A Parametric Study of Jet Fuel Thermal Stability

by

Sara Khan

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Graduate Department of Institute for Aerospace Studies
University of Toronto

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Thermal stability and degradation of a commercial jet fuel were studied using an experimental rig developed recently. The apparatus consists of a fuel pre-heating section to simulate the usage of fuel as a coolant on aircraft, and a test section to simulate operating conditions found in aircraft injector fuel nozzles. Two independent diagnostic techniques, pressure drop measurements and carbon burn-off, were applied to assess carbon deposition produced within a test section. Parametric studies were performed to investigate the effect of test section inner diameter, test section material, and test section inlet temperature gradient on carbon deposition within the test section. It was determined that as the inner diameter of a test section increased, carbon deposition generally decreased. The axial location of maximum surface deposition moved downstream in a narrower test section. It was also found that solid carbon deposition decreased by increasing the test section inlet temperature gradient.
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Chapter 1

Introduction

1.1 Motivation

Modern aviation gas turbine engines use petroleum distillate to propel commercial and military aircrafts. Jet fuel is a middle-distillate product of the crude oil refinery process. Predominant types of commercial jet fuel used worldwide are kerosene base; namely, Jet A and Jet A-1 [1]. Fuel in aircrafts not only serve as an energy source but also as a coolant before entering the combustion process. Systems such as hydraulics, gear boxes, avionics, and electrical systems are sources of heat which use fuel as a coolant in view of the fuel’s high heat capacity [2].

Thermal heat management in aircrafts is crucial since new engines are designed to be more powerful, fuel efficient, and environmentally friendly [3, 4]. Fuel undergoes the most thermal stress during idle and flight descent conditions, reaching temperatures in excess of 200°C at fuel injector nozzles [5]. Although the fuel flow rate is lower in these modes, the fuel is still required to absorb high heat sources. The past few decades have shown an exponential increase in cooling requirements of aircrafts and this trend is expected to continue [6]. Exposure of fuel to high temperatures in aircraft poses a problem as fuel begins to breakdown and change structure with temperatures exceeding
150°C [7]. By exposing fuel to even higher temperatures, carbon deposition is promoted which hinders the performance of aircraft engines. A buildup of deposits in fuel lines restrict the flow, resulting in decreased heat exchanger effectiveness and an increase in pumping power requirements to maintain the same flow rate [8]. Deposits formed near fuel injector nozzles can also cause problems by producing undesirable spray patterns and subsequently causing thermal stress and degradation to combustors and turbines [8].

Due to coking of jet fuel under thermal stress, research to tackle thermal stability of aviation fuels has been ongoing since the 1950s. Various facilities have been developed to thermally stress fuel under different operating conditions. Engine problems related to inadequate fuel thermal stability typically become evident only after hundreds or thousands of hours of operation. The long time and the large volume of fuel consumed makes it impractical to test fuel thermal stability under conditions identical to those that exist in engines [1]. Therefore, facilities for assessing thermal degradation of aviation fuels, are designed to see measurable effects in a reasonable period of time. The most well-known standardized test utilizes the jet fuel thermal oxidation tester (JFTOT) [9]. There are also various testing apparatus, commonly deployed in university laboratories, to study thermal stability of jet fuel. These studies can range from understanding chemistry of formation of carbon deposits to studying effects of numerous parameters on deposition. These various studies performed on jet fuel will be discussed in further detail within the following sections.

1.2 Thermal Stability Chemistry and Mechanisms

Extensive literature review and many experiments performed show that there are a variety of factors which affect thermal stability of jet fuel [10]. However, the most important parameter in thermal stability is considered to be temperature. Generally, as fuel is subjected to high temperatures, the propensity of thermal degradation of the fuel increases.
An increase in temperature does not necessarily result in an increase in carbon deposit formation [11,12]. A general trend of the effect of temperature on deposition is outlined in Figure 1.1. As shown, temperatures between 175°C and 325°C as well as temperatures greater than 450°C display a steady increase in carbon deposition. However, temperatures ranging from 325°C to 450°C yield a rapid decline in deposition. The figure also illustrates that removal of dissolved oxygen from fuel suppresses the quantity of deposits over temperatures of 175°C to 475°C. This will be discussed further in Section 1.3.1.

Figure 1.1: Expected trend of deposit formation as the temperature is increased. The removal of oxygen also suppresses carbon deposition [13].

Carbon deposition is one of the many consequences of fuel degrading under thermal stress, which is influenced by various parameters. At times, carbon deposition is considered to be a side branch of fuel degradation reactions since the amount of deposits produced in comparison to the bulk fuel is insignificant [14, p.78]. In one study [14, p.96],
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It was concluded that the mass fraction of fuel converted to deposits was only $0.1 \times 10^{-6}$ (0.1 ppm by mass). Similar to the previous study, Venkataraman et al. [15] proposed that an insignificant mass fraction of $13 \times 10^{-6}$ (13 ppm by mass) of Jet A deposited on wall surfaces. Although fuel degradation refers to all the changes in the composition of the fuel under thermal stress, carbon deposits, even at small amounts, are the most detrimental product of thermal stressing.

Thermal degradation of fuel can be broken down into three regimes. The autoxidation regime exists between temperatures from $150^\circ$C to $350^\circ$C. Moving above temperatures of $400^\circ$C, the pyrolytic regime is in place. In between these two regimes is the transition regime [16, 17]. In all three regimes, it is believed that free radical reactions are key components in the chemical reaction mechanisms [18]. The mechanisms for the autoxidation and pyrolysis regimes are well studied and will be discussed in the next sections. However, little research is conducted to shed light on the transition regime. It is also uncertain whether the transition regime contains aspects of autoxidation or pyrolytic mechanisms or a combination of both.

1.2.1 Autoxidation Mechanism

There have been many hypotheses to describe the process of autoxidation. The regime can be explained by two main chemical mechanisms. The first mechanism suggests that when dissolved oxygen reacts with fuel compounds, they produce free radicals. This is known as the first chain initiating step in autoxidation. As a result, hydroperoxide is the next key intermediate species formed [19] [14, p.69,93]. By continuing to oxidize, hydroperoxides form insoluble gums which inevitably leads to carbon deposits [11,20].

The second mechanism is known as the soluble macro-molecular oxidatively reactive species (SMORS). Hardy and Wechter [21] proposed this concept when researching carbon deposition in long term storage of diesel fuel. The study, and proposed mechanism, was later found to be applicable to jet fuel [11,17]. The hypothesis states that polar com-
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5 pounds, such as phenols, are the precursors in forming deposits [17]. Phenol, commonly found in jet fuel, autoxidizes to create a compound called quinone. This molecule then bonds with electron-rich aromatics to produce a low molecular weight SMORS [22]. By itself, SMORS can be made to precipitate out of the fuel in the presence of methanol, otherwise it is insoluble [20,22,23].

The two main chemical mechanisms discussed above focused on how insoluble particles precipitate from jet fuel. After precipitation, there are three routes for insolubles to form surface deposits (Figure 1.2) [24]:

1. Insoluble particles still contained within the bulk fuel diffuse towards the hot surface, attach, and then agglomerate.

2. The metal surface catalyzes reactions for insoluble particles to form directly on hot surfaces.

3. A combination of the above two routes occur.

![Figure 1.2: Deposition pathways from insoluble particle to surface deposit. The insoluble particle can either precipitate out of the liquid, diffuse to the surface, adhere and agglomerate, or directly form on the surface through a surface catalyzed reaction [24].](image)

The route in which surface deposition is formed depends on the concentration of precursor species in the bulk fuel [24]. If the precursor concentration is large, deposition
happens by the first route and corresponds to the SMORS mechanism. However, if the precursor concentration is low, a high surface temperature will allow deposition through the second route. Also in this case, a combination of the two routes of surface deposition is possible [11].

1.2.2 Pyrolysis Mechanism

The pyrolytic temperature regime involves different mechanisms compared to the autoxidation regime. The most dominant feature in this regime is decomposition over time since the deposition rate increases nearly exponentially as temperature increases [25]. Oxygen is not an important factor since it is assumed to be completely consumed during the autoxidation mechanism [14, p.94]. The main pyrolytic mechanism is initiated with the formation of various length alkyl radicals. This occurs either with (i) thermolysis (chain scission) or (ii) catalytic cracking of weak C-C bonds [10, 26] [7, p.195] [27, p.22]. After the initiating step, a pressure-dependent chain propagation reaction occurs on the alkyl radicals. At high pressures, bi-molecular hydrogen abstraction occurs since there is a greater chance of molecular collisions [27, p.22]. At lower pressures, unimolecular decomposition of the alkyl radical by $\beta$-scission propagates the free radical species while forming an alkene [27, p.23]. The free radicals, alkenes, alkyl cycloalkanes and alkyl cycloalkenes then undergo cyclization and dehydrogenation to form alkyl aromatic compounds such as alkylbenzenes and 2-ring aromatic structures like naphthalene [10,16] [27, p.26]. Condensation of the alkyl aromatic compounds eventually form six or seven ring polycyclic aromatic hydrocarbons (PAH) which precipitate out of the fuel in the form of solid deposits. Due to the difference in nature of deposits formed by autoxidation and pyrolysis, test rigs are designed to study both these regimes to further expand the understanding of chemical mechanisms involved.
1.3 Effects of Operating Parameters

1.3.1 Dissolved Oxygen

The significant factors that affect carbon deposition are oxygen concentration, temperature and fuel composition [12]. Among these three factors, concentration of dissolved oxygen is the most important when it comes to determining the concentration of carbon deposits formed in the autoxidation regime. Oxygen is found both dissolved within liquid fuel and chemically bonded to the fuel hydrocarbons. To directly study the effects on carbon deposition, oxygen content in liquid fuel can be removed (de-oxygenated) or added (air saturated). Beaver et al. [17] performed a study which concluded that jet fuel, de-oxygenated by nitrogen, produced relatively less carbon deposits compared to air-saturated fuel in a 90 cm long tube. The results are shown in Figure 1.3. Studies have shown that in order to observe these effects on carbon deposition, the mass fraction of dissolved oxygen must fall within a certain range. Specially, between $1 \times 10^{-6}$ to $30 \times 10^{-6}$. As seen in Figure 1.4, mass fractions greater than this show no observable change in deposition rate. While mass fractions less than the stated range result in insufficient oxygen to produce carbon deposits [28,29] [7, p.10]. Multiple studies have concluded that the removal of dissolved oxygen from liquid fuel decreases the total amount of deposits formed [28,29] [14, p.101].

The amount of dissolved oxygen in the fuel can also effect the induction time: the period before deposits first form on surfaces. Venkataraman et al. [15] varied experimental times from five minutes to ten hours and concluded that the minimum induction time for initial surface deposit formation was approximately 15 minutes. Another study carried out by Watkinson and Wilson [24] showed that the induction period decreased as the dissolved oxygen content of the fuel was increased. Furthermore, induction time is also linked to catalytic reactions between fuel and fresh metal surfaces vacant of carbon deposits [30]. This indicates that other factors may be affecting the studies above.
Figure 1.3: Carbon deposition profile of deoxygenated and air-saturated JP-8 along length of a heated tube in PSUs flow reactor. Increasing axial distance is representative of increasing fuel temperature, with the region between 0 cm to 50 cm as the autoxidation regime and the remaining as pyrolytic [17].

Figure 1.4: Effect of dissolved oxygen mass fraction on deposition rate in the Aero Propulsion Laboratory JFTOT experiment [14, p.101].
1.3.2 Fuel Storage

The main concern for thermal stability in jet fuel is during flight conditions since the fuel is exposed to high temperatures and shorter residence time (known as the duration of time which fuel is exposed to specific experimental conditions). However, the fuel may also degrade when it is placed in storage over long periods of time ranging from weeks to months from initial procurement [1]. Multiple studies [31–34] analyzed various aged fuels, from weeks to years old, and found either insignificant or no effect of aging on thermal stability. A study conducted by Pande and Hardy [35], thermally stressed JP-5 at temperatures of 20°C and 90°C with additives such as, copper (II) ethyl acetoacetate ($\text{C}_{12}\text{H}_{18}\text{CuO}_6$). The fuel was stored over a range of time from 24 hours to 6 months. The study concluded that carbon deposits increased with the presence of copper additives, high temperatures, and longer storage times.

