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Rhenium Material Properties

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RHENIUM MATERIAL PROPERTIES

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Abstract

Tensile data were obtained from four different types of rhenium at ambient and elevated temperatures. The four types of rhenium included chemical vapor deposition (CVD) and three powder metallurgy (PM) types, i.e., rolled sheet and pressed and sintered bars, with and without hot isostatic pressure (HIP) treatment. Results revealed a wide range of values with ultimate strengths at ambient temperatures varying from 663 MPa for CVD rhenium to 943 MPa for rolled sheet. A similar spread was also obtained for material tested at 1088 K and 1644 K. The wide variance observed with the different materials indicated that the rhenium manufacturing process, material composition and prior handling strongly dictated its properties. In addition to tensile properties, CVD, pressed and sintered material and HIP rhenium successfully completed 100 cycles of low cycle fatigue. Creep data were also obtained showing that CVD and pressed and sintered rhenium could sustain five hours of testing under a tension of 27.5 MPa at 1922 K.

Introduction

The On-Board Propulsion Branch of NASA Lewis Research Center (LeRC) and the aerospace industry are currently investigating a series of 22 N to 890 N thrust chambers (refs. 1, 2) made from rhenium material coated with iridium for oxidation protection. The longest test time on a single chamber so far has been 39 hours at NASA LeRC with a oxide coated iridium/rhenium 22N chamber (ref. 3) and

6.3 hours with a flight type 440 N thrust chamber (ref. 4). The operating temperature of these chambers were in excess of 2000 K yielding 8 percent improvement in performance over conventional chambers. This excellent performance means less film cooling, greater life, and increased payloads into orbit with space vehicles using rhenium thrust chambers. However, the lack of reliable material properties data are hindering their full acceptance. Current properties data are very sparse and scattered throughout the literature. Furthermore, property data are often reported for forms of rhenium that are not used for chamber manufacture or where the material history is not reported. Property data for various forms of rhenium are reported in references 5 to 8. The data they report are within the scatter of the results obtained here.

This paper presents the material properties of rhenium in manufacturing forms that were considered prime candidates for thrust chamber development. Reproducible data, obtained in controlled environments, are required before rhenium can be considered as a viable candidate for thrust chamber development. In particular the tensile strength, low cycle fatigue, and creep properties were determined for up to four different types of manufactured rhenium. The four different types of rhenium included: chemical vapor deposition (CVD), and powder metallurgy (PM) in the forms of rolled sheet, pressed and sintered bars, and hot isostatic pressure (HIP) treated material. Using these four material varieties, a program plan was developed to determine the properties of 59 rhenium samples. This

large number of test samples was selected to insure that any large variation from established trends could easily be identified and further investigated for their deviation.

Background

Current on-board propulsion systems use a high temperature alloy C-103 with a disilicide oxide coating. These chambers are limited by wall temperature to 1593 K operation. In order to maintain wall temperature below this limit, 30 to 40 percent of the fuel is usually required as a film cooling barrier. This large film cooling requirement extracts a penalty in performance since operation of the chamber is kept below levels that would yield optimum specific impulse. A leading candidate to replace C-103 is rhenium with iridium used as an oxidation resistant coating. Rhenium has a high melting temperature of 3453 K along with high temperature strength and thermal shock resistance. Performance gains of up to 20 seconds specific impulse are demonstrated with rockets using earth storable fuel and rhenium chambers (ref. 9). These gains are achieved through designs which eliminated film cooling and improved combustion efficiency. CVD and PM are two of the prime material preparation techniques for this rhenium material. The successful performance of these materials generates a need to know their physical properties in more detail. These material data are especially important in the design process for determining the durability of thrusters during launch vibration and repeated firing on-orbit.

In order to generate the needed information, LeRC conducted a rhenium materials development program under the Space Storable Rocket Technology Program (SSRT) with TRW and a parallel effort in-house. The SSRT program investigated the properties of 37 samples made by CVD and PM produced from pressed and sintered material. LeRC investigated 22 samples, including CVD, HIP, and rolled sheet rhenium.

CVD rhenium The CVD material used in this investigation was obtained from Ultramet. The CVD process produces a thick structural deposit by the chemical reaction of a vapor at a surface on a heated substrate. To form structures of 0.132 cm thickness required in this study, the deposition was done in several layers. The first layer formed at nucleation sites. After the substrate is fully covered growth continued on the crystal faces of the deposit until the desired thickness was reached. The process was then halted as many times as needed to prevent large crystal formation. The surface was machined smooth and the process restarted until the final desired thickness was reached.

