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Young's Moduli of Cold and Vacuum Plasma Sprayed Metallic Coatings

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Abstract

Monolithic metallic copper alloy and NiCrAlY coatings were fabricated by either the cold spray (CS) or the vacuum plasma spray (VPS) deposition processes. Dynamic elastic modulus property measurements were conducted on these monolithic coating specimens between 300 K and 1273 K using the impulse excitation technique. The Young's moduli decreased almost linearly with increasing temperature at all temperatures except in the case of the CS Cu-23%Cr-5%Al and VPS NiCrAlY, where deviations from linearity were observed above a critical temperature. It was observed that the Young's moduli for VPS Cu-8%Cr were larger than literature data compiled for Cu. The addition of 1%Al to Cu-8%Cr significantly increased its Young's modulus by 12 to 17% presumably due to a solid solution effect. Comparisons of the Young's moduli data between two different measurements on the same CS Cu-23%Cr-5%Al specimen revealed that the values measured in the first run were about 10% higher than those in the second run. It is suggested that this observation is due to annealing of the initial cold work microstructure resulting form the cold spray deposition process.

1.0 Introduction

Combustion liner materials in a liquid hydrogen (LH_2) fueled rocket engine experience extreme conditions due to a combination of environmental and thermo-mechanical effects, where the combustion flame temperatures in the chamber interior are about 3600 K whereas the backside of the 1 mm thick liner wall experiences cryogenic temperatures of 20 K [1,2,3,4,5,6]. Copper and its alloys have been traditionally used as combustor liner materials in these regenerative rocket engines because of their high thermal conductivity to enable efficient heat transfer from the combustion flame to preheat the cryogenic LH_2 flowing in the cooling channels. It is anticipated that the design of the next generation of reusable launch vehicles (RLVs) would use GRCop-84 (Cu-8(at.%)Cr-4%Nb) copper alloy liners due to its superior properties compared to other conventional copper alloys, such as NARloy-Z [7,8,9]. However, uncoated copper alloy liners undergo environmental degradation due to a combination of the spallation of the copper oxide scale and "blanching", which consists of repeated oxidation of the copper matrix and subsequent reduction of the oxide scale [6].

The application of protective coatings on GRCop-84 and other copper alloy substrates can either minimize or eliminate many of the problems experienced by uncoated liners and significantly extend their operational lives in RLVs. This factor potentially translates to increased component reliability, shorter depot maintenance turn around time and lower operational cost. In addition, the use of a suitable top coat to act as a thermal barrier can allow the engine to run at higher temperatures thereby resulting in its

increased thermal efficiency. As a result, several types of ceramic [1,5] and metallic [10,11,12,13,14,15] coatings have been advocated as protective coatings for copper alloy liners. However, differences in the mechanical and thermophysical properties between the coatings and copper alloy substrates can lead to the development of large residual stresses and coating spallation as the coated liner experiences variations in temperature during processing and engine operation [16].

Recently, it was demonstrated that CuCrAl and NiCrAlY coatings deposited either by the cold spray (CS) or the vacuum plasma spray (VPS) techniques are potentially viable coatings for GRCop-84 combustion liners [14,15]. However, elastic moduli and thermophysical data for these sprayed coatings are either limited or nonexistent in the temperature range of interest for use in RLVs. Although some data on VPS alloys have been previously reported in the literature [17,18,19], it is noted that these properties are sensitive to compositional and processing variables. Thus, it is essential that thermophysical data be generated on coatings sprayed under processing conditions and for compositions similar to those developed for spraying the GRCop-84 liners in order to ensure reliable design models to be developed.

The specific objectives of this paper are to report the temperature dependence of the dynamic, E_D, and static, E_S, Young's moduli of CS and VPS monolithic Cu-Cr, CuCrAl, and NiCrAlY coating alloys between 300 and 1273 K.