1.3.3 Pressure

With the various operating pressures within an aircraft fuel and injector system, the effect of pressure on thermal degradation of fuel is of importance; effecting the Reynolds number, residence time, and subsequently the amount of carbon deposition. When jet fuel is in the state of an incompressible liquid, pressure has little effect on these three parameters in the autoxidation regime [36]. However, in the pyrolytic regime, lighter hydrocarbons can vaporize forming a two-phase fluid when exposed to high pressures [37]. Previous studies [24, 38] experimented with three different Jet A fuels and determined that increasing the absolute pressure, up to 2.17 MPa (315 psia), caused a decrease in the amount of deposits formed on the test section’s surface for both de-oxygenated and air-saturated fuel. Furthermore, this increase in pressure resulted in an axial shift of carbon deposits downstream in the cylindrical test section. However, between pressures 2.17 MPa and 4.24 MPa (615 psia), the deposition was independent of pressure. Szetela et al. also found that deposition rate was independent of pressure between 2.76 MPa and
5.51 MPa for fuels thermally stressed between 177°C and 482°C [39]. A study performed by Marteney and Spadaccini tested JP-5 at three different pressures of 1.8, 2.9, and 5.6 MPa (250, 400, and 800 psig respectively) which showed no influence of pressure on carbon deposition [40]. Lastly, Ervin and colleagues determined that raising the pressure increased the amount of deposits formed in the pyrolytic regime, as shown in Figure 1.5 [41]. Overall, the studies which showed a dependence of deposition formation on pressure were completed in high pressure and high temperature environments. Exposure to extreme environments would cause the fuel to be a supercritical fluid. Hence, the deposit formation may have been a result of other factors and not pressure. From an experimental perspective, the operating system pressure must not exceed the vaporization pressure since the phase change will yield anomalous deposition patterns.

![Figure 1.5: Effect of pressure on thermal-oxidative and pyrolytic deposition for treated and untreated test sections [41].](image-url)
1.3.4 Fuel Flow Regime

It is crucial to measure the effect of different flow regimes on deposit formation as the fuel experiences both laminar and turbulent regimes within a gas turbine engine. Spadaccini et al. [36] researched the effect of space velocity (the ratio of the volumetric flow rate to the total test passage volume) on carbon deposition. The study found that as space velocity was increased, carbon deposition increased linearly as well. The effect of Reynolds number and residence time on carbon deposition was also studied. It was determined that at higher Reynolds numbers the quantity of carbon deposits increased. However, the residence time of this study was also allowed to vary, suggesting the observation may not have been solely the result of varying Reynolds number. Furthermore, the authors attribute the increase in carbon deposits due to the enhanced heat and mass transfer resulting from the turbulent flow regime. Jones et al. [42] presented that surface deposition increased at lower Reynolds numbers, when the flow was laminar and residence time was long. Chin and Lefebvre [43] analyzed the effect of Reynolds number on the rate of deposition by changing the test section diameter. It was shown that an increase in Reynolds number produced an increase in deposition rate, as seen in Figure 1.6. This increase was attributed to (i) high heat transfers between the wall and fuel and (ii) large transverse velocity components. These flow characteristics allowed the material to be effectively transported from the bulk fuel flow to the test section walls.

Pande et al. [35] conducted tests on three different rigs (one which was turbulent) to determine the effect of fuel flow velocity on carbon deposition. Due to the relation of Reynolds number with temperature and residence time, it was deemed difficult to isolate the sole impact of Reynolds number on carbon deposits. Ultimately, the study found the amount of deposits in the laminar regime slightly exceeded those found in the turbulent regime. Carbon deposits in the laminar regime were caused by reactant depletion due to temperature gradients in the fuel. Therefore, it was concluded that Reynolds number was not a critical factor in influencing thermal stability in jet fuel.
Figure 1.6: Increase in deposition rate by increasing Reynolds number via the tube inner diameter [43].

1.3.5 Material and Geometry of Test Section

During engine operation, jet fuel encounters flow passages inside the fuel system designed with various geometries and materials. The interaction of fuel with the surface material of the test section, along with the geometry, has been known to affect heat transfer rates to the fuel [14].

Spadaccini and colleagues [36] performed experiments on test sections of different diameters. The results indicated that increasing the passage diameter reduced the amount of carbon deposits. By reducing the diameter, diffusion was promoted and subsequently carbon deposition was enhanced. The axial location of maximum deposition was also
shown to be shifted downstream in smaller diameters. A study conducted by Alborzi et al. [44] researched thermal stability in test sections with a sudden expansion and contraction as shown in Figure 1.7. These results were compared with a uniform cylindrical test section. The results showed a sharp rise in temperature, 50°C higher than the uniform cylindrical test section, occurring at the contraction location. The temperature rise was due to a recirculation zone allowing fuel to be exposed to heat for a longer period of time. This particular area was also shown to have the largest concentration of carbon deposits. However, a study by Pei and Hou [45] contradicted the previous study. They assessed thermal stability of secondary flow in a bent pipe. As displayed in Figure 1.8, the flow was re-directed 180° to produce the desired secondary flow. The results showed that deposits were relatively low in the bends compared to other parts of the test section. The bend induced secondary flow, promoting high heat transfer and radial mixing, which lead to lower temperatures and the observed decrease in carbon deposits. Furthermore, the presence of a recirculation zone at the bend also hindered wall deposition by eliminating the laminar sublayer.

Figure 1.7: Schematic depicting nominal cylindrical tube above the expansion/contraction tube. Thermocouples were placed at locations A, B, C, D, and E while "RF" is the radio frequency induction heating coil. Dimensions are given in mm [44].
Many studies performed have shown an influence of material on coking propensity of jet fuel. Faith et al. [46] performed a study on heated test sections fabricated from 14 different materials and alloys. The test sections were analyzed in the JFTOT. The results found that aluminum 6061, nickel 200, and stainless steel (SS) 446 exhibited the least amount of deposits. Inconel 600 and SS 316 were next in performance, producing six times more deposits than the aforementioned materials. Taylor [47] reported that aluminum, SS 304, and pure titanium gave equivalent deposition rates with air-saturated fuel while copper performed the worst among all the metals tested. Bradley et al. [48] assessed the effect of surface roughness on carbon deposition. They determined that a SS test section with a significantly smoother surface, as indicated by a micro-photograph, exhibited a much lower rate of deposition during the first period of testing compared to a rough surface SS test section. However, after 30 hours of testing, the deposition rates became equal. Altin and Eser [49] evaluated five different test sections constructed of SS 316, SS 304, nickel, Silcosteel, and glass-lined SS. In the results, nickel produced the most deposits followed by SS 316 and then SS 304 due to high catalytic activity of these surfaces. The inert materials, Silcosteel and glass-lined SS, produced the least amount of deposits. This showed the coatings effectively inhibited catalytic deposition and therefore minimized carbon deposits, overall. Another study performed by Tang et al. [50] presented that titanium nitride (TiN), titanium dioxide (TiO$_2$), and titanium carbide (TiC) coatings had an obvious anti-coking effect compared to a standard SS 304 test section. It was also shown that TiN and TiC coatings performed equally well and above the TiO$_2$ in terms of anti-coking abilities.
1.4 Research Objectives

The objective of this work is to investigate the effect of various operating parameters on coking propensity of Jet A-1. Experiments are carried out on a coking test rig designed and built by Wong and Liang [57,58] at the University of Toronto Institute for Aerospace Studies (UTIAS). Two independent diagnostic techniques, pressure drop measurements and carbon burn-off, are utilized to assess carbon deposition. Further discussion of the experimental outline will be presented later in the thesis. Overall, three experimental sets are performed to assess the following objectives:

1. Investigating the effect of inner diameter and material type on coking propensity while maintaining a constant Reynolds number.

2. Investigating the effect of inner diameter and material type on coking propensity while maintaining a constant residence time.

3. Investigating the effect of inlet temperature gradient on coking propensity.
Chapter 2

Experimental Apparatus

Prior to outlining the experimental apparatus utilized in this thesis, a brief overview of existing thermal stability test rigs and various diagnostic techniques is presented.

2.1 Existing Experimental Apparatus

Various testing rigs have been designed to research and explore thermal stability of aviation fuels. These experimental apparatus fall under two categories: i) continuous flow dynamic tests, and ii) pressurized static tests.

Dynamic tests have a continuous supply of fresh fuel through a heated test section. This allows for a steady fuel temperature profile and shortened fuel residence times within the test section. These tests have a greater application in studying thermal stability of aviation fuel during flight conditions since they mimic the movement of fuel through an aircraft fuel system. In contrast, static tests produce longer residence times by subjecting the fuel to a constant temperature in a stagnant environment [7, p.115]. Priority is given to dynamic testing rigs in this literature review since static tests are mainly used as a supplementary check [51].

Along with test rigs in various universities and laboratories, a standardized dynamic test rig also exists. The Coordinating Research Council (CRC) coker and the subsequent
JFTOT are the standardized apparatus for measuring thermal stability of jet fuel. In 1959, the ASTM D-1660 CRC coker was designed with a 330 mm long electrically heated aluminum tube within a cylindrical casing. Downstream of this assembly was a 25 μm porosity filter in an electric heated housing [14, p.14-17]. Laminar fuel flowed in between the aluminum tube and the cylindrical casing. The outlet was set to a constant temperature of 149°C and the filter temperature to 205°C. The purpose of the filter was to simulate the tiny fuel passage within a fuel spray nozzle. A constant flow rate of 60 mL/min was maintained for 5 hours at a system pressure of 1.0 MPa and residence time of 10 seconds. The maximum allowable pressure drop across the filter was 10 kPa. The diagnostic technique was based on visual discoloration of the aluminum tube from surface carbon deposition, as shown in Figure 2.1.

![Image of color code for ASTM JFTOT](image)

Figure 2.1: The visual reference of discoloration used on the standardized thermal stability testing rig [14].

In 1973, the JFTOT replaced the CRC coker with ASTM D-3241 [9,52] [14, p.16-17]. JFTOT reduced the quantity of fuel required by decreasing the flow rate to 3 mL/min over a period of 2.5 hours. Furthermore, the outlet temperature was increased to 260°C. Various other changes such as increasing the system pressure to 3.45 MPa, utilizing a non-heated filter housing, and reducing the filter pore size to 17 μm were present in the new apparatus. Visual discoloration remained as the diagnostic technique for measuring
carbon deposition with a maximum allowable pressure drop across the filter reduced to 3.3 kPa. Unfortunately, the JFTOT has major limitations from a research perspective; having a maximum temperature capacity of 260°C, while modern studies desired much higher temperatures [7, p.28] [27, p.6,9]. Furthermore, Morris et al. argued that the low fuel flow rate in the JFTOT only produced laminar flow which contrasted a real aircraft engine where turbulent flow may also be experienced [53]. Other limitations come from the qualitative and subjective nature of the diagnostic technique used to measure the carbon deposition.

Due to shortcomings of the JFTOT as a research tool, various research rigs were designed and built in several laboratories. Most of the rigs consist of single tube heat exchangers which utilize cylindrical test sections of various lengths, diameters, wall thickness, and materials. The test sections can also be heated by various methods of heat transfer. The United Technologies Research Center and Phoenix Rig conductively heated test sections with isothermal copper resistor blocks surrounding the test section [13,17,36,54]. Australian Airframes and Australian Defense Science and Technology Organization also conductively heated a submerged test section by means of a fluidized sand bath [55,56]. Alternatively, Pennsylvania State University’s (PSU) flow reactor radiatively heated a test section placed at the center of an electrically heated furnace [3, 10]. UTIAS also applies a similar method of heating the fuel test section [57–59] which will be further discussed in Section 2.3.1.

### 2.2 Common Diagnostic Techniques

Diagnostic techniques for assessing thermal stability, commonly found in literature, can be broken into two classes. The first class quantifies carbon deposition on test section surfaces while the other detects chemical changes in the fuel due to thermal stressing. Examples of these techniques are given in this section.
2.2.1 Temperature Programmed Oxidation

Temperature programmed oxidation is the most commonly used diagnostic technique for determining carbon deposition inside a metal fuel flow passage. Many researchers [15, 17, 36, 60–62] in the past have used this technique in studying thermal stability of jet fuel. In this technique, also known as carbon burn-off, the test section in question is fully dried and placed in a furnace with a constant flow of oxygen. As the test section is heated, the solid carbon deposits oxidize to produce carbon dioxide, as a consequence of the following global reaction,

\[ C_{\text{solid}} + O_2 \rightarrow CO_2 \]  \hspace{1cm} (2.1)

The apparatus then yields a value of carbon deposits in the specimen by relating it to the amount of carbon dioxide measured. This technique has been used at UTIAS in the past [57, 59]. Further discussion of the carbon burn-off apparatus used in this thesis is discussed in Section 2.3.2.

