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## Paper Session I-B - Robust Low Cost Aerospike/RLV Combustion Chamber By Advanced Vacuum Plasma Process

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# Robust Low Cost Aerospike/RLV Combustion Chamber By Advanced Vacuum Plasma Process

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## Introduction

Next-generation, regeneratively cooled rocket engines will require materials that can withstand high temperatures while retaining high thermal conductivity. At the same time, fabrication techniques must be cost efficient so that engine components can be manufactured within the constraints of a shrinking NASA budget. In recent years, combustion chambers of equivalent size to the Aerospike chamber have been fabricated at NASA-Marshall Space Flight Center (MSFC) using innovative, relatively low-cost, vacuum-plasma-spray (VPS) techniques (1,2). Typically, such combustion chambers are made of the copper alloy NARloy-Z. However, current research and development conducted by NASA-Lewis Research Center (LeRC) has identified a Cu-8Cr-4Nb alloy which possesses excellent high-temperature strength, creep resistance, and low cycle fatigue behavior combined with exceptional thermal stability. In fact, researchers at NASA-LeRC have demonstrated that powder metallurgy (P/M) Cu-8Cr-4Nb exhibits better mechanical properties at 650°C (1200°F) than NARloy-Z does at 538°C (1000°F). The objective of this program is to develop and demonstrate the technology to fabricate high-performance, robust, inexpensive combustion chambers for advanced propulsion systems such as Lockheed-Martin's VentureStar and NASA's Reusable Launch Vehicle (RLV) using the VPS process. The VPS Cu-8Cr-4Nb had mechanical properties that match or exceed those of P/M Cu-8Cr-4Nb. In addition, oxidation resistant and thermal barrier coatings can be incorporated as an integral part of the hot wall of the liner during the VPS process, significantly extending the life/performance. Tensile properties of Cu-8Cr-4Nb material produced by VPS are reviewed and compared to material produced previously by extrusion. VPS formed combustion chamber liners have also been prepared and will be reported on, following scheduled hot firing tests at NASA-LeRC.

## Experimental Procedure

### Powder Production and Consolidation

Powder was purchased by MSFC from Crucible Research. Two lots were procured. Their chemistries are given in Table 1. The first lot was powder with an oxygen content of 1355 ppm. This powder was used to establish VPS parameters. The second lot of powder had significantly less oxygen, 805 ppm. This powder lot was more comparable to the powder previously supplied by Special Metals and tested at LeRC (3,4,5). The chemistry of the Special Metals powder is also included in Table 1.

Based on prior experience with NARloy-Z, a vacuum anneal at 926°C (1700°F) for 4 h was done on the VPS material to further consolidate the material and remove some of the oxygen. After consultation with Rocketdyne, the temperature was raised to 954°C (1750°F). Two different vacuum anneal furnaces were used. One allowed for a much faster cooling rate.

The slower furnace cooling was designated "Furnace Cool" (FC) and the faster cooling was designated "Rapid Cool" (RC).

For full consolidation, hot isostatic pressing (HIPing) was done on the VPS material. Two different times were used; 1 h to take into account the already high density of the vacuum annealed material and 4 h to replicate the HIPing done by Rocketdyne on their rocket engine combustion liners. In both cases a temperature of 954°C (1750°F) and a pressure of 208 MPa (30 ksi) were used. As another comparison to prior testing, one set of VPS samples was directly HIPed for 4 h without a vacuum anneal. This is approximately equivalent to prior Rocketdyne consolidation of powder via HIPing.

**Table 1 - Chemistries of Cu-8 Cr-4 Nb Powders**

	Cr (wt.% / at.%)	Nb (wt.% / at.%)	O (wt.%)
MSFC Powder Lot 1	6.45 / 8.00	5.61 / 3.90	0.1355
MSFC Powder Lot 2	6.79 / 8.33	5.99 / 4.11	0.0805
Special Metals Powder Lot 2	6.35 / 7.79	5.75 / 3.95	0.0468

### **Tensile Testing**

Tensile testing was conducted at MSFC. Unlike previous LeRC work, MSFC used a high pressure gaseous helium atmosphere to protect the samples from oxidation. All tests including room temperature were run with a 17.3 MPa (2.5 ksi) gaseous He atmosphere. The high pressure environment would tend to raise the ultimate tensile strength (UTS) and increase the ductilities of the samples slightly. The strain rate chosen was 0.0067/min to correspond to previous LeRC testing. Testing was conducted at room temperature, 538°C (1000°F) and 649°C (1200°F) as samples permitted. A base line temperature of 538°C was chosen for all samples since it fell within the range of prior test temperatures and is comparable to a rocket engine combustion chamber environment.