2.0 Experimental Procedures

2.1 Alloy Composition and Processing

Gas atomized copper alloy powders were procured from Crucible Research, Inc., Pittsburgh, Pennsylvania, whereas the NiAl and NiCrAlY powders were obtained from Homogenous Metals, Inc., New York, and Praxair, Indianapolis, Indiana, respectively. The nominal compositions of the alloy powders were Cu-8(wt.%)Cr, Cu-26(wt.%)Cr, Cu-8(wt.%)Cr-1%Al, Cu-23(wt.%)Cr-5%Al, and Ni-17(wt.%)Cr-6%Al-0.5%Y. Monolithic cylindrical coatings, typically 175-250 mm long and 19 to 25 mm thick, were fabricated by spraying the powders on rotating aluminum or steel mandrels by either CS or VPS. The Cu-23%Cr-5%Al coatings were cold sprayed at ASB Industries, Inc., Barberton, Ohio [20]. The Cu-8%Cr, Cu-26%Cr, Cu-8%Cr-1%Al, and NiCrAlY coatings were deposited by the vacuum plasma spray method at Plasma Processes, Inc., Huntsville, Alabama. The coated mandrels were hot isostatically pressed (HIP) between 1073 and 1273 K under argon gas pressures varying between 100 and 210 MPa for times varying between 1 and 4 hr.

Specimens were machined from the sprayed cylinders by electrodischarge machining (EDM). Dynamic Young's moduli measurements were made on specimens with dimensions 50x4x3 mm by the impulse excitation technique (IET) [21,22,23] using a commercially available GrindoSonic MK5I test equipment equipped with a furnace and an environmental test chamber. The theoretical foundations of this technique for measuring elastic constants are described elsewhere [21,24,25]. The measurements were conducted between room temperature and 1273 K under flowing Ar. The bar specimen was supported at two points corresponding to its vibrational nodes, which occur at distances of 0.224 L, where L is the specimen length, from each end of the specimen. A small ceramic projectile propelled by low pressure Ar was used to lightly apply a mechanical impulse load on the specimen, and the generated out-of-plane fundamental resonant frequency, f_R, of the bar in flexure at absolute temperature, T, was recorded by an acoustic microphone at the desired rate. The specimen temperature was increased at 100 K/hr, and the resonant frequency, time and temperature were automatically recorded every 5 K by a computerized data acquisition system. The uncorrected Young' modulus was determined from the equation [21,22,24]

$$E_D = [0.9465 * \{M*(f_R)^2/w\}]\ (t/L)^3\ T_1 \tag{1}$$

where M is the mass of the specimen in gm, and w and t are the width and thickness, respectively, of the specimen in mm, and T_1 is a correction factor related to the Poisson's ratio, ν, through

$$T_1 = 1 + 6.585\left(1 + 0.0752\nu + 0.8109\nu^2\right)\left(\frac{t}{L}\right)^2 - 0.868\left(\frac{t}{L}\right)^4 - \left[\frac{8.340\left(1 + 0.2023\nu + 2.173\nu^2\right)\left(\frac{t}{L}\right)^4}{1 + 6.338\left(1 + 0.1408\nu + 1.536\nu^2\right)\left(\frac{t}{L}\right)^2}\right] \quad (2)$$

A value of $\nu = 0.33$ was assumed in evaluating T_1. It has been recommended that the values of E_D be corrected for effects due to thermal expansion using [22]

$$E_{DT} = E_{D0}\left(\frac{f_R}{f_0}\right)\left[\frac{1}{\left(1 + \alpha_T \Delta T\right)}\right] \quad (3)$$

where E_{DT} and E_{D0} are the Young's moduli are at test temperature and room temperature, respectively, f_0 is the resonant frequencies at room temperature, respectively, α_T is the average coefficient of thermal expansion (CTE) and ΔT is the temperature differential between test temperature and room temperature. The values of α_T in eq. (3) represent the average linear thermal expansion between room temperature and test temperature. Based on experimental measurements of α_T [26], applying the temperature correction given be eq. (3) to the experimental data resulted in a maximum decrease in the magnitude of E_D by about 1.5 to 2.0% at the highest test temperature, which is insignificant in most applications. Thus, the values of E_D reported in this paper do not include corrections for CTE.

Dynamic and static Young's moduli measurements were conducted on sprayed monolithic coating alloys. The static moduli were determined on round tensile NiCrAlY specimens with a gage length of 25 mm and gage diameter of 6.3 mm using point contact extensometers under engineering strain rates varying between 10^{-6} to 10^{-4} s^{-1}. Since the magnitudes of E_S were independent of strain rate, the strain rates at which static moduli data were generated are not distinguished in this paper. These tests were conducted either in air in the case of NiCrAlY or under flowing Ar in the case of the copper alloys between room temperature and 1273 K. There was insufficient material of the near full density Cu-26%Cr (V2-03-524) batch to produce specimens of sufficient length for dynamic Young's modulus measurements.