Powder Metallurgy All three types of powder metallurgy samples were made from a high green strength 200 mesh powder that was 99.99 percent pure. Rhenium Alloys Inc. provided all the PM samples. The pressed and sintered bars measured 95 to 96.3 percent dense. The rolled sheet was obtained from an ingot that was rolled in the longitudinal direction to produce a sheet that was nominally 8 cm wide by 38 cm long and 0.16 cm thick. Density of the rolled sheet was greater than 99 percent. The HIP treated samples were obtained from the outer perimeter of a large cylindrical ingot that was used to manufacture a 440 N thrust chamber. Density of these samples was also greater than 99 percent.

Test Procedure

All sample bars, both those required in the SSRT program and LeRC in-house investigations were sent for testing to Energy Material Testing Laboratory (EMTL). This was done to assure consistency in test procedures and measured results. The flat bars which measured approximately 2.2 cm wide by 10.1 cm long, were electron discharged machined (EDM) using copper wire into the dog bone shape shown in Figure 1. The dog bone samples were then acid scrubbed to remove any melt or recrust area formed by machining. The test pieces were then acid washed several times to

remove any contaminants. Any test piece not annealed by the manufacture was then annealed. For the LeRC in-house samples, only the HIP material had to be annealed at 1922 K for 30 minutes. Data, from rhenium material that has been annealed, are important as it simulates the condition of the thrust chamber after hot rocket acceptance testing. After annealing, Rockwell hardness measurements were made and were recorded for the LeRC samples in Table I. All samples were then weighed, photographed and visually inspected before testing. Test samples were placed in a chamber that was first purged with argon which was then out gassed until a oxygen sensor dropped below 50 ppm. Samples to be tested were heated at a rate of 13 K per minute until target temperature was reached. Tensile testing of all samples was then conducted at a load rate cross head velocity of 0.13 cm per minute until the sample failed. Upon the occurrence of failure all data were saved and the sample redimensioned to determine any length change due to plastic deformation. The testing facility, EMTL then prepared and submitted a detailed report to LeRC (ref. 10).

Test Results

The data for rhenium tensile samples are presented in Table II and includes values for unannealed and annealed bars. Test data are presented for tensile yields of 0.2 and 0.5 percent ,ultimate strength, elastic modulus, and percent strain to failure ratio. Figure 2 plots ultimate strength and the 0.2 percent yield data. Comparison of the data shows that rhenium tensile strength has a wide range of values based on the anneal, the method of material preparation, and the test temperature. For ease of comparison, powder metallurgy results are plotted separately in Figure 3 and CVD results in Figure 4. In addition to tensile data, creep data is presented in Table III for CVD and PM pressed and sintered rhenium while low cycle fatigue data is presented for CVD, PM pressed and sintered and HIP rhenium in Table IV.

Rolled Sheet. From Figure 2 it is seen the rolled sheet produced some of the highest as well as the most consistent data of all the rhenium materials tested. Maximum yield strength at room temperature was 590.1 MPa with a ultimate strength of 943.1 MPa, elastic modulus 434.4 GPa and a strain to failure of 17.2 percent. When the test temperature was raised to 1644 K , yield was reduced to 370.1 MPa, ultimate to 443.3 MPa with a elastic modulus of 195.1 GPa and a strain to failure ratio of 1.38 percent. The rolled sheet was the only PM material that showed the typical characteristic of decreasing yield strength with increasing temperature.

Electron microscope photographs in Figure 5 further detail some of the unique characteristics of rolled rhenium. Figure 5 shows that failure was preceded by elongation or necking of the specimen followed by rupture. In several cases this failure is along a 45 degree angle with the load. This type of failure is characteristic of ductile materials and is a good indication that intergranular shear forces are primary responsible for failure. Scanning electron microscope (SEM) photographs taken at times sixty magnification (Figure 6) indicate that failure is due to intergranular ductile rupture at room temperature with some dimple rupture evident at the 1644 K temperature. Observations of the samples after testing showed that three of them exhibited signs of plastic deformation on the sample surface due to material yield. This behavior was observed outside of the rupture location No low cycle fatigue or creep tests were conducted with the rolled sheet.

Pressed and sintered The pressed and sintered samples were investigated under the SSRT program and are reported in detail in Reference 11. Results presented here are meant to summarize the materials performance and show how it compared to the other forms of manufactured rhenium samples. The pressed and sintered PM material produced the lowest ultimate strength levels of any PM sample tested. Figure 4 shows that yield strengths were below any other tested rhenium sample.

These low values were attributed to the low density of the material, 95 to 96 percent. Table II shows that unannealed test samples have lower ultimate strength than annealed. This difference was opposite to what was anticipated and was attributed to repressing of some of the samples after sintering and annealing. This repressing or straightening was done on samples that showed signs of being warped and as a result added work hardening to the material. The sintered PM was one of three types of rhenium that was tested for low cycle fatigue as shown in Table IV. Results indicated that in tension and compression PM sintered rhenium had sufficient strength to successfully sustain 100 low fatigue cycles based upon yield strengths. Another sample of this same material completed a five hour creep test at 1922 K and 27.5 MPa. The creep test temperature and pressure were values selected that represent the worst case scenario for 440 to 880 N thrust class rockets.

