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Deposit Formation in Hydrocarbon Rocket Fuels

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Deposit Formation in Hydrocarbon Rocket Fuels

TABLE OF CONTENTS

	Page
SUMMARY	1
INTRODUCTION	2
TECHNICAL APPROACH	4
Fuels Characterization	4
Test Facility and Test Hardware Test Matrix and Operating Procedures	5
Data Analysis	9 11
EXPERIMENTAL RESULTS AND DISCUSSION	14
Calibration Tests	14
RP-1 Tests	17
JP-7 Tests	21
Propane Tests	24
Tests With Nickel-Plated Tubes	27
Deposit Morphology	28 29
CONCLUDING REMARKS	34
REFERENCES	37
TABLES	
FIGURES	
APPENDIX A - TABULATED TEST DATA	۸ – 1

Deposit Formation in Hydrocarbon Rocket Fuels

Richard Roback Eugene J. Szetela Louis J. Spadaccini

SUMMARY

An experimental research program was undertaken to evaluate the thermal stability characteristics of hydrocarbon rocket fuels under conditions that simulate high-pressure, hydrocarbon-fueled rocket cooling systems. The thermal decomposition (coking) limits and rates of deposition in heated copper tubes for two hydrocarbon fuels, RP-1 and propane, were determined using a continuous flow test apparatus which permitted independent variation and evaluation of the effect of wall temperature, pressure, and fluid velocity on fuel thermal stability. In addition, tests were conducted to investigate the effects of further refining of these fuels, to reduce the concentration levels of deposit forming precursors, on improving their thermal stability.

Parametric tests to map the thermal stability characteristics of RP-1 and commercial-grade propane, were conducted at pressures of 136 atm to 340 atm, bulk fluid velocities in the range 6 to 30 m/sec, and tube wall temperatures in the range 422 to 811K. Selected tests were also performed using de-oxygenated JP-7 and chemically-pure propane as being representative of more refined cuts of the standard rocket fuels. In addition, the effect of the inside wall material on deposit formation was evaluated in selected comparative tests which were conducted using nickel-plated tubes.

The results of the tests indicated that substantial deposit formation occurs with RP-1 fuel at wall temperatures between 600 and 800K, with peak deposit formation occurring near 700K. No improvements were obtained when de-oxygenated JP-7 fuel was substituted for RP-1. For both fuels, carbon deposition rates ranged from approximately 400 to 600 $\mu g/cm^2$ -hr at wall temperatures of 500 to 800K. Examination of deposits obtained with propane fuels indicated heavier, blacker and more uniform deposits than those observed with the kerosene-type fuels and there appeared to be little difference between commercial-grade and chemically-pure propane with regard to type and quantity of deposit. The carbon deposition rates for the propane fuels were generally higher than those obtained for either of the kerosene fuels at any given wall temperature and ranged from 400 to 600 $\mu g/cm^2$ -hr at wall temperatures as low as 400 to 500K. The results of tests conducted with RP-1 at pressures of 136atm to 340 atm indicated that the rate of deposit formation increased slightly with pressure over the range tested. Finally, plating the inside wall of the tubes with nickel was found to significantly reduce carbon deposition rates for RP-1 fuel.

INTRODUCTION

In an effort to increase the performance of hydrocarbon/LOX rocket engines for space booster or orbit transportation systems, i.e. to reduce weight and increase specific impulse, combustion pressures as high as practical are desirable. However, increased combustion pressure leads to a nearly proportionate increase in wall heat flux in the thrust chamber, and therefore, greater stress is placed on the design of the regenerative cooling system. To some extent, the increased heat fluxes can be accommodated by increasing coolant-side heat transfer rates by the expedients of reducing coolant channel flow area, and/or increasing coolant flow velocity, etc., but the implementation of these expedients is not without cost. Therefore, it is desirable to establish upper wall temperature limits to maximize the heat flux into the propellant coolant. Regenerative cooling with hydrocarbon fuels is feasible up to a point where the coolant wall temperature reaches a limit defined by a thermal decomposition or "coking" temperature. Deposit formation on the coolant wall surface, which usually occurs when the thermal decomposition temperature is reached, causes an increased thermal resistance, a progressively increasing wall temperature and, ultimately, failure. Therefore, the rocket engine designer needs to know what maximum heat fluxes can be accommodated under a variety of coolant flow conditions and, especially in the case of the reusable engines, when fuel deposits will be incurred, their nature, (as it affects heat transfer limits) and what rates of formation will prevail.

Hydrocarbon fuel stability and deposit formation (coking) has been the subject of investigation for many years (Refs. 1 to 19). Although the exact mechanism of deposit formation has not been clearly defined, it usually results from the pyrolysis of organic molecules which make up the fuel. Free radicals are generated thermally and, because of their affinity for atoms such as nitrogen, oxygen, and sulfur which might be in the fuel, stable complex solids are formed (Refs. 20 to 25). Studies have shown that additional processing of the fuel to remove these deposit forming precursors has improved fuel thermal stability and decreased deposit formation (Refs. 26 to 31). Fuel decomposition rates and subsequent deposit formation rates have been found to be a function of temperature, pressure, velocity and the composition and physical state of the fuel.

Because of its superior thermal conductivity, copper has been the preferred material for forming the regenerative cooling passages in the high-heat-flux regions of high-pressure rocket thrust chambers. However, studies of the effect of wall materials on deposit formation, Refs. 6, 10 and 32, indicated that deposit rates on copper can be very high. No data was available with regard to deposit formation of rocket fuels such as RP-1 and propane at conditions simulating high-pressure rocket operation. Results deduced from low-pressure coking tests for jet fuels on stainless steel indicated that a coolant-side wall temperature limit in the range 600 to 700K may exist for typical kerosene-type fuels.

However, the effects of the use of copper as a wall material and the effects of fuel pressure and velocity were not determined in the previous investigations. Therefore, to permit more accurate determinations of the maximum allowable coolant-side wall temperatures, a fuel coking test apparatus was designed and developed and was used for parametric evaluation of fuel thermal stability under conditions that simulate high-pressure, hydrocarbon-fueled rocket cooling systems. Using the apparatus developed, experiments were directed toward (1) evaluating the thermal decomposition (coking) limits and rates of deposition in heated copper tubes for two hydrocarbon fuels, RP-1 and propane, and (2) investigating the effect of further refining of these fuels, to reduce the concentration levels of deposit-forming precursors, on improving their thermal stability. Tests were conducted using RP-1 and commercial-grade propane as the standard hydrocarbon rocket fuels and de-oxygenated JP-7 and chemically-pure propane as being representative of more refined cuts of these fuels. A parametric evaluation of fuel thermal stability was performed at pressures of 136 atm to 340 atm, bulk fuel velocities in the range 6 to 30 m/sec, and tube wall temperatures in the range 422 to 811K. In addition, the effect of the inside wall material on deposit formation was evaluated in selected comparative tests which were conducted using nickel-plated tubes.

TECHNICAL APPROACH

As stated above, the objective of the present investigation was to determine the character and rate of formation of fuel deposits under conditions representative of advanced hydrocarbon/oxygen rocket engine cooling systems, and to investigate the effect of increased fuel purity on mitigating deposit formation rates. The overall approach adopted to accomplish these objectives consisted of: characterizing the test fuels as to their chemical composition and physical properties, designing and fabricating appropriate test apparatus, and utilizing this equipment in an experimental study of deposit formation in electrically-heated copper tubes. The fuel properties, test equipment and operating procedures are discussed in detail in the following sections.

Fuels Characterization

The standard fuels which were tested during this program were RP-1 rocket fuel (MIL-P-25576), supplied by the Government, and a commercial-grade propane, purchased from a local supplier. In addition to the primary objectives of this program, to determine the coking temperature limit and rate of carbon deposition for the as-delivered fuels, the merits of further refining of these fuels relative to improving their thermal stabilities were also investigated. To this end, additional quantities of refined quality fuels, which met the specifications listed in Table I, were obtained. Since refinement of a relatively small quantity of the RP-1 fuel was neither practical nor cost effective, it was decided, with NASA approval, to utilize JP-7 (MIL-T-38219) fuel to simulate refined quality RP-1 fuel. Because JP-7 fuel is severely hydrotreated to meet a thermal stability specification, it generally has an order of magnitude less sulfur than RP-1 fuel. In all other respects, the JP-7 fuel tested met the RP-1 specification. Additional on-line treatment of the JP-7 by filtration through a molecular sieve to remove water and by nitrogen sparging to reduce the dissolved oxygen content was required to provide a fuel of sufficiently refined quality to meet the specifications listed in Table I and to adequately establish the effect of fuel refinement on deposit formation. The use of chemically pure propane (minimum 99% pure in liquid phase), purchased directly, was determined to be the most cost effective way to obtain refined-quality propane.

Each fuel chosen for testing was characterized as to selected chemical and physical properties. These properties determinations were obtained utilizing a combination of in-house, Government and independent analytical laboratory facilities; API and ASTM standard correlational procedures (Refs. 33 and 34); supplier's certified analyses (Refs. 35 and 36); and published data (Refs. 37 to 43). Typical physical properties of the test fuels are shown in Table II, while the variation of selected physical properties with temperature is shown in Table III for RP-1 and JP-7 fuels and in Table IV for the propane fuels. Certificates of analysis for the RP-1 and JP-7 fuels used in this program were

provided by the fuel suppliers. Selected chemical analyses of constituents not generally included in the military specifications were performed either in-house or by an independent testing laboratory. A summary of these analyses is shown in Table V.

Since commercial-grade propane is distributed in bulk, a certificate of analysis is not normally available. However, the commercial-grade propane used in this test program was certified by the supplier as meeting the ASTM standard for motor-grade propane. This fuel contains a minimum of 90% propane, a maximum of 5 percent propylene and the remainder comprises a mixture of other hydrocarbons such as butane, butylene, and ethane. The chemically-pure propane was certified as being 99% propane. Typical analyses of the commercial-grade propane and the chemically-pure propane utilized in this test program are shown in Table VI.

Test Facility and Test Hardware

Description of Apparatus

The test program was conducted in a self-contained combustion test facility which consists of a concrete test cell and a separate control room for operating personnel. The test facility is capable of continuous operation with fuel flowing at a velocity up to 36.5 m/sec and at pressures as high as 340 atm. An electrical power supply, which is capable of providing 40 KVA AC and 4000 amps, was used to provide power for electrically heating the copper test tube up to the maximum temperature of 866K specified for the test program.

The test apparatus, shown schematically in Fig. 1, consisted of the following major components: (1) a fuel supply tank, (2) a run tank to which is connected a zeolite-type molecular sieve water-absorber and a porous metal sparging element for reducing water and dissolved oxygen concentrations to the levels specified for the refined fuels, (3) a fuel delivery system consisting of two piston-type accumulators which are pressurized and used to drive fuel through the test section, (4) a venturi flowmeter, (5) a resistance-heated test tube connected to a 40 KVA high-amperage power supply, (6) an in-line filter for collecting any solid particles which might form in the bulk flow or breakoff from the test tube wall during test, (7) a fuel cooler, (8) an electrically-driven metering valve which was used to control the fuel flow through the test section, (9) a turbine-type flowmeter, and (10) a fuel dump tank.

The original design of the test apparatus included a high-pressure (340 atm), variable displacement axial piston pump, which was to be used to raise the pressure

of the test fuel to the desired level and pump it through the test section. However, after several pump failures occurred, it was concluded that the pump was not compatible with low viscosity fluids, such as RP-1 and propane, and therefore, the fuel delivery system was modified to utilize two high-pressure piston type accumulators as fluid transfer barriers. For the low-pressure tests (136 atm) the accumulators were installed in parallel (as shown in the inset in Fig. 1), and one side of each accumulator was precharged with test fuel while the opposite side was pressurized with 156 atm nitrogen which actuated the pistons and forced the test fuel through the test section. A dome-loaded pressure regulator was used to regulate the nitrogen driver gas pressure. With this configuration, a total of approximately 48 gallons of fuel could be expended before the system had to be refilled.

Because the facility was not capable of supplying a high flow rate of nitrogen at pressures in excess of 156 atm, this configuration could not be used for higher pressure tests and therefore, additional modifications were made to the fuel delivery system to permit the use of high-viscosity hydraulic fluid, pumped by the existing high-pressure piston pump, as the accumulator driver fluid. This system, also shown in the Fig. 1 inset, consists of two piston accumulators in series; the first accumulator contained hydraulic fluid on one side of piston and test fuel on the other side, and the second accumulator (which empties through the test section) contained test fuel on both sides of the piston. With this arrangement, the hydraulic fluid and test fuel are separated by a relatively large volume of contained fuel, thus eliminating the potential for contamination of the test fuel by hydraulic fluid left on the wall of the accumulator or by hydraulic fluid leaking across the piston seals. However, this arrangement limited the fuel capacity to 23 gallons and it was therefore necessary to refill more frequently between tests.

Test Tube Fabrication and Characteristics

Since test data at pressures up to 340 atm was desired, all tube test elements were designed to withstand this pressure at tube wall temperatures up to 1000K. A duplex tube wall configuration was selected to meet this requirement and to also provide a better match of the specific electrical resistance of the tube elements to the capabilities of the electric power supply. In the duplex tube configuration, an inner wall of an oxygen free-high conductivity copper (No. 102; 99.95% pure; electrical conductivity = 0.586 Megmho-cm) provided the desired test surface for studying the rates of deposit formation on copper while an outer wall of Inconel 600 provided the necessary high-temperature tensile strength.

The individual test tube elements comprised an inner 0.254-cm ID x 0.366-cm OD copper tube surrounded by a 0.366-cm ID x 0.478-cm OD Inconel tube. This configuration had the advantage that, while the structural load was carried by the outer sheath, the majority of the power (\sim 95%) was generated in the copper and, as a result, the tube radial temperature gradient was small. The duplex tube was manufactured by threading the 0.254-cm ID x 0.366-cm OD copper tube into an

oversized Inconel outer sheath, and subsequently drawing the Inconel tube through a die to obtain the proper sheath thickness and OD of the duplex tube. This process ensured a good bond, i.e., intimate wall contact between the Inconel and copper surfaces which was subsequently verified by various tests described later in this subsection.

To preclude any significant electrical or thermal resistance at the interface of the two metals, stemming from oxidation, contamination or local separation, the tubes were cleaned and inspected before and after fabrication. Tests to evaluate the effectiveness of several candidate procedures for cleaning the copper and Inconel tubing prior to fabricating the duplex test tubes indicated that a thorough degreasing was sufficient for the Inconel tube, since Inconel is relatively corrosion resistant and is not easily oxidized. 10 percent nitric acid solution appeared to clean the copper surface adequately; however, a water slurry of a commercially-available powdered copper cleaner containing oxalic acid was just as effective and easier to use. Based on the results of these tests, the cleaning procedure adopted consisted of (a) immersion of both the copper and Inconel tubing in a hot degreasing solution, followed by a thorough water rinse; and (b) application of the commercial copper cleaner to the outer surface of the copper tubing with a fine brush, removal with a clean sponge and water, followed by a final water-rinse and degreasing. Handling of all materials was done with lint-free gloves.

Prior to fabricating the required quantity of tubing, a sample length was manufactured according to the procedure described above, and the sample was subjected to several tests and examinations to evaluate the quality of the mechanical bond between the Inconel and copper tubes. The test procedures included metallographic examination, shear tests, and thermal cycling in a high-temperature oven followed by sectioning and microscopic examination.

Metallographic examination of tube samples, including scanning electron-microprobe analysis at the Inconel/copper interface, indicated that the copper closely followed the contour of the Inconel surface and the interface was free of contamination (i.e., no oxides or elements other than the parent metals were present) and/or air gaps. A maximum separation of approximately 0.00025-cm was observed between the copper and Inconel surfaces, which was attributed primarily to the sample preparation procedures (i.e., cutting, mounting and polishing).

The shear test was performed in an experiment in which an axial load was applied to the inner copper tube in an attempt to push the copper out of the Inconel sheath. Several tube samples having different lengths (0.2, 0.51 and 0.64 cm) were tested and it was found that an average shear load of 57 atm on the Inconel/copper interface was required for initial movement of the copper; an average shear load of 516 atm was required to extrude the copper through the Inconel tube.

A thermal cycling test was performed in which a sample tube was heated to 811K in an oven, air cooled to room temperature and then reheated and recooled. This test was designed to aggrevate any tendency to separate at the bond surface because of the difference in thermal expansion between copper and Inconel and evaluate any possible separation on cooling, which, if encountered, would preclude reuse of tubes in which no deposits were formed. The sample tube was sectioned and microscopic examination indicated that no apparent separation of the copper from the Inconel had occurred.

The results of the various tests indicated that the mechanical bond between the Inconel and copper sections of the duplex tube was of very high quality, and that any tendency for separation at the Inconel/copper interface would be opposed during testing by the combined action of internal pressure forces and the higher rate of thermal expansion of copper relative to Inconel. Therefore, having validated the manufacturing process, the entire length of tubing required for all tests was manufactured in a single run.

The test tube assembly is shown schematically in Fig. 2 and by the photograph in Fig. 3. The test tube was silver soldered to a copper bus ring which in turn was bolted to copper ring adaptors. A high temperature silver solder (890K) was used during fabrication of the initial set of test tube assemblies to insure that no problems with tube attachment would occur during testing at the highest wall temperature. These tubes had to be discarded because longitudinal cracks developed in the Inconel outer sheath at the points of attachment to the copper bus rings, apparently as the result of excessive thermal stressing of the tube which occurred during the application of the high temperature silver solder. A detailed heat transfer analysis of the test tube assembly, which will be described in a later section, indicated that the temperature of the bus ring and adapter would not exceed 425K for the highest test tube temperature; therefore, a lower melting temperature silver solder (650K) was used for subsequent tubes and the problem was eliminated.

Ten thermocouples were spotwelded to the outer tube wall of each assembled test piece to monitor the outside tube wall temperature during a test. These thermocouples were placed at equal spacings of 2.54-cm starting at a location of 1.27-cm from the bus rings. The placement of the thermocouples was also determined from the heat transfer analysis which will be discussed later. The surface of the tube was coated with Sauereisen at the thermocouple junctions, to electrically insulate the thermocouple wire from the tube, and the wire was wrapped once around the tube and coated with additional Sauereisen cement to insure good thermal contact. By use of an AC power supply, thermocouple errors resulting from a voltage drop across the thermocouple bead were minimized.

The test section mounting arrangement is shown in the schematic of Fig. 4 and the photograph in Fig. 5. The test tube assembly is supported on teflon-lined cradles that were designed to accommodate the bus rings and to permit thermal expansion by providing a low coefficient of sliding friction. The teflon also acts as an electrical insulator and prevents grounding of the test tube. In

addition, non-conductive flexible hose was installed at the entrance and exit of the test tube assembly to allow thermal expansion and to electrically isolate the tube from the other components of the test apparatus. Connections to the power supply were made using a solid connector and a flexible water-cooled cable. Adjustable wall anchors and transformer connections were provided to assist in aligning the tube assembly prior to testing.

Data Acquisition and Control System

All test data were recorded utilizing a microprocessor-controlled, data acquisition/reduction system. The data system converted outputs from thermocouples, current transmitters, pressure transducers, etc., to precisely scaled DC voltages for measurement, and displayed the data in engineering units. The reduced data was logged on paper tape via a programmed logging format or through demand logging by push-button control. Specific groups of data channels were scanned and/or logged at specific intervals, or continuously. The system enabled scanning up to 70 channels at a scan rate of 35 channels per second on standard resolution, and 10 channels per second on high resolution. Data was logged on paper tape at the rate of 6 lines per second.

A built-in cathode ray tube (CRT) was used to display key operating parameters such as fuel pressure, temperature and flow rate; tube wall temperatures; and input voltage and current. The display provided a continuous visual check of the thermocouple outputs and test control parameters. An alarm function is also included and was utilized to provide a continuous check of each thermocouple output to ascertain that prescribed maximum temperature limits were not exceeded during a test run.

The primary fuel flow measurement was made with a turbine-type flow meter which was located downstream of the test section. In addition, a redundant fuel flow measurement system, consisting of a venturi-type flow meter and a differential pressure transducer, was installed upstream of the test section. Pressures were measured both with strain-gauge type pressure transducers and conventional Bourdon type pressure gauges. All instrumentation received frequent routine calibrations against laboratory standards.

Test Matrix and Operating Procedures

The experimental program began with a sequence of shakedown tests to verify the structural integrity of the system and to optimize procedures for testing and data acquisition. The shakedown period also included tests directed toward verifying the absence of a significant thermal resistance at the copper-Inconel interface, resulting from poor metal-to-metal contact or from impurities at the Inconel/copper interface. For this purpose, special tests were conducted (a) using high-pressure water in place of fuel, and (b) using a specially instrumented

test tube which allowed direct temperature measurement at the Inconel/copper interface as well as at the Inconel outer wall. After qualification of the experimental hardware and test procedures, a series of parametric tests was conducted to document the coking limits and decomposition rates of RP-1, JP-7, commercial-grade propane and chemically-pure propane. The full matrix of test conditions included fluid inlet velocities of 6.1, 12.2, 18.3, 24.4, 30.5 m/sec; tube entrance static pressures from 136 to 340 atm; and tube wall temperatures of 422, 589, 700 and 811K. The duration of each test was to be ten minutes; unless, during a test any tube wall temperature exceeded the maximum allowable limit of 866K, in which case the test was terminated.