2.2.2 Pressure Drop Measurements

Another method to quantify the deposition as a result of thermally stressing fuel is to measure the pressure drop across a test section. The correlation between the pressure drop and pipe diameter within a cylindrical test section is derived from the Hagen-Poiseuille Law:

\[ \Delta P = \frac{8\mu LQ}{\pi r^4} \]  \hspace{1cm} (2.2)

Where \( \Delta P \) is the pressure drop across the length of the pipe, \( \mu \) is the dynamic viscosity, \( L \) is the length of the pipe, \( Q \) is the volumetric flow rate, and \( r \) is the radius of the pipe. If \( \Delta P_i \) and \( \Delta P_f \) are taken to be the initial and final pressure drops across the test section before and after thermal stressing, respectively, and \( R_i \) and \( R_f \) the initial and final radii,
respectively, by holding $\mu$, $L$, and $Q$ constant, the following relation between pressure drop and radii can be derived:

$$\frac{\Delta P_i}{\Delta P_f} = \frac{R_f^4}{R_i^4}$$  \hspace{1cm} (2.3)

From Equation 2.3, it is apparent that a larger pressure drop corresponds to a greater reduction in the test section diameter due to a buildup of carbon deposits on the inner wall surface of the test section. However, this relation may only be used for laminar flow since it will be invalid for turbulent flow. It is also important to note that the calculated reduction in test section diameter is an estimated average of the carbon deposit thickness. It is assumed that the thickness of carbon deposits is axially and radially uniform throughout the test section, which may not be correct [63]. A study by Yuen et al. used pressure drop measurements to monitor deposit buildup across a test section [64]. The study utilized the Hagen-Pouiseuille flow equation to calculate the apparent reduction in test section diameter as a result of deposition. It was determined that the pressure-drop method is an effective and non-intrusive technique that allows for continuous monitoring of deposit buildup. It was also concluded that the technique produced repeatable results, as shown in Figure 2.2, which is crucial in minimizing random errors. Therefore, this diagnostic technique was also used along with carbon burn-off in this thesis.
2.2.3 Spectroscopic Techniques for Chemical Analysis

Spectroscopic techniques can be very useful in determining changes in a fuel’s chemical composition due to thermal stressing. Specifically, UV-visible spectroscopy analyzes the absorbency and florescence of chemical species. Commodo et al. confirmed that these methods are sensitive to chemical changes which occur as a result of autoxidative reactions [65]. The researchers also studied high temperature formation of PAH in stressed fuel by using three-dimensional (3-D) UV-visible fluorescence [66]. Furthermore, the 3-D fluorescence spectra was also used to evaluate the effect of dissolved oxygen on fuel instability over a range temperatures to cover the autoxidative regime [67]. Electrospray ionization mass spectroscopy, which is highly selective to polar species, was another diagnostic technique used to analyze changes in polar species due to thermal degradation in the fuel [68].

Another chemical analysis method used for assessing thermal stability of fuels is
nuclear magnetic resonance (NMR). This spectroscopic technique operates by detecting a specific frequency of an atom caused by the nuclei when subjected to an external magnetic field. The specific frequency then allows determination of the type of bond present as well as its concentration in the sample. Various species that may be present in the sample, such as aromatics and paraffins, are then classified by the type of bond. Young applied this diagnostic technique in his study to determine the change in composition due to thermally stressing alternative fuel blends [59]. Fuels were analyzed by NMR before and after being thermally stressed and degradation of fuel was based on the concentration of aromatics.

Since fuel composition was not the prime focus of this thesis, spectroscopic techniques and NMR were not adopted in analyzing thermal stability of jet fuel.

2.3 Experimental Apparatus

To assess the coking propensity of jet fuel, an experimental apparatus previously developed at UTIAS by Wong [57], was used to thermally stress fuel. Initially, the setup utilized a single stage heating mechanism to stress jet fuel. Fuel was thermally stressed inside a 91 cm (36 in) long test section with an inner diameter of 1.8 mm (0.069 in). The fuel was pumped at a constant volumetric flow rate through the test section. Here, the stainless steel 304 test section was heated within a tube furnace. A 0.5 μm filter was placed downstream of the test section to capture any dislodged particulates. The carbon deposition within the test section, due to thermal stressing of fuel, was analyzed by an infrared analyzer. Similar to the JFTOT, fuel was subjected to single stage heating in Wong’s design and that did not accurately represent fuel conditions on-board an aircraft. Hence, the experimental apparatus at UTIAS was redesigned by Liang [58]. As it was impractical to design a test rig that produces the various temperatures present in an aircraft fuel system, a simplified two stage heating mechanism was used to simulate
the complex environment. The new and current set-up by Liang provided more control over temperature profiles which allowed one to simulate a similar environment that fuel experiences while being used as a coolant and energy source.

The current coking test rig consists of two sections where the temperature is controlled independently: (i) a pre-heating section and (ii) a test section. The pre-heating section simulates the heat exchange between the fuel and surrounding aircraft components before entering the injector. The test section simulates fuel in a fuel nozzle being heated from the radiation of a combustion chamber. The time scales experienced in the pre-heating and test section were chosen to represent engine idle and descent conditions, since fuel undergoes the most thermal stress during those stages of flight [5]. Hence, the time scale to which fuel is subjected to heat in the pre-heating section and test section is on the order of one minute and 0.2 seconds, respectively [14]. Furthermore, time scales regarding the duration of aircraft engine operation also hold importance since they affect the accumulation of carbon deposits within an injector. This time scale is on the order of hours for aircrafts, however, taking into consideration continuous experiment monitoring and fuel capacity within a laboratory environment, five hours was deemed appropriate as the third time scale.

The coking test rig redesigned by Liang was used in this thesis to thermally stress Jet A-1 in accordance to the aforementioned three time scales. Pressure drop and carbon burn-off were used as the diagnostic techniques to assess carbon deposits accumulated during thermal stressing. An in-depth description of the coking test rig and carbon burn-off apparatus are given in Section 2.3.1 and 2.3.2, respectively.

2.3.1 Coking Test Rig

The coking test rig used to thermally stress Jet A-1 is depicted in Figure 2.3.
Fuel is continuously pumped, at a constant flow rate, from an 8 L stainless steel supply tank via an automated syringe pump (Teledyne Isco). The 1 m long fuel line, with a 0.25 in (6.35 mm) outer diameter and 0.125 in (3.18 mm) inner diameter, is submerged in a pre-heating oil bath. The fuel then passes through a 3.25 in (82.55 mm) long stainless steel test section with an outer diameter of 0.125 in (3.18 mm). The internal diameter of the test section is varied during this study and will be outlined in more detail later. A nozzle band heater contains an assembly of the test section encased in a brass block. Heat is transferred to the test section from the nozzle band heater via conduction through the brass block.

There are a total of six thermocouples within the apparatus, as depicted in Figure 2.3. Two are located immediately upstream and downstream of the test section to measure the inlet and outlet temperatures of the fuel, respectively. The remaining four are equally spaced axially along the test section, in contact with the test section’s outer wall. These serve to extract the axial temperature profile of the fuel during testing. To prevent heat
loss to the ambient environment, non-heated components are insulated with multiple layers of ceramic strip insulation and aluminum foil tape, as shown in Figure 2.4.

![Insulating layers around the non-heated segments of the test section.](image)

When the fuel flowing in the test section is heated to temperatures above 150°C, carbon deposits begin to form. These deposits either adhere to the test section’s inner wall or flow with the bulk fuel. To ensure that all deposits from the coking process are captured, a sintered filter is placed downstream of the test section prior to the cooling system to capture all particulates within the bulk flow. Further downstream, a regulator controls the back flow pressure of Jet A-1 before entering a stainless steel waste tank. A description of the main components and their operational characteristics is provided below.

**Fuel Pump**

To provide continuous flow within the coking apparatus, a syringe pump (Teledyne Isco, 500D) system is used. This dual system consists of two 500 mL pumps connected together by a D-series controller. The controller allows one pump to refill while fuel is delivered to the rig by the secondary pump. The transfer occurs by a set of pneumatic valves operating from a compressed air source of 80 psig (552 kPa). Automatic switch-over from one pump to another is smooth, which ensures that pressure fluctuations are minimized. Two operating conditions are available in the syringe pump, namely, (i) con-
stant pressure mode and (ii) constant flow rate mode. In this study, the pump system operated in constant flow rate mode since pressure can be controlled via a back pressure regulator (Swagelok, KPБ1G0A4A5A20000).

**Fuel Pre-heater**

To address the multiple sources of heat on an aircraft seen from the hydraulics, gear boxes, and pumps, fuel is used as a coolant due to its high heat capacity [14, p.8]. However, usage of fuel as coolant increases its temperature before entering the injectors. To simulate the previously mentioned heat exchange, a pre-heater is used to raise the temperature of fuel prior to entering the test section.

An oil-bath (Memmert model ONE), displayed in Figure 2.5, is used as a pre-heater in the coking test rig. The oil is maintained at a uniform temperature via resistance heaters. With a resolution of 0.1°C and a maximum temperature of 200°C, this oil-bath allows inlet temperatures of the test section to be varied with precision.

![Figure 2.5: Memmert model ONE oil-bath heater used as a heat exchanger for jet fuel during experiments [58].](image)
Test Section and Heater

The test section simulates fuel conditions through a spray nozzle facing a combustion chamber, hence its wetted-wall temperature (temperature jet fuel is exposed to when in contact with a test section inner wall surface) is set higher than the oil-bath. Schematic of a test section is displayed in Figure 2.6.

![Swagelok Ferrules](image)

Figure 2.6: Test section simulating fuel injector nozzles with ferrules attached to the inlet and the outlet.

The test section assembly consists of a test section encased in solid brass blocks with an outer diameter of 1.5 in (3.81 cm) and a length of 2.0 in (5.08 cm), as shown in Figure 2.7. The two halves of the brass block are held together by a 300 W electric band heater, which is regulated by a temperature controller (Omega, CN7523). Four stainless steel fittings are attached to the brass block, by silver soldering, for thermocouple access to the outside wall of the test section in order to measure the corresponding surface temperature.
Figure 2.7: Brass heater block encasing the test section with thermocouples inserted. Four holes of 0.125 in (3.18 mm) diameter were drilled through the top half of the block and the steel thermocouple fittings are soldered on the brass block [58].

**Filter and Fuel Cooling System**

In order to capture all the solid particulates that pass through the test section, a 0.5 µm porosity sintered stainless steel filter (Swagelok, SS-4TF-0.5) is installed downstream of the test section. Post-test analysis of filters containing insolubles allow measurement of the amount of carbon precipitates which formed during the experiment but did not agglomerate to a test section surface. This ultimately provides a better understanding of the total amount of coke produced during a test. Furthermore, the filter ensures all insoluble particles flowing within the bulk fuel are captured to reduce contamination of the cooling system.

After the filtering stage, the heated fuel passes through coiled tubing with an outer
diameter of 0.125 in (3.18 mm) encased in a 1.5 in (38.1 mm) outer diameter tubing. Cold water, acting as a coolant, is fed around the encased coiled tube in the direction of fuel flow by a recirculating chiller (Lytron Kodiak, RC022J03BE2).

**Temperature and Pressure Drop Measurements**

Six Type-K thermocouples with an accuracy of $\pm 2.2^\circ\text{C}$ are used to monitor the temperature of the test section, as displayed in Figure 2.8. Two thermocouples are placed 5.2 cm upstream and downstream of the test section for measurement of fuel inlet ($T_{in}$) and outlet ($T_{out}$) temperature, respectively. Four thermocouples are spaced 1.3 cm apart along the test section, labeled $T_{ts,1}$ to $T_{ts,4}$, to measure outer wall temperature of the test section. The dual-element thermocouple, $T_{ts,1}$, also provides controller feedback to the electric band heater for setting test section temperature. A computerized data acquisition system (Omega, OM-CP-OCTTEMP2000) monitors and collects the temperature data in intervals of 30 seconds.

![Diagram of thermocouple placement](image)

**Figure 2.8**: Placement of the six thermocouples that measure outside wall temperature of the inlet, outlet, and along the test section [58].

