Mechanical and fatigue properties of diamond-reinforced Cu and Al metal matrix composites prepared by cold spray

O. Kovarik¹, J. Cizek², S. Yin³, R. Lupoi³, M. Janovska⁴, J. Capek¹, J. Siegl¹, T. Chraska²

¹ *Faculty of Nuclear Sciences and Physical Engineering, Czech Technical University in Prague, Prague, Czech Republic*

² *Institute of Plasma Physics of the Czech Academy of Sciences, Prague, Czech Republic*

³ *Trinity College Dublin, The University of Dublin, Dublin, Ireland*

⁴ *Institute of Thermomechanics of the Czech Academy of Sciences, Prague, Czech Republic*

Abstract

Diamond-reinforced composites prepared by cold spray are emerging materials simultaneously featuring outstanding thermal conductivity and wear resistance. Their mechanical and fatigue properties relevant to perspective engineering applications were investigated using miniature bending specimens. Cold sprayed specimens with two different mass concentrations of diamond 20% and 50% in two metallic matrices (Al – lighter than diamond, Cu – heavier than diamond) were compared with the respective pure metal deposits. These pure metal coatings showed rather limited ductility. The diamond addition slightly improved ductility and fracture toughness of the Cu-based composites, having a small effect also on the fatigue crack growth resistance. In case of the Al composites, the ductility as well as fatigue crack growth resistance and fracture toughness have improved significantly. The static and fatigue failure mechanisms were fractographically analyzed and related to the microstructure of the coatings, observing that particle decohesion is the primary failure mechanism for both static and fatigue fracture.

Introduction

Diamond-based metal matrix composites (DMMC) combine properties of a ductile metal matrix with the high hardness and thermal conductivity of the diamond reinforcement. This combination predetermines their application as superior thermally conducting abrasives (i.e., materials that can absorb the friction-generated heat easily), used, e.g., in cutting and grinding devices. Such applications demand good mechanical and fracture properties of the DMMC. These properties are defined by the qualities of the matrix, matrix-particle interface, and the reinforcement particles. There are several production techniques for DMMC, including sintering techniques [1], pressure infiltration [2], and cold spray deposition [3,4]. The former two methods require high temperature processing that may lead to high residual thermal stress development or even diamond graphitization. Cold spray can deposit DMMCs without exposing the materials to high temperatures, thereby avoiding the graphitization, tensile residual stresses

development, and limiting several other drawbacks inherently associated with the high-temperature methods. The difficulty of this approach lies in the fact that diamond-based coatings are not easily deposited by cold spray. However, recent studies showed that cladding of the diamond particles significantly increases their deposition efficiency and prevents the diamond phase fragmentation [3,4], enabling cold spraying of thick, high-diamond content coatings by this method.

This paper explores the relation between the architecture of cold sprayed DMMC composite deposits and their engineering properties. The double-clad diamond particles are sprayed together with lighter Al or heavier Cu matrices. For each, three types of deposits with different matrix to particles density ratio are investigated. The characterization of basic stress-strain, fracture mechanics, as well as fatigue properties is complemented with residual stress measurement and a fractographic study in order to unveil the contribution of the individual DMMC phases to its final performance.

Experimental

The feedstock powders consisted of a mechanical mixture of either Al (-58+15 µm, Valimet, USA) or Cu (-38+15 µm, Safina, Czech Republic) powders with a double-clad diamond powder. The cladded particles (-53+45 µm, PDA C50, Element-Six, Ireland) consisted of a diamond core cladded with thin Ni interlayer and Cu shell. The weight ratio of the cladding-to-diamond was approximately 1:1, resulting in a cladding layer thickness of 2-5 µm. Aside from two pure metals used as reference (denoted as Al100, Cu100), four composites containing 20 wt.% and 50 wt.% of the diamond phase were deposited (denoted as Al80, Cu80, and Al50, Cu50). The coatings were deposited by an in-house developed cold spray system (Trinity College Dublin, Ireland). The system consists of high-pressure nitrogen/helium gas from cylinders, gas heater, powder feeder, CNC working platform for controlling the substrate movement, de-Laval nozzle, and computer control system. The nozzle used for spraying has a throat diameter and an exit diameter of 2 mm and 6 mm, respectively, with a total length of 210 mm. The used deposition parameters listed in Table 1 were successfully tested in the previous study [3],

where a high deposition efficiency and no diamond graphitization was observed.

Table 1. Deposition parameters of the investigated cold sprayed DMMC coatings.