Fuels testing at a pressure of 136 atm was started with standard, as-delivered RP-1 fuel and then proceeded to JP-7 fuel. Appropriate changes were made to the test apparatus to accommodate liquid propane and testing was continued with commercial-grade propane and, finally, chemically-pure propane. A limited number of tests were then performed with RP-1 fuel at pressures up to 340 atm to determine the effect of increased pressure on deposit formation. Finally the effect of inside wall material on deposit formation was evaluated in selected tests which were conducted using nickel-plated tubes.

Prior to each test the inside of the test tube was cleaned with a degreasing solution and a water slurry of commercial powdered copper cleaner, rinsed with water and dried with nitrogen, and then installed in the test apparatus. In addition, the in-line fuel filter located downstream of the test tube was replaced. If tests using kerosene fuel of refined quality were conducted, sparging and water filtration of the fuel were performed to reduce the oxygen and water concentrations to the desired levels. The piston type accumulators were then charged with the appropriate fuel and pressurized with nitrogen driver gas for the 136 atm tests, or with hydraulic fluid supplied by the high pressure pump for the higher pressure tests.

The test was initiated by opening the accumulator valve and then adjusting the downstream metering valve until the desired flow rate was obtained. The electrical powerstats were activated and set for a low power level at which a relatively flat wall temperature distribution was obtained (e.g., at wall temperatures of approximately 350 to 450K). Data was recorded at this initial power setting; any deviation from a flat temperature profile was noted as being indicative of improper thermocouple attachment.

The electrical powerstats were then advanced to a predetermined position to achieve the desired wall temperature (transient time was usually less than 6 seconds) and the corresponding input power was maintained as the test was continued for ten minutes or until a maximum wall temperature of 866K was reached. The data logger continuously monitored and recorded test data at one minute intervals, or more frequently if large variations in data were occurring. After the test was completed, the test tube was removed and set aside for sectioning and deposit analysis. The fuel filter was also removed and set aside for post-test examination.

Data Analysis

During testing, coking was detected by a change in the tube axial wall temperature distribution when the system pressure, fluid velocity, and tube heating rate were held constant. After each test in which there was a positive indication of coking, qualitative and quantative analysis of the deposits formed was performed and the results were correlated with the initial wall temperature and test operating parameters.

Calculation of Inner Wall Temperature

The test tube inner wall temperatures were calculated from the measured outer wall temperatures using a computerized heat transfer analysis. In this analysis (TCAL), a finite difference representation of the heat conduction equation (a time-dependent version of Laplace's equation) is solved by a relaxation technique. This solution is applied to any one-, two-, or three-dimensional model in order to calculate the steady-state or transient temperature distribution for all elements of the model. The required inputs to the computer program included a geometric description of the test tube assembly, the physical properties of the duplex tube and bus ring materials, the transport properties of the test fuels and the ambient environment, and the heat generated (electrically) within the apparatus. Material and fluid properties are permitted to vary with temperature. A preprocessor was used to convert coarse dimensions of two-dimensional or axisymmetric models into TCAL input. The output is the steady-state (or transient) temperature of the elements, including the local temperature of the fluid.

A 152 element model was utilized to describe the tube/bus ring configuration used in the tests and to calculate the axial and radial wall temperature gradients for RP-1 fuel flowing at a velocity of 30.5 m/sec through a tube having a maximum inner wall temperature of 811K. This was the most severe test condition with respect to the radial temperature gradient. The fuel inlet temperature was assumed to be 311K. The elemental description of the tube/bus ring configuration and the calculated local wall and bulk fuel temperatures are shown in Fig. 6. It should be noted that the TCAL analysis indicated that less than 1 percent of the electrical energy dissipated in the tube wall was lost to the surroundings, and that the balance of the electrical energy input to the system (more than 99 percent) appeared as heat conducted into the test fluid. Therefore, knowing the radial origin of the electrical energy input (from the relative electrical resistance of the Inconel and copper wall materials), the wall temperature drop (from the external point of temperature measurement to the inside copper wall) and the fluid temperature rise was calculated using the TCAL computer program. It can be seen from the figure, that the bulk temperature of the fuel was increased by 71K as it flowed through the heated tube and that the maximum

temperature difference between the outer and inner walls is 15K. Because the relatively massive bus-ring/connector assembly remained essentially at ambient temperature during a test, significant local cooling and steep temperature gradients occurred at the ends of the tube. Therefore, to avoid placement of thermocouples at locations which could be affected by tube end cooling, the grid elements adjacent to either bus ring were subdivided into smaller elements (not shown in Fig. 6) and a more accurate calculation of the local temperature distribution in this region was performed. This calculation indicated that placement of the thermocouples at least 1.27-cm from the bus ring would avoid the tube end effects.

The results of the TCAL calculation are also shown graphically in Fig. 7, along with the axial variation of heat transfer coefficient. The equation used for calculating the local heat transfer coefficients was obtained from Ref. 44. It will be shown later that this equation matches measured data with reasonable accuracy. An unanticipated result of the calculation was the prediction that the maximum wall temperature would occur near the inlet of the tube and not at the exit where intuitively it would be expected. This seemingly anomalous behavior can be explained by examining the axial variation of the heat transfer coefficient along the inner wall. The variation of bulk fuel properties with temperature results in a local heat transfer coefficient which is lowest at the tube entrance, where the fuel is cold, and increases as the fuel is heated. As a result, in the case of the kerosene type fuels, the highest wall temperatures occur near the tube entrance and the wall temperature decreases along the length of the tube.

These calculations, which were performed for one extreme in the matrix of test conditions (i.e., the highest wall temperature and highest velocity), indicated a temperature difference between the outer and inner walls of only 13 to 15K. The TCAL results were used to predict the wall temperature difference (inner vs outer) over the entire range of test conditions, see Fig. 8. The differences ranged from approximately 3 to 13K at the highest wall temperature of 811K and 1 to 3K at the lowest wall temperature of 422K. These temperature differences were, for the most part, within the expected experimental accuracy of the temperature measurement and, therefore, the measured outer (Inconel) wall temperature was considered representative of the inner (copper) wall temperature.

Deposit Characterization

After each test in which there was a positive indication of coking, the tube was sectioned. As shown in Fig. 9, five 1.27-cm long sections were cut from the 25-cm length of tubing and from each of these sections, a longitudinal and transverse section was prepared for microscopic examination. The transverse

sections were mounted in plastic, metallographically polished, and photomicrographs were taken to estimate deposit thickness. The longitudinal sections were used for qualitative analysis of the deposits, in terms of type and uniformity. The remaining four sections, each approximately 3.8-cm long, were used in burnoff tests where a quantitative estimate of the total carbon deposit was made by determining the quantity of CO₂ evolved.

Depending upon the type and amount of deposit present, several methods were considered to determine the quantity of deposit on the tube surface. These procedures included (1) direct measurement of deposit thickness, (2) tube weighing before and after deposit burnoff, (3) deposit burnoff in air with analysis of evolved gases and (4) surface analysis using ion spectroscopy. Examination of the deposits obtained early in the tests with RP-1 indicated that the deposits were of sufficient quantity, i.e., > 0.1 mg, to permit the determination of deposit rates by burnoff in air with analysis of the evolved CO₂. Tube weighing before and after deposit burnoff was not performed because of anticipated inaccuracy in weighing such small quantities and because the weighing procedure would be complicated by the fact that the copper surface could be oxidized during burnoff. The direct measurement of the deposit thickness from photomicrographs of transverse sections of the tube was also undertaken but the results were inconclusive because of the non-uniform deposition of material along the tube.

EXPERIMENTAL RESULTS AND DISCUSSION

The experimental test program comprised (a) two special calibration tests to verify that there was no significant thermal resistance at the Inconel/copper tube interface; (b) an extensive series of tests designed to investigate the deposit formation tendencies of RP-1, JP-7, commercial-grade propane and chemically pure propane at a pressure of 136 atm and over a range of velocities and wall temperatures; (c) tests to determine the effect of pressure on deposit formation; and (d) a short series of tests to evaluate the effect of the tube inside wall material on deposit formation. A discussion of the results of these experiments is included in the following sections. A tabulation of all the test data, including calculated parameters (e.g., velocity, Reynolds No., discharge coefficient and deposit thermal resistance), is presented in Appendix A.

Calibration Tests

A special calibration test using high-pressure water in place of fuel was conducted to determine if there was any significant thermal resistance at the Inconel/copper interface which could result from poor metal-to-metal contact or from impurities at the interface. If a significant interfacial resistance was present, the theoretical heat transfer prediction would have to be modified and the input power and output wall temperature settings revised. Water was used for these tests, since its thermal properties are known with greater accuracy than any other candidate test fluid. Testing with water ensured that the contribution of uncertainties in the basic fluid transport characteristics to any differences between the TCAL predictions and the actual temperature measurements would be minimized. Therefore, if the measured wall temperature profile closely matched the TCAL temperature profile predictions for water, the absence of a significant thermal resistance at the Inconel/copper tube interface would be verified.

Three well-known heat transfer correlations described in Ref. 45, (i.e., the classical Dittus-Boelter equation, the Sieder-Tate equation and the Colburn equation) were used to predict the axial temperature distribution along the outer wall of the heated tube. These equations were chosen because they appeared to be best suited for experiments conducted with high-pressure water. The Dittus-Boelter and Sieder-Tate equations utilize fluid bulk properties and/or corrections for wall to bulk temperature differences. With the exception of specific heat, film properties are used in the Colburn equation. For water flowing at a pressure of 64 atm and a velocity of 7.5 m/sec, through a tube heated with 17.6 kW of electrical power, the three equations predicted temperatures which were higher than the experimental values. The Colburn equation gave the best correlation, perhaps because the conditions of the experiment better matched the conditions for which the equation was derived. A comparison between the Colburn prediction and the experimental results is shown in Fig. The shaded area represents a \pm 5 percent difference in power input, and reflects the differences in the heat balance obtained when the measured input power was compared with the measured sensible heat added to the water. Since

no Inconel-copper interfacial resistance was assumed in the theoretical analysis and since, as stated above, the predicted temperatures were higher than the measured wall temperatures, (the converse would be expected if a significant interfacial thermal resistance were present) the absence of any significant resistance present at the Inconel/copper interface of the tube was inferred.

A test was also performed using a special tube which was instrumented with thermocouples attached to the copper tube outer wall through holes drilled in the Inconel sheath. This tube had been prepared by NASA/LeRC to aid in correlating experimental data with the theoretical heat transfer predictions, and to verify the integrity of the mechanical bond at the Inconel/copper interface. Pretest inspection of the tube revealed that there were very fine cracks in the Inconel sheath, close to the copper bus rings. These cracks were similar to ones observed previously at UTRC, stemming from overheating of the tube during application of the high-temperature silver solder for attaching the bus rings to the tubing. This special tube was instrumented with additional thermocouples on the outside Inconel wall and then tested at operating conditions of reduced severity. This was done because the results of earlier experiments had shown that regions of locally high temperatures would develop around the cracks and, thereby, limit the maximum allowable power input to the tube.

Tests were performed with RP-1 fuel at a pressure of approximately 136 atm, fluid velocity of 6.1 m/sec and wall temperatures ranging from 350 to 750K. At the highest wall temperature, a leak developed around one of the thermocouples attached to the copper and therefore, the test was terminated and no additional testing with the tube was attempted. A comparison of the temperature distributions measured along the outer walls of the copper and Inconel sections of the tube is shown in Fig. 11. It can be seen that there is excellent agreement between the two wall temperature distributions, (except at the peak temperature where the largest difference is only 10K), thereby verifying, in a general sense, the TCAL temperature predictions as well as confirming the absence of a significant resistance at the interface between the two metals. The relatively high temperature reading of the thermocouple located at the tube exit (i.e., the 23-cm station) was attributed to cracks in the outer tube.

Special tests were also conducted with RP-1 in which the fluid velocity was fixed and the power level into a single tube was varied sequentially to produce peak wall temperatures within the range 400 to 900K. Operating conditions were held constant only momentarily, so that data could be recorded at each power setting and in the absence of significant deposit formation. The tests were repeated for the full range of fluid velocities. The temperature distributions obtained at the maximum and minimum test velocities (i.e., 7.1 and 30.5 m/sec) are shown in Fig. 12. These results indicated that substantial changes in the heat transfer processes were incurred as the flow, power and wall temperature were varied. At low velocity conditions, and particularly at high power, the wall temperature increased with length near the tube entrance,

reached a maximum value at approximately 20-30 percent of the tube length, and thereafter decreased continuously to the end of the tube. In contrast, at high fluid velocity the wall temperature distribution did not show a peak; instead the wall temperatures gradually decreased from the tube entrance to the tube exit.

The wall temperature distribution noted at low velocity appears to indicate laminar-like flow at the tube entrance, followed by a transition to turbulent flow. In laminar flow, an initial high heat transfer coefficient is expected until an appreciable thermal boundary layer thickness has accumulated. Thereafter, the heat transfer coefficient diminishes rapidly to a minimum value as the thermal boundary layer grows and then it rises again as transition to a fullydeveloped turbulent flow condition ensues. This entrance effect could be expected to be minimized at the higher Reynolds numbers associated with the higher flow rates. The inlet Reynolds number at the low velocity condition is approximately 7000 and, therefore, turbulent flow would be expected to exist in the tube. However, there is theoretical and experimental evidence (Ref. 46) which indicates that in the case of liquid flow in smooth pipes, heating tends to stabilize a laminar boundary layer and to lower the liquid viscosity and thus delay the transition to fully developed turbulent flow. At 20 to 30 percent of the tube length, the Reynolds number has apparently increased sufficiently to permit the transition to turbulent flow.

Wall temperatures measured in the special tests described above were used to calculate experimental heat transfer coefficients which were compared with theoretical heat transfer coefficients calculated from the following correlations:

Dittus-Boelter: Nu = 0.023 Re^{0.8}Pr^{0.4} (Ref. 45)

Sieder-Tate Nu = 0.023 Re^{0.8}Pr^{0.4}
$$\left(\frac{\mu_R}{\mu_W}\right)^{0.14}$$
 (Ref. 45)

Rocketdyne: Nu = 0.0056 Re^{0.95}Pr^{0.4} (Ref. 44)

Rocketdyne: Nu =
$$0.0056 \text{ Re}^{0.95} \text{Pr}^{0.4}$$
 (Ref. 44)

Where Nu, Re, Pr are the Nusselt, Reynolds and Prandlt numbers, respectively, and μ_R and μ_U are viscosities at the bulk and wall temperatures, respectively.

The experimentally-determined heat transfer coefficients calculated for a flow velocity of 18.3 m/sec are compared with the theoretical heat transfer coefficients in Fig. 13. It can be seen that all the experimental data fall slightly above the theoretical predictions. Since the test conditions were held constant only momentarily, so that no significant deposit formation would occur, the differences between the experimental data and the theoretical curves cannot be attributed to carbon formation. Furthermore, at the velocity of 18.3 m/sec, the tube entrance effects were

not appreciable and should not have had a significant effect on the heat transfer. However, it is possible that the higher experimental heat transfer coefficients noted could have been caused by a slightly higher than usual tube roughness which resulted during tube fabrication.

RP-1 Tests

Since the special calibration tests corroborated the results of the physical tests performed on the tubing, it was concluded that a good mechanical bond, free of contamination existed at the metal interface and that the radial temperature gradient across the tube was negligible for the purposes of the intended tests. Therefore, more detailed testing with RP-1 fuel was initiated; a summary of the tests performed is shown in Table VII. RP-1 testing began at a fluid velocity of 6.1 m/sec and a wall temperature of 422K. However, no significant temperature rise was observed along the tube during the ten minute test duration, suggesting the absence of significant deposit formation. The tube used in this test was sectioned and microscopic inspection of the inner copper surface confirmed that deposits had not been formed. In appearance, the surface was only slightly stained with no evidence of black, carbon-like deposits which were subsequently obtained at higher temperatures. Based on these results, no additional testing was done at a wall temperature of 422K.

Outer tube wall temperatures were measured in tests within a range of fluid velocities from 6.1 to 30.5 m/sec. The input heat flux to the tube was varied between 173 and 1460 Watt/cm² so that wall temperatures in the desired range from 583 to 811K could be achieved. The wall temperature distributions are shown in Figs. 14 through 16 where each condition shown is a composite of data obtained from a different test. The results shown in Figs. 14 through 16 indicate that the temperature rise that was obtained during the ten-minute duration was lowest at the lowest wall temperature, increased with increasing wall temperature and reached a maximum value at locations which were initially at temperatures between 700 and 750K. Since it was expected that a wall temperature rise indicated deposit formation, this behavior suggested that the rate of deposit formation increased with increasing temperature, reached a maximum at a temperature of 700 to 750K and fell off thereafter. Also, the first indication of a significant local temperature rise generally occurred at the tube entrance (region of highest wall temperature) and eventually spread to other locations all along the tube.

The effect of the initial wall temperature on temperature rise with time is shown more clearly in Fig. 17, where the tube temperature rise data are plotted against the initial wall temperature. It can be seen that a maximum temperature rise of approximately 80K occurred at a location on the tube where the initial wall temperature was between 700 and 750K. There appears to be no appreciable effect of fluid velocity, except possibly to produce a slight shift in the location of the peak temperature rise.

Heat transfer across a deposit layer formed and the wall is governed by the following:

$$Q/A = \frac{k}{T} \Delta T_w$$

where Q/A is the heat flux, k is the thermal conductivity of the deposit, τ is the deposit thickness and $\Delta T_{\pmb{w}}$ is the wall temperature rise. If the thermal conductivity of the deposit is assumed to be constant and the deposit thickness increases during the test, then for the constant heating rate, maintained during the test, the tube wall temperature would be expected to increase in proportion to accumulated deposit thickness. Similarly, if the deposit rate is assumed to be independent of fluid velocity (as a first order assumption), but the power input to the tube is increased to maintain a given initial wall temperature when the fuel flow rate (and therefore velocity) is increased, then the wall temperature rise observed should be higher at the higher velocities (i.e., equal deposit thickness, but proportionately increased heat flux). However, since the wall temperature rise was observed to be essentially independent of velocity for the RP-1 tests (see Fig. 17), either the rate of deposit formation decreases with increased fluid velocity, or other factors, such as a synergistic effect of deposit roughness may contribute to an enhanced local heat transfer coefficient.

For each test which gave a positive indication of deposit formation, i.e., a significant local wall temperature rise with time, the test tube was sectioned and the tube sections were prepared for microscopic examination and deposit burnoff. Microscopic examination of the inside surfaces of longitudinal sections of the tubes revealed that the deposit coverage was generally very non-uniform and ranged from specks, to connected islands of deposits, to essentially full coverage. No particular pattern could be established with test conditions and the non-uniform deposit coverage made a determination of the point of incipient deposit formation impossible.

Also, the deposits observed were multi-colored and took on various shades of red, black and sometimes gray. All three colors could sometimes be observed on samples taken from a single tube; however, it was difficult to associate color with a particular run condition or wall temperature. The deposits appeared to vary in degree of roughness but were generally hard and did not break loose from the tube surface very easily. From the general appearance of the surfaces of the tubes, it was concluded that the formation of deposits on copper is a very complex process leading to various intermediate compounds which can take on various colors and textures.

Transverse cross sections of the tubes were also taken at approximately the same location as the longitudinal sections. These cross-sections were potted in plastic and polished to produce a flat surface for subsequent microscopic examination. Photomicrographs of these surfaces were taken and an attempt was made

to determine the deposit thickness. The deposits observed in these photomicrographs were usually not of constant thickness and, because of the non-uniformity of the deposit formation, covered only a small portion of the surface. However, on the average, they ranged from 0.0002 to 0.001-cm in thickness.

Because of the high magnification of the small surface area which is observed in the transverse sections, the photomicrographs of the tube transverse sections appeared to indicate even greater non-uniformity of deposit formation than the longitudinal sections had shown. In fact, in many cases, the transverse sections showed little or no deposit formation whereas the longitudinal sections taken from approximately the same locations on the tube and viewed at lower magnification indicated significant deposit coverage. For these reasons, deposit rate estimates obtained from the photomicrographs were not used as the primary measurement of deposition rate. These data were instead inferred from the results of the burnoff tests to be discussed below.

A typical photomicrograph of a tube cross-section (with deposits) corresponding to a test with RP-1 is shown in Fig. 18. The dark area shown in the photomicrographs is the plastic potting material and the bright area is the copper. The thin line of material between the dark and bright areas is deposit. From this figure, there appears to be an indication that the deposit is thicker for the low velocity condition. The particular sections shown represent some of the more uniform deposits; however, many of the samples revealed deposits having a much more irregular pattern, from which consistent trends in either deposit nature or thickness with test conditions could not be derived.

The primary measure of the deposit formation rate with the various test operating conditions was made by burning off the tube deposits and measuring the quantity of CO2 evolved. A special laboratory bench-type apparatus was employed for this task, wherein a metered flow of air was passed at a constant rate through heated sections of the test tube. The product gases resulting from the burnoff were subsequently passed through a nondispersive infrared analyzer which measured the concentration of CO2 in the effluent gas. This instrument was calibrated before each series of burnoff tests using air for setting the instrument zero level and a N2/CO2 gas mixture of certified CO2 concentration for establishing the scale factor. Also, prior to initiating the tube deposit burnoff tests, a special calibration run was made in which pre-weighed samples of instrument grade graphite were burned off. Agreement between the weight of carbon calculated from the CO2 evolved and the actual weight of graphite was within two percent. No special post-test procedures were employed prior to deposit burnoff to remove any residual fuel which might remain in the tubes. It was felt that since several days elapsed between testing, tube sectioning, and deposit burnoff, there was adequate time for the tubes to dry. Furthermore, since the burnoff procedure involved a gradual heating of the tube sections, there should have been sufficient time for vaporization of any residual liquid fuel prior to oxidation of carbonaceous deposits. However, in order to check if significant residual fuel remained in the tubes, alternate sections of several tubes were washed in solvent, blown dry with nitrogen, and heated in a vacuum oven to remove any traces of residual fuel. Comparison of rate data determined for these specially treated sections with that determined for adjacent untreated sections indicated that any effect of residual fuel was insignificant.