HIP The HIP treated PM rhenium samples were second only to the rolled sheet in ultimate strength when evaluated at ambient and 1088 K. When the test temperature reached 1644 K values of ultimate strength as with sintered PM were below that of the CVD samples. Yield strengths at all temperatures were the second lowest of all tested material. The HIP material also exhibited the unique property of increased yield strength as the test temperature was raised from ambient to 1088 K (Figure 3).

Examination of the ruptured samples in Figure 5 shows that in all cases the break produced almost no reduction or necking of the sample. This lack of necking and the transverse breaks are a good indication that HIP rhenium is more brittle in nature than was seen in the previous samples. Figure 6 shows the HIP rhenium had a very fine almost sand like structure rather than a stringy or rock candy like texture. The sand like structure indicates that failure was through transgranular rupture rather than intergranular that was characteristic of the other PM materials. PM HIP material thus behaves more like a structure that fractures

across a fine grain surface. Observations of the samples after testing showed no noticeable surface plastic deformation. Table IV shows that HIP rhenium possesses the ability to withstand 100 low fatigue cycles at both 1088 K and 1644 K. The one sample that failed to reach this goal was accidentally pretested to 400 MPa and in addition was warped across the width direction. This sample was warped due to stress relieving after annealing and was sufficient to prevent correct alignment in the test apparatus.

CVD Chemical vapor deposition results are presented for tensile data obtained under the SSRT program and the LERC in-house effort. The SSRT data are reported in detail in Reference 11. The SSRT and LERC samples were obtained from the same manufacturing run. Since both sets of material were from the same run and identical procedures were used to obtain tensile data, they are treated in this report as if coming from one source. Results of tensile tests indicate good agreement with yield strengths at ambient conditions but a wide variation as the temperature was raised to 1088 K and 1644 K. The CVD samples also showed the unique property of increased yield strength, as noted with PM material, when the temperature was increased from ambient to 1088 K. Ultimate strengths showed good agreement at ambient and 1088 K but a wide variation at 1644 K. Figure 5 and 6 reveal some of the unique characteristics of the CVD material as far as rupture and grain structure. In Figure 5, in the majority of cases there is a noticeable elongation in the area of the rupture followed by a very jagged and irregular tearing type break across the sample. This type of very coarse rupture reveals several layers of the CVD material indicating that rupture points occurred in different planes. The elongation of the samples and the ragged angular breaks identified the CVD material as being ductile in nature. It is this elongation 0.4 percent for sample 6-CVD and 3.1 percent 5-CVD that is responsible for the large spread in data at 1644 K (Figure 4). In addition to the elongation and jagged breaks, Figure 6

shows that CVD material had a different grain structure than was seen with any of the other manufactured rhenium samples. The grain structure consisted more of large columnar or rock candy surface and that failure was by intergranular fracture modes. As with the SSRT samples, there was no indication of laminar separation. Observations of the samples after testing showed that all but one had numerous small cracks on the edge of the test piece. These cracks extended only slightly into the gage section. No noticeable plastic deformation was observed with the CVD samples.

The CVD material was also tested for low cycle fatigue and the ability to withstand creep. CVD rhenium showed the ability to complete 100 cycles of 137.9 MPa tension and 68.9 MPa compression but suffered from plastic deformation. The CVD rhenium also showed no creep failure at 1922 K and 27.5 MPa.

Summary/Conclusions

- (1) Tensile tests of four different rhenium materials revealed a wide range of values that strongly suggest rhenium strength is process controlled and that selection of a rhenium manufacturing process and prior handling will strongly dictate its properties.
- (2) HIP rhenium demonstrated some of the highest levels of strength along with the ability to withstand 100 low fatigue cycles based upon yield strengths. Its failure was characterized by a sharp brittle like break.
- (3) Rolled sheet demonstrated the highest level of strength. Its failure was by intergranular and dimpled rupture indicative of a ductile material.
- (4) CVD material demonstrated a wide range of tensile strengths failing by intergranular shear forces. Tensile data was within the scatter of previously reported results.
- (5) Pressed and sintered rhenium showed the lowest level of strength for PM samples tested but was sufficiently strong enough to pass the low cycle fatigue and creep tests.