	Gas pressure [MPa]	Gas temperature [°C]	Standoff distance [mm]	Torch traversal speed [mm/s]
Al-based coatings	3.0	400	35	50
Cu-based coatings	3.0	600	35	50

The elastic constants of the deposits were determined by resonant ultrasound spectroscopy (RUS). Small prismatic samples (approximately $1 \times 1 \times 1 \text{ mm}^3$) were cut from each deposit. The density of the samples was determined from weight and dimensions of the samples. The upper face of each sample was mirror polished to enable scanning by laser vibrometer. A fully non-contact setup described in detail in [5] was used. The sample was placed in an evacuated chamber with low nitrogen pressure (20 mbar) which enabled precise temperature control ($\pm 0.1 \text{ }^\circ\text{C}$) of the measured sample. The vibrations were excited at the bottom face of the sample face by infrared Nd:YAG laser (Quantel ULTRA, nominal wavelength $1.064 \text{ }\mu\text{m}$, pulse duration 8 ns). The upper face of the sample was scanned by scanning laser vibrometer (MSA-500) to obtain frequencies and modal shapes of individual resonant modes. Resonant spectra of the free vibrating samples were recorded in frequency range 0.1 - 1 MHz, which covered between 10 to 20 from the first 40 resonant peaks. These resonant frequencies together with the velocities of longitudinal waves in direction perpendicular to sample faces were involved in inverse procedure to determine the elastic constants. An isotropic symmetry described by two independent constants (c_{11} and c_{44}) corresponding to Young's and shear modulus was assumed.

Mechanical testing of the deposits, i.e., the stress-strain measurements, fatigue crack growth rate tests, as well as fracture toughness measurements used the unified rectangular specimen geometry of $3 \times 4 \times 32 \text{ mm}^3$, described e.g. in [6]. The specimens were cut from the 5 mm-thick coatings using electric discharge machining. The specimen orientation denoted as L-T (analogous nomenclature to ASTM E399 standard) shown in Fig. 1 was used. This means that in all performed bending tests, the bending axis was in the spray direction (S). For fracture mechanical tests it indicates that the crack plane was perpendicular to longitudinal direction of the spray pattern (L), whereas the crack growth direction was parallel to its transversal direction (T).

The stress-strain properties in both tension and compression were obtained from bending tests following the method of Herbert [7] and the setup described in [8]. The method uses four-point bending fixture with an outer span $L = 27 \text{ mm}$ and an inner span $l = L/2$. A crosshead speed of $u = 1 \times 10^{-6} \text{ m/s}$ was used. The acting force was measured by 1 kN-force transducer and the deformation at the upper and the lower specimens'

surfaces was measured using digital image correlation (DIC) method using ncorr software [9] and Blackfly (5-megapixel monochrome camera FLIR systems, Wilsonville, OR, USA) equipped with in-line illuminated telecentric lens.

Figure 1. Schematic drawing of specimen orientation. Specimens denoted L-T are described in this paper.

The fatigue crack growth rates were measured using a resonance technique based on ASTM E647 standard [10] at a stress ratio $R \sim -1$. The specimens are loaded in pure bending and the actual crack length is obtained using differential compliance method. The details of the technique are given in [11]. A straight notch of approximately 0.5 mm length was pre-cut to the specimens using a low-speed diamond saw. The experiment was performed in rate-control mode, where the crack growth rate da/dN is prescribed as a function of crack length a and the load is adjusted by the control loop accordingly [11]. The crack was first grown to crack length $a = 1.1 \text{ mm}$ at crack growth rate decreasing from $da/dN \sim 10^{-9} \text{ m}$ to $da/dN \sim 5 \times 10^{-11} \text{ m/cycle}$. The da/dN vs. K curve was then measured with da/dN increasing exponentially from $da/dN \sim 5 \times 10^{-11} \text{ m/cycle}$ to $da/dN \sim 1 \times 10^{-6} \text{ m/cycle}$.

The fracture toughness measurement on fatigue pre-cracked specimens followed the ASTM E1820 standard. The pre-cracking was performed at crack growth rate of $da/dN \sim 10^{-9} \text{ m}$. The crack length and crack opening displacement (COD) was evaluated from the displacement field obtained by digital image correlation (DIC) by a method described in [8]. The DIC approach further simplifies the COD measurement and makes it more robust compared to videoextensometer-based measurement used e.g. in [12]. Using the DIC data to evaluate the J -integral directly by a method similar to [13] and [14] would be even simpler, however it was unsuccessful in the present case due to the out-of-plane movement of the specimen that would require a 3D DIC system. The detailed description of the used fracture toughness measurement can be found in [8]. The critical value J_{IC} corresponding to J -integral at the onset of a stable crack growth was estimated from the R -curves describing the dependence of J -integral on crack extension Δa from fatigue precrack. The J -integral was converted to the fracture toughness using the plane stress formula $K_{IC} = \sqrt{E J_{IC}}$.