To measure pressure drop across the test section, two static pressure taps are installed at the inlet and outlet thermocouple locations, as displayed in Figure 2.9. Pressure drop is measured by a differential pressure transducer with a range of 1 psi (6.89 kPa) and
an accuracy of ±0.08% full scale (Omega, MMDWU001V5P3A0T1A1). A multifunction data acquisition device (National Instruments, USB-6210) collects the pressure drop data. By using MATLAB as an interface, pressure drop measurements are recorded in intervals of 30 seconds.

As previously mentioned in Section 2.2.2, the Hagen-Pouiseuille relation can be used to measure the average thickness of carbon deposits within a test section. Therefore, pressure drop data are collected to monitor and measure carbon deposits formed within the test section. However, since Equation (2.3) assumes carbon deposit thickness is axially and radially uniform throughout the test section, which may not be correct [63], carbon burn-off analysis is used as a second diagnostic.

![Diagram of pressure tap locations](image)

Figure 2.9: Placement of the pressure taps just upstream and downstream of the test section to measure the pressure drop across the test section [58].

### 2.3.2 Carbon Burn-off Apparatus

Carbon burn-off is the second diagnostic technique used (first being pressure drop measurements) to determine the amount of carbon precipitate during an experiment. The apparatus utilized is a surface carbon determinator (ELTRA, SurfaceC-800) with a mea-
suring range of 1 to 1000 µg of carbon per cm² and an accuracy of ±1% of nominal value. A carbon burn-off measurement is started by entering the surface area of the specimen into the calculating software of the carbon burn-off apparatus. Afterwards, the specimen is placed into the carbon burn-off resistance-heated furnace for analysis. Once analysis is initiated, an oxygen stream, at a temperature of 700°C, oxidizes carbon deposits adhered to the specimen’s surface. The combustion products are led through a copper oxide catalyst and infrared cells are used to detect the concentration of CO₂. Results are then displayed on an adjacent computer in units of mgC/dm² (milligrams of carbon per unit area). Carbon burn-off is capable of higher precision and accuracy in comparison to pressure drop measurements. The profile of carbon deposition along a test section can also be determined by carbon burn-off which will be discussed in Section 3.4.1.
Chapter 3

Experimental Procedure

3.1 Experimental Conditions

Based on the literature review conducted (Section 1.3.4), studies by Spadaccini et al. determined that decreasing a test section’s inner diameter produced more carbon deposits [36]. The authors later suggest that the results may be a direct consequence of higher Reynolds number and shorter fuel residence time, rather than the test section’s inner diameter alone. However, other studies have shown contradicting findings. A study by Jones et al. determined that a lower Reynolds number and a longer fuel residence time produced higher amounts of deposition [62]. Furthermore, another study deemed it difficult to isolate the sole impact of Reynolds number on carbon deposits due to the relation of Reynolds number with temperature and residence time [35]. Hence, the goal of this study is to identify the exclusive impact of test section inner diameter on carbon deposition while controlling for the unestablished effects of Reynolds number and residence time.

After conducting experiments which controlled Reynolds number and fuel residence time, a trend in the profile of carbon deposits along the test section was observed (will be presented and discussed in Section 4). It was hypothesized that the test section
inlet temperature gradient (the difference between the average wetted-wall temperature within the test section and the inlet temperature of the fuel) was responsible for the trend. Therefore, the effect of test section inlet temperature gradient on carbon deposition was also studied in this thesis.

Lastly, multiple studies observed an effect of fuel passageway material on coking propensity of jet fuel [46–50]. Therefore, this thesis studies the effect of test section material on carbon deposition in order to determine the optimal material to use for diminishing deposits within fuel injectors.

Three sets of experimental conditions are studied in this thesis based on the discussion above. Namely, the relation between carbon deposition in Jet A-1 and (i) test section inner diameter, (ii) test section material, and (iii) test section inlet temperature gradient. Each experimental set is outlined briefly below with more detail and justification following in Section 3.2 and a summary of the test conditions in Table 3.1, 3.2, and 3.3.

**Experimental Set 1: Constant Reynolds Number**

The first experimental set assessed coking propensity of three test section inner diameters and two test section materials while maintaining a constant Reynolds number of 1500. Based on the inner diameter of the test section being studied, the fuel flow rate was adjusted accordingly to ensure a laminar flow with constant Reynolds number within the test section, across all test conditions. The acronym defining an experiment with a fixed Reynolds number utilizing a stainless steel 304 test section of inner diameter 0.027 in is CRN304-27 as shown in Table 3.1.

**Experimental Set 2: Constant Residence Time**

The second experimental set assessed coking propensity of three test section inner diameters and two test section materials while maintaining a constant residence time of 0.2 seconds within the test section. This value was chosen to represent similar residence
times seen within aircraft fuel injectors. Similar to the previous experimental set, the fuel flow rate was varied accordingly to produce a constant residence time across all test conditions, based on the selected test section inner diameter. The set flow rate and residence time also ensured the fuel within the test section remained in the laminar flow regime. The acronym defining an experiment with a fixed residence time utilizing a stainless steel 304 test section of inner diameter 0.027 in is CRT304-27 as shown in Table 3.2.

**Experimental Set 3: Temperature Gradient**

This experimental set consisted of test conditions with various inlet temperature gradients at a constant Reynolds number and residence time of 774 and 0.18 seconds within the test section, respectively. By assessing the effect of various temperature gradients, while keeping all other parameters fixed, it can be determined whether inlet and outlet temperatures play an important role in carbon deposition within a test section. The acronym defining an experiment with a temperature gradient of 215°C between the inlet and outlet is TG215 as shown in Table 3.3.
Table 3.1: Test conditions for experiments with a constant Reynolds number. The average wetted-wall temperature was 340°C for all experiments.

<table>
<thead>
<tr>
<th>Experiment Code</th>
<th>Inner Diameter cm (in)</th>
<th>Flow Rate mL/min</th>
<th>Residence Time s</th>
<th>Material</th>
<th>Reynolds Number</th>
</tr>
</thead>
<tbody>
<tr>
<td>CRN304-27</td>
<td>0.069 (0.027)</td>
<td>19.39</td>
<td>0.096</td>
<td>Stainless Steel 304</td>
<td></td>
</tr>
<tr>
<td>CRN304-55</td>
<td>0.140 (0.055)</td>
<td>39.50</td>
<td>0.19</td>
<td></td>
<td></td>
</tr>
<tr>
<td>CRN304-69</td>
<td>0.178 (0.069)</td>
<td>49.55</td>
<td>0.25</td>
<td>Stainless Steel 304</td>
<td>1500</td>
</tr>
<tr>
<td>CRN316-27</td>
<td>0.069 (0.027)</td>
<td>19.39</td>
<td>0.096</td>
<td></td>
<td></td>
</tr>
<tr>
<td>CRN316-55</td>
<td>0.140 (0.055)</td>
<td>39.50</td>
<td>0.19</td>
<td>Stainless Steel 316</td>
<td></td>
</tr>
<tr>
<td>CRN316-69</td>
<td>0.178 (0.069)</td>
<td>49.55</td>
<td>0.25</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table 3.2: Test conditions for experiments with a constant residence time. The average wetted-wall temperature was 340°C for all experiments.

<table>
<thead>
<tr>
<th>Experiment Code</th>
<th>Inner Diameter cm (in)</th>
<th>Flow Rate mL/min</th>
<th>Reynolds Number</th>
<th>Material</th>
<th>Residence Time s</th>
</tr>
</thead>
<tbody>
<tr>
<td>CRT304-27</td>
<td>0.069 (0.027)</td>
<td>9.149</td>
<td>707.7</td>
<td>Stainless Steel 304</td>
<td></td>
</tr>
<tr>
<td>CRT304-55</td>
<td>0.140 (0.055)</td>
<td>37.96</td>
<td>1442</td>
<td></td>
<td>0.2</td>
</tr>
<tr>
<td>CRT304-69</td>
<td>0.178 (0.069)</td>
<td>59.74</td>
<td>1808</td>
<td></td>
<td></td>
</tr>
<tr>
<td>CRT316-27</td>
<td>0.069 (0.027)</td>
<td>9.149</td>
<td>707.7</td>
<td></td>
<td></td>
</tr>
<tr>
<td>CRT316-55</td>
<td>0.140 (0.055)</td>
<td>37.96</td>
<td>1442</td>
<td>Stainless Steel 316</td>
<td>0.2</td>
</tr>
<tr>
<td>CRT316-69</td>
<td>0.178 (0.069)</td>
<td>59.74</td>
<td>1808</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Table 3.3: Test conditions for experiments with various temperature gradients with fixed test section inner diameter, fuel flow rate, and test section material. Throughout all test conditions, Reynolds number and residence time are fixed at 774 and 0.18 seconds, respectively.

<table>
<thead>
<tr>
<th>Experimental Code</th>
<th>Inlet Temperature °C</th>
<th>Average Wetted Wall Temperature °C</th>
<th>Temperature Gradient °C</th>
<th>Inner Diameter cm (in)</th>
<th>Flow Rate mL/min</th>
<th>Test Section Material</th>
</tr>
</thead>
<tbody>
<tr>
<td>TG175</td>
<td>165</td>
<td>340</td>
<td>175</td>
<td>0.069</td>
<td>10.0</td>
<td>Stainless Steel 304</td>
</tr>
<tr>
<td>TG195</td>
<td>145</td>
<td>340</td>
<td>195</td>
<td>(0.027)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>TG215</td>
<td>125</td>
<td>340</td>
<td>215</td>
<td></td>
<td></td>
<td>Steel 304</td>
</tr>
</tbody>
</table>
3.2 Preparation for Pressure Drop Experiments

To ensure a constant Reynolds number and residence time was achieved across the corresponding experiment sets, a variety of experimental operating parameters and components were calculated and fabricated, respectively, prior to thermal stressing the fuel. The following sections detail the procedure of each.

3.2.1 Fuel Viscosity

It is important to determine kinematic viscosity of jet fuel as it is a function of temperature. As the fuel’s temperature is increased, viscosity of the fuel decreases. A lower viscosity will allow Reynolds number to increase, which may significantly affect carbon deposition due to enhanced diffusion. This is especially important in the autoxidation regime, where insoluble particles are deposited onto a hot surface via diffusion [37]. Unlike gases where viscosity can be estimated from theoretical formulations, liquid viscosity data are largely empirical [69]. Therefore, empirical data from handbooks and a semi-empirical formula were applied to determine the viscosity of Jet A-1.

Both Varagaftik et al. and the CRC handbook provide dynamic viscosity data for Jet A-1 at different temperatures [70,71]. In addition to this data, Lewis-Squires relation is used as the semi-empirical formula [69],

\[
\mu^{-0.2661} = \mu_{T_1}^{-0.2661} + \frac{T - T_1}{233} \tag{3.1}
\]

where \(\mu\) is an unknown dynamic viscosity at temperature \(T\), and \(\mu_{T_1}\) is a known dynamic viscosity of a liquid at temperature, \(T_1\). Applying Equation (3.1) and the maximum specified viscosity limit of Jet A-1 in Canada (8.0 mm²/s at -20°C) [72], an approximate viscosity-temperature curve can be plotted. Resulting viscosity predictions from these three approaches were provided by Liang [58] and are displayed in Figure 3.1. Based on the results in Figure 3.1, for test section temperatures exceeding 200 °C, a dynamic
viscosity of 0.20 mPa-s was used during this study.

Figure 3.1: Comparison between the dynamic viscosity of jet fuel, including handbook data from Vargaftik et al. (T-1 kerosene fuel) [70], the Coordinating Research Council (Jet A-1) [71], and a calculated curve using the Lewis-Squires approximation for liquid viscosity (Eq. 3.1) [69]. Figure credits to Liang [58].

3.2.2 Fuel Flow Regimes

All the experiments in this thesis are conducted in a laminar flow regime. A flow regime is considered laminar when Reynolds number in a pipe is approximately less than 2000 [73]. A Reynolds number between 2000 and 4000 refers to the transition regime while the turbulent regime resides above 4000. With the fuel viscosity determined based on the operating temperature of the test section, the Reynolds number can be evaluated by the following equation,

\[ Re = \frac{4 \dot{Q}}{\pi d \nu} \]  

(3.2)

where \( \dot{Q} \) is volumetric flow rate of fuel, \( d \) is pipe diameter, and \( \nu \) is kinematic viscosity. Furthermore, the residence time within a test section, of length \( l \), can be evaluated by,
\[ t_r = \frac{\pi d^2 l}{4Q} \]  

(3.3)

Both Equations (3.2) and (3.3) can be easily rearranged to determine the volumetric flow rate for a given Reynolds number and residence time, respectively. The flow rate for Experimental Set 3 was selected as 10 mL/min for two reasons: (i) to keep the flow laminar within the selected pipe diameter, and (ii) to have a residence time on the same scale as the experiments in Experimental Set 1 and Experimental Set 2.