3.0 Results and Discussion

3.1 Density Measurements

Table 1 gives the bulk density, $\rho_{experimental}$, for the different coatings[1] measured at room temperature. Microstructural observations of Cu-26Cr (V2-02-27B) revealed that it had a relatively higher amount of porosity compared to the other sprayed coatings, which were nearly 100% dense. The porosity content of this batch was determined to be about 35% from a comparison of the bulk and immersion densities, where the latter was determined to be 8337 Kg/m^3. The theoretical density, $\rho_{theoretical}$, was estimated to be 8410 Kg/m^3. This high level of porosity was attributed to non-optimized processing conditions for this alloy. Although the density of NiCrAlY (V2-02-27E) is similar to the experimental values of 6900 to 7500 Kg/m^3 [18,27] and theoretical value of 7000 Kg/m^3 reported for plasma sprayed NiCrAlY [27], this batch had a larger amount of porosity than NiCrAlY (V2-03-528), which was 100% dense. The porosity

[1]The bulk density measurements were conducted at the Thermophysical Properties Research Laboratory, Inc. (TPRL), West Lafayette, Indiana.

content of the NiCrAlY batch V2-02-27E was estimated to be about 7% based on a comparison of its density with that of the NiCrAlY batch, V2-03-528.

TABLE 1.—MAGNITUDES OF ROOM TEMPERATURE BULK DENSITY OF DIFFERENT SPRAYED MONOLITHIC COATINGS[a]

Nominal coating composition[b]	Batch I.D.	Processed condition	Bulk density [c] (kg/m^3)
Cu-8%Cr-4%Nb	GRCop-84	Extruded	8945
Cu-8%Cr	V2-03-134	VPS	8598
Cu-26%Cr[d]	V2-02-27B	VPS	5450
Cu-8%Cr-1%Al	V2-05-27	VPS	8546
Cu-23%Cr-5%Al	Cu23Cr5Al	CS	7575
NiCrAlY[d]	V2-02-27E	VPS	7161
NiCrAlY	V2-03-528	VPS	7711

[a]The bulk density measurements were conducted at the Thermophysical Properties Research Laboratory, Inc. (TPRL), West Lafayette, Indiana
[b]All compositions are in wt.%.
[c]Calculated from the ratio of the mass to the geometric volume of the specimen.
[d]These specimens had various amounts of porosity. All other sprayed specimens were close to 100% density.

3.2 Vacuum Plasma Sprayed Coatings

3.2.1 Cu-Cr Coatings

Figure 1 shows the decrease in E_D with increasing T for the Cu-8Cr and Cu-26Cr monolithic coatings. Owing to the limited solid solubility of Cr in Cu [28], the Cu-Cr alloy can be considered to be a mechanical mixture. Fig. 1 also shows the predicted values, E_{ROM}, calculated from the rule of mixtures (ROM) model:

$$E_{ROM} = V_{Cr}\, E_{Cr} + (1 - V_{Cr})\, E_{Cu} \tag{4}$$

where V_{Cr} is the volume fraction of the Cr phase, and E_{Cr} and E_{Cu} are the Young's moduli of pure Cr and Cu, respectively. The temperature dependence of E_{Cr} and E_{Cu} were calculated from the equations published by Frost and Ashby [29] and Raj and Langdon [30] for the Young's moduli of Cr and Cu, respectively. The density-corrected data for Cu-26Cr are also shown, where the experimental values were corrected by multiplying them by ($\rho_{theoretical}/\rho_{experimental}$).

The Young's moduli for the two alloys decrease almost linearly with increasing temperature similar to other materials [29,31]. The density-corrected and uncorrected experimental data could be well represented by the equation [32]

$$E_D = E_0 - (\partial E_D/\partial T)\, T \tag{5}$$

where E_0 is the extrapolated Young's modulus to absolute zero[2] and ($\partial E_D/\partial T$) is the rate of change of Young's modulus with absolute temperature. The magnitudes of E_0 and ($\partial E_D/\partial T$) determined from linear regression analyses of the experimental data between 300 and 750 K are given in Table 2, where R_d^2 is the coefficient of determination. Above 750 K, the data deviated from the regressed lines to lower values presumably due to the effects of significant atomic diffusion. The presence of Cr particles in Cu-8%Cr increases the magnitude of E_D above that for Cu [30] by about 12% at room temperature but their effect decreases with increasing temperature so that the E_D for both materials are comparable above 900 K.