The X'Pert PRO MPD (Malvern Panalytical, Malvern, UK) diffractometer was used to measure lattice deformations of the Al and Cu based matrix in L-direction (see Fig. 1). The

measurement was performed on metallographically prepared surfaces using chromium and the manganese radiation with the average effective penetration depths 5–11 μm and 3–5 μm , respectively. Diffraction angles $2\theta^{\text{hkl}}$ were taken as a center of gravity of the $\{311\}$ diffraction doublet. The values of $\sin^2\psi$ corresponding to positive and negative values of angles ψ were 0, 0.15, 0.3, 0.45, 0.6. To determine residual stresses, the $\sin^2\psi$ method and X-ray elastic constants $1/2s_2 = 19.05 \text{ TPa}^{-1}$, $s_1 = -4.89 \text{ TPa}^{-1}$ and $1/2s_2 = 11.74 \text{ TPa}^{-1}$, $s_1 = -3.12 \text{ TPa}^{-1}$ were used for the Al and the Cu phase, respectively. Pinholes of $4 \times 0.3 \text{ mm}^2$ size determining the irradiated area were used.

Results

The results of the performed experiments are summarized in Table 2 and include density, stress-strain properties, fracture mechanical properties and residual stress values in the matrix. In the table, the investigated deposits are complemented with reference coatings deposited using a high pressure cold spray system [8], but characterized by the same methods. These references are included to see the obtained results in a broader context, defined by the optimized deposition performed by a state-of-the-art commercial system.

The relative coating density ρ/ρ_b of the Al100 and Cu100 coatings was around 93% for the coatings deposited in this study and around 99% for the reference coatings from [8]. The addition of the clad diamond phase increased the density of the Al-based coatings as both densities of the core diamond ($\sim 6.3 \text{ g/cm}^3$) and the Ni cladding ($\sim 8.9 \text{ g/cm}^3$) have significantly higher density than bulk Al ($\rho_b = 2.7 \text{ g/cm}^3$). Naturally, an opposite trend was observed for the heavier Cu matrix ($\rho_b = 8.96 \text{ g/cm}^3$). The matrix-to-reinforcement density ratio predetermines the effect of the diamond particles. Assumedly, the heavier particles can add a significant peening effect and improve the inter-particle bonding, thereby increasing the mechanical properties. Indeed, the mechanical properties increased significantly with the addition of the clad diamond to the Al matrix (Table 2). This effect was less pronounced for the Cu-based samples as the densities of Cu and diamond are closer. Here, only a slight increase of fracture mechanics properties was detected, whereas the stress-strain properties actually decreased slightly (Table 2).

The four-point bending test (4PB) results are presented in Fig. 2. Both pure metal coatings showed brittle behavior with ductility below 0.2% in tension. Therefore the yield strength $R_{p0.2}$ could only be evaluated for some specimens. The addition of diamonds increased the ductility; slightly for the Cu-based and significantly for the Al-based coatings. Further, the Cu80 and Cu50 coatings showed slightly lower tension strength R_{mt} (Table 2, Fig. 2) than the pure copper coating Cu100, whereas the tension strength of Al50 and Al80 coatings was significantly higher than that of the Al100 pure metal aluminum coating. The compression and tension curves for pure metals were similar, whereas the presence of the diamonds caused the compressive curves to be higher than the tensile stress-strain curves. These differences are also reflected in the values of elastic moduli and are most probably caused by the loss of inter-particle contact in the deposit. Such contact loss is prominent in the Cu-based

coatings and is indicated by the large difference of tension and compression modulus $E_t < E_c$. This follows from the particle adhesion mechanism described in [15]. The particles can detach in the weakly bonded polar area of the impact crater enclosed by the contact rim. This area is pressed into contact by the residual stresses, however it detaches even under small imposed tension when residual stresses are low enough. This effect is very similar to modulus difference observed in thermal spray coatings [16]. It is difficult to observe this effect in the pure coatings due to noise in the stress-strain curve. However, the presence of diamonds brings a very stiff element with elastic modulus of more than 1000 GPa to the coating structure. This stiffens the deposit in compression, whereas in tension, the stress will be concentrated around it, and the polar contact areas will be opened even sooner. In the Cu-based coatings, the compressive stress-strain curve followed the pure metal baseline, while the tensile curve was lower. In the Al-based coatings, an opposite effect was observed: the tensile curve followed the pure metal baseline, whereas the compressive curve was higher. This seems to be somehow connected with the peening stress imposed by the double-clad diamond particles. Comparing the density of the double-clad diamond with the density of Al and Cu, one can anticipate higher peening effect on the Al-based coatings and a lower one for the Cu-based coatings.