As a comparison to prior LeRC work, three tensile samples made from material extruded at LeRC were also tested under identical conditions at 538°C.

## **Results**

### **VPS Cu-8 Cr-4 Nb Density And Hardness**

As can be seen in Table 2, the VPS method produced a material with high densities and good hardnesses. The density of extruded Cu-8 Cr-4 Nb is 8.66 g/cm<sup>3</sup> with a 95% confidence interval of ±0.13 g/cm<sup>3</sup>. Average hardnesses of the as-extruded samples range from 73 to 78 on the Rockwell Hardness B (R<sub>B</sub>) scale.

### **Tensile Properties**

The tensile strengths are shown in Figure 1. The VPS material was given a 4 hour vacuum anneal followed by a 1 h HIP cycle. The LeRC samples tested at 538°C (1000°F) are also included for comparison. The results indicate that the material retains good strength over the temperature range tested.

The results for the elongation and reduction in areas are presented in Figure 2. The ductility of the material was still significant, but some data points had lower values than have been previously observed. The scatter of the data also appears to be greater than previously observed. The high oxygen materials retained better than expected ductility, but they were less ductile than the low oxygen samples.

**Table 2 - Hardness and Density of VPS Samples**

Condition	Hardness (R <sub>B</sub> )	Density (g/cm <sup>3</sup> )
As-Sprayed	62.6	8.48
4h Vacuum Anneal @ 954°C (1750°F)	72.3	8.60
4h Vacuum Anneal + HIP 1h/954°C (1750°F) / 208 MPa (30 ksi)	76.8	8.73
HIP only 4h/954°C (1750°F) / 208 MPa (30 ksi)	69.3	8.73

The moduli of the samples were also measured. The results appear in Figure 3. For comparison, at room temperature the modulus of OFHC copper is 117.3 GPa (17 ksi) and at 500°C it decreases to 100.0 GPa (14.5 Msi) (6).

There were only sufficient samples for the samples HIPed directly without a vacuum anneal and the material extruded at LeRC to conduct testing at 538°C. A summary of the results is presented in Table 3

## Discussion

### Effect of Processing on VPS Cu-8 Cr-4 Nb Hardness and Density

As can be seen in Table 3, the hardness and density increased as a result of vacuum annealing. The vacuum anneal appears to sinter the material. Unlike the vacuum anneal of NARloy-Z (1), it is not expected that significant reductions in oxygen content will be observed. The chromium and niobium oxides are much more stable than the copper and silver oxides found in NARloy-Z. Exposure to vacuum at these temperatures will not reduce them.

The 1 h HIP cycle increased the density and hardness to the maximum values observed. The density was higher than the value for extruded material, but within the 95% confidence interval for the extruded density. **Table 3 - MSFC Tensile Test Results for VPS + HIP and Extruded**

#### Cu-8 Cr-4 Nb Samples

Processing		0.2% Yield (MPa / ksi)	UTS (MPa / ksi)	Modulus (GPa / Msi)	Elongation (%)	Reduction In Area (%)
VPS + HIP 4 h / 954°C	Avg.	179.4	197.1	87.4	19.0	26.8
	$\sigma$	2.8	1.4	4.4	1.6	1.7
LeRC Extruded	Avg.	165.6	183.5	78.4	28.3	44.2
	$\sigma$	1.8	2.5	7.4	3.0	3.9

The 4 h HIP cycle densified the alloy more than the vacuum anneal, but the extra time at temperature appears to have coarsened the Cr<sub>2</sub>Nb precipitates and led to a lower hardness. There was no significant difference in the densities of the 1 h and 4 h HIPed material.

These results indicate that shortening the HIP time from the current 4 h to 1 h would be beneficial. Eliminating the vacuum anneal step since it cannot reduce the oxides but does coarsen the precipitates may also be beneficial.