Output from the meter was continuously recorded to give a time trace of percentage CO_2 evolved during burnoff of the deposit. Integration of the data over the total burnoff time gave the total volume of CO_2 evolved, from which a carbon weight and deposition rate were calculated. As shown in Fig. 9, four equally-spaced sections (each approximately 4-cm long) were cut from each test tube and used for the burnoff tests. The four tube sections represented approximately 60 percent of the total surface area of the tube; the remainder of the tube was used for the longitudinal and transverse sections. The results of the burnoff tests for RP-1 are shown in Table VIII.

The data listed in Table VIII are presented graphically in Fig. 19, in the form of plots of the rates of carbon deposition as a function of the average initial wall temperatures. The open symbols represent the data obtained from individual tube sections (i.e., 15 percent of the total tube length) while the closed symbols represent an average of the four sections of the tube (60 percent of the total tube length). It can be seen that there is considerable scatter in the data; however, some general trends can be observed. The data scatter is believed to result from the combined effects of experimental error and the non-uniformity of the deposits on the tubes. The deposit rate data appear to substantiate the conclusions drawn previously from the wall temperature distributions (see Fig. 17); i.e., the rate of carbon increases with increasing temperature, reaches a maximum and then falls off as temperature is increased further. This maximum deposition rate occurs at a wall temperature of approximately 600K at the lowest fluid velocity and appears to shift to higher temperatures (700 to 750K) as the fluid velocity is increased. For each velocity, the maximum deposition rate occurred at a lower temperature than was inferred from the temperature rise data shown in Fig. 17.

The rate of carbon deposition also appears to decrease as the fluid velocity increases. In order to explore this apparent trend, data was taken from Fig. 19 and replotted in Fig. 20 to illustrate the dependency of the rate of carbon deposition on velocity. It can be seen that the rate of deposit formation decreases as the fluid velocity is increased up to a wall temperature of 700K; whereupon, the rate of carbon deposition appears to reverse trend and increase with flow velocity.

Another measure of the rate of carbon deposition which occurred during a test is the deposit thermal resistance build up rate (R_c) , which is defined as:

$$R_{c} = \frac{\Delta T_{w}}{(O/A)(t)}$$

where $\Delta T_{\mathbf{w}}$ is the wall temperature rise (deg K) observed during the test, Q/A is the heat flux (Watt/cm 2), and t is the test duration (min.). The thermal resistance growth rates calculated for tube sections which exhibited temperature rises during testing with RP-1 fuel are shown in Fig. 21. These rates were calculated for the full test duration of 10 minutes. It should be noted that the thermal resistance buildup rates generally decreased with time as shown in the data tabulations for RP-1 in Appendix A. The data presented in Fig. 21 indicates that the resistance buildup rate reached a maximum at tube locations where the initial wall temperature was approximately 700K. In addition, the magnitude of the peak appears to decrease as the fluid velocity is increased, a trend which is in agreement with the deposit burnoff data discussed above. Also, as the fluid velocity is increased, the initial wall temperature at which the maximum rate occurs appears to shift to a lower value. The deposit thermal resistance data were also plotted against the reciprocal of the initial wall temperature (i.e., the Arrhenius form) and are shown in Fig. 22. Straight lines were fit through the data on either side of the peak buildup rates and are also shown in the figure. A comparison of the thermal resistance buildup rate data obtained for the highest fluid velocity considered in this program (V = 30.5m/sec) with experimental data for RP-1 (Ref. 49.) obtained at velocities in the range 46-76 m/sec is also shown. It can be seen that there is good agreement in the magnitude of the rates over the temperature range indicated.

As indicated previously in the description of the test apparatus, a 0.45 µm nylon-membrane filter was placed downstream of the test section to collect any particles which might form in the fuel or break off from the test tube wall during testing. Post-test examination of the filters indicated that varying amounts of grey-to-black material was collected from test to test. No particular pattern was evident either in the amount of deposit or the visual appearance of the filter paper as test conditions were varied. However, there was some indication that more material was collected at the higher test velocities, which would be consistent with increased deposit erosion which might be expected at higher velocities. This conclusion is complicated, however, by the fact that deposit was also found to collect on the inside surface of the flexible hose and intermediate tubing downstream of the test section. Since it was not possible to determine how much of this material was carried over from a previous test, no attempt was made to analyze the material collected on the filter for subsequent correlation with test conditions.

JP-7 Tests

Since significant deposit formation was obtained in the tests with RP-1 fuels, testing proceeded to JP-7 fuel to investigate the effect of utilizing lower sulfur

content fuel on deposit formation rates. A summary of the actual tests which were performed with JP-7 fuel is also shown in Table VII. Prior to each test, the JP-7 fuel was sparged with nitrogen in order to reduce the dissolved oxygen content to less than 10 ppm. Measurements of the dissolved oxygen content were made with a Beckman dissolved oxygen analyzer. It was found that the JP-7 fuel in the as-delivered condition generally contained less than 20 ppm oxygen and that a 30 minute sparging of the fuel was sufficient to reduce the oxygen content to less than 5 ppm. Axial wall temperature distributions obtained with the JP-7 are shown in Fig. 23. In this figure, data are presented for tests at fluid velocities of 6.1, 18.3 and 30.5 m/sec, all with an initial wall temperature of approximately 700K. The temperature distributions obtained with JP-7 are similar to those obtained with RP-1, in that the peak temperature occurs near the tube inlet, and entrance effects which are apparent at the low velocity, disappear as the fluid velocity is increased. One significant difference in these distributions is that the magnitude of the temperature rise noted during the test appears to increase with velocity, whereas it appeared to be independent of velocity for RP-1 fuel (see Fig. 17). Also shown in Fig. 23 is the wall temperature distribution obtained when the test duration was extended to 20 minutes. It can be seen that the increase in temperature observed during the last ten minutes of the test is not appreciably different from that observed after the first ten minutes. If the rate of deposition had remained constant as the test progressed, the deposit thickness would have increased with time and the magnitude of the temperature rise over the last ten minutes would have also increased as the run progressed, suggesting that the rates of deposit formation for JP-7 fuel are not constant with time. The implied decrease in deposit formation rate may also be attributed to deposits breaking off the wall surface after reaching a critical thickness or to passivation of the copper surface as the test proceeded.

The axial temperature distributions obtained as the wall temperature was increased for a fixed fluid velocity of 18.3 m/sec are shown in Fig. 24. Although the results are similar to those obtained with RP-1 fuel; (i.e., significant deposit formation, as evidenced by a wall temperature rise, occurs only after the initial wall temperature was increased to approximately 700K) the magnitude of the temperature rise was appreciably less than that with RP-1. This trend is also shown in the next two figures, Figs. 25 and 26, wherein the axial wall temperature distributions obtained with JP-7 are compared directly to those obtained with RP-1 for the same run conditions. The temperature rises obtained with JP-7 fuel are generally less than those obtained with RP-1 fuel for most of the conditions shown. However, at a fluid velocity of 30.5 m/sec and tube wall temperature of approximately 700K there appeared to be more deposit formation with the JP-7 fuel than with RP-1, as indicated by a general temperature rise along the entire length of tube.

Since the temperature rises obtained during the tests with JP-7 were generally much lower than those obtained with RP-1, it was expected that less deposit would be found with JP-7. When the tubes were sectioned, microscopic examination revealed that there was significant deposit formation with JP-7 and that the deposits appeared darker and more uniform than the RP-1 deposits. This apparent anomaly of the lower temperature rise with test time (inconsistent with the observation of increased deposit rates) may be due to a different character of the deposits formed with the JP-7 fuel. Relatively heavy deposits with JP-7 were not anticipated, due to the reduced sulfur content, and it was hypothesized that the presence of antioxidant and lubricity improving additives, in the absence of dissolved oxygen in the fuel, might have promoted higher rates of deposit formation. Therefore, a test was conducted with unsparged JP-7 fuel (Run No. 32) to determine if dissolved oxygen might affect deposit formation. When the results of this test also indicated heavy deposit formation, additional testing with JP-7 was suspended because the JP-7 did not appear to offer any benefit in terms of increased thermal stability.

Since JP-7 fuel must satisfy a special thermal stability requirement, which is not included in the RP-1 specification, it was assumed that JP-7 would have better thermal stability and; therefore, lower deposit formation at a given test condition than RP-1 fuel. Also, the severe hydrotreatment required to produce JP-7 fuel of sufficient quality to meet the thermal stability specification usually results in production of a fuel containing very low concentrations of sulfur compounds, which are known to promote deposit formation. Examination of previous certificates of analysis for typical batches of the two fuels indicated that the total sulfur content of JP-7 was generally an order of magnitude less than that for RP-1. However, comparison of the certificates of analysis of the RP-1 and JP-7 fuels (Table V) actually tested in this program revealed that the difference in sulfur content between the two fuels was only a factor of 2.3, and not the order of magnitude expected. This smaller difference in sulfur content could contribute to the unexpectedly small difference in deposit rates observed in the experiments.

Furthermore, in order to characterize the actual difference in thermal stability between the two fuels, samples were sent to an industrial laboratory where high-temperature stability of the two fuels was measured with a Jet Fuel Thermal Oxidation Tester (JFTOT) according to ASTM procedure D3241, (Ref. 48). The results of these evaluations, which are shown in Table IX, indicate that not only do the RP-1 and JP-7 both meet the thermal stability specification for JP-7, but that the RP-1 is even more stable than the nominally higher quality JP-7 fuel. For JP-7, the wall temperature that caused deposits which exceeded the thermal stability specification (the breakpoint temperature) was 638K. On the other hand, RP-1 was heated to 653K, the temperature limit for the test apparatus, and the deposits observed were still within the specification limit. Therefore, based on this result, it can be concluded that the particular batches of RP-1 and JP-7 fuels tested in this program were not significantly different, and that there probably would be a slightly lower rate of carbon deposition for RP-1.

Photomicrographs of transverse sections of tubes tested with RP-1 and JP-7 at a fluid velocity of 6.1 m/sec and a wall temperature of approximately 811K are compared in Fig. 27. It can be seen that for this test condition, the deposits obtained with JP-7 appear to be more uniform and heavier than those shown for RP-1 fuel.

Deposit burnoff tests were also conducted on the tube sections obtained from the JP-7 tests. A summary of the results of these tests is given in Table X. Since only a limited number of tests were performed with JP-7, not enough test data was available to graphically indicate the trends with test conditions. However, the tabular data do indicate that the deposit formation rates are generally of the same magnitude as was determined for RP-1. However, unlike the results obtained with the RP-1 fuel there does not appear to be a significant effect of fluid velocity on the deposit rate. This result is also in agreement with the temperature rise indications noted previously in the discussion of the JP-7 tests.

Propane Tests

All deposit formation tests with propane were conducted at a pressure of 136 atm. For commerical-grade propane, test data was obtained for fluid velocities ranging from 6.1 to 36.6 m/sec and for wall temperatures ranging from 422 to 811K. Most of the tests using commerical-grade propane at the higher temperatures (700-811K) had to be terminated prematurely; i.e., before the full ten minute test time was achieved because the wall temperatures near the fluid exit end of the tube fluctuated excessively and eventually exceeded the maximum allowable level (866K). Since this behavior, which will be discussed in detail below, was expected to also occur with chemically-pure propane, subsequent tests with chemically-pure propane were limited to wall temperatures between 422 and 589K. A summary of run conditions for both commercial-grade propane and chemically-pure propane is shown in Table XI.

The tube outer wall temperature distributions at the start of test that were obtained for propane and RP-1 over a range of electrical power input levels are compared in Fig. 28. It can be seen that unlike the results obtained for the distillate fuels RP-1 and JP-7, the wall temperatures observed in the propane tests at the start of the test run exhibited a more or less monotonic increase in temperature from the inlet end to the exit end of the heated tube. This behavior would be expected from a fluid whose local heat transfer coefficient was nearly constant over the range of bulk temperatures from tube inlet to exit. All the required transport properties data for propane are not available at the temperature and pressure of the tests, and therefore, a detailed heat transfer analysis (TCAL) of the variation of wall temperature and heat transfer coefficient along the tube length could not be made. However, low-temperature property data for propane indicates that the expected variation of heat transfer coefficient at higher temperatures would be much less than that shown for RP-1

in Fig. 7, and therefore, the wall temperature distributions obtained with propane are not unexpected. Also the entrance effect which was observed in the RP-1 and JP-7 wall temperature distributions is not apparent in the propane distributions shown in Fig. 28. This result is not surprising since even at the lowest flow velocity of 6.1 m/sec, the flow Reynolds number for propane was substantially higher than that for the RP-1 and JP-7 fuels.

An anomaly which can be seen in Fig. 28, is a sharp increase in wall temperature near the tube exit at the highest power setting $(T_{wall} \simeq 700 \text{K})$. Further, at the higher power settings, the wall temperature readings were observed to be very unsteady with increasing test time. An example of this behavior is shown in Fig. 29, where even though the variation of test conditions with time was small (i.e., < 2 percent) the wall temperature at the tube exit fluctuated over a 200K temperature range. This behavior was consistent and was observed in the majority of tests where the initial wall temperature was set at 700 or 811K. One explanation could be the formation and breakoff of flake-like deposit accumulations. However, this character of deposits is not consistent with the physical nature of the deposits observed on the tube surfaces when the relevant tubes were sectioned and examined. Also, the amount and appearance of the material trapped in the downstream fuel filter was not significantly different from that observed after the low temperature tests or after tests with RP-1 and JP-7. Of the eleven propane tests that were conducted at wall temperatures of 700K and above, ten had to be terminated before the full ten minute test duration was reached because the wall temperature oscillations became so severe that the maximum over-temperature condition of 866K was exceeded. Fluctuations in wall temperature were also observed during many of the tests which were conducted at temperatures of 589K, although they generally were not as severe and did not lead to premature shutdown.

It should be noted that when the test tube wall reaches 589K, the bulk temperature of the propane exceeds the critical point (366K), suggesting that perhaps the temperature fluctuations may be due to a change in character of the propane when the critical temperature is exceeded. Moreover, when the wall temperature exceeded 700K, and severe temperature instabilities were observed, the measured bulk fluid temperature was generally in the range 400 to 500K. In this temperature range, the specific heat of propane at a pressure of 136 atm changes very rapidly with temperature and passes through a maximum at 440K (Ref. 43). Therefore, it would appear likely that other properties of propane such as density, viscosity, and thermal conductivity may also be changing very rapidly and that these properties changes could lead to the unusual heat transfer characteristics that were observed for propane. Instabilities have been observed in other experiments in which propane was flowed through heated tubes, particularly at higher wall temperatures (Ref. 50).

The wall temperature distributions obtained for both grades of propane are not appreciably different and are compared in Figs. 30 and 31. Data obtained for a maximum wall temperature of approximately 422K (Fig. 30) indicate that wall temperature increased from the tube entrance to the exit and the local

temperature measurements were essentially constant over the ten minute test duration. At a maximum wall temperature of 589K (Fig. 31), the wall temperature again increased along the length of the tube, but the local wall temperature varied with time as the test proceeded. In contrast to the test runs conducted with the kerosene fuels, a continuous drop in local wall temperature with increasing test time was frequently noted with the propane fuels, suggesting that deposit formation may have occurred but that the deposits were rough enough to significantly increase the turbulence level in the flow, and thereby, increase the heat transfer to the fuel. The drop in temperature appears to decrease as the velocity increases, or consistent with the hypothesis above, the "rough wall" augmentation in local heat transfer characteristics diminished at the higher Reynolds numbers.

The deposit burnoff data for the propane fuels are tabulated in Table XII. Since most of the high-wall temperature (> 589K) tests with propane were terminated prematurely, the burnoff data could not be used in a graphical presentation of the dependence of the rates of carbon deposition on wall temperature. The rate data shown in Table XII for wall temperatures of 422 and 589K indicate that the deposit rates for both grades of propane fall in the range 400 to $600~\mu g/cm^2$ -hr, and generally overlap. Therefore, the rate data for the two fuels were combined to correlate the rates of deposit formation with fluid velocity. The average rate of deposit formation calculated for each test tube at wall temperatures of 422 and 589K is shown in Fig. 32. Although there is some scatter in the data, the carbon deposition rate for propane fuel appears to decrease slightly with increasing fluid velocity for each wall temperature condition. As is shown in the figure, at low velocities, the deposit rates for a wall temperature of 589K are higher than the deposit rates for a wall temperature of 422K; however, at the higher fluid velocities, no significant difference in the deposit rates is discernible.

Microscopic examination of the deposits obtained with both types of propane indicated heavier, blacker and more uniform deposits than those observed with the kerosene-type fuels, especially at the higher tube temperatures. The carbon deposition rates determined for propane in the burnoff tests confirmed this observation, and the deposit levels are generally higher than those obtained for either of the kerosene fuels at any given tube wall temperature. The carbon deposition rate for propane generally ranged from 400 to 600 $\mu \rm g/cm^2-hr$ over wall temperatures as low as 394 to 533K; whereas comparable rates were not observed for the kerosene fuels until the wall temperatures reached 589 to 700K.

One test with chemically-pure propane, (Run No. 53), was repeated (Run No. 60) but purposely terminated after a duration of 3 minutes to determine the effect of test time on deposition rate. The average deposit rate for the short test was 918 $\mu g/cm^2$ -hr whereas the rate calculated for the full ten minute run was 352 $\mu g/cm^2$ -hr; a decrease in deposition rate by a factor of 2.6. Also, two tests with commercial-grade propane (Run Nos. 33 and 34) were conducted at essentially identical test conditions but for different test durations, i.e., 2 and 5 minutes,

respectively. The burnoff data also indicated a decrease in the deposition rate with time by a factor of 2.7 (i.e., $2021~\mu\text{g/cm}^2$ -hr for the 2 minute test vs. 738 $\mu\text{g/cm}^2$ -hr for the 5 minute test). For the limited number of results obtained it appears that there may be a nearly linear decrease of deposition rate with time and therefore, test duration may be an important factor to consider in deposit rate correlations.

Photomicrographs of representative transverse sections of tubes which were tested with the propane fuels at a velocity of 30.5 m/sec and wall temperatures of 589K are shown in Fig. 33. It can be seen that the deposits are generally thicker and more uniform than those obtained with the kerosene fuels (cf., Fig. 27). Also, it appears that for this test condition, there may be less deposit for chemically-pure propane than for commercial-grade propane.

An interesting phenomenon that was observed in many of the higher wall temperature propane deposits was the appearance of dendritic or tree-like formations in which the deposits appeared to grow out from the copper surface as filaments. This stucture is apparent in Fig. 34. Scanning electron microprobe analysis, discussed in a later section, revealed that the filament composition was primarily copper, with some carbon concentrated at the base of the tree-like structure. The dendrites were quite tenaceous and although they could be removed by hard scraping, they could not easily be brushed or blown from the copper surface.

Filament deposits have been observed in various studies of carbon deposition on metal surfaces and several mechanisms have been proposed to explain filamentary carbon growth (Ref. 51). One explanation is that after a carbon particle is deposited on metallic surface, it diffuses into the bulk metal, either by dissolving in the metal or by diffusion through the surface and grain boundaries, and aggregates at some active sites along grain boundaries to form carbon nuclei. These nuclei grow to microscopic carbon through the continued supply of carbon atoms and push the metal grains out of the substrate. Copper does not dissolve carbon to any extent, but it does form filamentary deposits from acetylene (Ref. 52). These filamentary deposits on copper are quite different from carbon filaments. They grow erratically and once formed, the filaments appear to be reactive toward one another, and sometimes fuse together to form a solid mass in which the original filaments are indistinguishable.

High Pressure Tests

A limited number of tests were conducted with RP-1 fuel at pressures above 136 atm using the modified fuel delivery system described previously. Briefly, this system consisted of two piston type accumulators in series; the first accumulator contained hydraulic fluid on one side of the piston and test fuel on the other side, and the second accumlator (which emptied through the test section) contained test fuel on both sides of the piston (see Fig. 1). With this arrangement, the hydraulic fluid and the test fuel were separated by a fluid volume, thus eliminating possible contamination of the test fuel by any hydraulic fluid left on the wall of the accumulator or by leaking across the piston seals. A high-pressure piston pump was used to supply the accumulator

with hydraulic fluid. A handwheel located on the pump controlled the flow rate of hydraulic fluid through the pump and was set at the appropriate rate prior to initiation of the test. Pressure was regulated by bypassing excess hydraulic fluid through a pressure-relief bypass. During testing with this high-pressure system, it was found that the flow rate of hydraulic fluid through the pump tended to drift during the test. Therefore, compared to the low-pressure system, more frequent adjustment of the flow rate through the test section and the electrical power into the test tube was required to maintain the desired fuel velocity and wall temperature.