[2]It is important to note that the magnitude of E_0 is likely to be higher than the actual value of E_D at 0 K [31].

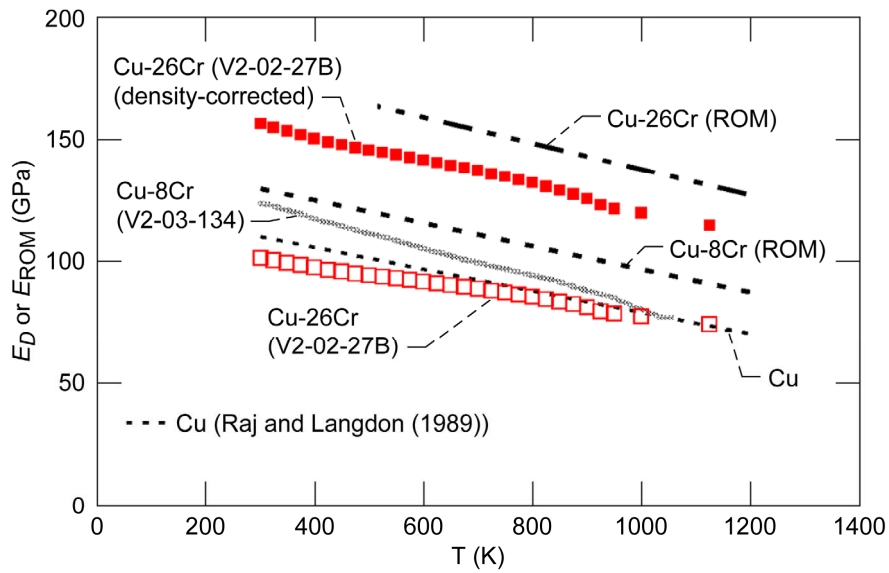

Figure 1.—Comparison of the experimental dynamic Young's moduli for VPS Cu-8%Cr and Cu-26%Cr with compiled data for Cu [30] and predicted values determined by the rule of mixtures.

TABLE 2.—REGRESSION PARAMETERS DESCRIBING THE TEMPERATURE DEPENDENCE OF THE YOUNG'S MODULI FOR MONOLITHIC COATINGS

Nominal coating composition[a]	Batch I.D.	Processed condition	E_0 (GPa)	$\partial E_D/\partial T$ (GPa/K)	R_d[b]
Cu-8%Cr	V2-03-134	VPS	142.0	0.060	0.997
Cu-26%Cr[b]	V2-02-27B	VPS	110.0	0.030	0.995
Cu-26%Cr[c]	V2-02-27B	VPS	169.8	0.047	0.998
Cu-8%Cr-1%Al	V2-05-27	VPS	158.7	0.065	0.988
Cu-23%Cr-5%Al	Cu23Cr5Al (specimen 3)	CS (Run 1)	166.0	0.071	0.989
Cu-23%Cr-5%Al	Cu23Cr5Al (specimen 3)	CS (Run 2)	147.3	0.059	0.995
NiCrAlY[b]	V2-02-27E	VPS	221.8	0.057	0.999
NiCrAlY	V2-03-528	VPS	232.7	0.060	0.998

[a]All compositions are in wt.%.
[b]These specimens had various amounts of porosity. All other sprayed specimens were close to 100% density.
[c]Density-corrected equation.

The uncorrected values of E_D for Cu-26%Cr are similar to the mean regression data for pure Cu [30] and lie below the experimental data for Cu-8%Cr due to the fact that this batch had a considerable amount of porosity. However, the density-corrected values for Cu-26%Cr with a calculated value of V_{Cr} of about 30 vol.% Cr are higher than those for Cu-8%Cr for which the calculated $V_{Cr} \sim 9$ vol.%. An examination of Fig.1 shows that the values of E_D predicted by the rule of mixtures are significantly higher than the experimental data especially for Cu-26%Cr.