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TABLE I. RHENIUM HARDNESS RESULTS

MATERIAL	SAMPLE	ROCKWELL "B" HARDNESS
PM ROLLED SHEET	RS-1	105
	RS-2	105
	RS-3	103
	RS-4	104
	RS-5	105
	RS-6	102
CHEMICAL VAPOR DEPOSITION	1-CVD	99
	2-CVD	99
	3-CVD	102
	4-CVD	103
	5-CVD	103
	6-CVD	102
PM HOT ISOSTATIC PRESSURE	Hiped-1	88
	Hiped-2	87
	Hiped-3	87
	Hiped-4	88
	Hiped-5	87
	Hiped-6	89
	Hiped-7	89
	Hiped-8	89
	Hiped-9	87
	Hiped-10	88

TABLE II. RHENIUM TENSILE DATA

SAMPLE NUMBER	TEST TEMP (K)	ANNEAL TEMP (K) TIME HR.	0.2 % YIELD STRENGTH MPa	0.5 % YIELD STRENGTH MPa	ULTIMATE STRENGTH MPa	ELASTIC MODULUS, GPa	% STRAIN TO FAILURE RATIO
HOT ISOSTATIC PRESSURE TREATED PM RHENIUM							
HIPED-1 (LeRC)	204	1922/0.5	298.5	279.9	910.8	407.5	17.2
HIPED-2	204	1922/0.5	232.4	277.2	918.6	428.9	18.5
HIPED-3	1088	1922/0.5	284.4	284.2	561.9	180.2	26.0
HIPED-4	1088	1922/0.5	284.1	273.6	497.8	180.7	12.3
HIPED-5	1644	1922/0.5	179.9	202.0	215.8	145.5	2.28
HIPED-6	1644	1922/0.5	191.0	222.0	251.6	137.2	4.49
PRESSED AND SINTERED PM RHENIUM							
PM-1 (TRW)	204	NONE	227.5	253.1	677.8	430.9	9.8
PM-2	204	1755/0.5	256.8	220.6	757.8	411.6	13.2
PM-3	204	NONE	213.7	234.4	713.6	405.4	11.2
PM-4	204	1755/0.5	234.4	283.9	841.2	392.3	16.2
PM-5	1088	1755/0.5	206.8	227.5	462.0	181.0	18.9
PM-6	1088	1755/0.5	220.6	234.4	482.6	188.9	25.0
PM-7	1644	1755/0.5	151.7	179.3	208.2	146.8	4.2
PM-8	1644	1755/0.5	144.8	172.4	202.0	148.9	4.3
CHEMICAL VAPOR DEPOSITION RHENIUM							
1-CVD (LeRC)	204	1992/1.0	306.8	339.9	688.1	418.5	21.9
2-CVD	204	1992/1.0	284.5	330.9	697.8	430.9	19.4
3-CVD	1088	1992/1.0	399.9	418.2	464.5	330.9	17.4
4-CVD	1088	1992/1.0	390.2	400.8	444.7	364.7	9.02
5-CVD	1644	1992/1.0	344.7	394.7	455.9	228.8	3.11
6-CVD	1644	1992/1.0	288.8	290.0	299.6	224.8	0.41
CHEMICAL VAPOR DEPOSITION RHENIUM							
CVD-1 (TRW)	204	NONE	310.3	351.2	673.6	461.3	15.9
CVD-2 #	204	1755/0.5	124.1	131.0	674.3	380.6	27.6
CVD-3	204	NONE	296.5	324.1	663.3	424.0	19.0
CVD-4	1088	1755/0.5	358.5	372.3	429.6	288.8	15.9
CVD-5	1088	1755/0.5	317.2	372.3	439.9	288.8	9.3
CVD-6	1644	1755/0.5	199.9	289.6	368.8	184.1	1.9
CVD-7 #	1644	1755/0.5	137.9	131.0	143.5	168.9	0.3
CVD-8	1644	1755/0.5	193.1	269.9	344.1	142.7	1.5
ROLLED SHEET PM RHENIUM							
RS-1 (LeRC)	204	1755/0.5	566.1	593.6	921.9	420.6	16.4
RS-2	204	1755/0.5	590.9	614.1	943.2	434.4	17.2
RS-3	1088	1755/0.5	632.9	541.9	810.9	245.5	21.2
RS-4	1088	1755/0.5	518.4	637.1	612.3	242.0	21.7
RS-5	1644	1755/0.5	369.9	414.4	419.2	191.0	0.80
RS-6	1644	1755/0.5	370.1	418.4	443.3	198.1	1.38

(LeRC) Data obtained by NASA Lewis.

(TRW) Data obtained under Space Storable Rocket Technology Program by TRW.

Data unreliable due to brittle failure and test procedure.