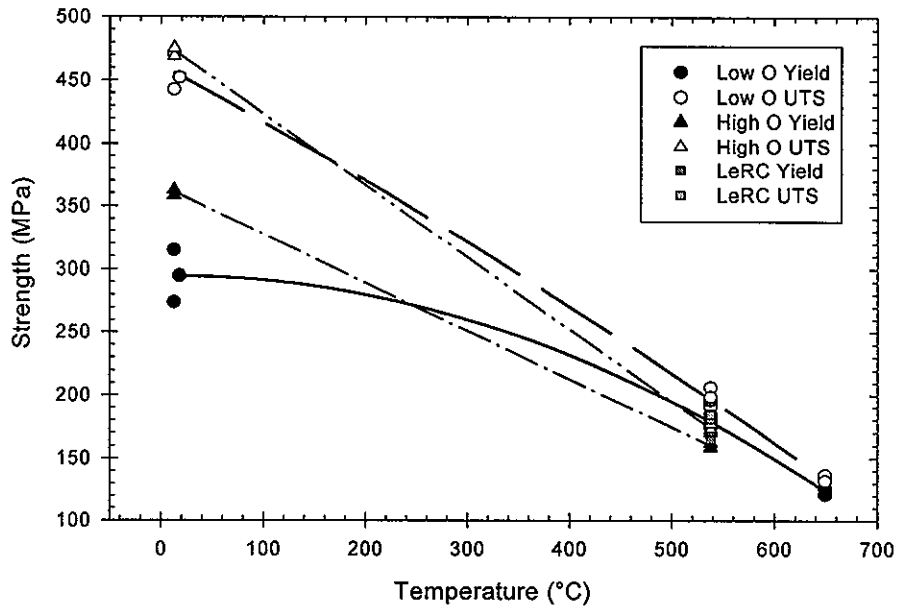


Figure 1 - VPS Cu-8 Cr-4 Nb Tensile Strengths

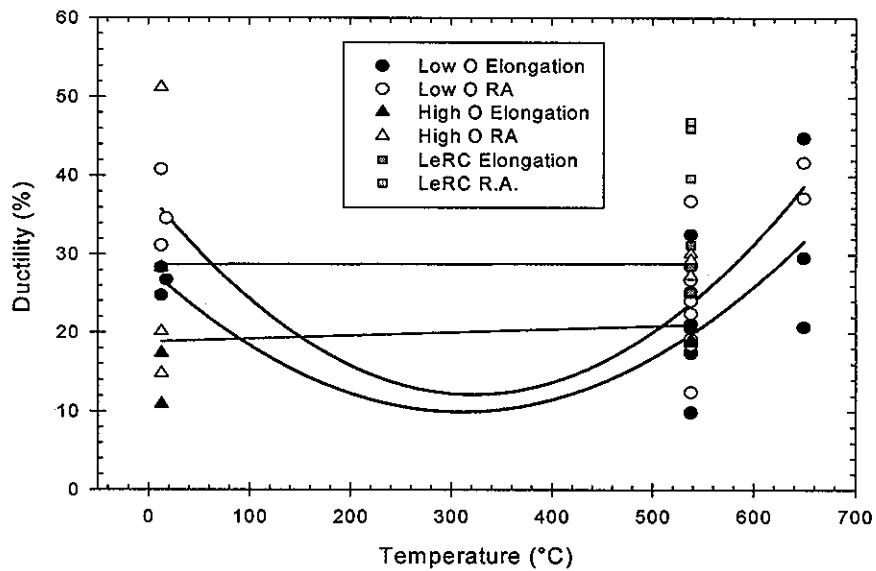
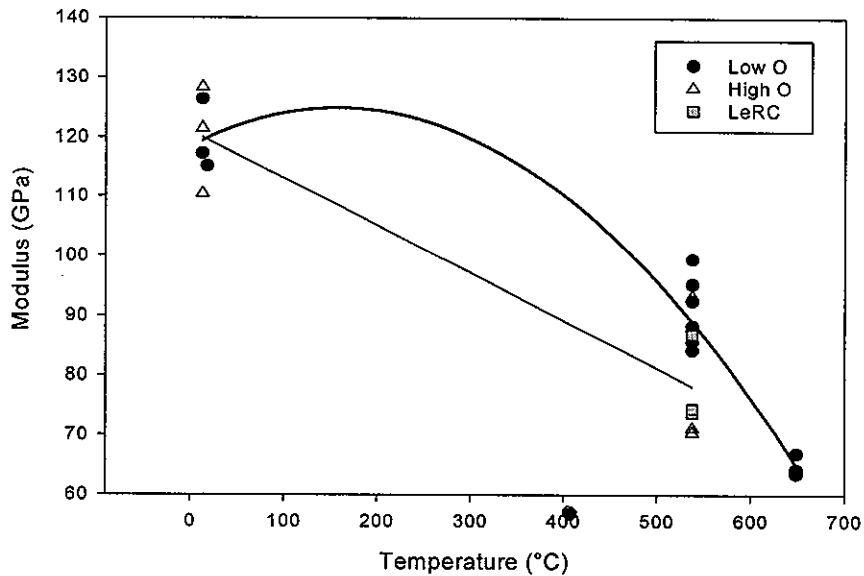