A summary of the run conditions for the high-pressure tests is shown in Table XIII. The tube wall temperature distributions obtained at pressures of 136, 204, and 340 atm are presented in Fig. 35. As can be seen in the figure, there are some differences in the wall temperature distributions and temperature rises obtained at each pressure. Much of these differences can be attributed to the variations in the test conditions, noted on the figure, which occurred because of the difficulties discussed above. Therefore, it would appear that there is no significant change in the wall temperatures as a consequence of increasing pressure, suggesting that the rate of deposit formation is relatively independent of pressure over the pressure range of 136 to 340 atm. Microscopic examination of the deposits obtained from these tests also revealed that there was no substantial difference in deposit appearance over the range of pressures tested; that is, the deposits were generally brownish-red in color, occasionally intermixed with black and/or grey-metallic streaks or specks.

A summary of the deposit burnoff data for the high-pressure tests is presented in Table XIV, and the overall rates of carbon deposition which are plotted in Fig. 36 fall within a relatively narrow band. A least squares fit of this data produced the straight line shown in the figure and indicates that the deposit formation rate increases slightly with pressure.

Tests With Nickel-Plated Tubes

Copper is prevalent in rocket engine cooling systems because of its superior thermal conductivity. However, previous experimental studies of the effect of wall materials on deposit formation, Refs. 6, 10, and 32, have indicated that carbon deposit rates on copper can be very high. Also, the results of the present experiments for kerosene fuels and propane fuels discussed in the previous sections appear to corroborate and extend the findings of the earlier experimental studies. It would appear that the copper surface probably promotes deposit formation to as great an extent as any deposit forming precursor contained in the fuels tested. Therefore, in order to obtain an indication of the importance of the tube wall material on deposit formation, five tests were conducted using tubes in which the inside (copper) surface had been plated with nickel by means of an electroless process. All of the tests were run using RP-1 fuel at a pressure of 136 atm. A summary of the test conditions is shown in Table XIII. The tube wall temperature distributions obtained with the nickel-plated tubes are compared with those obtained with the

copper tubes in Fig. 37. It can be seen from the figure that the temperature distributions are very similar and that the temperature rise obtained with the nickel-plated tube is significantly less than that obtained with copper, suggesting a substantial decrease in deposit formation.

The tubes used in the nickel-plated tube tests were sectioned and subjected to microscopic examination and deposit burnoff measurements. Initial microscopic examination of the virgin tubing indicated that the appearance of the nickel surface varied from rough granular to smooth fine-grained, perhaps due to variations in plating solution concentrations stemming from lack of solution agitation/circulation or solution instability. However, because of the differences in grain size and color as viewed through the microscope, (varied from black to metallic grey), it was difficult to distinguish black carbon deposits. Photomicrographs of transverse sections of the test tubes are shown in Fig. 38. They indicate that the nickel coating was generally of good quality and of uniform thickness (.0002-.0005 cm). These photomicrographs (at 500X magnification) do not indicate any significant deposit formation.

A scanning electron microprobe analysis was made of the five sections taken from the tube which was tested at a wall temperature of 700K and fluid velocity of 6.1 m/sec (the test condition which gave the highest deposition rate on copper). Photomicrographs taken at a magnification of 1600X revealed no deposits, and an elemental analysis indicated the presence of nickel and phosphorus (the major constituents in the electroless plating solution) but no copper, carbon, oxygen or sulfur. Also, the results of the burnoff tests of the sectioned nickel-plated tubes, presented in Table XV, indicate that very little material was deposited on the nickel surface during testing. Since the average rates of deposition ranged from 40 to 80 $\mu g/cm^2$ -hr, it can be concluded that a substantial decrease in deposit formation occurred when the copper tubes were replaced with nickel-plated tubes.

Deposit Morphology

In order to characterize the complex structure of the deposits observed during this program, a scanning-electron microscope (SEM) was used to study the deposits. The SEM is particularly useful for examining solid specimens whose surface structures are rough, because it has considerably greater depth of focus than a conventional reflected-light microscope. As an aid in interpreting the SEM photomicrographs obtained in this program, a brief discussion of the morphology of the carbonaceous material (Ref. 53) is useful. Carbon lends itself to the formation of stable complex solids because of its chemical valance of four and its readiness to combine with itself and with other atoms such as hydrogen, nitrogen, oxygen, and sulfur. In such carbonaceous materials, the carbon atoms are bound together by strong covalent bonds to form the main units of molecular structure which can consist of chains or rings or both. Molecules with random three-dimensional networks and no particular microstructure may also be formed.

When viewed with a SEM, amorphous materials, such as asphalt, show no particular form because the molecular structure lack any order. On the other hand, crytalline materials, such as graphite have sharp geometric outlines because both atoms and molecules are arranged in a very high degree of three-dimensional order. Carbonized materials such as coke, however, are distinctly different from other carbonaceous materials. While they are neither amorphous or crystalline, these carbon residues do have a limited organization and orientation which gives them characteristic features. Generally, the carbon atoms tend to group together in tightly packed aggregates of spherical particles.

A SEM analysis was performed on four samples of deposit, one for each fuel, which were obtained in tests conducted with copper tubes (Run No. 3, 29, 40, and 55). SEM photomicrographs of the inside wall of a test tube at locations near the tube entrance (1.9 cm), in the middle (12.1 cm) and near the exit end (21.2 cm) are shown in Figs. 39 through 42. For reference, a photomicrograph of the surface of an unused tube is shown to the left in each figure.

The microstructure of RP-1 deposits (tube wall temperature of 700K, velocity of 7.2 m/sec-Run No. 3) is shown in Fig. 39. Near the entrance of the tube (1.9 cm), there appears to be a continuous film of deposit on the surface of the copper substrate. At higher magnification, it can be seen that the deposit is made up of agglomerated particles, which are very small ($\sim 0.5~\mu m$) and spherical in shape. In the photomicrographs taken at the 12.1 cm location, the microstructure of tightly packed aggregate of spherical particles is more evident. It appears that the top film of deposit fractured, exposing a highly-fused substrate which has been overlayed with the tightly packed spherical agglomerates. The photomicrograph taken at a location near the exit of the tube reveals another type of microstructure that has a more vitreous and amorphous appearance. Again, it appears that fracture of the deposit had taken place and the fused substrate is evident.

SEM photographs of JP-7 fuel deposits (tube wall temperature of 700K, velocity of 17.3 m/sec-Run No. 29) are shown in Fig. 40. Again, the microstructure of tightly packed spherical agglomerates typical of coke deposits is evident. In the photograph taken near the entrance of the tube, there are areas where the deposits appear to be more flocculent and porous. The photograph taken of the section from the middle of the tube shows a knobby surface which appears to have been formed by fusion of the spherical particles. The photograph taken at the end of the tube indicates a deposit which is vitreous in appearance. Not as much deposit fracturing is evident as was observed in the SEM analysis of the RP-1 deposits. The JP-7 deposits also appear to be generally more uniform than the RP-1 deposits.

The microstructure of commercial-grade propane deposits (wall temperature of 700K, velocity of 30.5 m/sec-Run No. 40) is shown in the SEM photomicrograph presented in Fig. 41. The sections near the entrance and at the middle of the tube show the typical dendritic formation. The dendrites were dispersed randomly along the entire length of the tube. Close-ups of these

areas do not show the aggregates of spherical particles that were observed with the kerosene type fuels and that are characteristic of coke formation; The highly-magnified photos of the dendrites show a distinctly smooth, finger-like structure. The microstructure of the deposits at the exit of the tube where the temperature was highest, is more dendritic in appearance and the finger-like deposits are more uniform and cover larger areas of the tube rather than being randomly clustered.

The microstructure of deposits obtained with chemically-pure propane (wall temperature of 589K, velocity of 18.3 m/sec-Run No. 55) is shown in Fig. 42. It can be seen that the dendritic formation is not as obvious at this wall temperature condition and that clusters of packed particles are spread across the length of the tube. These clusters appear to overlay a fused layer of deposits and are made up of packed spherical particles similar to the microstructure of the deposits obtained with the kerosene fuels. A SEM analysis was not made of deposits obtained in tests with commercial-grade propane at low wall temperatures. However, samples of deposits from tests with both grades of propane at various test conditions were examined microscopically at magnifications up to 400X and no apparent differences in deposit appearance was discerned.

These photomicrographs, shown in Figs. 39 through 42, indicate that the deposits accumulated during the heated tube tests are generally not formed on the tube surfaces as smooth, continuous films of uniform structure and composition. Instead, they indicate that discrete particles, spherical or dendritic in shape, accumulate over a fused substrate to produce a highly variable three-dimensional structure. The deposit surface appears to be sufficiently rough to significantly increase turbulence and thereby affect heat transfer. However, there also appears to be areas of the tube in which the surface has become smoother during deposit formation. From the SEM photomicrographs, it is obvious that surface roughness and deposit homogeneity can be expected to change along the length of a test tube and may significantly affect the local heat transfer characteristics.

As indicated in the discussion of the results obtained in tests with nickel-plated tubes, it was difficult to visually identify any deposits on the nickel surface using the relatively low-magnification reflected-light microscopes. A SEM analysis was made of a nickel-plate tube which has been tested with RP-1 fuel at conditions which gave substantial deposits in copper tubes (wall temperature of 700K, velocity of 6.1 m/sec-Run No. 70). The SEM photomicrographs of the surface of this tube are shown in Fig. 43. The photomicrograph shown at the extreme left shows the microstructure of a virgin tube and indicates a corncob appearance which is typical of an electroless-plated surface. The photomicrograph taken of the surface of the test tube near the fluid entrance end (1.9 cm) shows no appreciable difference in appearance from the unused tube. There is evidence of some deposit-like structure at the middle (12.1 cm) and near the end (21.2 cm) of the test tube, but they appear to be more random and widely dispersed than the deposits obtained in the kerosene fuel and propane tests.

A limited qualitative elemental analysis of the deposits was made utilizing a Scanning Electron Microprobe (SEMP). The SEMP incorporates an x-ray energy-dispersive spectrometer (EDS) which produces a qualitative scan of elements which are present in the deposit within a detectability limit of approximately 200 ppm. Because of poor sensitivity for elements with atomic numbers less than twelve, the EDS scan excludes likely constituents of deposits such as hydrogen, carbon, oxygen, and nitrogen. Also, x-ray emission from a thin conducting layer of gold, which coats the specimen to prevent charging, may mask the emission from elements such as sulfur. However, a selective wavelength spectrometer can be used with the SEMP which will allow characteristic x-ray mapping of selected elements. An image of the x-ray emission for the selected element is produced and matches exactly the standard photomicrograph of the sample. The presence of the element is indicated by clusters of white dots on a dark background to allow easy identification of the areas of local concentration of the particular element.

A SEMP analysis was made of samples from the same five test tubes which had undergone SEM analysis. The EDS scans of these samples indicated that besides the gold overlay and the parent materials in the inner tube surface; i.e., copper and nickel/phosphorus, other elements such as silicon, aluminum, potassium, calcium, and chlorine were present. It is believed that these materials were introduced during the cleaning and polishing required in the sample preparation for the SEMP analysis.

The results of the selected-wavelength analysis made with the SEMP are shown in Fig. 44. In the figure, the upper series of photographs correspond to a tube deposit obtained with JP-7 fuel. The first photograph shows a SEM photomicrograph of the deposit sample. This region was selectively scanned for the presence of copper, carbon, oxygen and sulfur and, if present, these elements would be indicated by an agglomeration of white dots against the dark background. It can be seen that the copper surface of the tube is clearly outlined but that no significant copper is contained in the area occupied by the deposit. The deposit, however, contains a heavy concentration of carbon and a smaller concentration of sulfur. The SEMP analysis of RP-1 fuel deposits gave essentially the same result as the JP-7 analysis except that a small concentration of oxygen was also present.

The bottom set of photographs represent a SEMP analysis of the microstructure of deposits obtained with propane at a high tube wall temperature (~700K) condition and shows a typical dendrite formation. It can be seen that the dendrite contains a high concentration of copper, suggesting that tube material was forced up and away from the surface. Most of the carbon image results from that contained in the potting material but some carbon is also evident at the base of the tree-like structure. A very small concentration of sulfur is also indicated but no oxygen was observed. A less detailed SEMP analysis of a tube deposit obtained with

chemically-pure propane at a lower wall temperature (~589K) was also performed (not shown in the figure). No obvious dendritic formations were observed in this particular sample although the deposit material contained significant amounts of copper, the presence of which may account for the observed decrease in wall temperature with increasing test time (see Fig. 31). However, the carbon content of the deposit was substantially more than that observed in the dendritic structure.

A SEMP analysis was also performed on five separate areas along the length of a nickel-plated tube which was tested at a wall temperature of 700K with RP-1 flowing at a velocity of 6.1 m/sec (the test condition which gave the highest deposition rate with copper). Photomicrographs at a magnification of 1600X revealed no deposits and an elemental analysis indicated the presence of nickel and phosphorus (the major constituents in the electroless plating solution) but no copper, carbon, oxygen, or sulfur.

CONCLUDING REMARKS

The thermal decomposition (coking) limits and rates of deposition in heated copper tubes for two standard hydrocarbon rocket fuels, RP-1 and commercial-grade propane, have been investigated. In addition, tests were conducted using decoxygenated JP-7 and chemically-pure propane as representative of more refined cuts of the standard fuels to determine the effect of improving thermal stability by reducing the concentration levels of deposit-forming precursors. The apparatus developed for these tests permitted independent variation and control of tube wall temperature, fluid pressure, and fluid velocity in order that the effects of each parameter could be investigated independently.

The results of the experiments with RP-1 fuel were as expected, in that there was previous evidence that copper promotes deposit formation in kerosene-type fuels. However, the relatively high deposition rates of between 400 and 600 $\mu g/cm^2-hr$ at temperatures of 500 to 800K for only a ten minute test duration were not anticipated. Peak deposit formation occurred near 700K, which is consistent with results obtained with kerosene-type aviation fuels. The deposit coverage was generally non-uniform and ranged from specks, to connected islands of deposits, to essentially full coverage. No particular pattern could be established with test conditions and the non-uniformity of deposit coverage made a determination of the point of incipient deposit formation impossible.

It was believed that JP-7, with a lower sulfur content (typically an order of magnitude lower) and a stringent thermal stability specification, would be a good simulator of refined quality RP-1 and demonstrate improved thermal stability. However, no benefit in terms of increased stability was realized with JP-7, since the results of tests indicated heavier deposit formation even though lower tube temperature rises were observed. Subsequent evaluation of the actual difference in the thermal stability between the two fuels was made in a series of tests using a Jet Fuel Thermal Oxidation Testor (JFTOT) to determine the breakpoint temperature. The results of this evaluation revealed that both the JP-7 and the RP-1 met the thermal stability specification for JP-7, and the RP-1 was even more stable than the normally high quality JP-7 fuel. Also, certified analyses of the two fuels indicated that the actual difference in sulfur contents was only a factor of two, and not the order of magnitude expected. Therefore, it can be concluded that there is little difference in carbon deposition rates and probably no particular advantage in using JP-7.

Another unexpected result of the study was that deposits obtained with propane fuels were heavier, blacker and more uniform than those observed with the kerosene-type fuels and there appeared to be little difference between commercial-grade and chemically-pure propane with regard to type and quantity of deposit. The carbon deposition rates for the propane fuels were generally higher than those obtained for either of the kerosene fuels at any given wall temperature. Two interesting phenomena were observed during testing with propane. The first phenomenon consisted of

unusual wall temperature instabilities whenever the bulk fluid temperature exceeded the critical temperature of propane. These temperature fluctuations were especially severe at wall temperature of 700K and above. For those wall temperatures, the bulk fluid temperature was in the range of 400 to 500K and corresponded to a temperature regime in which the specific heat of propane is known to vary rapidly with small changes in temperature. It is likely that other properties of propane, such as density, viscosity, thermal conductivity, etc. may also be changing rapidly and that these properties changes lead to unusual heat transfer characteristics above the critical temperature. The second phenomenon was observed in most of the deposits recovered from tests conducted with propane at the higher wall temperatures. These deposits contained dendritic or tree-like formations which appeared to grow out from the copper surface as filaments. The filament composition was primarily copper, with some carbon concentrated at the base of the tree-like stucture.

Results of tests with RP-1 over a range of pressures from 136 to 340 atm revealed that there was no substantial difference in deposit appearance; that is, the deposits were generally brownish-red in color, occasionally intermixed with black and/or gray-metallic streaks or specks. Also, there was only a very slight increase in deposit formation rate with pressure. For both RP-1 and propane fuels, the rate of deposition appeared to decrease with increasing fluid velocity. However, for RP-1, wall temperatures above 700K, the rate of carbon deposition appeared to reverse trend and increased with increasing flow velocity.

Copper was specified for the tube inner surface in this experimental program because of its superior thermal conductivity, even though previous experimental studies had shown that deposit rates on copper can be very high. On the other hand, nickel, which has good thermal conductivity, did not appear to promote deposit formation with kerosene-type jet fuels. This conclusion was corroborated by the results of tests conducted in this experimental program which indicated a substantial reduction in deposit-formation when copper tubes were replaced with nickel-plated tubes.

Another observation made during the performance of this test program was that the deposition rate appeared to change with time and, therefore, test duration is an important factor to consider in deposit rate correlations. Also, post-test photomicrographic examination of the tube surfaces indicated that the deposits were generally not formed as smooth, continuous films of uniform structure and composition. Discrete particles, spherical or dendritic in shape, accumulated over a fused substrate to produce a highly variable three-dimensional microstructure. The deposit surface appeared to be sufficiently rough to significantly increase turbulence and thereby affect heat transfer. However, there were instances where the surface became smoother

during deposit formation. It was obvious from the photomicrographic analysis of the deposits that surface roughness and deposit homogeneity can be expected to change along the length of a test tube and may significantly affect the local heat transfer characteristics.

The results obtained in this experimental study suggest areas in which further work may be desirable to more firmly establish the trends observed. The result of the JFTOT analysis indicated that RP-1 fuel is very stable relative to standard jet fuels. Since it is known that deoxygenation of fuel significantly improves its thermal stability, additional parametric tests for de-oxygenated RP-1 are desirable to determine the minimum coking limits and deposition rates on copper. Also, since the substitution of the copper tubes with the nickel-plated tubes resulted in a substantial reduction in deposit formation, several different candidate high-thermal-conductivity tube materials (e.g., nickel, gold or silver platings, copper/nickel alloys, etc) should be investigated to determine their effect on deposit formation with RP-1 fuel. Furthermore, because the different techniques employed in manufacturing the regenerative cooling passages of high-pressure rocket thrust chambers result in surfaces of varying degrees of roughness, tests should be conducted to determine if there is an effect of surface roughness on deposit formation.

Finally, some of the trends observed in the present experimental study were not conclusive and therefore the range of test conditions should be extended to permit evaluation of the effects of run time, periodicity, and increased velocity.

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TABLE I
Fuels Contaminant Specification

<u>RP-1</u>		Propane	
Oxygen	5 ppm by wt.	Oxygen	5 ppm by wt.
Sulfur	report	Sulfur	report
Sulfur Compounds	report	Sulfur Compounds	report
Nitrogen	0.30 wt%	Carbon Dioxide	0.10 vo1%
Water	10 ppm by wt.	Ethane	0.30 vo1%
		Nitrogen	0.30 wt%
		Water	10 ppm by wt.