3.2.2 Effect of Al addition

Figure 2 compares the temperature dependence of E_D for Cu-8%Cr-1%Al with those for Cu [30] and Cu-8%Cr. The data for Cu-8%Cr-1%Al shown in Fig. 2 represent the average values of measurements made on two specimens. It is noted that these two sets of data almost overlapped each other between 300 and 1000 K thereby indicating excellent reproducibility in the measurements. The magnitudes of E_D for the alloys exhibit an inverse linear dependence on absolute temperature, where the values of E_0 and

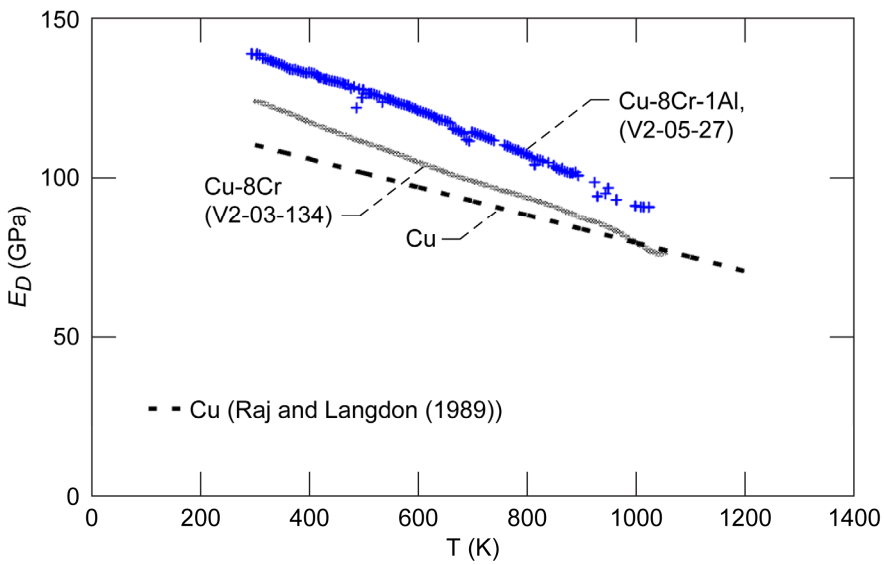

Figure 2.—Comparison of the experimental dynamic Young's moduli for VPS Cu-8%Cr-1%Al and Cu-8%Cr with compiled data for Cu [30].

($\partial E_D/\partial T$) are given in Table 1. It is clear from Fig. 2 that the addition of 1% Al to Cu-8%Cr results in a significant increase in the elastic modulus by 12 to 17% between 300 and 1000 K through its influence on E_0; its effect on ($\partial E_D/\partial T$) is relatively insignificant (Table 2). Since Al exists in solid solution in Cu at this relatively low compositional level, this increase in E_D can be attributed entirely due to a solid solution effect. Noting that the densities of Cu-8%Cr and Cu8%Cr-1%Al are similar (Table 1), an examination of eq. (1) suggests that the lattice distortion due to the presence of Al atoms in the Cu lattice most likely increased the magnitude of f_R over the base alloy.

3.2.3 NiCrAlY Coatings

Figure 3 compares the magnitudes of dynamic and static Young's moduli for the two batches of NiCrAlY coatings. The data of Cook et al. [19,33] for an alloy of similar composition are also shown in the figure for comparison. The data are well represented by eq. (5) between 300 and 1000 K. The two sets of data for batch V2-03-528, which are nearly identical, lie above the data for batch V2-02-27E consistent with their higher density (Table 1). The differences in the magnitudes of Young's moduli determined for batches V2-02-27E and V2-03-528 varied between 2.5 and 7.5% in the temperature range 300 to 1150 K primarily reflected in the magnitudes of E_0 rather than ($\partial E_D/\partial T$). The values reported by Cook et al. [19,33] are in excellent agreement with the magnitudes of E_D determined for batch V2-02-27E. The static moduli values are lower than the dynamic moduli and exhibit more scatter especially at the higher temperatures.