TABLE III. RHENIUM CREEP DATA

SAMPLE	TEMPERATURE (K)	STRESS MPa TENSION/COMPRESSION	% STRAIN
CHEMICAL VAPOR DEPOSITION			
CVD-15	1922	27.5	0.22
CVD-16	1922	27.5	0.24
PM PRESSED AND SINTERED			
PM-16	1922	27.5	3.60
PM-17	1922	27.5	1.75
PM-18	1922	27.5	1.17
PM-19	1922	27.5	1.80

TABLE - IV. LOW CYCLE FATIGUE FOR RHENIUM

SAMPLE #	TEMPERATURE (K)	STRESS MPa TENSION/COMPRESSION	CYCLES COMPLETED
Chemical Vapor Deposition CVD-9	RT	151.7/151.7	100
CVD-10	RT	151.7/151.7	100
CVD-11	1088	275.8/1793	FAILED ON 1ST CYCLE
CVD-12	1088	275.8/137.9	100
CVD-13	1088	275.8/137.9	11
CVD-14	1088	275.8/137.9	12
CVD-15	1644	137.9/68.9	85
CVD-16	1644	137.9/68.9	37
PM Pressed and Sintered PM-9	RT	206.9/206.9	100
PM-10	RT	189.6/189.6	100
PM-11	1088	193.0/193.0	100
PM-12	1088	206.8/206.8	100
PM-13	1644	137.9/137.9	23
PM-14	1644	103.4/103.4	100
PM-15	1644	103.4/103.4	100
PM Hot Isostatic Pressure HIPED-7	1088	137.9/68.9	103
HIPED-8	1088	127.6/62.1	20*
HIPED-9	1644	131.0/62.1	100
HIPED-10	1644	137.9/68.9	100

* Sample was accidentally loaded to 248.2 MPa in a pretest run and exhibited a plastic deformation of 1.2 percent when removed. The sample was also warped in the width direction due to stress relieving during the annealing cycle.

