recent studies have suggested that the Poisson’s ratio in nanocrystalline metals could be higher than for polycrystalline metals. An analysis of strains in x and y directions could potentially reveal this phenomenon. However, such an analysis should be done at a lower strain rate (e.g. at $10^{-3}$ – $10^{-5}$/s) to ensure sufficient data acquisition in the elastic region.
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8. Appendix

8.1 Appendix A

Figures 8.1 to 8.3 show the basic principles on the computation of the 2D DIC method by comparing the digital images of the test object surface at different states [Pan et al. 2009].

Figure 8. 1: (a) Reference image, the imposed square is the subset used for tracking the motion and the intersection points of the yellow grid denote the points to be calculated; (b) the calculated displacement vectors imposed on the deformed image [Pan et al. 2009]

Figure 8. 2: Schematic illustration of a reference square subset before deformation and a target (or deformed) subset after deformation [Pan et al. 2009]
Figure 8. 3: Schematic figure of local discrete correlation coefficient distribution obtained from an integer pixel displacement scheme [Pan et al. 2009]
8.2 Appendix B

Figure 8.4 shows an example of flow stress on a stress-strain curve required to calculate the Taylor-Quinney coefficient $\beta$ for different materials.

![Figure 8.4: Example of flow stress on a stress-strain curve](image-url)