3.3 Cold sprayed coatings

The magnitudes of E_D decreased linearly with increasing absolute temperature for cold sprayed Cu-23Cr-5Al alloys between 300 and 700 K (Fig. 4). The data for VPS Cu-8Cr-1Al are shown for comparison. Deviation from linearity was observed above 700 K. Interestingly, the Young's moduli were higher for specimens tested in the first run, "Run 1", compared to measurements made in the second run, "Run 2". As shown in Fig. 4, these differences were quite reproducible in repeat tests. Noting that the cold spray process involves extensive deformation of the powder particles during the deposition process [34], the higher values of E_D observed in "Run 1" can be attributed to a highly cold worked state of the coating. Thus, the decrease in the magnitudes of E_D in Run 2 can be attributed to the effects of annealing

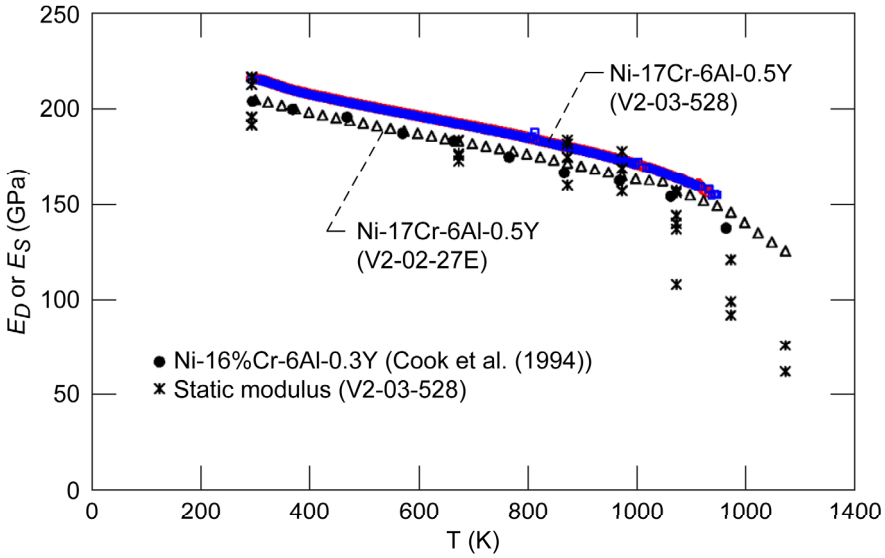

Figure 3.—Comparison of the experimental dynamic and static Young's moduli data for two batches of VPS Ni-17%Cr-6%Al-0.5%Y coating. Data determined from two measurements for NiCr AlY (V2-03-528) are shown. Literature data on Ni-16%Cr-6Al-0.3Y are shown foe comparison [19, 33].

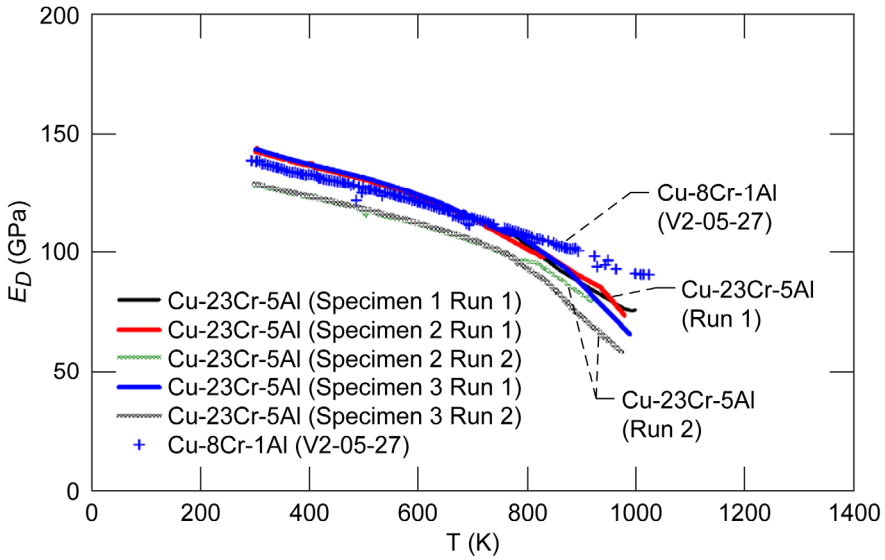

Figure 4.—Comparison of the experimental dynamic Young's moduli data for CS Cu-23%Cr-5%Al with that for VPS Cu-8%Cr-1%Al coatings. The first set of measurements conducted on a Cu-23%Cr-5%Al specimen corresponding to the as-received material is termed "Run 1" while the repeat set of measurements are termed "Run 2". Three sets of data are shown for Run 1 and two sets are shown for Run 2.