### 3.2.3 Test Section Temperature

To ensure the wetted-wall temperature for all three experiment sets remained constant at 340°C, both the oil-bath and test section temperatures were changed for each experiment. This was done to compensate for the thermal offset caused by the varying flow rates among the test conditions. However, prior to selecting a wetted-wall temperature, three parameters were considered: 1) oil-bath temperature, 2) fuel’s boiling point, and 3) electric band heater.

**Oil-Bath Temperature**

The oil-bath pre-heats jet fuel to an assigned temperature. However, as fuel travels away from the oil-bath towards the test section, heat is transferred to the ambient environment despite the quality of insulation applied. Benchmark tests were conducted at various oil-bath settings to determine the corresponding inlet temperature. The results are displayed in Figure 3.2.
As shown in Figure 3.2, the test section inlet temperature is approximately 20-30°C cooler than the oil-bath temperature. Due to linearity of the relation, a simple equation can be produced to determine the test section inlet temperature from the given oil-bath temperature. However, during thermal stressing, conduction of heat across the test section caused a rise in inlet temperature. Therefore, the oil-bath temperature was periodically adjusted to maintain a constant inlet temperature.

**Fuel’s Boiling Point**

Jet fuel should be thermally stressed at temperatures higher than 150°C [14] for degradation and carbon deposition to occur. However, using temperatures too high can cause the fuel to boil. As a consequence, pressure drop data may be inaccurate due to high pressure fluctuations. To eliminate this problem, the testing apparatus was pressurized
to 100 psig (689 kPa). Young determined 346°C to be the maximum temperature at 100 psig which the fuel could be heated to before the fuel started to boil [59]. Therefore, adding a factor of safety, 340°C was selected as the wetted-wall temperature within the test section to simulate conditions inside a fuel injector.

**Electric Band Heater**

The electric band heater was limited to setting the test section’s outer wall temperature due to position of the feedback thermocouple. However, it is important to note that the test section’s outer wall temperature is higher than the test section’s wetted-wall temperature. Therefore, a MATLAB code developed by Liu was used to relate the test section’s outer wall temperature to the wetted-wall temperature [74]. This code takes into consideration the test section’s inner diameter, flow rate, and inlet temperature. Insertion of these parameters into the code outputs the test section’s axial distribution of wetted-wall temperature.

### 3.2.4 Test Section and Filter

Commercially available seamless stainless steel 304 and 316 pipes were procured for in-house fabrication of test sections. Various wall thicknesses were selected to attain a range of inner diameters of 0.027 in (0.69 mm), 0.055 in (1.40 mm), and 0.069 in (1.75 mm) while maintaining a constant outer diameter of 0.125 in. A test section was fabricated by cutting the pipes into lengths of 3.25 in (82.26 mm) with a hacksaw blade containing 24 teeth per inch. Thereafter, the test sections were filed down to eliminate all rough edges that were created from the cutting process. The inlet and outlet of the test section were deburred using a cobalt steel countersink, single flute, 90° angle with a 0.125 in (3.175 mm) body diameter.

Each test section was fitted with a stainless steel ferrule (Swagelok, SS-200-SET) at the inlet and outlet. Prior to attaching ferrules, the test section was purged with
compressed air to ensure no metal filings remained inside. Ferrules, along with the respective fittings, were installed to accommodate the test section in the coking test rig. Thereafter, gage pins with an accuracy of \( \pm 0.0005 \) in \( (\pm 0.0127 \text{ mm}) \) were inserted into the inlet and outlet of the test section. This ensured the inner diameter was uniform throughout the length of the test section.

The test section was placed within the two halves of a brass block and secured by an electric band heater wrapped around the brass block. An average wetted-wall temperature of \( 340^\circ \text{C} \) was achieved inside the test section via conduction through the heated brass block assembly. Four thermocouples were installed into the top half of the brass block to monitor the temperature of the test section’s outer wall.

After the test section assembly was completed, it was placed into the coking test rig. Insulation consisting of multiple layers of insulating tape and aluminum foil tape was wrapped around the exposed inlet and outlet of the test section to minimize heat loss to the ambient environment.

The filter was locked into a T-type housing by applying \( 62.5 \text{ N} \cdot \text{m} \) of torque with a 1 in torque wrench. Thereon, the assembled filter was installed downstream of the test section, as shown in Figure 2.3. Once all the components were incorporated into the test rig, experiments were ready to begin.

### 3.3 Pressure Drop Experiments

The pressure drop experiments consists of three phases in which Jet A-1 was thermally stressed and data were acquired:

1. **Pre-steady state phase**: to derive the initial average internal diameter of the test section with pressure drop measurements, while under thermal expansion.

2. **Steady state phase**: to monitor pressure drop in test section while fuel is being thermally stressed and coking occurs.
3. **Post-steady state phase**: to derive the final average internal diameter of the test section with pressure drop measurements, while under thermal expansion and after deposition has occurred.

### 3.3.1 Phase 1: Pre-Steady State Phase

The first phase commenced when the oil-bath began heating up to a set temperature. After two hours, when the oil-bath reached target temperature, fuel flow was initiated by assigning a flow rate to the syringe pump. Both the cooling system and temperature data acquisition were activated immediately after.

Once fuel began discharging into the waste tank and the syringe pump had completed equilibrating, the back pressure was slowly increased to 100 psig (689 kPa).

As the pre-heated fuel moved through the rig, the temperature of the test section increased which caused the test section to expand. The temperature decayed after an hour of initial fuel flow. However, the decay time and temperature were completely dependent on the ambient temperature, oil bath temperature, fuel flow rate, and quality of insulation applied.

The test section temperature was assumed as steady when temperature rise was less than 0.1°C per minute. It is important to note that no coking occurred in this phase since the temperature always decayed to less than 80°C. At this point, pressure drop data was recorded every 30 seconds for a 15 minute period.

### 3.3.2 Phase 2: Steady State Phase

After completion of Phase 1, the electric band heater was set at a desired temperature to heat the test section. Once the target temperature was reached, pressure drop monitoring was initiated for a duration of five hours. During this phase thermal stressing of jet fuel occurred. As the fuel was stressed, carbon deposits agglomerated inside the test section. Over time, the syringe pump detected a gradual increase in pressure, although the back pressure regulator was maintained at a constant pressure. This increase in pressure
was due to solid carbon adhering on the inside wall of the test section and narrowing the fuel’s passage. To compensate for this affect, the back pressure regulator was periodically adjusted to maintain 100 psig (689 kPa) throughout the test rig.

The temperature data recorded in this phase were purely for monitoring purposes. As discussed in Section 3.2.1, if temperature is not uniform, it will have an effect on the viscosity of the fuel and thus the pressure drop. As shown in Figure 3.3, the temperature fluctuates throughout the five hour run. These fluctuations may be a result of errors within various components such as the oil-bath, test section heater, and thermocouples during the five hour experimental run. However, since the fluctuations are no greater than 0.5°C, it is assumed they have no significant effect on carbon deposition.

Figure 3.3: Temperatures recorded for an experimental run of five hours by the four thermocouples placed within the test section assembly.
After the completion of the five hour data acquisition, the oil-bath heater and electric band heater were both switched off. Back pressure was reduced to 0 psig prior to stopping the syringe pump and cooling system. The test rig was then left overnight to cool down for Phase 3, occurring the next day.

3.3.3 Phase 3: Post-Steady State Phase

The last phase of the full experimental run was a repetition of Phase 1. However, rather than allowing the temperature to decay to a steady point, pressure drop was monitored for 15 minutes at the steady temperature of Phase 1. Before and after Phase 2 (where thermal stressing of fuel occurs), pressure drop measurements during the 15 minute period were recorded at the same steady temperature for two reasons: (i) to ensure same thermal expansion of test section during pressure drop measurements; (ii) to ensure same fuel viscosity during pressure measurements. This was crucial when calculating an average radial reduction within the test section by using Equation 2.3. After pressure monitoring was completed, the test rig was switched off following the same procedure as Phase 2.

3.4 Carbon Burn-off Procedure

Once the coking test rig had cooled down, the thermally stressed test section and filter were removed to be analyzed later in the carbon burn-off apparatus. However, prior to diagnostics, the test section and filter required preparation.

3.4.1 Test Section and Filter Preparation

A pipe cutter was used to cut the test section into multiple segments with minimal intrusion to the carbon deposits within. Performing carbon burn-off on segments of the test section, rather than on the whole, allowed determination of the axial distribution of carbon deposits.
Chapter 3. Experimental Procedure

The test sections used in Experimental Sets 1 and 2 were cut in a fashion depicted in Figure 3.4. The unequal lengths of segments were due to limitation of the tools available. Interference between the pipe cutter’s lining rollers and the test section’s ferrules inhibited the inlet and outlet to be cut into shorter segments. Also, a minimal length of 0.5 cm (0.2 in) was required for a vice grip to restrain the test section into position whilst being cut.

Figure 3.4: Lengths of test section segments (TSS) used for carbon burn-off in Experimental Sets 1 and 2.

Test sections utilized for the temperature gradient experiments (Experimental Set 3) were cut into sections of different lengths as shown in Figure 3.5. These lengths were chosen to better assess the deposition profile at the inlet and outlet due to temperature gradients present at those regions. A method to remove ferrules carefully from a test section was determined which utilized a vice grip and bench vice. This allowed the inlet and outlet to be cut into shorter segments.

Figure 3.5: Lengths of test section segments (TSS) used for carbon burn-off in Experimental Set 3.

After a test section was cut, the adjacent filter and test section segments (TSS) were gently rinsed with n-hexane ($\text{C}_6\text{H}_{14}$) to remove any remaining liquid fuel while preserving solid carbon deposits. The TSS and filter were then dried for at least eight hours in a vacuum oven at a gage pressure of -90 kPa (-13 psi) and temperature of 120°C.
This procedure ensured all liquid inside the TSS and filter were vaporized and no liquid compounds influenced the detected carbon during analysis.

3.4.2 Carbon Burn-off Apparatus

Carbon burn-off analysis commenced by allowing infrared cells to attain a stable temperature. This required approximately one hour. Once the infrared cells were ready, the carbon burn-off apparatus was assigned a temperature of 700°C. This temperature ensured carbon deposits within a specimen (TSS or filter) were completely oxidized during diagnostics. Once the target temperature was obtained, oxygen of 99.5% purity was fed into the apparatus at a pressure of 50 psig (345 kPa). Along with oxygen, carbon dioxide of 99.99% purity was delivered at a pressure of 15 psig (100 kPa). Thereafter, the apparatus was calibrated while the mass of each specimens were measured and recorded by use of a scale with an accuracy of ± 0.0001 g.

After completion of calibration, a single specimen was placed onto a quartz boat and inserted into the apparatus for analysis. Once carbon deposition in the specimen was analyzed, results were displayed on an adjacent computer three to four minutes later. Afterwards, the specimen was removed from the apparatus and its final mass was recorded.
Chapter 4

Results and Discussion

There were two diagnostic techniques used to measure the coking propensity of Jet A-1 in order to validate the results obtained from each technique. The first technique was the pressure drop measurements as discussed in Section 2.2.2. A pressure transducer was set up to measure the pressure drop between the inlet and outlet of the test section. This data set provided the reduction in radial thickness of the test section, since an increasing pressure drop indicated a decreasing passage diameter due to carbon deposits. The second technique was the carbon burn-off apparatus as described in Section 2.2.1. This method measured the mass of carbon deposition from thermally stressing jet fuel within a specimen (TSS or filter) by oxidizing all carbon deposits on the surface of the specimen.

4.1 Experimental Set 1: Constant Reynolds Number

4.1.1 Pressure Drop Results

In this experimental set, the Reynolds number within the test section was kept constant for all cases. This ensured the exclusive impact of test section inner diameter on carbon deposition was measured. Furthermore, the effect of test section material on carbon
deposition was also assessed.