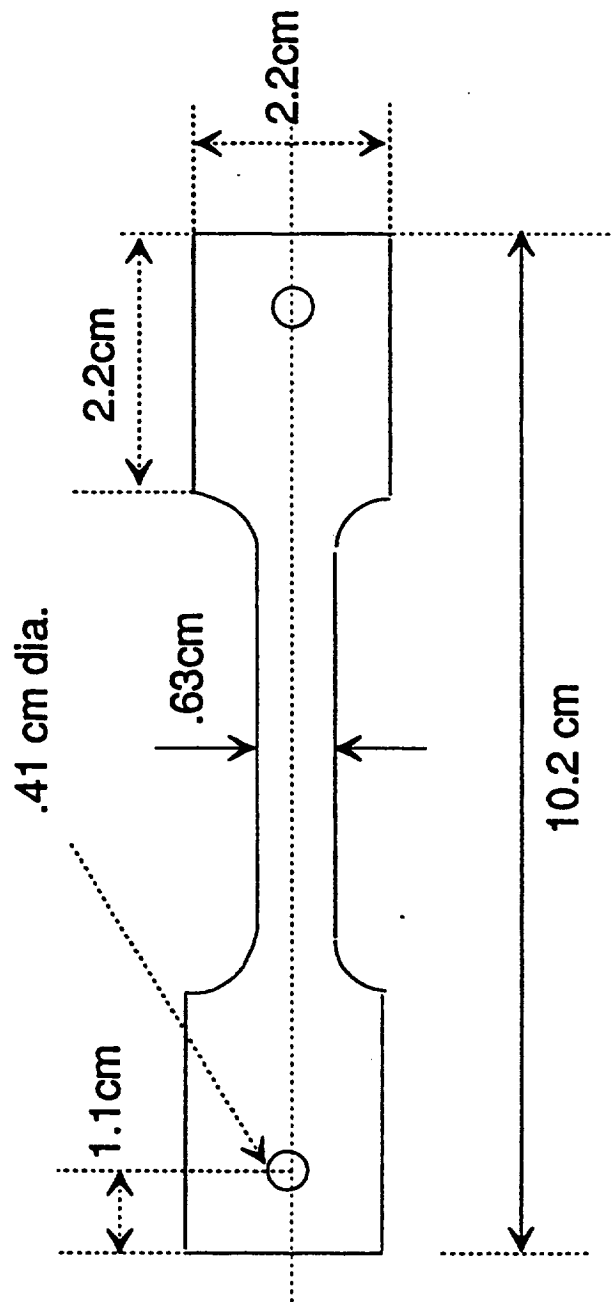


Figure 1. - 0.16 cm Thick Tensile Samples

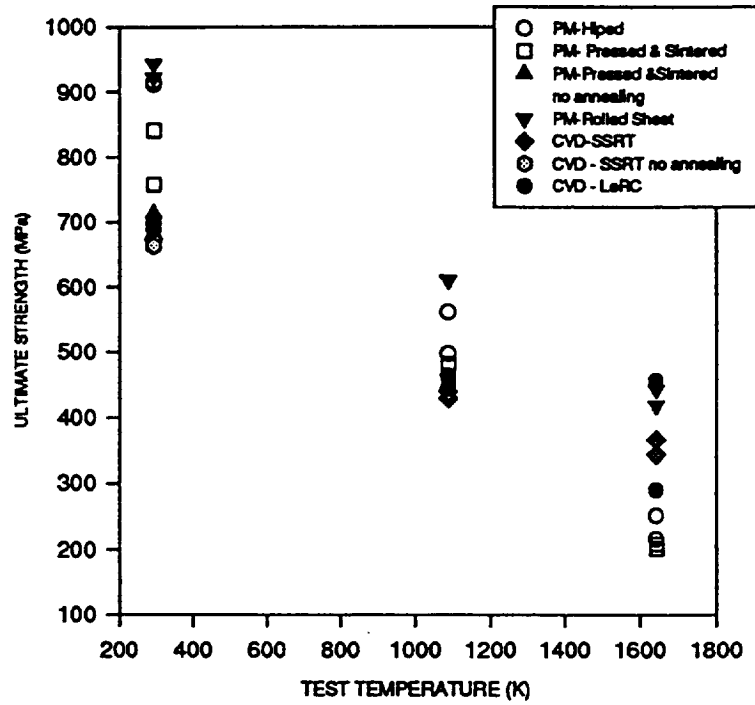


Figure 2-a. RHENIUM ULTIMATE STRENGTH VS TEMPERATURE (K)

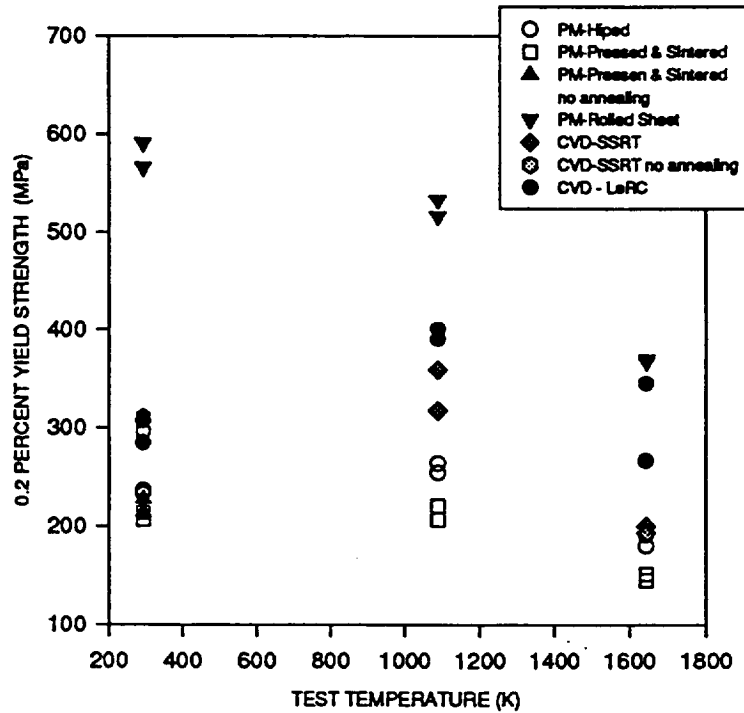


FIGURE 2-b. RHENIUM YIELD STRENGTH VS TEMPERATURE (K)

FIGURE 2. ULTIMATE AND YIELD STRENGTH VS TEMPERATURE (K)

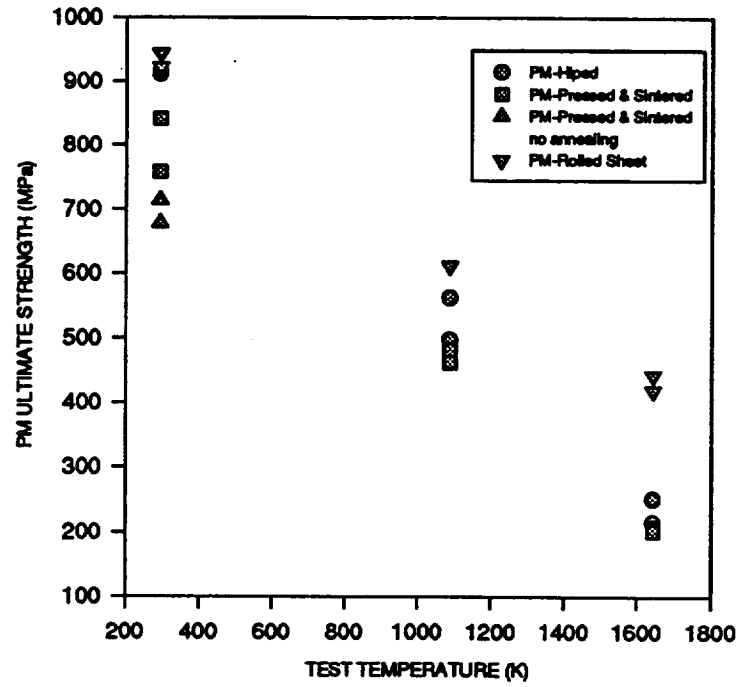


FIGURE 3-a. PM RHENIUM ULTIMATE STRENGTH VS TEMPERATURE (K)