Figure 2 - VPS Cu-8 Cr-4 Nb Tensile Elongations and Reduction In Areas



**Figure 3 - Modulus of VPS Cu-8 Cr-4 Nb  
Effects of Processing and Oxygen Content on 538°C Tensile Properties**

Insufficient samples were available to test all materials at all temperatures. As a result, a base line temperature of 538°C (1000°F) was chosen to approximate the hot wall temperature of future liners. Samples from all processing routes were tested at this temperature.

Two suppliers of powder were used for making the samples. Special Metals has produced all powder for samples previously tested by LeRC. The same source of powder was used for the LeRC supplied extruded material tested by MSFC. Crucible Research has recently started to produce Cu-Cr-Nb powders. All VPS material from MSFC was made from the Crucible Research powder. Due to the small number of samples and conditions, it is impossible to determine if the difference in powder suppliers had any effect.

The averages for the 538°C tensile tests for all materials and processing conditions are presented in Figures 4 and 5. Error bars corresponding to one standard deviation are also included in the plots. To determine if the differences in the average strengths are statistically significant, the data was analyzed using SigmaStat 2.0<sup>1</sup>. The general procedure was to use an ANOVA test first to determine if the averages were statistically different followed by a Student-Newman-Keuls (SNK) pairwise comparison to determine the relative ranks of the averages. In all cases, a 95% confidence was used for the statistical analyses. When a comparison is made (equal to, greater than, less than), it means that there was a statistically significant difference at the 95% confidence limit.

For the yield strength, the analysis showed that the averages were different at a greater than 99.9% level. All five data sets passed the tests for normality and equal variance. The SNK analysis showed that the low oxygen VPS samples subjected to different cooling rates had equal yield strengths. This indicates that the effects of a furnace cool (FC) versus a rapid cool (RC) on the alloy were not important.

<sup>1</sup> SPSS Software, 233 S. Wacker Drive, 11th floor, Chicago, IL 60606-6307

The lack of difference in the yield strength when comparing a 1 h HIP to a 4 h HIP indicates that within the times tested, the effect of HIP time on the yield strength is insignificant. This is most likely a direct result of the good resistance to coarsening exhibited by the  $\text{Cr}_2\text{Nb}$  precipitates and their ability to pin copper grains and stop coarsening. It may be possible to not vacuum anneal Cu-8 Cr-4 Nb and lower the HIP time from 4 h to 1 h and achieve the same or better mechanical properties.

The low oxygen VPS material had a yield strength greater than the LeRC extruded material tested under the same conditions. The high oxygen material had the lowest yield point. It was somewhat surprising that the high oxygen material had the lowest yield point. Many fine oxides had been observed in the material, and any dissolved oxygen remaining in the copper matrix was expected to have a strengthening effect. Other factors not examined in this study may have overwhelmed the strengthening effects of oxygen.

The data sets for the UTS also passed the tests for normality and equal variance. Analysis showed that the low oxygen samples had equal average UTS averages. The LeRC extruded specimens had a lower UTS than the low oxygen material but a higher UTS than the high oxygen samples.

These results mirror the results for the yield strength and show that overall the cooling rate after the vacuum anneal did not matter but the oxygen content did. For samples with low oxygen contents, the VPS processing produced materials with a 13.8 MPa (2 ksi) higher strength compared to the extruded material.

The data for the modulus showed that the data sets were all normal and had equal variance. The ANOVA analysis revealed that there was no statistically significant difference in the average of the moduli ( $P=0.178$ ).

The elongation data sets failed the test for normality. A Kruskal-Wallis one-way ANOVA of ranks was used to determine if there was a statistically significant difference in the averages of the elongations. The results indicated that there was no differences ( $P=0.294$ ).

Finally, the data for reduction in area were analyzed. The data sets again failed the test for normality, so the Kruskal-Wallis one-way ANOVA of ranks was used to determine if there was a statistically significant difference in the averages. It was determined that there were differences. SNK analysis showed that the VPS material had equal average reductions in area, but all four conditions tested resulted in lower reductions in area than the LeRC extruded material.

In summary, the low oxygen VPS material had the highest strength at 538°C but a lowered reduction in area compared to extruded material. Elongations and moduli were equal for all materials tested. Extruded materials had a slightly lower strength, but the best reduction in area. High oxygen VPS material was the worst material tested. For future processing, VPS spraying of low O Cu-8 Cr-4 Nb with a direct 1 h HIP shows promise of producing the best tensile strengths while retaining good ductility.