The experiments were carried out in three phases. Phase 1 was conducted to derive the initial average internal diameter of the test section. In Phase 2, the pressure drop across the test section was monitored while the fuel was thermally stressed and coking occurred. Lastly, Phase 3 was conducted to derive the final average internal diameter of the test section after deposition had occurred.

Pressure drop measurements were taken every 30 seconds in Phase 1, 2 and 3 of the experiments. However, the data acquisition for Phase 2 was for control purposes and Phase 1 and 3 average pressure drop data were used in Equation 2.3 to calculate the total thickness of carbon deposits in a test section. With a constant average wetted-wall temperature (WWT) of 340°C inlet temperature of 162°C, the fuel was thermally stressed for five hours and produced the results in Table 4.1.

On a first glance at Table 4.1, negative values are listed which indicate the test section’s inner diameter increased. This is not a valid result and is most likely due to random errors. Also, carbon deposits on the filter will make it more difficult for jet fuel to pass through the filter. Hence, there will be higher pressure before the filter. This high pressure region can propagate towards the outlet of the test section and influence the pressure transducer to register a lower pressure drop across the test section. Consequently, a lower final pressure drop can result in a negative value. Another cause of the negative values may be the metal reacting with the fuel and provoking material to flush away from the inner surface of the test section. Presence of microbial growth, organic acids, or mercaptans within the fuel can instigate corrosion of the test section’s inner surface [1]. The mass of carbon deposits collected in the test section, from thermally stressing Jet A-1, may also have detached from the inner wall surface after Phase 2, when the rig was left to cool overnight prior to Phase 3. If this is the case, the precipitates will be captured by the filter.
Table 4.1: Thickness of carbon deposits accumulated inside the test section during Experimental Set 1. Data are taken from the average pressure drop across the test section in Phase 1 and 3 of experiments with a constant average WWT of 340°C and inlet temperature of 162°C. The flow’s Reynolds number is kept constant at 1500 in all cases. In the experimental code, 304 and 316 refers to stainless steel 304 and 306, respectively.

<table>
<thead>
<tr>
<th>Experimental Code</th>
<th>Inner Diameter cm (in)</th>
<th>Flow Rate mL/min</th>
<th>Residence Time s</th>
<th>Carbon Deposit Thickness µm</th>
</tr>
</thead>
<tbody>
<tr>
<td>CRN304-27</td>
<td>0.069 (0.027)</td>
<td>19.39</td>
<td>0.096</td>
<td>1.32 ± 0.07</td>
</tr>
<tr>
<td>CRN304-55</td>
<td>0.140 (0.055)</td>
<td>39.50</td>
<td>0.19</td>
<td>0.27 ± 0.01</td>
</tr>
<tr>
<td>CRN304-69</td>
<td>0.178 (0.069)</td>
<td>49.55</td>
<td>0.25</td>
<td>2.32 ± 0.12</td>
</tr>
<tr>
<td>CRN316-27</td>
<td>0.069 (0.027)</td>
<td>19.39</td>
<td>0.096</td>
<td>-1.33 ± 0.07</td>
</tr>
<tr>
<td>CRN316-55</td>
<td>0.140 (0.055)</td>
<td>39.50</td>
<td>0.19</td>
<td>15.58 ± 0.78</td>
</tr>
<tr>
<td>CRN316-69</td>
<td>0.178 (0.069)</td>
<td>49.55</td>
<td>0.25</td>
<td>-1.07 ± 0.05</td>
</tr>
</tbody>
</table>

Due to the negative values in SS 316, a general trend cannot be specified. For experiments with SS 304, test cases CRN304-69, CRN304-27, and CRN304-55 yielded the most to least carbon deposits. Furthermore, the pressure drop readings from Phase 2 display a similar trend for both materials, as shown in Figure 4.1.
Figure 4.1: Pressure drop data across the test section during five hour thermal stressing in Experimental Set 1. Test sections are made of (a) stainless steel 304 and (b) stainless steel 316.
For both SS 304 and SS 316, it is shown in Figure 4.1 that as the inner diameter of the test section decreases the pressure drop increases with time. This is especially the case for test sections with an inner diameter of 0.027 in (smallest diameter) since there is rise of 0.13 kPa and 0.17 kPa for SS 304 and SS 316, respectively. The larger inner diameters of 0.055 in and 0.069 in are nearly horizontal, indicating no layer of carbon deposit adhered to the inner surface. However, at the very beginning there is a slight increase in pressure drop. A sharp increase in deposition, for CRN304-27 and CRN316-27 within 40 minutes of the five hour run, may be the result of a carbon deposit layer forming on the inner surface wall when the fuel is exposed to the high WWT of 340°C. This layer can act as an insulation for the rest of the bulk fuel passing through and discouraging deposition. Therefore, once the layer is formed the rate of deposition slows down and the increase in pressure drop is shallower.

There are also pressure fluctuations throughout the five hour run in Figure 4.1. These are a consequence of the syringe pump’s pneumatic action when it refills and/or switches pumps to maintain a continuous flow operation mode. However, CRN316-27 has a 0.2 kPa pressure fluctuation which is not seen in any of the other cases. The pressure drop data following the fluctuation decreases by almost 0.1 kPa. This may indicate a large agglomerate detaching from the inside wall and passing through the test section.

Furthermore, comparing the results for the two materials (SS 304 and SS 316), it is not evident that one material is more susceptible to coking. Although CRN304-27 has an average pressure drop 31% lower than CRN316-27, the pressure drop over time is similar. This may indicate that CRN316-27 had a rougher inner surface.

4.1.2 Carbon Burn-off Results

During Phase 2, jet fuel was thermally stressed within a test section allowing carbon insolubles to attach to the inner surface of the test section or flush out and gather on the filter. After completing all three phases of an experiment, the test sections were cut,
cleaned and dried in preparation for carbon burn-off. The results of carbon-burn-off are shown in Figure 4.2.

Figure 4.2: Mass of carbon deposits in test sections retrieved from carbon burn-off apparatus after Experimental Set 1 thermal stressing. Error bars are removed for clarity. Each point presents the average carbon deposits in a single TSS. Test sections are made of (a) stainless steel 304 and (b) stainless steel 316.
The common trend that is most visible in the two graphs is the large amount of deposits at the inlet and outlet compared to the middle TSS, creating a U-shaped curve. However, keeping in mind Figure 3.4 and how the test section was cut, the middle segments were 20% shorter than the inlet and outlet. The longer segments provided more surface area for carbon deposits to agglomerate. Since internal surface area scales linearly with length, the mass of carbon deposits can be reduced by 20% at the inlet and outlet (first and last data point of each experiment) as an approximate way of correcting for differences in lengths. If the deposits at the inlet and outlet points are reduced by 20%, a U-shaped trend is still the most common among the data. The greater amount of carbon deposits in the inlet and outlet may be due to the temperature gradient the fuel is exposed to when it enters and exits the heated test section. This hypothesis will be explored more in detail in Section 4.3. Although the smallest inner diameter also yields the largest mass of deposits at the inlet and outlet, the U-shaped trend is not visible like it is in diameters of 0.055 in and 0.069 in. Instead the carbon deposits are greater in the middle segments of the smallest diameter as well. This supports the works of Spadaccini et al. who concluded a small flow passage shifts carbon deposition further downstream in a test section [28].

Another trend, especially visible in SS 304, is the mass of carbon deposits in CRN304-27 is greater than CRN304-55 and CRN304-69. This indicates that a smaller inner diameter is more susceptible to carbon deposit build up. Referring back to Figure 4.1, this result corroborates with the pressure drop measurements. However, this does not seem to be the trend when comparing inner diameters of 0.055 in and 0.069 in. In both cases, SS 304 and SS 316, an inner diameter of 0.055 in produces less carbon deposits than 0.069 in. Nonetheless, this result agrees with the carbon deposit thickness calculated for SS 304 presented in Table 4.1.

According to ASTM standards [75], the maximum allowable concentration of carbon in SS 304 and SS 316 is 0.07% and 0.08%, respectively. Therefore, it was important to
create a calibration base line with blank test sections. This determined the additional measured carbon resulting from carbon in the steel diffusing to the surface and oxidizing within the carbon burn-off apparatus. The mass of carbon detected in various test section lengths are shown in Figure 4.3.

![Figure 4.3: Mass of carbon detected via carbon burn-off apparatus of blank sections not undergone thermal stressing.](image_url)

It is evident from Figure 4.3 that as the length of a blank section increases, the amount of carbon detected also increases. The relationship is linear. The mass of carbon detected in a blank section of a given length was subtracted from the mass of carbon measured in a TSS of equal length which was thermally stressed. Therefore, some of the data points for inner diameters of 0.055 in and 0.069 in are below zero in Figure 4.2. These negative values indicate that more carbon was measured in a blank section than one that experienced thermal stressing. This may be the cause of a systematic error or it may be due to material of the test section’s inner wall being corroded by flow of Jet
A-1 during Phase 2. If the material is being corroded then the negative values on Table 4.1 are also supported.

Assessing Figure 4.4, the mass of carbon deposits are least to greatest in order of CRN304-55, CRN304-69, CRN316-55, CRN316-27, CRN304-27, and CRN316-69. This graph validates the results in Figure 4.2 by showing a greater mass of carbon deposits in test sections with an inner diameter of 0.069 in compared to 0.055 in. The trend of the smallest inner diameter producing the largest mass of carbon deposits is also present but only for CRN304-27. However, referring to the pressure drop results for CRN316-27 as displayed in Figure 4.1b, there was a high pressure fluctuation that may have indicated an agglomerate detaching from the test section. This hypothesis also explains the lower total carbon deposition in CRN316-27 in Figure 4.4. On the graph, SS 304 test sections generally have a lower amount of deposition compared to SS 316 test sections. These results are supported by Altin and Eser’s study in which SS 316 generated more deposition than SS 304 [49].

![Figure 4.4: Total carbon in a test section undergone thermal stressing during Experimental Set 1. Results are obtained from carbon burn-off apparatus.](image-url)
4.1.3 Filter Results

The filter for each experiment was lightly rinsed with n-hexane and vacuum dried prior to analysis in the carbon burn-off apparatus. The total mass of carbon deposits captured by the filter are shown in Table 4.2.

Table 4.2: Total mass of carbon collected in the filter from Experimental Set 1. Data produced by carbon burn-off.

<table>
<thead>
<tr>
<th>Experimental Code</th>
<th>Total Mass of Carbon Deposits in the Filter</th>
</tr>
</thead>
<tbody>
<tr>
<td>CRN304-27</td>
<td>Saturated (&gt;1000)</td>
</tr>
<tr>
<td>CRN304-55</td>
<td>447 ± 6.91</td>
</tr>
<tr>
<td>CRN304-69</td>
<td>336 ± 6.02</td>
</tr>
<tr>
<td>CRN316-27</td>
<td>Saturated (&gt;1000)</td>
</tr>
<tr>
<td>CRN316-55</td>
<td>450 ± 6.72</td>
</tr>
<tr>
<td>CRN316-69</td>
<td>373 ± 6.24</td>
</tr>
</tbody>
</table>

As the inner diameter of the test section increases, the mass of carbon deposits collected on its respective filter decreases. A list of possible explanations follow:

1. The mass of insoluble particles detaching from hot surfaces decrease with increasing test section diameter.

2. The mass of insoluble particles within the bulk fuel, not attaching to the hot surface and passing through the test section, decreases with increasing diameter.

3. A combination of the above two mechanisms occurs.

In Table 4.2 it is apparent that inner diameters of 0.027 in are saturated due to the large amounts of carbon deposited onto the filter. There is 25% decrease in filter deposition from CRN304-55 to CRN304-69. Whereas, from CRN316-55 to CRN316-69, the decrease is 17%. It is also shown in these results that experiments conducted with SS 316 test sections collected a larger mass of carbon deposits in their respective filters. The
increase of carbon deposits from SS 304 to SS 316 is 0.7% and 11% for inner diameters of 0.055 in and 0.069 in, respectively. This supports the similar trends that were seen with the carbon burn-off of the test section and pressure drop results.

### 4.2 Experimental Set 2: Constant Residence Time

#### 4.2.1 Pressure Drop Results

In this experimental set, the residence time was kept constant at 0.2 seconds while the Reynolds number was allowed to vary. The thickness of carbon deposits accumulated in the test section, after thermally stressing Jet A-1, was calculated using the pressure drop across the test section. The results are displayed in Table 4.3.