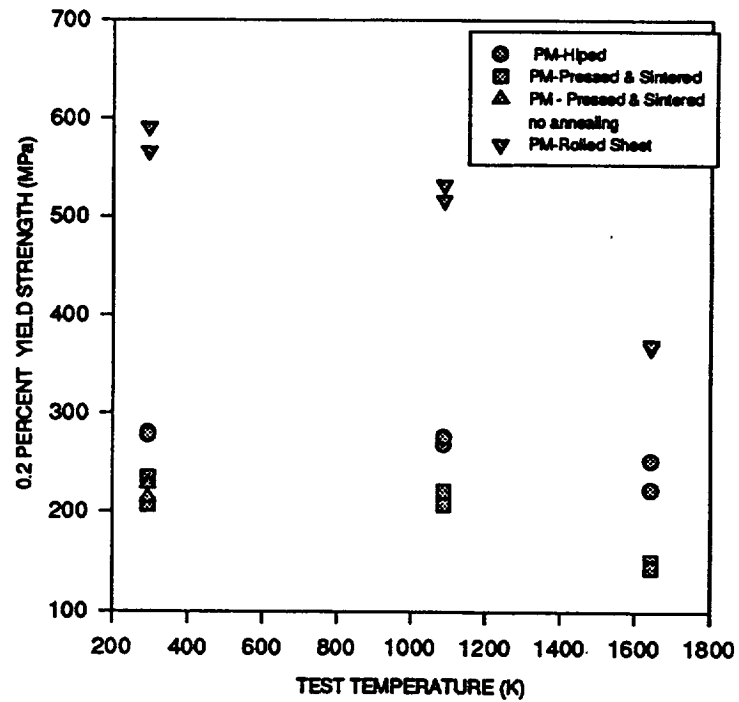


FIGURE 3-b. PM YIELD STRENGTH VS TEMPERATURE (K)

FIGURE 3. PM ULTIMATE AND YIELD STRENGTH VS TEMPERATURE (K)

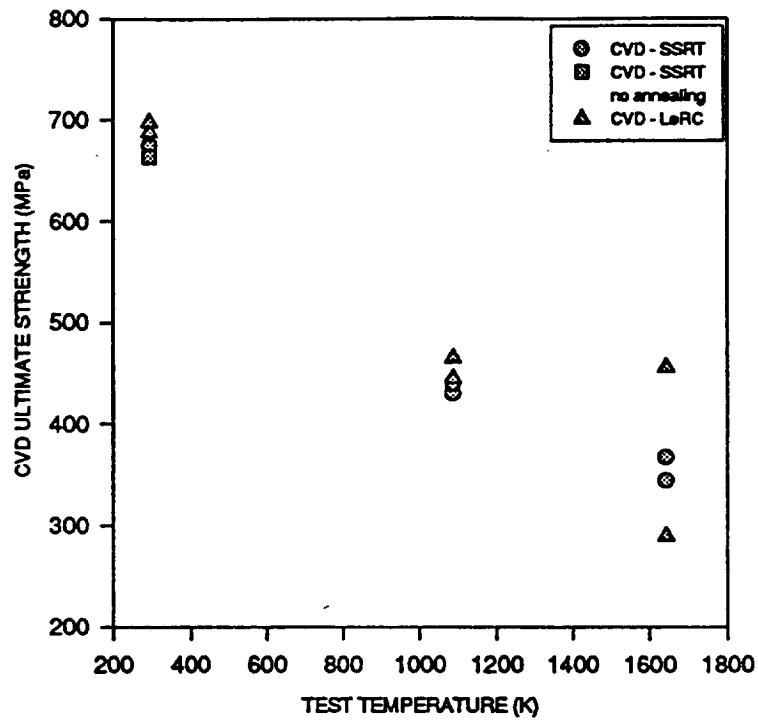


FIGURE 4-a. CVD ULTIMATE STRENGTH VS TEMPERATURE (K)