TABLE II

Typical Physical Properties of Test Fuels

	д 1-дд	TP-7	Chemically	Commercial
	T IN		ruie riopane	riopane
Molecular weight	167	175	44.1	44.7
Freezing point, deg K	< 235	250	85	< 135
Mean boiling point (MBP), deg K	787	490	231	233
ASTM distillation, deg K				
IBP	429	461	!	}
10%	697	476	i	}
20%	787	489	ł	ļ
206	508	513	ŀ	}
FBP	529	534	!	1
Specific gravity @ 289K	0.801	0.803	0.508	0.513
Critical temperature, deg K	999	670	370	370
Critical pressure, atm	21.7	20.8	42.0	41.9
Critical volume, cm ³ /gm	2.06	4.18	4.62	4.24
Heat of vaporization @ MBP, cal/gm	56.5	62.1	102	102
Heat of fusion, cal/gm	1	}	19.2	!
Heat of formation, cal/gm	-4 40	-430	-664	-620
Heat of combustion, cal/gm	10457	10486	11054	11045
Autoignition temperature, deg K	523	!	741	!
Flammability limits in air, vol. %	!	!	2.2-9.5	2.2-9.5
Flash point, deg K	337	336	ţ	1
Specific heat @ 298K, cal/gm-k	.477	787.	0.785	0.780
Thermal conductivity @ 298K, milliwatts/cm ^{2-k} /cm	0.880	0.770	0.090	0.093
Viscosity @ 298K, centipoise	1.56	1.47	0.12	0.13
Vapor pressure @ 311K, atm	4.1x10-4	3.4×10^{-4}	12.6	12.3

TABLE III

Temperature Dependence of Selected Physical Properties of RP-1 and JP \cdot 7

			RP-1					JP-7		
Temperature	Specific Heat	Density	Vapor Pressure	Viscosity	Thermal Conductivity	Specific Heat	Density	Vapor Pressure	Viscosity	Thermal
Deg K	Cal/gm-K	gm/cc	atm	Centipoise	mW/cm ² -K per cm	Cal/gm-K	gm/cc	atm	Centipoise	mW/cm ² -K
300	0.477	0.801	1.00 x 10 ⁻⁴	1.563	0.885	0.484	0.803	8.85 x 10 ⁻⁵	1.468	0.777
325	0.503	0.782	9.66 x 10 ⁻⁴	1.087	0.911	0.510	0.786	8.51×10^{-4}	1.091	0.800
350	0.527	0.763	5.87×10^{-3}	0.752	0.942	0.534	0.769	5.17×10^{-3}	0.798	0.827
375	0.563	0.744	0,016	0.542	0.967	0.560	0.750	0.014	0.579	0.848
400	0.577	0.724	0.048	0,426	0.974	0.586	0.727	0.042	0.442	0.855
425	0.603	0.704	0.127	0.351	0.982	0.612	0.702	0.112	0.355	0.862
450	0.628	0.682	0.297	0.298	0.971	0.637	0.681	0.262	3.285	0.853
475	0.653	099.0	0.653	0.256	0.958	0.663	0.655	0.575	0.235	0.841
200	0.679	0.637	1.251	0.223	0.942	0.687	0.631	1.102	0.199	0.827
525	0.705	0.612	2.124	0.190	0.922	0.717	909*0	1.871	0.167	0.810
550	0.732	0.583	3.330	0.174	868.0	0,740	0.577	2.934	0.142	0.788
575	0.758	0.552	5.261	0.157	0.871	0.767	0.551	4.635	0.121	0.765
009	0.784	0.515	7.963	0.136	0.837	0.793	0.513	7.016	0.101	0.735
625	0.812	0.472	11.59	0.121	0.798	0.822	0.468	10.21	980*0	0.701
650	0.837	0.405	16.61	0.104	0.730	0.848	0.404	14.63	690.0	0.641

TABLE IV Selected Physical Properties of Propane at Saturation

			Commercial	11				Chemically-Pure	.y-Pure	
Temperature	Specific Heat	Density	Vapor Pressure	Viscosity	Thermal Conductivity	Specific Heat	Density	Vapor Pressure	Viscosity	Thermal Conductivity
Deg K	Cal/gm-K	gm/cc	atm	Centipoise	mW/cm ² -K per cm	Cal/gm-K	co/mg	atm	Centipoise	mW/cm ² -K per cm
100	0,462	0.751	2.00×10^{-7}	00*9	2.47	097*0	0.745	2.36×10^{-7}	3.60	2.45
120	0.467	0.707	2.52×10^{-5}	1,810	2.21	997*0	0.701	2.95×10^{-5}	1.48	2.19
140	0.474	0.684	6.51 x 10 ⁻⁴	098.0	2.03	0.474	0.678	7.60×10^{-4}	0.841	1.98
160	0.482	0.664	7.00×10^{-3}	0.549	1.86	0.482	0.658	8.14×10^{-3}	0.532	1.83
180	0.492	799.0	4.26×10^{-2}	0.387	1.68	0.493	0.637	4.88×10^{-2}	0.374	1.66
200	0.504	0.622	0.174	0.290	1.53	905*0	0.615	0.197	0.281	1.50
220	0.520	0.602	0.536	0.225	1.39	0.522	0.593	0.595	0.219	1.36
240	0.575	0.578	1.337	0.180	1.25	0.572	0.570	1,461	0.176	1.23
260	0.630	0.552	2.854	0.145	1.13	0.632	0.546	3.071	0.143	1.11
280	0.702	0.527	5.390	0.119	1.02	0.705	0.519	5.752	0.117	1.00
300	0.789	0.497	9.296	660.0	0.94	0.792	0.489	9.866	0.095	0.91
320	0.919	0.461	14.93	0,081	0.85	0.922	977.0	15.82	0.077	0.82
340	1,115	0.419	22.75	0.063	0.77	1.120	0.412	24.04	090.0	0.73
360	-	0,351	41.52	0.043	0.67	-	0.345	35.08	0.042	0.66

TABLE V

Certified Analyses of RP-1 and JP-7 Fuels

TABLE VI

Typical Chemical Properties of Propane

Typical Analysis, Vol %	Commercial	Chemically Pure
Propane Propylene	> 90.0 < 5.0	99.39 0.01
Ethane N-Butane	< 5.0	0.05 0.05
I-Butane Sulfur, Wt %	< .015	0.50

Wall Temp.	Velocity	Test Duration
deg K	m/sec	min
	RP-1 Fuel	
		10
		10
		10
811	7.2	1
589	14.4	10
700	14.4	10
811	14.4	10
811	21.6	9
811	7.2	10
700	21.6	10
589	21.6	10
700	28.8	8
Varied	Varied	-
811	7.2	10
811	24.4	1
811	24.4	6
589	24.4	8
589	30.5	10
700	30.1	10
811	30.1	10
700	24.4	10
589	24.4	10
Varied	6.1	_
	JP-7 Fuel	
700	6.1	\$
811	6.1	10
811	18.3	10
700	18.3	20
811	30.5	10
700	30.5	12
811	12.2	7
589	18.3	10
700	30.5	10
	deg K 422 589 700 811 589 700 811 811 700 589 700 Varied 811 811 589 589 700 811 700 589 Varied 700 811 700 811 700 811 700 811 700 811 700 811 700 811 700	RP-1 Fuel 422

*Calibration Run

†NASA tube

TABLE VIII

Deposit Burnoff Data for RP-1 Fuel

						
Average Deposit Rate µg/cm ² -hr	630	977	429	523	186	314
Average Wall Temperature Deg K	572	628	550	634	716	662
Deposit Rate µg/cm²-hr	647 655 588	662 393 284	493 502 275 446	244 526 799 	130 226 161 225	362 244 371 277
Deposit Weight mg	.295	.287 .207 .131	.217 .226 .121 .200	.109	.061 .099 .070	.162
Section Surface Area cm ²	2.74 2.68 2.68 2.68	2.60 2.74 2.77 2.77	2.64 2.70 2.64 2.70	2.70 2.75 2.72 2.72	2.77 2.64 2.72 2.77	2.70 2.64 2.64 2.64
Initial Wall Temperature Deg K	601 613 545 530	672 661 605 572	573 568 534 526	685 633 616 603	800 711 686 666	694 661 650 642
Velocity m/sec	7.2	7.2	14.6	14.6	14.6	21.6
Run Time min	10	10	10	10	10	10
Run-Section	2 - A B C	3 - B B C C	5 - A B C	6 A C C C C C C C C C	7 – A B C	10 - A B C

TABLE VIII (Continued)

Average Deposit Rate Hg/cm ² -hr	320	186	355	188	241	371
Average Wall Temperature Deg K	634	726	770	560	634	734
Deposit Rate µg/cm ² -hr	269 350 339 322	180 193 212 161	502 304 360 255	212 192 147 200	207 246 243 267	191 564 358
Deposit Weight mg	.057 .124 .119	.083 .087 .094 .074	.137 .080 .097	.099 .084 .067	.098	.098
Section Surface Area cm ²	2.70 2.66 2.64 2.72	2.77 2.72 2.66 2.74	2.79 2.70 2.75 2.68	2.79 2.64 2.74 2.74	2.81 2.72 2.77 2.79	2.77 2.82 2.82 2.79
Initial Wall Temperature Deg K	655 627 623 633	822 744 686 650	801 769 755 753	569 557 561 554	661 630 619 625	766 733 716 722
Velocity m/sec	28.8	7.2	24.5	30.5	30.1	30.1
Run Time min	∞	10	9	10	10	10
Run-Section	12 – A B C D	14 – A B C D	16 - A B C D	18 – A B C C	19 – A B C D	20 - A B C D

TABLE VIII (Continued)

1	Average Deposit Rate µg/cm ² -hr	474	366
	Average Wall Temperature Deg K	631	557
	Deposit Rate µg/cm ² -hr	408 592 471 425	607 201 243 413
	Deposit Weight mg	.192 .272 .222 .194	.277 .091 .112
	Section Surface Area	2.83 2.75 2.83 2.74	2.74 2.70 2.75 2.70
	Initial Wall Temperature Deg K	648 633 623 620	574 558 550 545
	Velocity m/sec	24.4	24.4
	Run Time min	10	10
	Run-Section	21 - A B C D	22 – A B C D

TABLE IX

Results of JFTOT* Evaluation

Temperature	Pressure Drop	Deposit	Rating
Deg K	mm Hg	Visual	TDR
633	25	< 3	< 12
628	0	1	5
633	0	1	6
638	0	3	12
628	0	1	2
638	0	1	4
648	0	1	6
653	0	1	7
	Deg K 633 628 633 638 628 638 648	Deg K mm Hg 633 25 628 0 633 0 638 0 628 0 638 0 648 0	Deg K mm Hg Visual 633 25 < 3 628 0 1 633 0 1 638 0 3 628 0 1 638 0 1 648 0 1

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^{*}ASTM D3241-27 - Standard Test Method for Thermal Oxidation Stability of Aviation Turbine Fuel (JFTOT Procedure)

TABLE X

Deposit Burnoff Data for JP-7 Fuel

Average Deposit Rate µg/cm ² -hr	. 470	797	267	172	451	378
Average Wall Temperature Deg K	616	719	725	664	771	656
Deposit Rate µg/cm ² -hr	389 698 461 332	332 278 424 820	251 264 234 319	116 171 232 167	315 636 419 435	233 222 608 447
Deposit Weight mg	.152 .270 .196	.141 .129 .185	.106 .114 .099 .131	.099 .149 .201	.135 .288 .186	.119 .111 .304
Section Surface Area	2.60 2.58 2.83 2.62	2.54 2.79 2.62 2.79	2.54 2.58 2.54 2.47	2.56 2.68 2.60 2.60	2.56 2.71 2.66 2.85	2.60 2.54 2.54 2.58
Initial Wall Temperature Deg K	675 609 603 578	813 735 676 651	769 726 703 702	700 666 650 642	806 759 759 760	665 653 653 655
Velocity m/sec	6.1	6.1	18.3	18.3	30.5	30.5
Run Time min	6	10	10	20	10	12
Run-Section	24 - A B C D	25 - A B C C	26 - A B C D	27 – A B C D	28 - A B C C	29 - A B C D

TABLE X (Continued)

Average Deposit Rate µg/cm ² -hr	408	345	352
Average Wall Temperature Deg K	715	557	620
Deposit Rate µg/cm ² -hr	420 226 557 429	414 322 276 368	364 386 367 289
Deposit Weight mg	.129 .071 .175	.177 .145 .121	.162 .167 .162 .123
Section Surface Area	2.64 2.70 2.62	2.56 2.70 2.64 2.64	2.68 2.60 2.64 2.54
Initial Wall Temperature Deg K	794 703 675 686	568 552 550 557	631 604 606 639
Velocity m/sec	12.2	18.3	30.5
Run Time min	7	10	10
Run-Section	30 - A B C C	31 - A B C D	32 - A . B C

TABLE XI

Summary of Test Conditions for Propane Fuels

Pressure = 136 atm

Run No.	Wall Temp.	Velocity	Test Duration
	deg K	m/sec	min
	Commercial-Grad	e Propane	
33	700	6.1	2
34	700	6.1	5
35	700	18.3	5
36	589	18.3	10
37	589	6.1	10
38	422	6.1	10
39	589	30.5	9
40	700	30.5	10
41	811	30.5	1.
42	422	36.6	10
43	422	18.3	10
44	811	18.3	10
45	811	6.1	5
46	700	12.2	10
47	811	12.2	10
48	700	24.4	5
49	589	12.2	10
50	5 89	24.4	10
51	422	12.2	10
52	422	24.4	10
	Chemically-Pur	e Propane	
53	589	6.1	10
54	422	6.1	10
55	589	18.3	10
56	589	30.5	10
57	422	30.5	10
58	422	18.5	10
59	700	6.1	8
60	580	6.1	3

TABLE XII

Deposit Burnoff Data for Propane A. Commercial Grade Propane

Average Deposit Rate ug/cm ² -hr	2021	738	661	518	306	450
Average Wall Temperature Deg K	586	638	582	530	556	391
Deposit Rate µg/cm ² -hr	1755 1581 2191 2556	850 704 642 758	582 700 645 717	373 474 414 809	361 429 204 231	411 468 422 500
Deposit Weight mg	.114 .106 .144 .171	.194 .156 .142 .174	.115 .144 .123	.166 .212 .178 .369	.152 .193 .090	.181 .209 .188 .226
Section Surface Area	2.60 2.68 2.62 2.68	2.68 2.60 2.60 2.70	2.64 2.74 2.54 2.74	2.68 2.68 2.58 2.74	2.53 2.70 2.64 2.72	2.64 2.68 2.60 2.72
Initial Wall Temperature Deg K	511 543 624 666	596 631 653 673	575 571 577 603	493 516 541 570	513 535 553 623	368 383 387 426
Velocity m/sec	6.1	6.1	18.3	18.3	6.1	6.1
Run Time min	7	Ŋ	'n	10	10	10
Run-Section	33 - A B C C	34 - A B C D	35 - A B C D	36 - A B C C	37 – A B C D	38 - A B C D

TABLE XII (Continued)

Average Deposit Rate µg/cm ² -hr	439	995	3462	318	354	340	920
Average Wall Temperature Deg K	514	594	539	392	392	624	675
Deposit Rate µg/cm ² -hr	346 381 482 548	324 316 617 1005	2800 2870 3870 4310	296 362 238 377	260 201 142 814	222 324 282 531	1185 630 922 943
Deposit Weight mg	.138 .156 .190	.149 .140 .279 .464	.127 .126 .175	.130 .160 .105	.117 .092 .062 .384	.100 .140 .126	.264 .141 .200 .213
Section Surface Area cm ²	2.66 2.74 2.62 2.77	2.75 2.66 2.72 2.77	2.72 2.63 2.72 2.70	2.64 2.66 2.64 2.63	2.71 2.73 2.60 2.83	2.70 2.58 2.68 2.71	2.68 2.68 2.60 2.70
Initial Wall Temperature Deg K	489 498 523 548	593 583 587 612	506 518 544 588	380 385 397 408	379 385 396 408	661 600 610 624	619 620 674 787
Velocity m/sec	30.5	30.5	30.5	36.6	18.3	18.3	6.1
Run Time min	6	10	П	10	10	10	5
Run-Section	39 – A B C C	40 - A B C D	41 - A B C D	42 - A B C D	43 – A B C D	44 - A B C	45 - A B C D

TABLE XII (Continued)

Average Deposit Rate µg/cm²-hr	780	733	1272	571	616	424	454
Average Wall Temperature Deg K	650	569	592	519	539	393	389
Deposit Rate µg/cm ² -hr	464 798 1088 770	811 529 742 851	1887 826 1231 1145	576 450 736 522	593 617 486 770	404 406 442 443	572 549 349 348
Deposit Weight mg	.201 .349 .455	.360 .263 .309	.401 .183 .260	.259 .196 .324 .229	.263 .269 .211 .356	.182 .186 .199	.255 .241 .158
Section Surface Area	2.68 2.70 2.69 2.66	2.66 2.68 2.58 2.70	2.62 2.73 2.60 2.68	2.70 2.62 2.64 2.64	2.66 2.62 2.60 2.77	2.70 2.75 2.70 2.73	2.68 2.64 2.71 2.68
Initial Wall Temperature Deg K	659 681 609 649	537 548 565 626	588 581 584 615	501 503 521 550	495 530 560 572	380 387 396 408	376 383 393 405
Velocity m/sec	12.2	12.2	24.4	12.2	24.4	12.2	24.4
Run Time min	10	10	ر.	10	10	10	10
Run-Section	46 - A B C D	47 - A B C D	48 – A B C D	49 – A B C D	50 - A B C	51 - A B C	52 – A B C D

TABLE XII (Continued)

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Chemically Pure Propane

В.

Average Deposit Rate µg/cm²-hr	. 352	440	491	452	381	381
Average Wall Temperature Deg K	533	398	532	522	410	394
Deposit Rate μg/cm ² -hr	329 255 471	391 345 537 488	386 327 570 680	393 378 584	389 461 260 414	335 338 327 522
Deposit Weight mg	.147	.172 .154 .234 .218	.170 .146 .243	.178	.178 .2097 .113	.154 .150 .148
Section Surface Area cm ²	2.68 2.68 2.71 2.68	2.64 2.68 2.62 2.68	2.64 2.68 2.56 2.75	2.71 2.68 2.64 2.68	2.62 2.71 2.60 2.64	2.75 2.66 2.71 2.71
Initial Wall Temperature Deg K	505 510 543 575	384 391 401 416	501 511 543 575	491 509 530 558	391 411 414 422	380 385 400 412
Velocity m/sec	6.1	6.1	18.3	30.5	30.5	18.3
Run Time min	10	10	10	10	10	10
Run-Section	53 - A B C C	54 - A B C D	55 - A B C D	56 - A B C C	57 – A B C D	58 - A B C D

TABLE XII (Continued)

Average Deposit Rate µg/cm ² -hr	636	918
Average Wall Temperature Deg K	596	549
Deposit Rate µg/cm²-hr	613 692 615 622	735 417 1368 1150
Deposit Weight mg	.217 .251 .268 .268	.097 .057 .186
Section Surface Area	2.66 2.71 2.62 2.68	2.64 2.73 2.71 2.71
Initial Wall Temperature Deg K	554 585 620 624	518 529 564 584
Velocity m/sec	6.1	6.1
Run Time min	ω	m
Run-Section	59 - A B C D	60 - A B C D

TABLE XIII $\\ \text{Summary of Test Conditions for High Pressure and Nickel-Plated Tubes } \\ \text{RP-1}$

Run No.	Wall Temp.	Velocity	Pressure	Test Duration
	deg K	m/sec	atm	min
	High P	ressure Tests		
61	700	18.3	204	10
62	700	18.3	272	10
63	700	18.3	272	6
64	700	18.3	136	10
65	700	18.3	136	10
66	700	18.3	340	9
	Nickel	-Plated Tubes		
67	700 ·	18.3	136	10
68	811	18.3	136	3
69	589	18.3	136	10
70	700	6.1	136	10
71	589	6.1	136	10

TABLE XIV

Deposit Burnoff Data for High Pressure Tests Velocity = 18.3 m/sec

	Average Deposit Rate µg/cm ² -hr	483	631	752	440	766	533
	Average Wall Temperature Deg K	616	480	607	615	650	629
	Deposit Rate ug/cm ² -hr	544 657 249	688 691 516	830 756 805 615	473 373 428 484	638 358 402	417 517 666
U	Deposit Weight mg	.246 .293 	.311 .310 	.224 .206 .218 .165	.208 .157 .192 .218	.274	.173 .211 .269
Velocity = 18.3 m/sec	Section Surface Area cm ²	2.71 2.68 2.68 2.68	2.71 2.70 2.70 2.79	2.70 2.73 2.68 2.68	2.64 2.52 2.70 2.70	2.58 2.70 2.64 2.64	2.77 2.70 2.70 2.73
Veloc	Initial Wall Temperature Deg K	615 623 615 609	611 613 647 598	626 603 615 584	645 614 603 598	676 649 638 636	681 626 618 590
	Pressure	204	272	272	136	136	340
	Run Time min	10	10	v	10	10	6
	Run-Section	61 - A B C D	62 - A B C D	63 - A B C C	64 - A B C D	65 - A B C C	66 - A B C D

TABLE XV

Deposit Burnoff Data for Nickel-Plated Tube Tests

Average Deposit Rate µg/cm ² -hr	777	77	56	41
Average Wall Temperature Deg K	650	590	640	561
Deposit Rate µg/cm ² -hr	. 82 38 < 20 < 20	< 20 < 20 89 178	 108 39 < 20	104 < 20 < 20 < 20
Deposit Weight mg	.036 .017 < .01 < .01	.01.01.039.079	.047	.044 <.01 <.01 <.01
Section Surface Area cm ²	2.64 2.69 2.69 2.75	2.62 2.71 2.64 2.67	2.60 2.62 2.60 2.62	2.54 2.59 2.60 2.58
Initial Wall Temperature Deg K	661 683 644 614	594 583 589 594	672 680 612 497	555 605 561 523
Velocity m/sec	18.3	18.3	6.1	6.1
Run Time min	10	10	10	10
Run-Section	67 - A B C D	69 - A B C	70 – A B C D	71 – A B C D