Table 4.3: Thickness of carbon deposits accumulated inside the test section during Experimental Set 2. Data are taken from the average pressure drop across the test section in Phase 1 and 3 of experiments with a constant average WWT of 340°C and inlet temperature of 162°C. The residence time is kept constant at 0.2 seconds in all cases. In the experimental code, 304 and 316 refers to stainless steel 304 and 306, respectively.

<table>
<thead>
<tr>
<th>Experimental Code</th>
<th>Inner Diameter (cm (in))</th>
<th>Flow Rate (mL/min)</th>
<th>Reynolds Number</th>
<th>Carbon Deposit Thickness (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CRT304-27</td>
<td>0.069 (0.027)</td>
<td>9.149</td>
<td>707.7</td>
<td>4.52 ± 0.23</td>
</tr>
<tr>
<td>CRT304-55</td>
<td>0.140 (0.055)</td>
<td>37.96</td>
<td>1442</td>
<td>0.42 ± 0.02</td>
</tr>
<tr>
<td>CRT304-69</td>
<td>0.178 (0.069)</td>
<td>59.74</td>
<td>1808</td>
<td>9.99 ± 0.50</td>
</tr>
<tr>
<td>CRT316-27</td>
<td>0.069 (0.027)</td>
<td>9.149</td>
<td>707.7</td>
<td>2.33 ± 0.12</td>
</tr>
<tr>
<td>CRT316-55</td>
<td>0.140 (0.055)</td>
<td>37.96</td>
<td>1442</td>
<td>1.51 ± 0.08</td>
</tr>
<tr>
<td>CRT316-69</td>
<td>0.178 (0.069)</td>
<td>59.74</td>
<td>1808</td>
<td>-1.35 ± 0.07</td>
</tr>
</tbody>
</table>

The same trend for SS 304 test sections in Experimental Set 1 (refer to Table 4.1) is also depicted in Table 4.3. However, the measurements from Experimental Set 2 are significantly larger than Experimental Set 1 results for experiments with SS 304. A greater thickness of agglomerated carbon in CRT304-69 compared to CRT304-27 may be
attributed to the difference in Reynolds number. A higher Reynolds number in CRT304-69 may abet to greater coking than a lower Reynolds number in CRT304-27 as supported by the works of Alborzi et al. [44]. Nonetheless, CRT316-27 yields a 54% thicker layer of deposition in comparison to CRT316-55 despite an inner diameter of 0.055 in experiencing a greater Reynolds number. Furthermore, other general trends are not visible in the data set therefore, the pressure drop readings of Phase 2 are assessed for further analysis in Figure 4.5.

As the inner diameter of a test section increases, the pressure drop across the test section decreases. This is especially the case for CRT304-27 and CRT316-27 since the rise in pressure drop, during the five hour run, has an increase of 0.07 kPa and 0.1 kPa, respectively. Whereas, similar to Experimental Set 1, the pressure drops for inner diameters of 0.055 in and 0.069 in are nearly horizontal. However, the pressure drop for inner diameters of 0.027 in is lower than the pressure drop obtained in Experimental Set 1 (Figure 4.1) where the pressure drop was 0.13 kPa and 0.15 kPa for SS 304 and SS 316, respectively. This may be attributed to the 53% decrease in Reynolds number from Experimental Set 1 to Experimental Set 2. Here, the claim is verified by a study performed by Spadaccini in which a higher Reynolds number yielded larger concentrations of carbon deposition despite the shorter residence time [36].
Chapter 4. Results and Discussion

Figure 4.5: Pressure drop data across the test section during five hour thermal stressing in Experimental Set 2. Test sections are made of (a) stainless steel 304 and (b) stainless steel 316.
The pressure drop for CRT316-27 pulsates throughout the five hour run as shown in Figure 4.5b. A repeated cycle of a sharp increase in pressure drop is followed by a steady decrease. This trend may be the cause of carbon depositing on the inner surface detaching in fragments until another cluster of insolubles from the bulk fuel attaches to the wall. If this is the case, the filter will capture the deposits washed through the test section. Furthermore, Figure 4.5 displays evidence of SS 316 test sections instigating coking far more than SS 304 when analyzing the inner diameter of 0.027 in.

4.2.2 Carbon Burn-off Results

The test sections used for thermally stressing Jet A-1 in Experimental Set 2 were cut and prepared in a similar fashion as Experimental Set 1 for carbon burn-off diagnostics. The analysis obtained via carbon burn-off is shown in Figure 4.6.

Assessing both Figure 4.6a and 4.6b, it is apparent that the smallest inner diameter (0.027 in) generated the most carbon deposition along the test section. The inner diameter with the second most carbon deposits is depicted as 0.069 in for experiments conducted in a SS 304 test section. However, in the experiments with SS 316 test sections, it is unclear whether deposition in an inner diameter of 0.069 in is greater than 0.055 in.
Figure 4.6: Mass of carbon deposits in test sections retrieved from carbon burn-off apparatus after Experimental Set 2 thermal stressing. Each point represents the average carbon deposits in a single segment of a test section. Test sections are made of (a) stainless steel 304 and (b) stainless steel 316.
Comparing these results with Experimental Set 1 (referring to Figure 4.2), there seems to be no defined U-shaped trend with the exception of CRT304-55 and CRT304-69 experiments. It is also evident that the maximum carbon deposition is pushed downstream in CRT304-27 and CRT316-27. There is a 200% increase in maximum carbon deposition in SS 304 from Experimental Set 1 to Experimental Set 2. Whereas, the maximum deposition for SS 316 is approximately the same in both experimental sets. The lack of clear trends, when maintaining a constant residence time in various diameters, can be attributed to Reynolds number having a greater effect on deposition along a test section than residence time as concluded by Spadaccini et al. [28]. When the Reynolds number was constant in Experimental Set 1, defined trends in the deposit accumulation along the test section were depicted. However, in Experimental Set 2 the Reynolds number varies from 707 to 1808 and a trend is not apparent.

It is also important to consider the effects of thermal and hydraulic entrance lengths here. In all the test cases (including Experimental Set 1), excluding CRT304-27 and CRT316-27 in Experimental Set 2 due to their lower Reynolds number of 707, the temperature profile and the hydraulic profile are not fully developed. The entrance length exceeds the total length of the test section. It is known that the convective heat transfer in the entrance region, where the thermal boundary layers are not yet established, can be far superior to heat transfer in the fully developed regions. This is due to the thin thermal boundary layers having low thermal resistance [76]. Also, if the velocity profile of the fluid entering a heated passage is not developed, the entrance effect will differ from that where the velocity profile is fully developed at the thermal entrance. Generally, the convective heat transfer coefficient is much larger than that of purely thermal entrance [77]. The thermal and hydraulic entrance lengths for both CRT316-27 and CRT304-27 are 2.95 cm (1.16 in) and 3.12 cm (1.23 in), respectively. As shown in Figure 4.6, the mass of carbon deposits decreases for the above cases after 3.5 cm (1.38 in.) from inlet of the test section. This decrease in carbon deposition may be characterized by the thermal
and hydraulic profile being fully developed at that position.

The total mass of carbon deposition in all test cases were calculated using the carbon burn-off data for the test sections. The results are displayed in Figure 4.7.

![Figure 4.7: Total carbon in a test section undergone thermal stressing during Experimental Set 2. Results are obtained from carbon burn-off apparatus.](image)

As shown in Figure 4.7, test cases which yield the most to least deposition are in order of CRT304-27, CRT316-27, CRT304-69, CRT316-69, CRT316-55, and CRT304-55. Similar to the results produced in Experimental Set 1, CRT304-27 produced the greatest quantity of deposition accumulating almost 530% more deposits in comparison to CRT304-55 (lowest amount of deposition). Test sections with inner diameters of 0.055 in once again cultivated least amount of carbon deposition, indicating an ideal diameter for inhibiting coking. Contrary to Experimental Set 1 results, test sections of SS 304 exhibit a greater sensitivity to deposition in comparison to SS 316. Overall, the maximum total deposition in Experimental Set 2 exceeds Experimental Set 1 by 228%. However, the difference in total carbon deposits across inner diameters for both materials is much larger in comparison to Experimental Set 1. The maximum difference
of total carbon in Experimental Set 1 between two test cases was 130 µg, whereas in Experimental Set 2 the maximum difference is 800 µg. This identifies a greater effect of Reynolds number across test cases than residence time for the same testing conditions. It is also important to note that in Experimental Set 1 the flow rate across experiments changed by approximately 10 mL/min to sustain a Reynolds number of 1500, as listed in Table 3.1. However, the flow rate in Experimental Set 2 varies by approximately 20 mL/min across test cases to maintain a residence time of 0.2 seconds as shown in Table 3.2. The increased change in flow rate may also provide an explanation for the larger difference in deposition when comparing inner diameters.

### 4.2.3 Filter Results

As a product of thermally stressing Jet A-1, deposits were attached to the inner surface of the test section’s wall. Over time, fragments of the deposits can detach from the wall. These fragments are captured by a 0.5 µm porosity filter placed downstream of the test section. Another product of thermally stressing jet fuel is the formation of agglomerates within the bulk fuel not attaching to any surface and flowing with the bulk fuel towards the filter. The total mass of carbon deposits detected on the filter, via carbon burn-off, is shown in Table 4.4.

<table>
<thead>
<tr>
<th>Experimental Code</th>
<th>Total Mass of Carbon Deposits in the Filter</th>
<th>µg</th>
</tr>
</thead>
<tbody>
<tr>
<td>CRT304-27</td>
<td>Saturated (&gt;1000)</td>
<td></td>
</tr>
<tr>
<td>CRT304-55</td>
<td></td>
<td>741 ± 8.94</td>
</tr>
<tr>
<td>CRT304-69</td>
<td></td>
<td>593 ± 7.76</td>
</tr>
<tr>
<td>CRT316-27</td>
<td>Saturated (&gt;1000)</td>
<td></td>
</tr>
<tr>
<td>CRT316-55</td>
<td></td>
<td>590 ± 7.74</td>
</tr>
<tr>
<td>CRT316-69</td>
<td></td>
<td>481 ± 6.94</td>
</tr>
</tbody>
</table>
The filters associated with CRT304-27 and CRT316-27 are saturated in this experimental set as well. The same trend shown in Experimental Set 1, increasing test section’s inner diameter increases the carbon deposits collected by the filter, is also present in Experimental Set 2. The decrease in carbon deposition from an inner diameter of 0.055 in to 0.069 in is 19% and 18% for materials SS 304 and SS 316, respectively. However, the difference in deposition accumulated on the filter for SS 304 and SS 316 exceeds those shown in Experimental Set 1. Agreeing with the results depicted in carbon burn-off of the test sections, SS 304 has 26% and 23% more carbon deposits than SS 316 for inner diameters of 0.055 in and 0.069 in, respectively. Overall, the experiments in Experimental Set 2 produced at least 29% greater accumulation of deposits on the filter in comparison to Experimental Set 1.

4.3 Experimental Set 3: Temperature Gradient

The purpose of this experimental set is to analyze the effect of the test section temperature gradient (the temperature transition from the inlet to the wetted-wall) on coking propensity. Stainless steel 304 test sections with an inner diameter of 0.069 cm (0.027 in) were utilized as the flow passage of Jet A-1. The wetted-wall temperature of the test section was maintained at 340°C while the inlet temperature was varied in increments of 20°C. The fuel flow of Jet A-1 was constant at 10 mL/min which generated a Reynolds number of 774 and residence time of 0.18 seconds.

4.3.1 Pressure Drop Results

Pressure drop readings were taken in increments of 30 seconds across the test section during all three phases of an experimental run. The average pressure drop during Phase 1 and Phase 3 were used in calculation for the thickness of carbon deposition inside a test section. The results are shown in Table 4.5.
Chapter 4. Results and Discussion

Table 4.5: Thickness of carbon deposits accumulated inside the test section during Experimental Set 3. Stainless steel 304 test sections are used for the three experiments. Throughout all test conditions, wetted-wall temperature, Reynolds number, residence time are fixed at 340°C, 774, and 0.18 seconds, respectively.