### **Comparison of Tensile Strength at Other Temperatures**

Insufficient data points exist to make the careful statistical comparison of the VPS and extruded materials at all temperatures as was done with the 538°C data set. However, simplified comparisons can be made of the relative properties of the materials by plotting the low O data and the results of prior LeRC analysis.

Figure 6 shows the data generated for the low O VPS material. Prior LeRC work has resulted in a model to predict the effect of temperature on strength. The model also allows prediction of confidence intervals for the data.

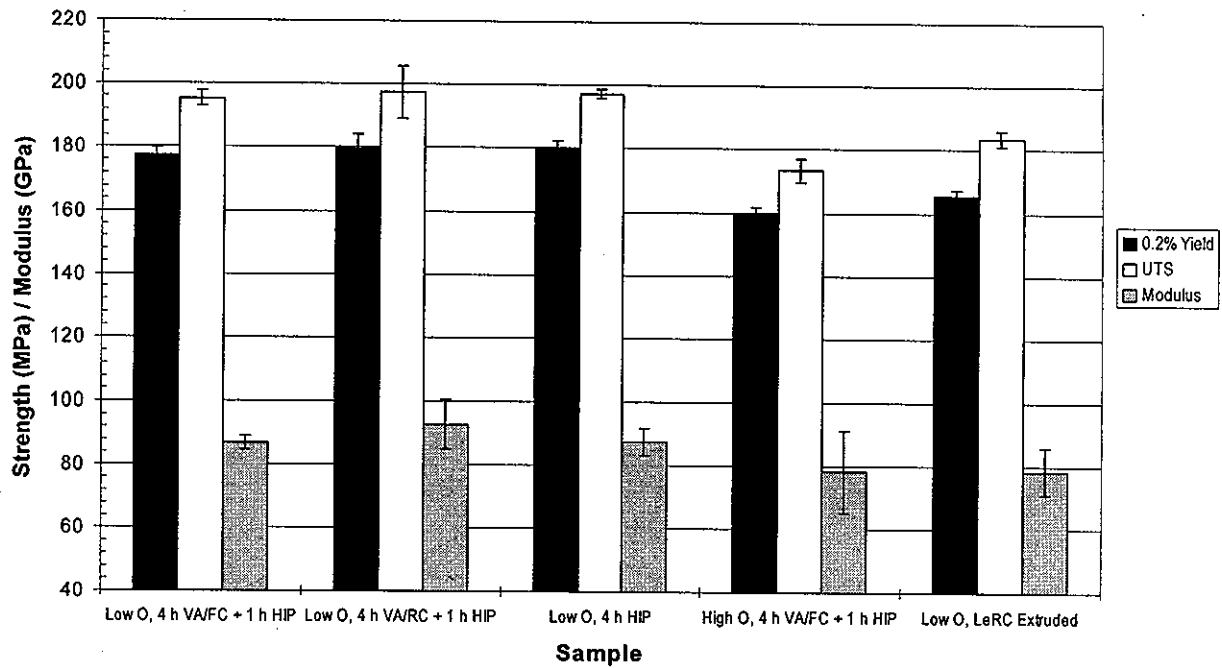


Figure 4 - Effect of Processing and Oxygen on 538°C Strength and Modulus

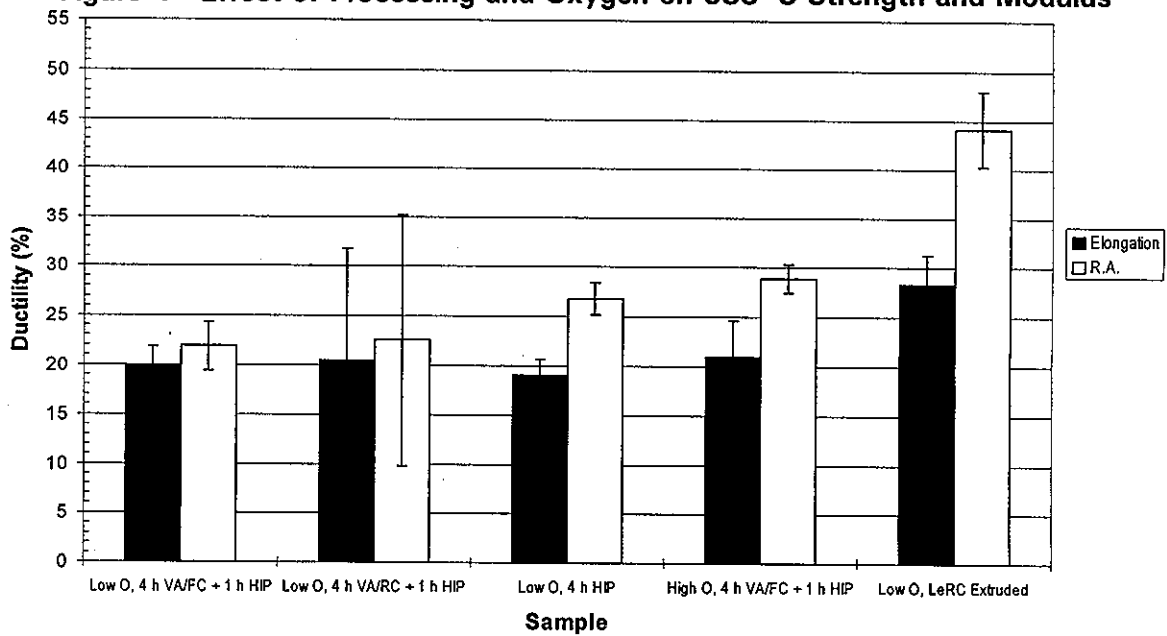
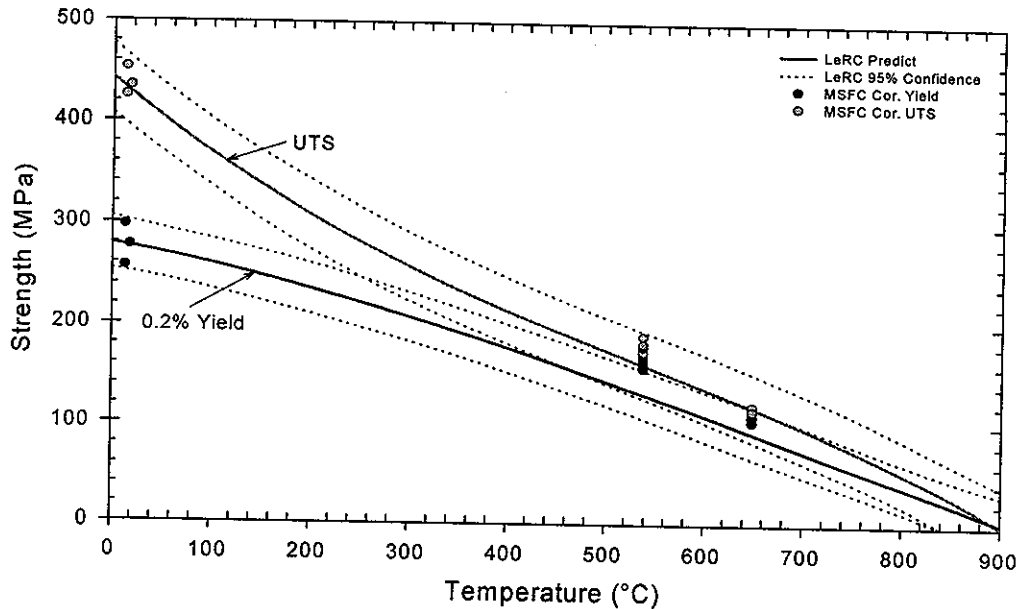


Figure 5 - Effect of Processing and Oxygen on 538°C Ductility





**Figure 6 - Comparison of Low Oxygen VPS Material to LeRC Strength Model**

Plotting the data shows that, with the exception of the 538°C yield strength, the VPS data falls within the 95% confidence interval for the previously tested extruded material. From this it is possible to conclude that there is no significant difference in the strength of material produced by the two processing routes except as noted.

## Summary and Conclusions

The low oxygen vacuum plasma sprayed (VPS) Cu-8 Cr-4 Nb exhibits a higher strength than Cu-8 Cr-4 Nb produced by extrusion at elevated temperatures and a comparable strength at room temperature. Moduli and ductilities were not significantly different.

The ability to produce parts to near-net shape and maintain the good elevated temperature tensile properties of the extruded Cu-8 Cr-4 Nb makes VPS an attractive processing method for fabricating rocket engine combustion liners.

## Future Testing

A combustion chamber liner spool piece has been fabricated will be hot fire tested at LeRC. Database development for extruded and HIPed material is currently underway at NASA LeRC as part of the RLV program.

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