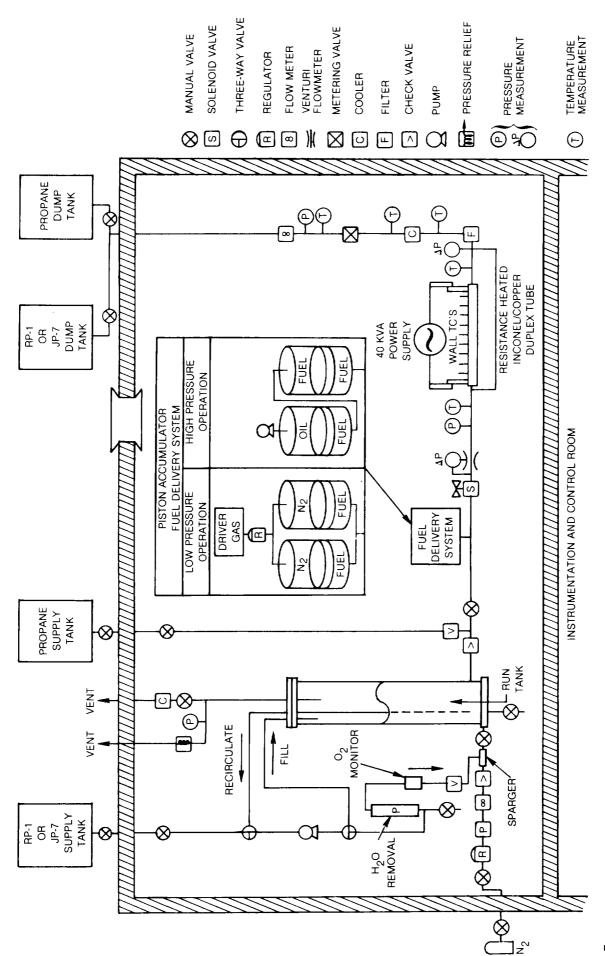


Figure 1. Deposit Formation of Hydrocarbon Rocket Fuels Test Apparatus

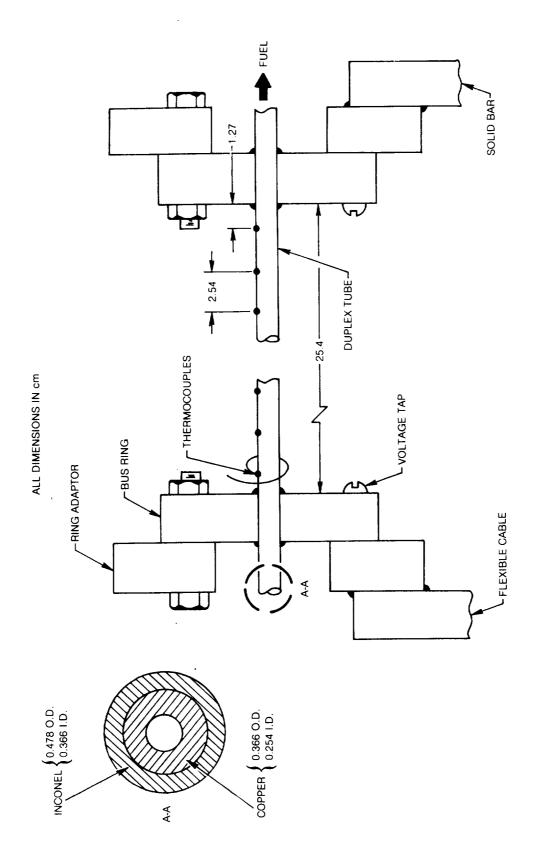


Figure 2. Test Tube Assembly

Figure 3. Test Tube Assembly

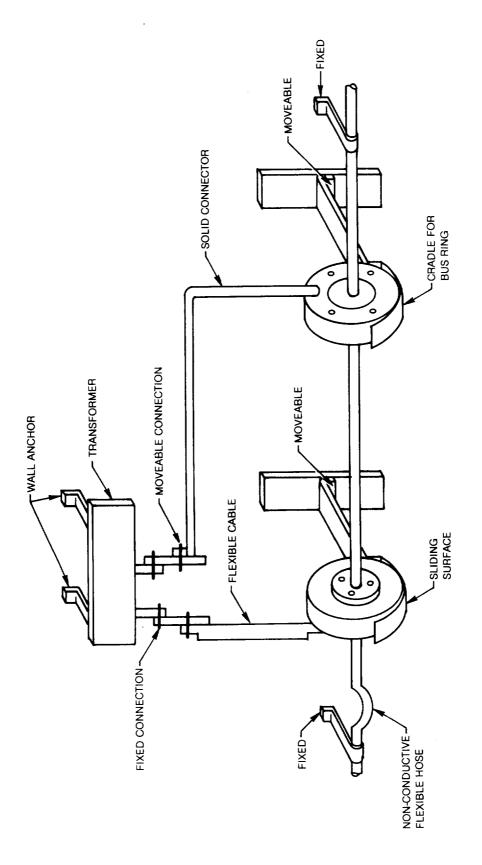


Figure 4. Test Section Mounting

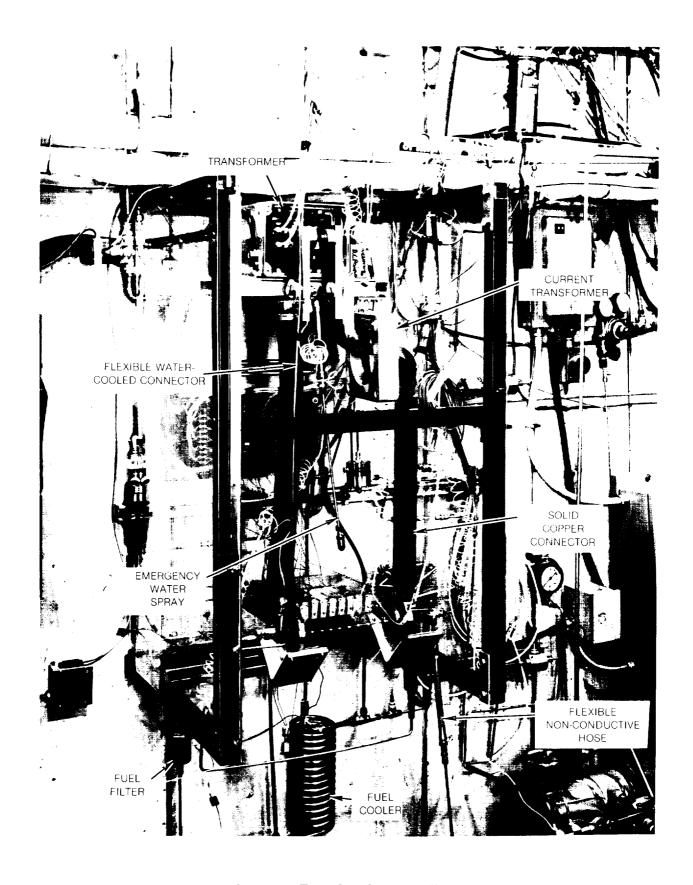


Figure 5. Test Section Installation

80-422-C

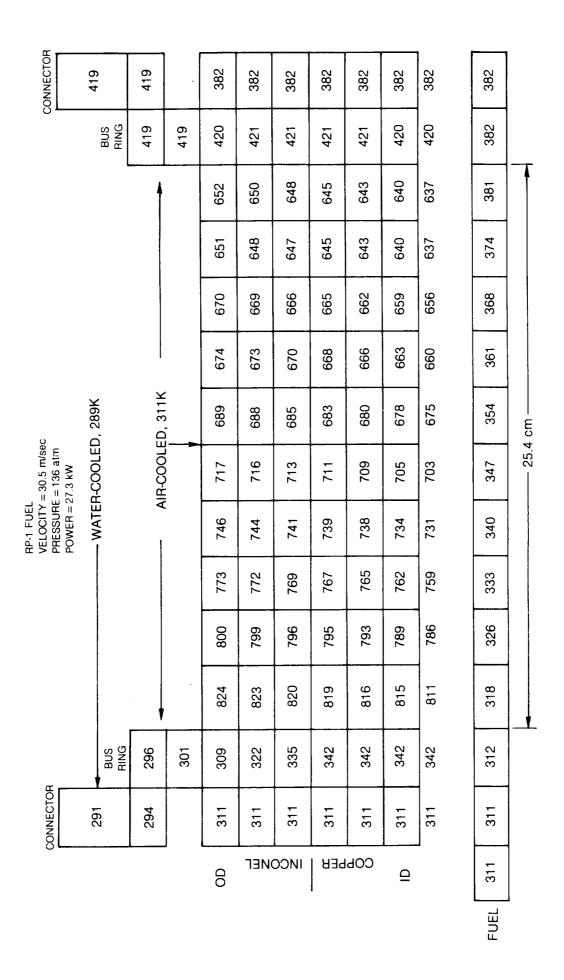


Figure 6. Tube and Bulk Fuel Temperature Distributions — TCAL Analysis

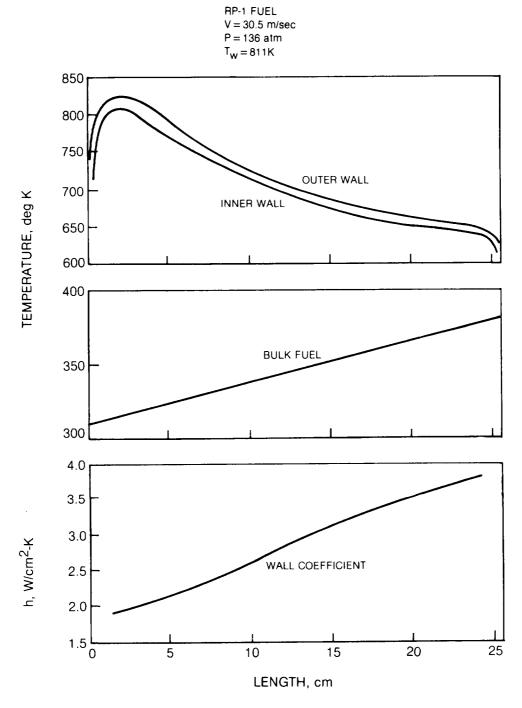


Figure 7. Axial Variation of Temperature and Heat Transfer

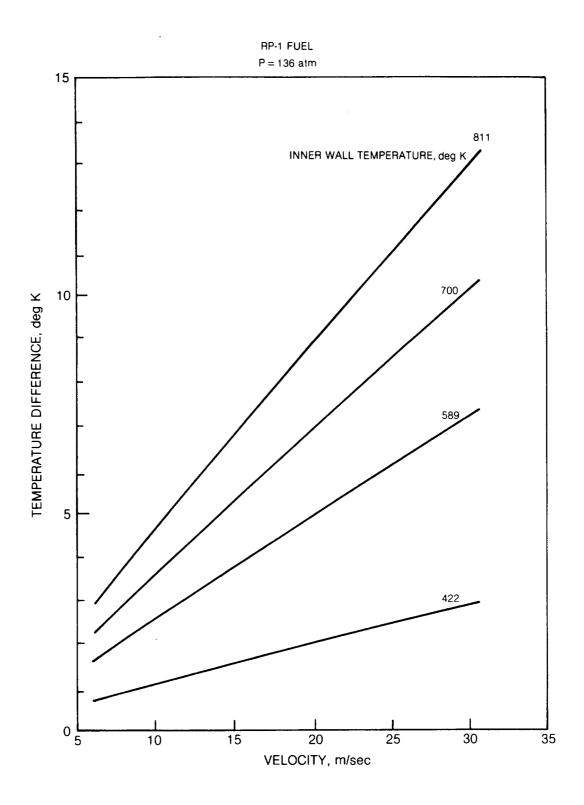


Figure 8. Temperature Difference Between Tube Outer and Inner Walls

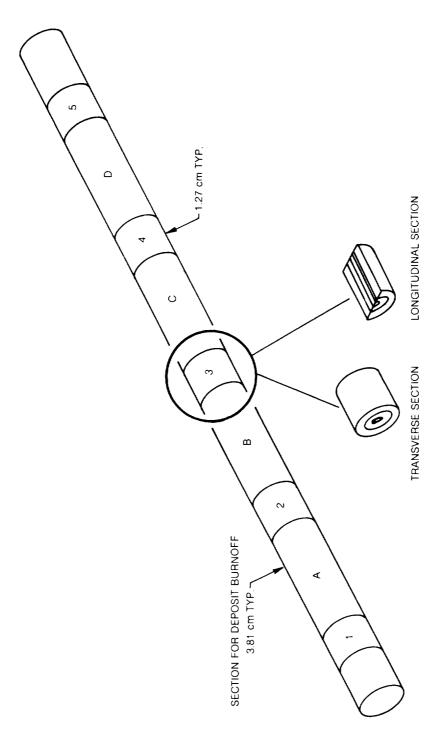


Figure 9. Tube Sectioning

81-6-6-7

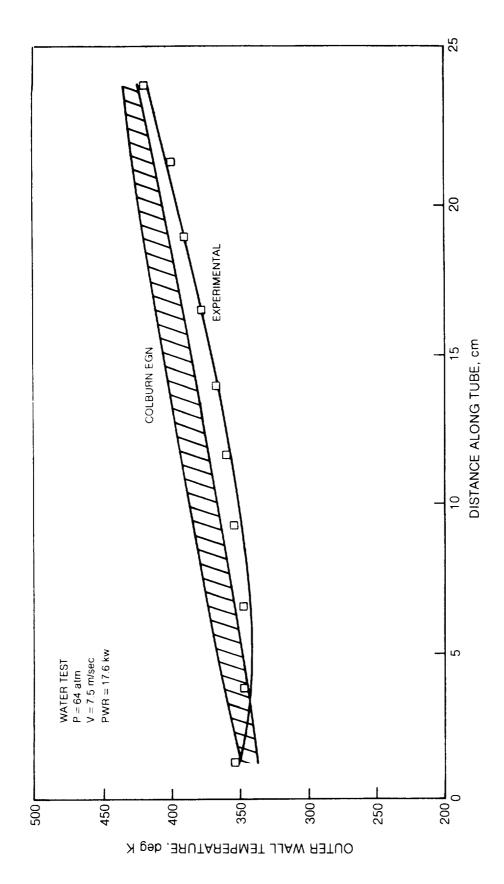


Figure 10. Comparison of TCAL Predictions with Experimental Data

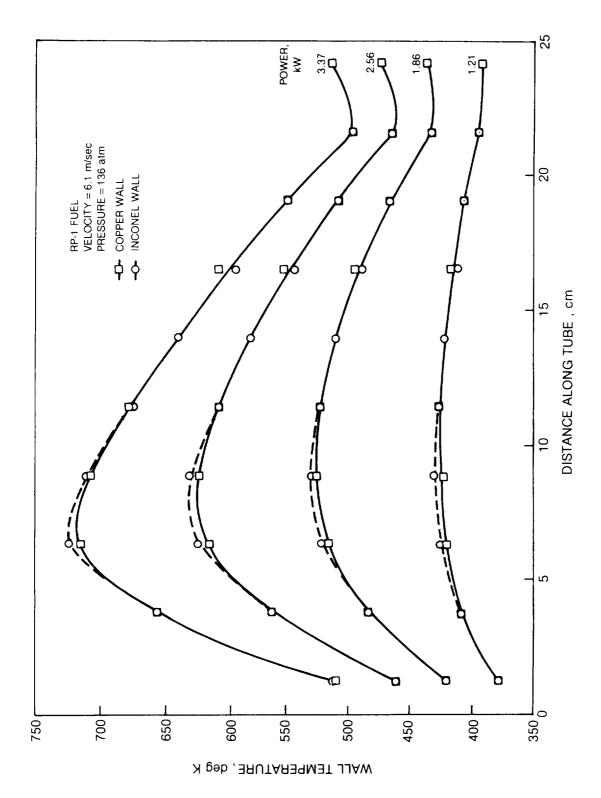


Figure 11. Calibration Tube Temperature Distributions

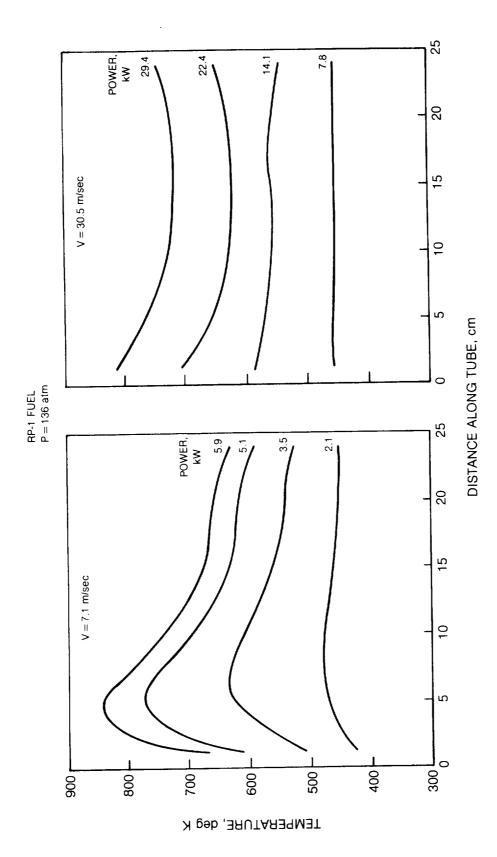


Figure 12. Variation of Axial Wall Temperature with Power

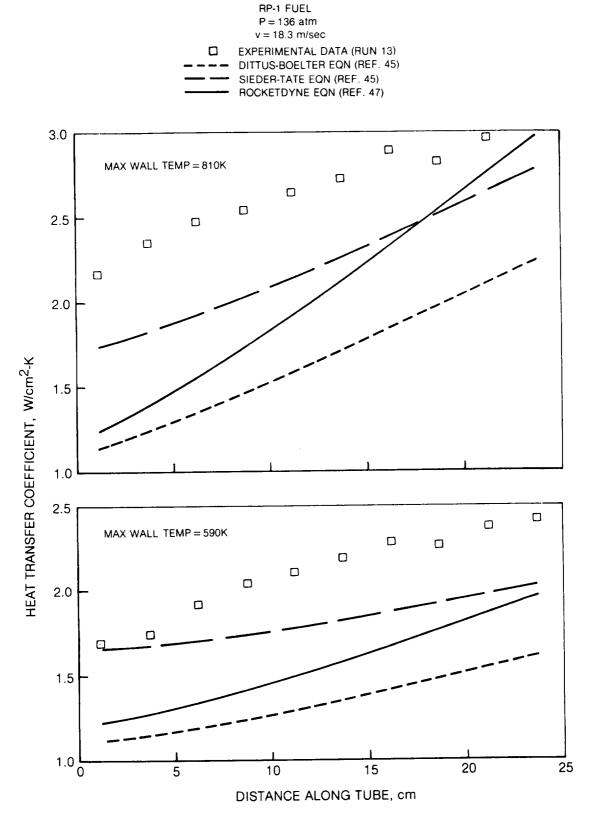


Figure 13. Variation of Heat Transfer Coefficient with Tube Length

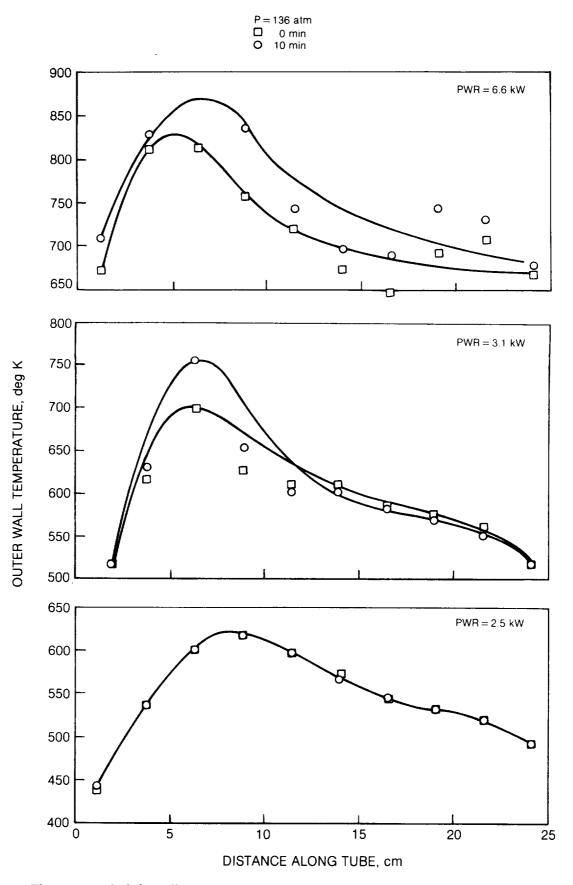


Figure 14. Axial Wall Temperature Variation for RP-1 Fuel, V = 7.3 m/sec

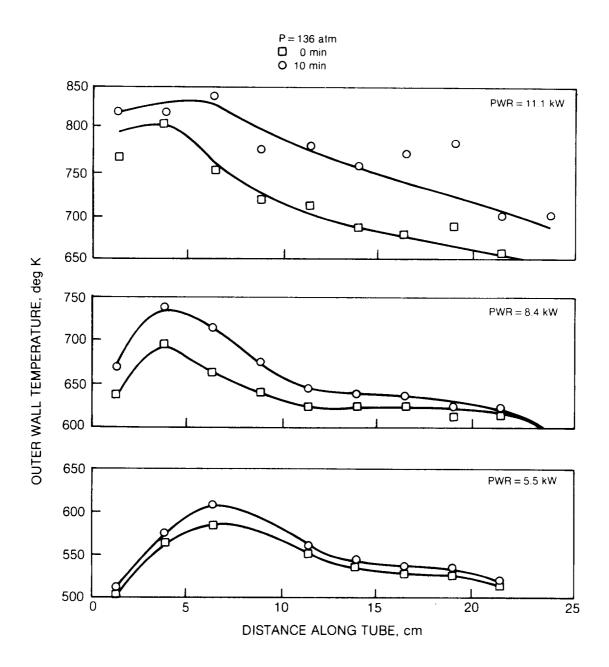


Figure 15. Axial Wall Temperature Distribution for RP-1 Fuel, V = 14.3 m/sec

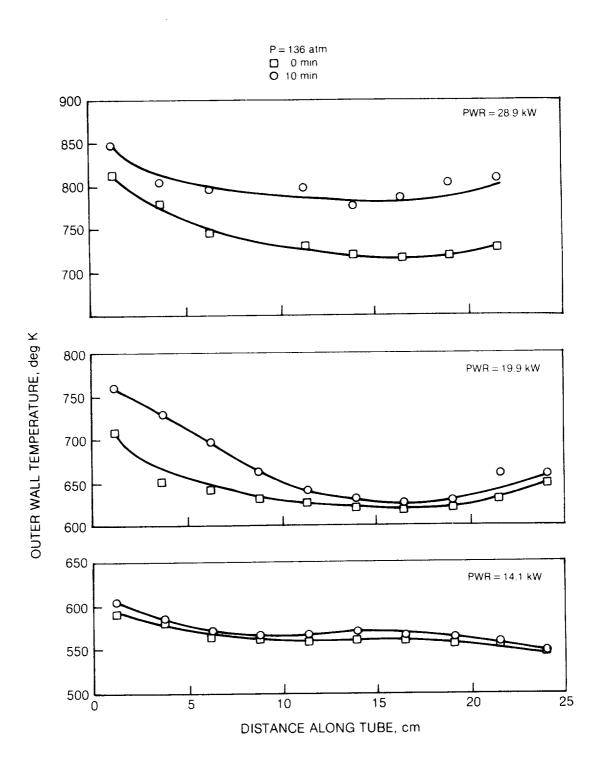


Figure 16. Axial Wall Temperature Distribution for RP-1 Fuel, V = 30.5 m/sec

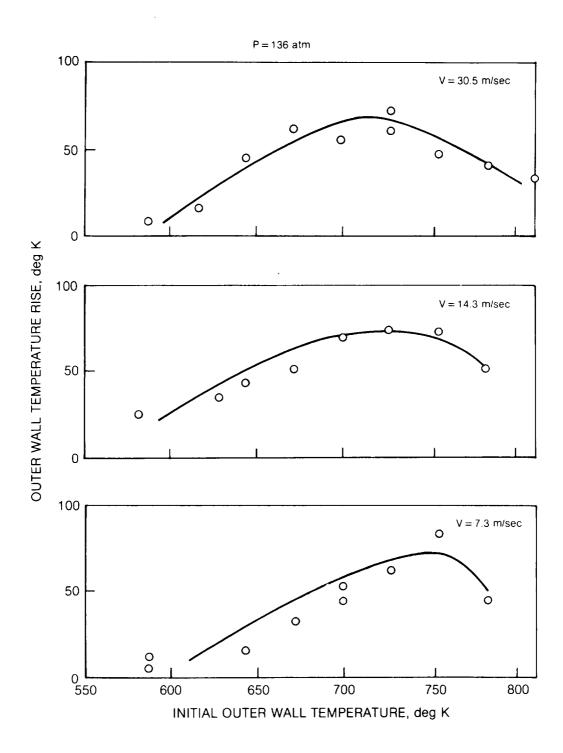
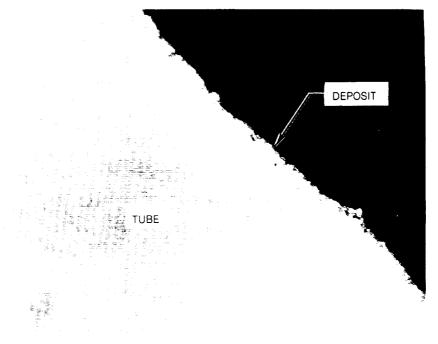
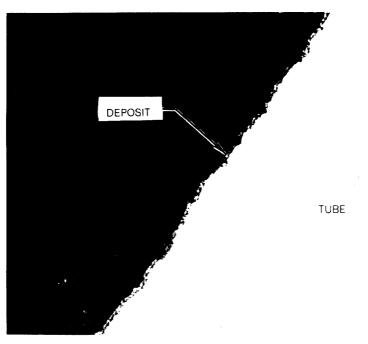