<table>
<thead>
<tr>
<th>Experimental Code</th>
<th>Inner Diameter (cm (in))</th>
<th>Flow Rate (mL/min)</th>
<th>Temperature Gradient (°C)</th>
<th>Carbon Deposit Thickness (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TG175</td>
<td>0.069 (0.027)</td>
<td>10</td>
<td>175</td>
<td>0.21 ± 0.01</td>
</tr>
<tr>
<td>TG195</td>
<td></td>
<td></td>
<td>195</td>
<td>1.45 ± 0.07</td>
</tr>
<tr>
<td>TG215</td>
<td></td>
<td></td>
<td>215</td>
<td>1.27 ± 0.06</td>
</tr>
</tbody>
</table>

As depicted in Table 4.5, there is a 590% increase in thickness of deposition from TG175 to TG195 and a 12% decrease from TG195 to TG215. Similar to most pressure drop calculations shown in the previous experimental sets, there is no specific trend between the operating parameters and thickness of carbon deposition. Therefore, it is important to consider the instantaneous pressure drop readings during five hour thermal stressing in Phase 2. Results for the three temperature gradients are shown in Figure 4.8.

![Figure 4.8: Pressure drop data across the test section during five hour thermal stressing in Experimental Set 3.](image-url)
In Figure 4.8, a more defined trend is visible between temperature gradients and carbon deposition. As the temperature gradient increases, the pressure drop across the test section decreases. This indicates that the test section produces a greater amount of surface deposits causing a higher pressure drop at lower temperature gradients. Also, the slope of pressure drop measurements (the test section constriction due to deposition as a function of time) decreases as the temperature gradient increases. This indicates that a larger temperature gradient only forms surface deposits within 30 minutes of thermal stressing whereas, a smaller temperature gradient will continue to form surface deposits for the duration the fuel is subjected to high heat. Although the temperature gradient is increased increments of 20°C, the pressure drop measurements are not linearly decreasing across the temperature gradients. Both TG175 and TG195 have a positive slope after the initial sharp increase and are close in measurements with an average pressure drop of 0.59 kPa and 0.58 kPa, respectively. However, TG215 data appears to have a slope of zero after the initial sharp increase and lies much lower at a pressure drop of 0.52 kPa. Furthermore, the three test conditions have similar fluctuations in the form of pulses in the data. These fluctuations may be result of the syringe pump or agglomerates detaching from the surface walls as mentioned in Section 4.2.1.

4.3.2 Carbon Burn-off Results

After the experimental run, the test sections were cut into segments as shown in Figure 3.5, cleaned and vacuum oven dried to evaporate any liquid fuel remaining inside the test section. Carbon burn-off analysis was performed on each test segment and produced the results depicted in Figure 4.9.
Displayed in Figure 4.9, as the temperature gradient increases, the mass of carbon deposits decrease. This supports the pressure drop data as both diagnostic techniques are producing the same results. Although the mass of carbon deposits in the inlet for the three test conditions are very similar and overlapping, this is not the case for the outlet or the middle segments. The general trend for the test cases is a greater accumulation of deposits in the middle of the test section. Recalling the lengths of each segment that the test section was cut into, the inlet and outlet were 71% shorter than the middle segments. Applying the same method as in Section 4.1.2, it can be determined whether the same trend is generated after correction of difference in TSS lengths. It is evident if the carbon deposits in the middle segments are reduced by 71% the trend remains. This trend is very similar to the previous trends in Experimental Set 1 and 2, where the smallest inner diameters (0.027 in) shifted maximum deposition downstream. Previously
in Experimental Set 2, an inner diameter of 0.027 in had a fully developed thermal and hydraulic profile. In this experimental set, all three test conditions have a fully developed thermal and hydraulic profile at 3.0 cm from the entrance of the test section. The mass of carbon deposits between adjacent segments, at 3.1 cm and 5.2 cm, increases by 50%, decreases by 1%, and decreases by 26% for TG175, TG195, and TG215, respectively. Therefore, as the temperature gradient increases, the mass of carbon deposition after the flow is fully developed decreases subsequently. This trend is carried through until the outlet where it is evident that a higher temperature gradient has lower deposition, whereas, the inlet data points overlapped.

In order to get a clear representation of the effect of increasing temperature gradients on the total mass of carbon accumulated inside a test section, the total carbon mass in each TSS were summed together. The results are shown in Figure 4.10.

Figure 4.10: Total carbon in a test section undergone thermal stressing during Experimental Set 3. Results are obtained from carbon burn-off apparatus.
Figure 4.10 displays a significant decrease in the mass of carbon as the temperature gradient is increased. This trend is explained by the works of Edwards [37]. He concluded in his study, as fuel is exposed to high temperature gradients, reactant depletion occurs which may inhibit carbon deposition. Furthermore, the relationship between carbon deposition and temperature gradient is not linear which was also shown in the pressure drop measurements. Although the temperature gradient is varied in increments of 20°C across the test cases, the decrease in total carbon deposits is 54% from TG175 to TG195 and 27% from TG195 to TG215. Recalling the temperature gradient of 178°C present in Experimental Set 1 and 2 (inlet was maintained at 162°C and wetted-wall temperature at 340°C), the claim of a temperature gradient abetting to large mass of carbon deposits at the inlet is verified. A similar temperature gradient in this experimental set, TG175, displayed the greatest amount of carbon deposition between all three testing conditions. Ultimately, it is evident that deposition can be decreased by subjecting Jet A-1 to higher temperature gradients.

### 4.4 Sources of Uncertainty

The accuracy of results were highly affected by the sources of uncertainty present throughout the experimental apparatus and procedure such as test section fabrication, pressure drop experiments, and carbon burn-off apparatus. They are discussed further in detail below.

#### 4.4.1 Test Section Fabrication

Stainless steel pipes were provided with an inner diameter uncertainty of $\pm 0.001$ in ($\pm 0.025$ mm). A standard ruler was utilized to measure and cut the required 82.55 mm (3.25 in) length test section. This added an uncertainty in the length of $\pm 0.5$ mm ($\pm 0.20$ in). After cutting and fitting the ferrules, the inner diameter of the test section was
measured once again with gage pins which had an uncertainty of ±0.0005 in (±0.0127 mm). However, to ensure the inner diameter remained constant within a test section, any test section in which the inlet and outlet differed by more than 0.0005 in (0.0127 mm) were rejected. Another method to ensure the uniformity of the inner diameter was to reject any test sections in which a gage pin could enter the inlet but not fall through the other end at the outlet.

4.4.2 Pressure Drop Experiments

The pressure drop experiments had multiple sources of error rooting from the pressure readings, temperature readings, and thermal expansion of the test section.

The pressure transducer had an accuracy of ±0.08% of its full range which equates to ±5.5 Pa (±0.8 × 10⁻⁴ psi). However, the larger source of error in the pressure measurements stems from the syringe pumps. When the pumps switched over to maintain continuous flow, the fluctuations in the pressure drop readings were as high as 10 Pa.

The temperature of the test section was measured by six thermocouples which had an accuracy of ±2.2°C. However, for the duration of a test, the temperature controllers for the oil-bath and the test section managed to keep temperature fluctuations within ±1°C. Another source of uncertainty comes from the quality of insulation applied to the inlet and outlet of the test section assembly. If there was heat loss changing the temperature by more than ±2.2°C, then the viscosity would be affected. A change in viscosity would affect the pressure drop readings taken during Phase 1 and Phase 3 for calculation of carbon deposit thickness in the test section.

When the test section was exerted to high temperatures, like most metals it expanded. The average thermal expansion coefficient for steel is 17.3 × 10⁻⁶ for temperatures in the range of 0°C to 315°C. By using the average thermal expansion coefficient, the increase in internal diameter due to heat is on the order of 0.5%. The brass blocks surrounding the test section mitigated any excessive increase in diameter by restraining the test section.
4.4.3 Carbon Burn-off Apparatus

The measurement error, as given by the manufacturer, is ±1%. However, as discussed in Section 4.1.2, the test section material itself has a concentration of carbon which adds to the carbon deposition detected. Multiple blank test sections of various lengths were analyzed in the carbon burn-off apparatus to determine the concentration of carbon. Although the results fell within a range of ±4.0 µg, none of the values were completely repeatable. Therefore, it is difficult to determine the random error produced by the carbon burn-off apparatus. It is also important to note that measuring the carbon added to the experimental results, from composition of the test sections, via blank sections may not be an accurate base line. The test sections used for thermally stressing fuel undergo high heats in a pressurized environment which may affect metal surface characteristics and either inhibit or support the detection of carbon during carbon burn-off. Furthermore, fuel passing through the test section may corrode the inner surface of the wall which is not comparable to a blank test section with its inner wall in original condition.
Chapter 5

Conclusions

An experimental apparatus was utilized in this thesis to study the coking propensity of Jet A-1. The apparatus simulated operating conditions found in aircraft injector fuel nozzles. The system was pressurized to 100 psig (689 kPa) and an average wetted-wall temperature of 340°C was maintained to thermally stress the fuel inside a test section. Parametric studies were performed to assess the effect of: (i) test section inner diameter, (ii) test section material, and (iii) test section inlet temperature gradient on deposition formed within the heated test section.

Two diagnostic techniques were used to analyze thermal stability of Jet A-1. The first technique used pressure drop measurements across a test section. By applying the Hagen-Poiseuille equation, the reduction in inner diameter of the test section, due to surface deposition on the hot walls, was calculated. The second technique utilized a carbon burn-off apparatus. Segments of the test section and the adjacent filter were placed into the 700°C furnace with a constant flow of oxygen to oxidize the solid carbon deposits within the specimen. This method evaluated the mass of carbon formed along the test section and within a filter with a 0.5 µm porosity downstream of the test section.
5.1 Experimental Sets

Experimental Set 1: Constant Reynolds Number

A total of six test conditions at a Reynolds number of 1500 were investigated in this experimental set. The first three test conditions were performed with a stainless steel 304 test section of inner diameters: 0.027 in, 0.055 in, and 0.069 in. The last three test conditions were performed with stainless steel 316 test sections with the same variation in inner diameter.

It was determined that as the inner diameter of a test section increased, surface deposition generally decreased when assessing the test section and filter deposits. There was a larger mass of carbon deposits at the inlet and outlet for inner diameters of 0.055 in and 0.069 in, which was attributed to a temperature gradient between the inlet and wetted-wall. However, for inner diameters of 0.027 in, the axial location of maximum surface deposition was pushed downstream in the test section. A test section with an inner diameter of 0.055 in produced the least amount of deposition among all cases. Also, stainless steel 304 test sections produced lower amounts of deposition compared to stainless steel 316.

Experimental Set 2: Constant Residence Time

The same parameters varied in Experimental Set 1 were also assessed in Experimental Set 2. However, the Reynolds number was allowed to vary while the residence time was maintained at 0.2 seconds within the test section.

It was concluded, similar to Experimental Set 1, as the inner diameter of a test section increased, the overall carbon deposition decreased. However, a specific trend of deposition along the length of the test section was not defined. This implied that changing Reynolds number had a greater effect on accumulation of deposits in comparison to changing residence time. An inner diameter of 0.055 in once again showed the least
amount of deposition. However, in this Experimental Set, stainless steel 304 test sections produced a greater amount of deposits compared to stainless steel 316.

**Experimental Set 3: Temperature Gradient**

Three test conditions were assessed in Experimental Set 3. While keeping test section inner diameter, test section material, Reynolds number, and residence time constant, the inlet temperature of the test section was varied. This produced temperature gradients between the inlet and wetted-wall of 175°C, 195°C, and 215°C.

It was concluded that as the temperature gradient increased, carbon deposition in the test section decreased. This was attributed to fuel being exposed to a high temperature gradient, which depleted necessary reactants required for carbon deposition formation, subsequently inhibiting coking. Furthermore, the relationship between coking propensity and temperature gradient appeared non-linear.

### 5.2 Recommendations for Future Work

The following are suggestions for expanding and improving a parametric study of jet fuel thermal stability:

1. Cut the test sections in equal lengths, prior to carbon burn-off. This will mitigate the effects of unequal lengths when studying deposition along the test section.

2. Add a pressure transducer to the coking test-rig to measure the pressure drop across the filter. This will allow assessment of the pressure between the test section outlet and filter.

3. Perform carbon burn-off on blank metal test sections subjected to various conditions. This will output an improved baseline by determining the effect of carbon concentration detected within a metal test section by heating and forcing pressurized flow within the test section.
4. Evaluate the effect on carbon deposition of various coatings and surface roughness within a heated fuel passage.

5. Include analysis of fuel composition before and after thermal stress tests to evaluate the chemical change in fuel due to thermal degradation.

6. Use non-evasive methods, such as laser scanning or micron-optic measurements, to assess the topology of surface carbon deposition within a test section.
Bibliography