as the specimens were heated from room temperature to about 1000 K in Run 1. Although the Young's moduli for Cu-23Cr-5Al are comparable to those for Cu-8Cr-1Al below 700 K in measurements made in Run 1, they exhibit a relatively steep drop with increasing temperature above 700 K dropping to values well below those for Cu-8Cr-1Al. Significantly, increasing the amount of Al from 1 to 5% and the Cr from 8 to 23% has a negligible effect on the magnitudes of E_D for these two alloys below 700 K.

The precise reason as to why E_D for Cu-23Cr-5Al decreases below that for Cu-8Cr-1Al above this temperature is still unclear but two possible causes could be considered. First, the increasing dissolution of the α-Cr precipitates with increasing temperature may have caused the observed decrease in E_D. However, since the maximum solid solubility of Cr in Cu is only about 0.89(at.%) [28], it does not appear probable that Cr dissolution can account for the present observations especially since VPS Cu-8Cr-1Al does not show a similar significant decrease in E_D with increasing temperature. Second, extensive secondary recrystallization of the (Cu,Al) matrix resulting from the prior deformation of the powder particles and the presence of the α-Cr phase may have resulted in strong annealed texture in the specimens. Since it is well known that elastic moduli are sensitive to texture, this explanation appears to be the most plausible in the present instance.

4.0 Summary and Conclusions

The temperature dependence of the dynamic Young's moduli of several copper alloy and NiCrAlY monolithic coating alloys fabricated either by the cold spray or vacuum plasma spray process were measured by the impulse excitation techniques between 300 and 1000 K. Tensile static moduli measurements were also conducted on the NiCrAlY coating in the same temperature range. The Young's moduli decrease with increasing temperature, where this decrease is linear at low and intermediate temperatures. The Young's moduli for Cu-8%Cr were higher than compiled data for pure Cu [30] below 1000 K but below the values predicted by the rule of mixtures. The experimental values of Cu-26%Cr coating exhibited values similar to the compiled literature values presumably due to a large amount of porosity. However, the density-corrected data were significantly higher than the values for pure Cu and Cu-8%Cr. The addition of 1%Al to Cu-8%Cr significantly increased the dynamic Young's modulus of the alloy presumably due to a solid solution effect. There was no significant difference in the Young's moduli data for the as-received cold sprayed Cu-23%Cr-5%Al and vacuum plasma sprayed Cu-8%Cr-1%Al coatings below 700 K thereby suggesting that the variations in the Al and Cr content between the two alloys did not affect E_D. However, the magnitudes of E_D for the cold sprayed Cu-23%Cr-5%Al determined in the second run of measurements were lower than the values obtained on the as-received material in the first run presumably due to the effects of annealing on the as-received cold worked microstructure of the alloy. The dynamic Young's moduli for the NiCrAlY coating were reproducible and larger than the static moduli for the coating.

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14. ABSTRACT
Monolithic metallic copper alloy and NiCrAlY coatings were fabricated by either the cold spray (CS) or the vacuum plasma spray (VPS) deposition processes. Dynamic elastic modulus property measurements were conducted on these monolithic coating specimens between 300 K and 1273 K using the impulse excitation technique. The Young's moduli decreased almost linearly with increasing temperature at all temperatures except in the case of the CS Cu-23%Cr-5%Al and VPS NiCrAlY, where deviations from linearity were observed above a critical temperature. It was observed that the Young's moduli for VPS Cu-8%Cr were larger than literature data compiled for Cu. The addition of 1%Al to Cu-8%Cr significantly increased its Young's modulus by 12 to 17% presumably due to a solid solution effect. Comparisons of the Young's moduli data between two different measurements on the same CS Cu-23%Cr-5%Al specimen revealed that the values measured in the first run were about 10% higher than those in the second run. It is suggested that this observation is due to annealing of the initial cold work microstructure resulting form the cold spray deposition process.

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Young's modulus; Cold sprayed coatings; Vacuum plasma sprayed coatings; NiCrAlY; Copper alloys; Launch vehicles

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