Figure 17. Outer Wall Temperature Rise for RP-1 Fuel





V = 7.3 m/sec



V = 30.5 m/sec

Figure 18. RP-1 Fuel Deposits

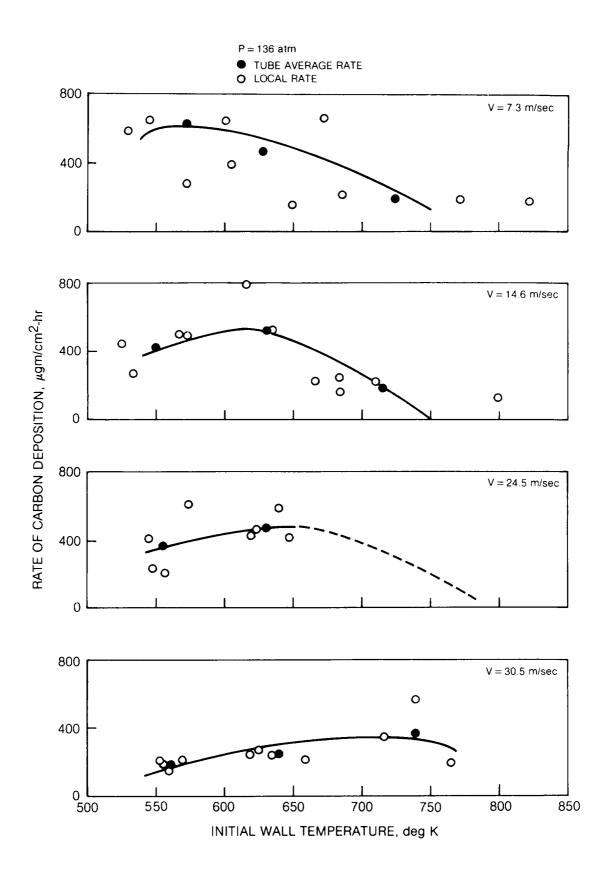


Figure 19. Rate of Carbon Deposition for RP-1 Fuel

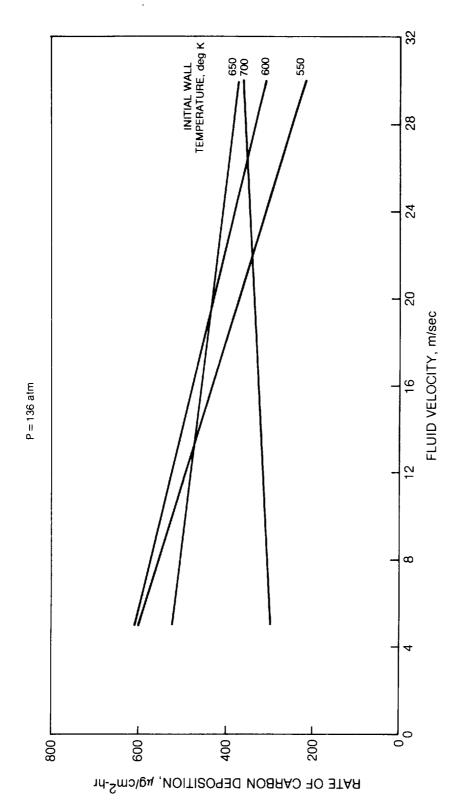


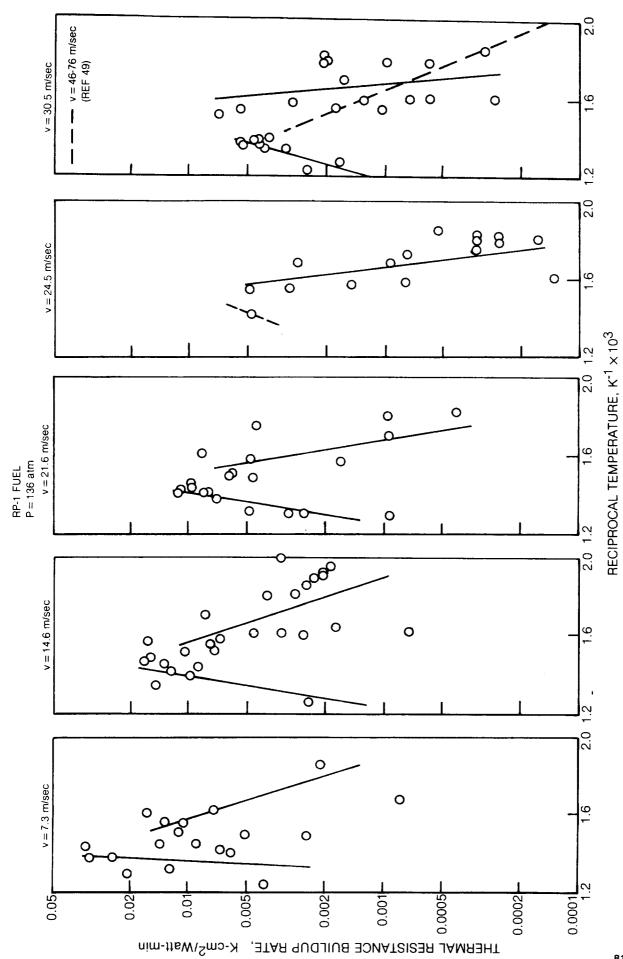
Figure 20. Effect of Velocity on Carbon Deposition Rate for RP-1 Fuel

RP-1 FUEL P = 136 atm

Figure 21. Variation of Thermal Resistance Buildup Rate with Wall Temperature



Figure 22. Deposit Thermal Resistance Buildup Rate



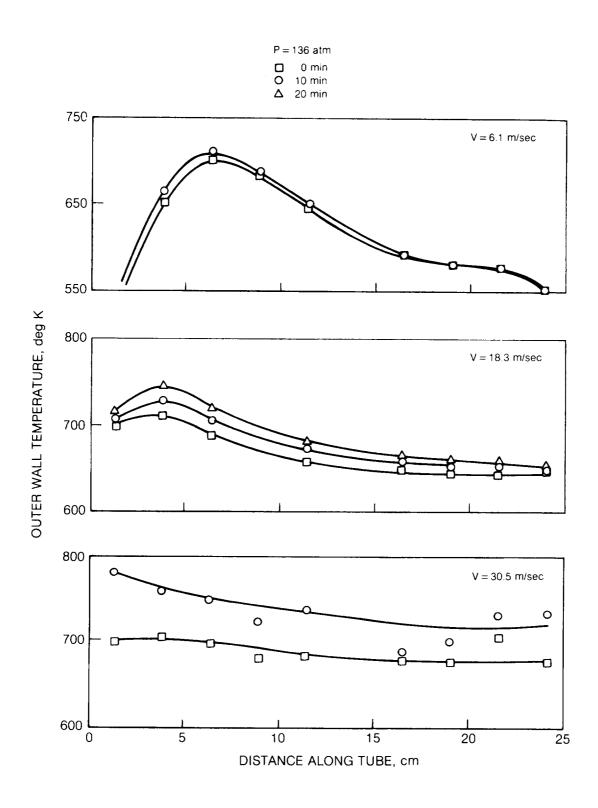


Figure 23. Axial Wall Temperature Distributions for JP-7

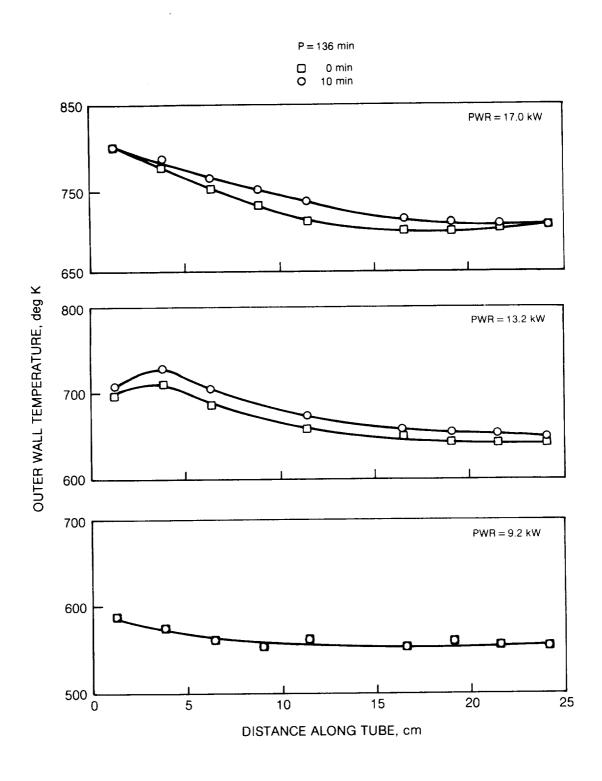


Figure 24. Axial Wall Temperature Distribution for JP-7 Fuel, V = 18.3 m/sec

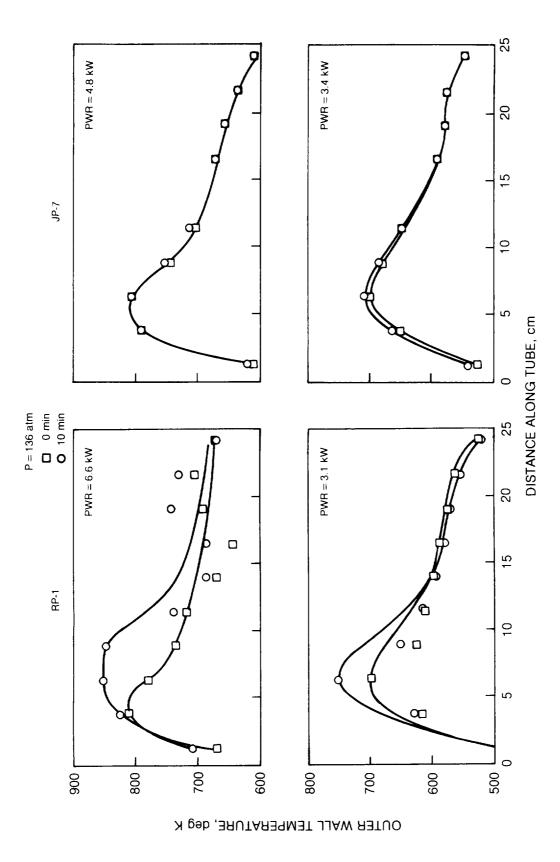


Figure 25. Comparison of Temperature Distribution for RP-1 and JP-7, V = 6.1 m/sec

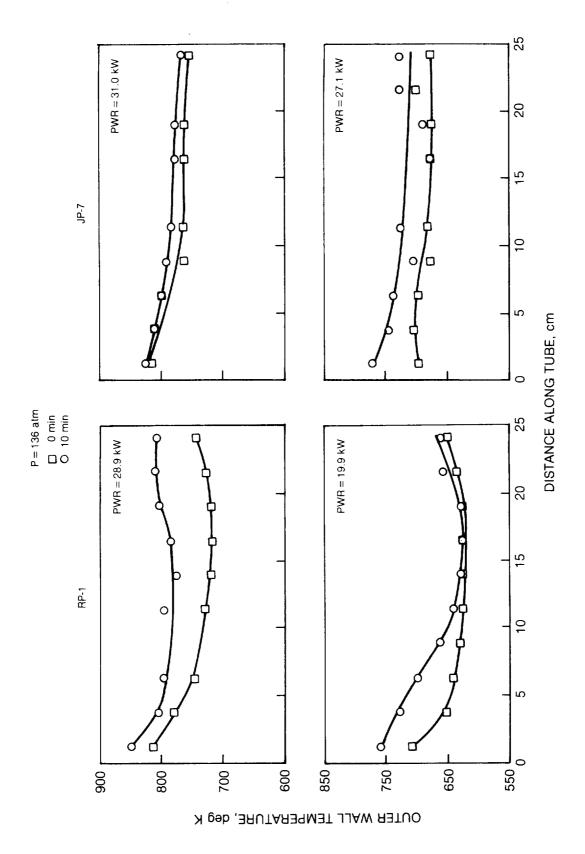
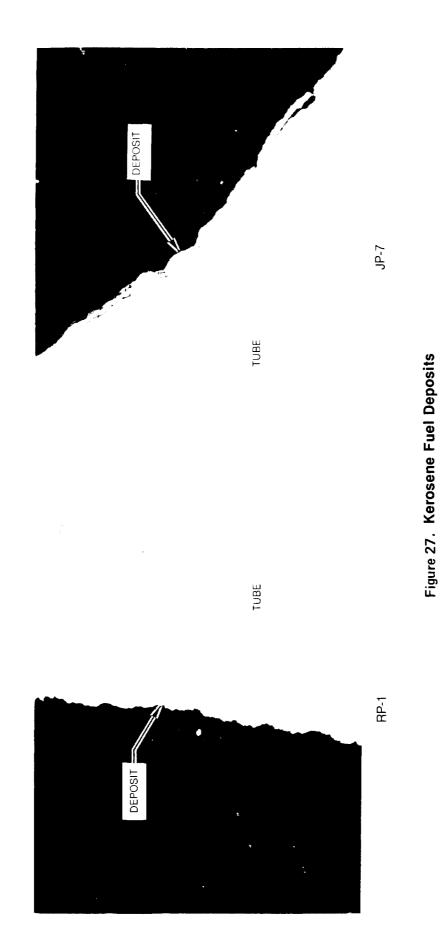


Figure 26. Comparison of Temperature Distributions for RP-1 and JP-7, V=30.5~m/sec