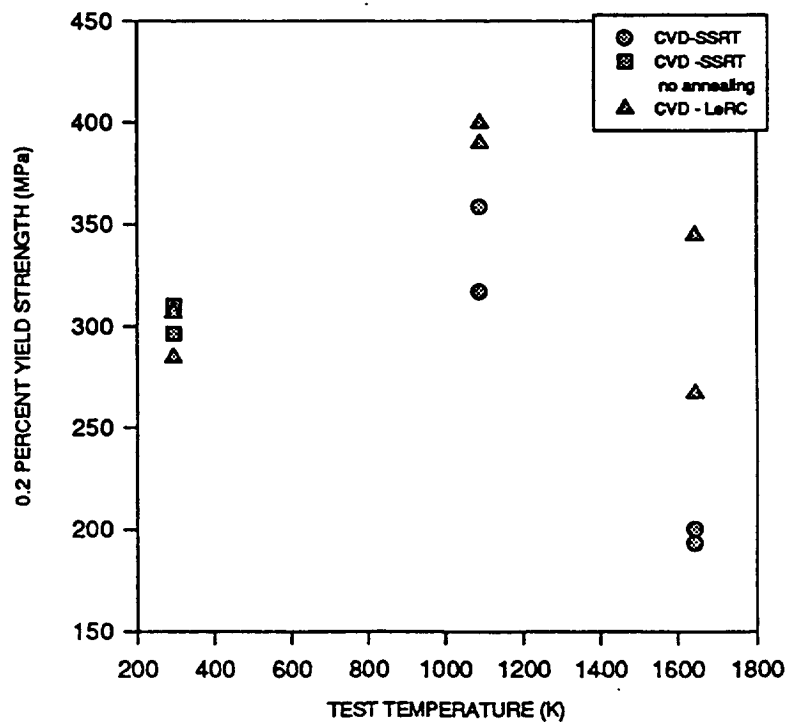


FIGURE 4-b. CVD YIELD STRENGTH VS TEMPERATURE (K)

FIGURE 4. CVD ULTIMATE AND YIELD STRENGTH VS TEMPERATURE (K)



PM rolled sheet



PM HIP rhenium

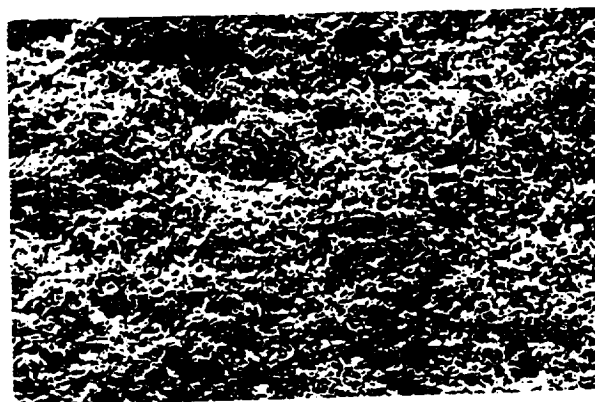


CVD rhenium

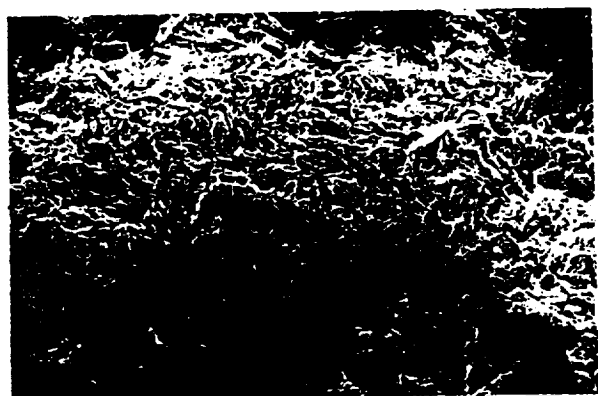
Figure 5. Typical tensile breaks for tested materials



Room Temperature HIP Material



1644 K HIP Material



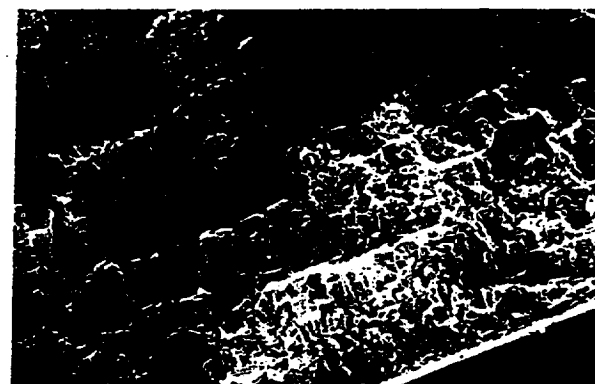
Room Temperature Rolled Sheet



1644 K Rolled Sheet



Room Temperature CVD



1644 K CVD

Figure 6.- Comparison of 60x electron microscope photographs of PM HIP, PM rolled sheet and CVD rhenium at room temperature and 1644 K.

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13. ABSTRACT (Maximum 200 words) Tensile data were obtained from four different types of rhenium at ambient and elevated temperatures. The four types of rhenium included chemical vapor deposition (CVD) and three powder metallurgy (PM) types, i.e., rolled sheet and pressed and sintered bars, with and without hot isostatic pressure (HIP) treatment. Results revealed a wide range of values with ultimate strengths at ambient temperatures varying from 663 MPa for CVD rhenium to 943 MPa for rolled sheet. A similar spread was also obtained for material tested at 1088 K and 1644 K. The wide variance observed with the different materials indicated that the rhenium manufacturing process, material composition and prior handling strongly dictated its properties. In addition to tensile properties, CVD, pressed and sintered material and HIP rhenium successfully completed 100 cycles of low cycle fatigue. Creep data were also obtained showing that CVD and pressed and sintered rhenium could sustain five hours of testing under a tension of 27.5 MPa at 1922 K.				
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