V = 6.1 m/sec $T_{WALL} = 811 \text{ K}$

200 ×

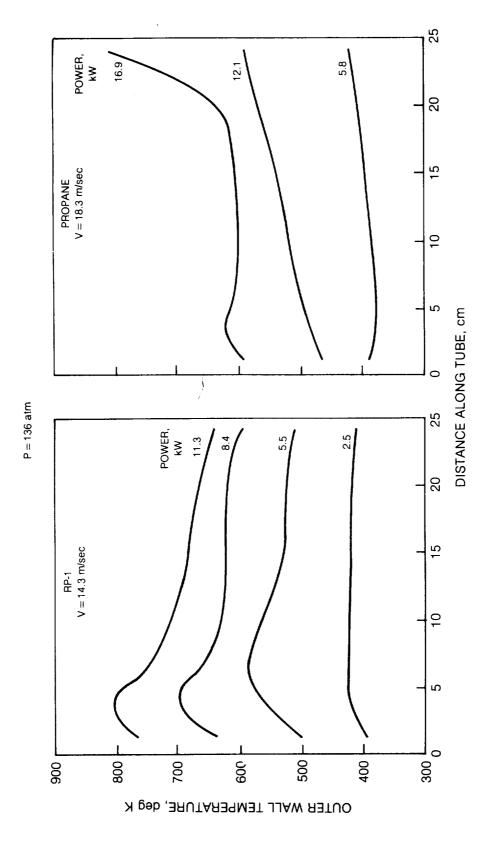


Figure 28. Comparison of RP-1 and Propane Temperature Distributions

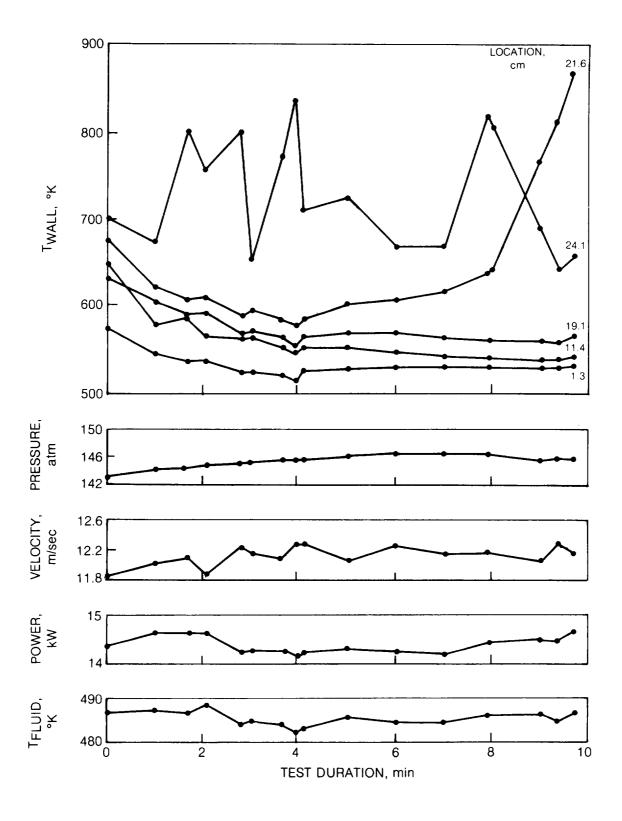


Figure 29. Variation of Propane Test Data with Time

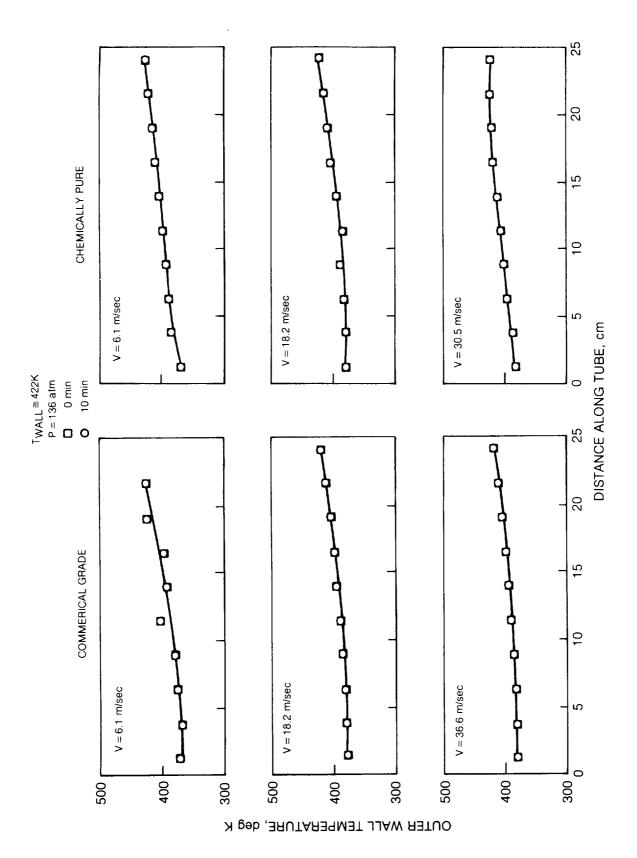


Figure 30. Comparison of Temperature Distributions for Propane

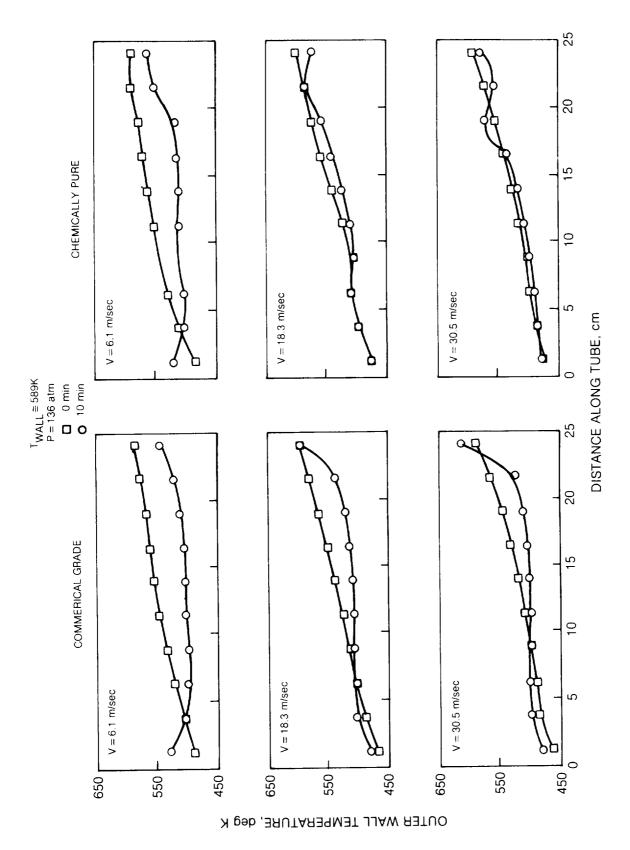


Figure 31. Comparison of Temperature Distributions for Propane

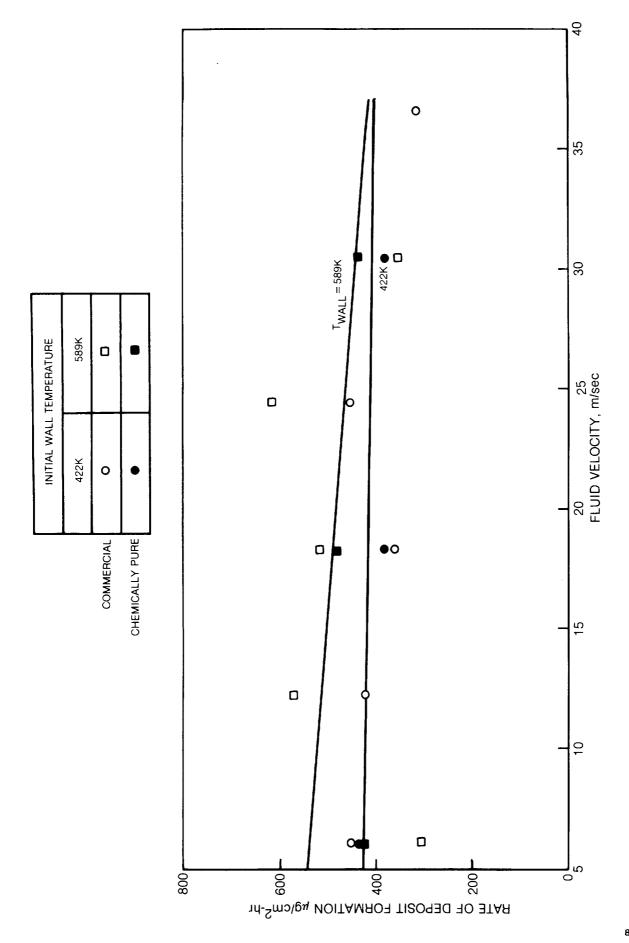
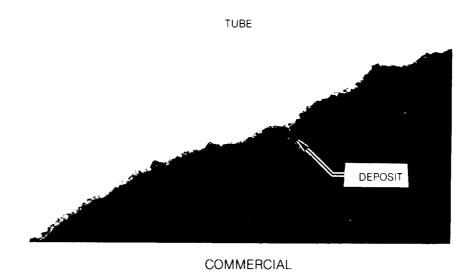
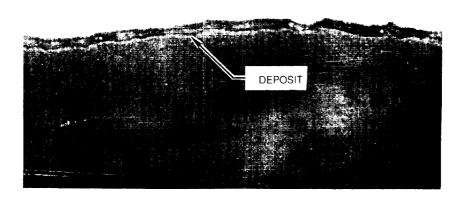


Figure 32. Effect of Velocity on Carbon Deposition Rate for Propane

V = 30.5 m/sec $T_{\text{WALL}} \cong 589 \text{ K}$ $500 \times$



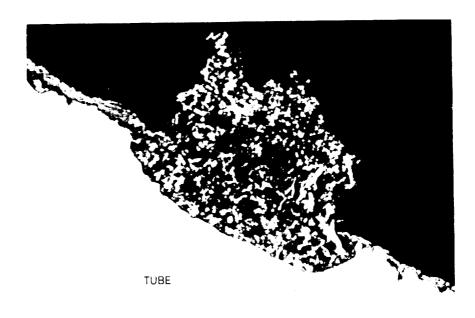




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Figure 33. Propane Fuel Deposits

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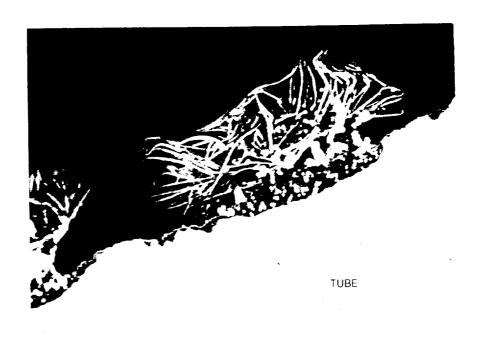


Figure 34. Propane Fuel Deposits

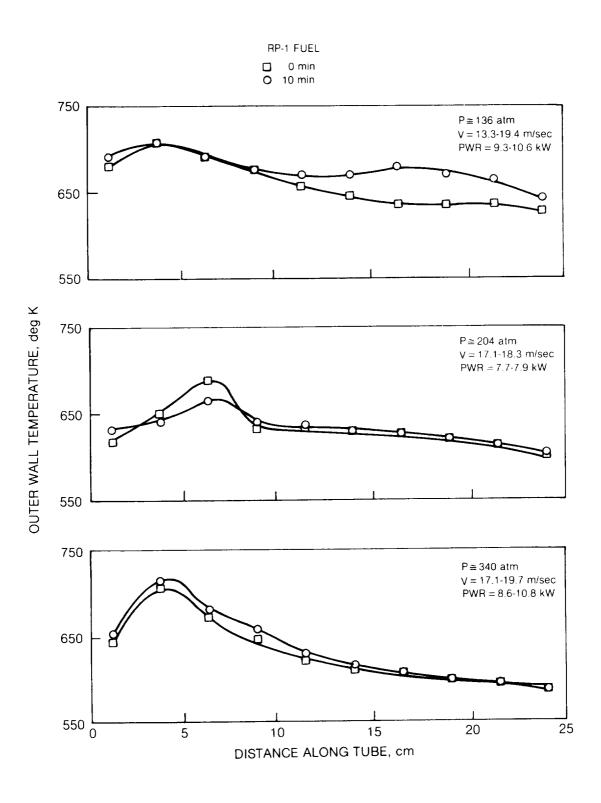


Figure 35. Variation of Wall Temperature with Pressure

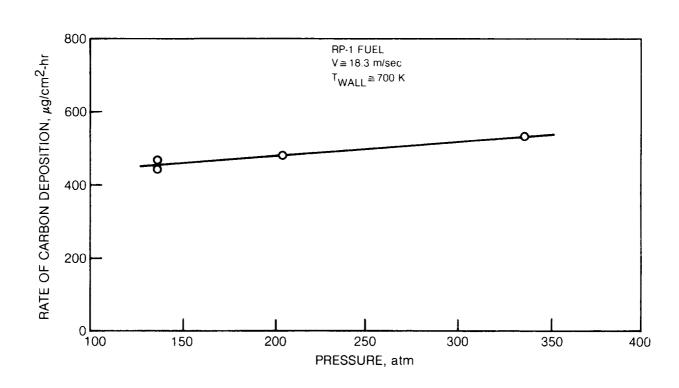


Figure 36. Variation of Deposit Rate with Pressure

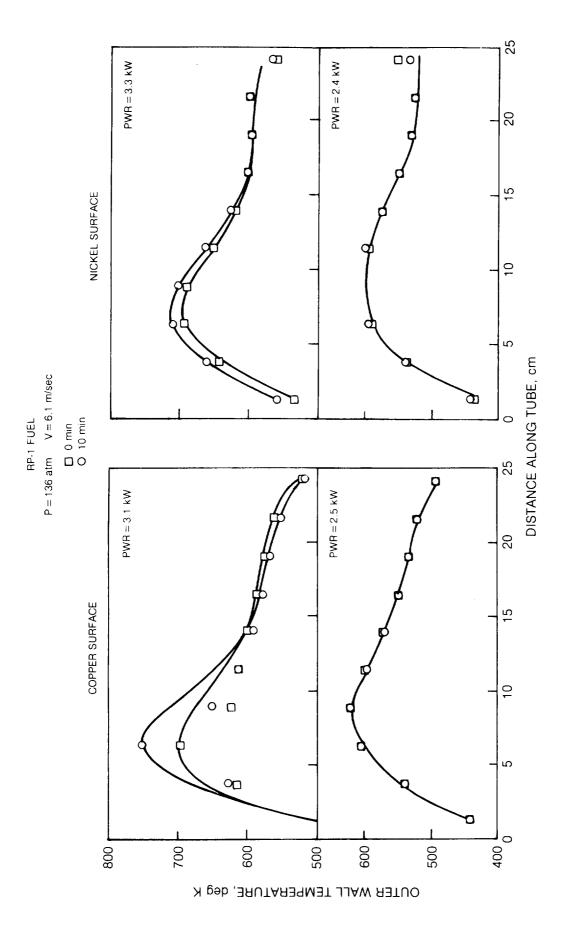
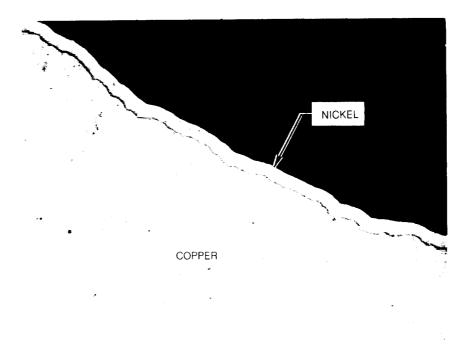
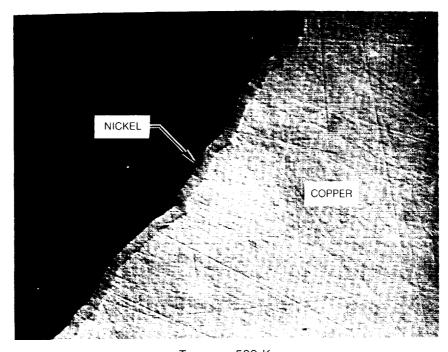


Figure 37. Comparison of Copper and Nickel Temperature Distributions



T_{WALL}≅700 K



T_{WALL}≅589 K

Figure 38. Sectioned Nickel Plated Tubes

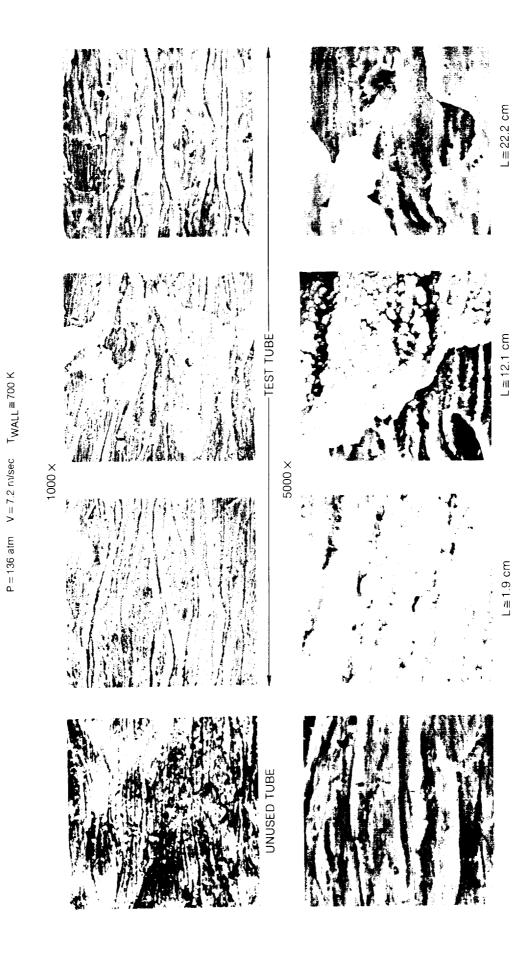


Figure 39 Microstructure of RP-1 Deposits on Copper

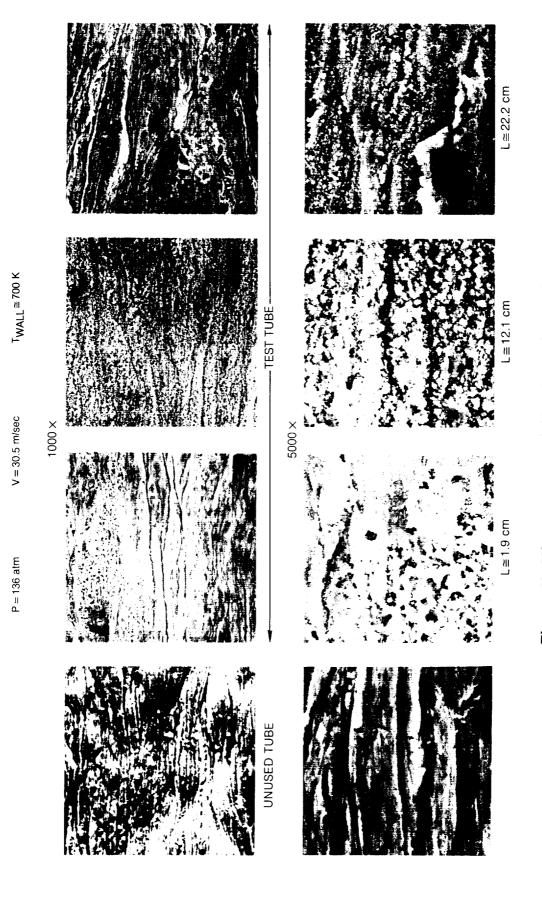
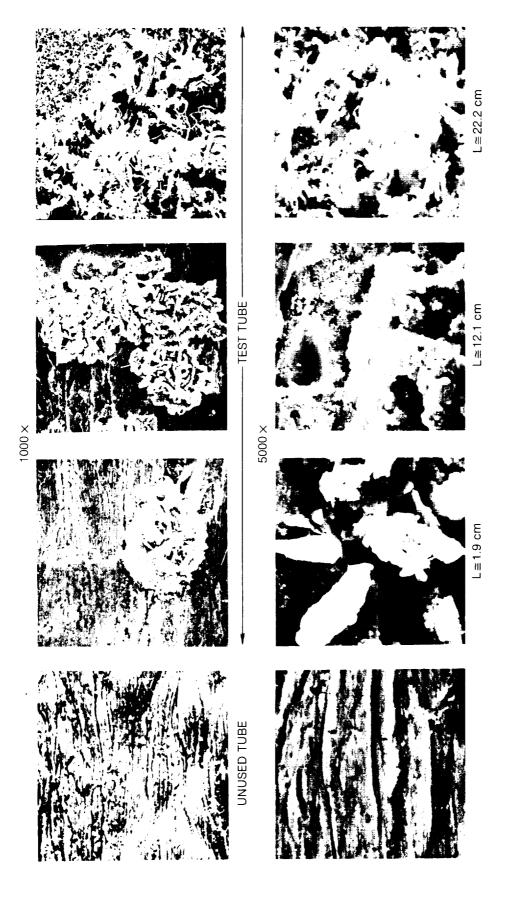


Figure 40. Microstructure of JP-7 Deposits on Copper



TWALL ≅ 644 K

V = 30.5 m/sec

P = 136 atm

Figure 41. Microstructure of Commerical Propane Deposits on Copper

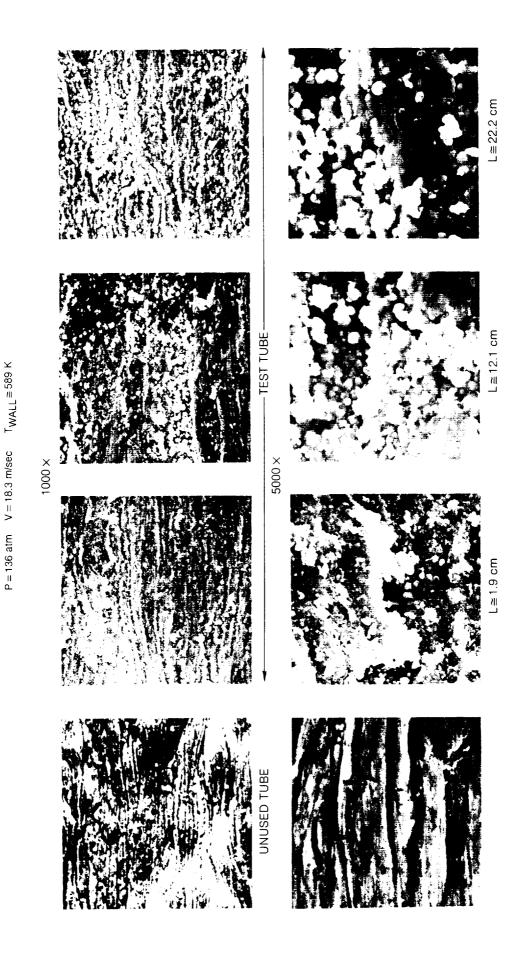


Figure 42. Microstructure of C.P. Propane Deposits on Copper

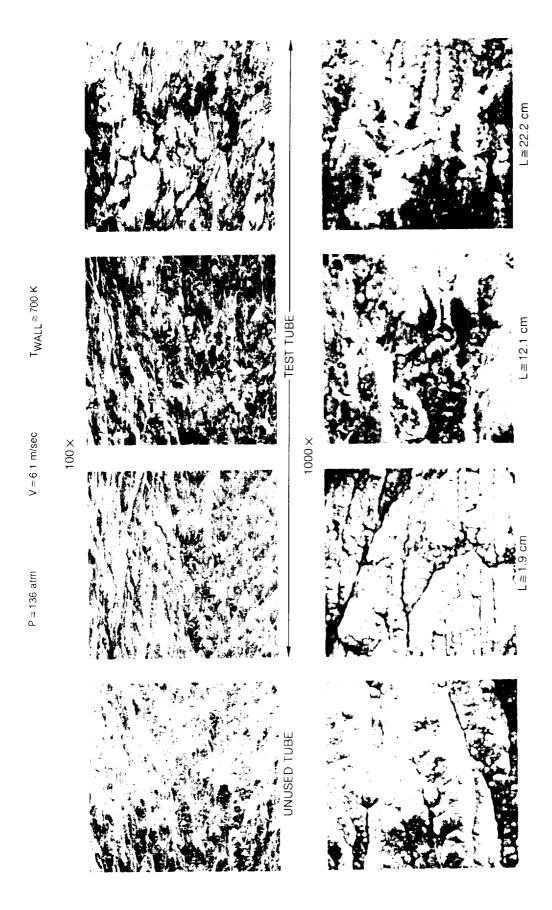


Figure 43. Microstructure of RP-1 Deposits on Nickel

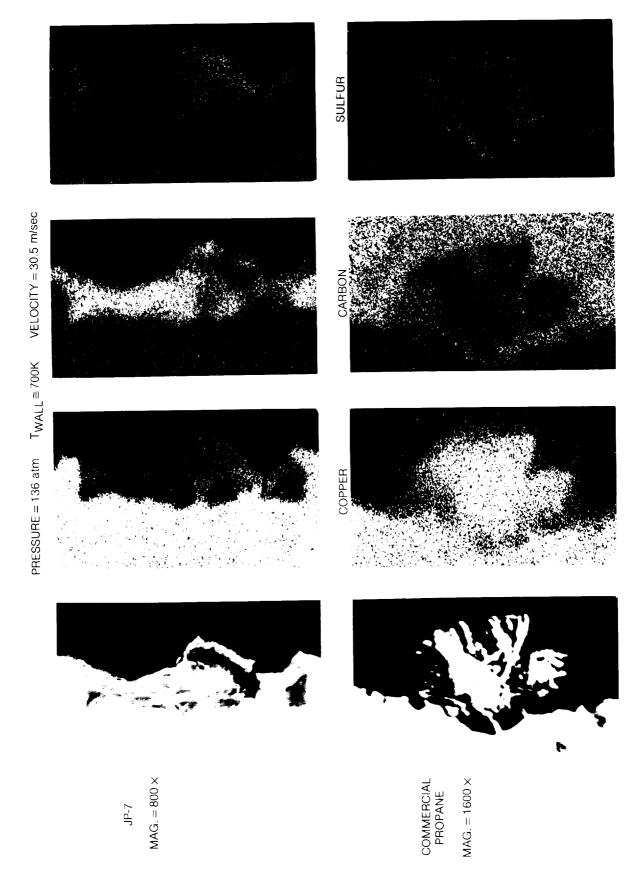


Figure 44. Scanning Electron Microprobe Analysis of Deposits

APPENDIX A

TABULATED TEST DATA

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TABLE A-1

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