The elastic modulus at low loads E_{RUS} was measured by RUS (Table 2). The obtained results are comparable with the compressive modulus E_c from 4PB, but differ significantly from the 4PB tensile modulus E_t . The explanation could dwell in the used loads: during the RUS testing, only a very small load is applied to the tested material, therefore the inter-particle contact loss does not decrease the modulus value.

The residual stress analyzed in the torch scanning direction (longitudinal axis of the torch pattern) by XRD using $\sin^2\psi$ method is presented in Table 2. The measurement is based on the elastic deformation of the metal matrix crystal lattice below the polished surface of the specimen (up to a depth of $\sim 10^{-6} \text{ m}$). As a self-standing deposit is measured, the average stress in the material should be zero, otherwise deformation will occur. The diffraction, however, does not take place equally in all parts of the deposited particles as they possess different levels and mechanisms of deformation. The deformation of a cold sprayed particle was modelled by several authors and even compared with FIB-DIC strain mapping results recently [17]. In the study, it was shown that any cold sprayed particle deforms plastically in the contact area due to inertia forces during impact. After the particle impact, the accumulated elastic deformation in the particle interior cannot be completely released due to the plastic strain in the contact area. Thus, elastic compressive residual stress is generated in the particle interior, balanced by tensile residual stress in the highly deformed contact area. The diffraction peaks originating from the plastically deformed contact areas are subject to peak broadening and loss of the diffraction intensity. On the contrary the relatively intact lattice in the particle interior diffracts well. Therefore, the analyzed compressive residual stress most probably comes from the particle interior. Its magnitude reflects the plasticity received by the particle upon impact, i.e. the peening effect of the impinging particles. Indirectly it may also relate to the particle cohesive

strength. This may be the reason why both Al- and Cu-based coatings with the highest measured residual stress (Al50 and Cu100*) showed the best properties in most of the evaluated parameters.

The results of the crack growth rate tests are presented as a relation of crack growth rate da/dN to a maximum value of stress intensity factor of the loading cycle K_{max} in Fig. 3. Fig. 4 presents a similar relationship, but for effective stress intensity factor range K_{eff} , i.e., for the part of the loading range with an open crack. The Al-based deposits show very steep crack

growth curves near the threshold, with an elbow at $da/dN \sim 10^{-8}$ m. Below the elbow, some oscillations probably caused by the loading history or coating inhomogeneity can be observed. The Cu-based deposits show steady linear crack growth rate curves down to 10^{-9} m with very indistinct near-threshold elbow at lower crack growth rates. The slopes of the linear part of the curves i.e. the exponent n in the Paris law $\frac{da}{dN} = CK^n$ are relatively high in the range from 7 for Al to 10 to Cu. This indicates that static mechanisms are probably active and contribute to the fatigue failure process.

Table 2. Mechanical properties of investigated cold sprayed DMMC. Asterisks denote reference samples from [6].

Deposit type	Density		RUS	4PB stress-strain					Fracture mechanics		Stress
	ρ [g/cm ³]	ρ/ρ_b [%]	E_{RUS} [GPa]	E_t [GPa]	E_c [GPa]	$R_{p0.2t}$ [MPa]	$R_{p0.2c}$ [MPa]	R_{mt} [MPa]	K_{IC} [MPa·m ^{0.5}]	$K_{th,eff}$ [MPa·m ^{0.5}]	$\sigma_{residual}$ [MPa]
Al50	3.14	116.3	78	54	82	146	210	155	7.1 ± 0.9	2.9	-23 ± 3
Al80	2.85	105.6	72	48	78	135	187	147	7.2 ± 0.3	2.6	-18 ± 2
Al100	2.51	93.0	54	50	44			81	3.8 ± 0.3	1.8	-1 ± 2
Al100*	2.66	98.5	66	75	69	69	70	>89	10.8 ± 1.6	1.4	-15 ± 3
Cu50	7.18	80.1	98	62	94			92	6.0 ± 1.4	2.5	-58 ± 8
Cu80	7.91	88.3	104	57	96			106	6.8 ± 0.5	2	-67 ± 4
Cu100	8.34	93.1	98	79	100			120	5.6 ± 0.1	1.7	-43 ± 3
Cu100*	8.85	98.8	121	100	113	343	-	348	8.7 ± 1.1	3	-103 ± 5

Figure 2. Stress-strain properties of Al and Cu based cold sprayed DMMC.

Figure 3. Crack growth rate da/dN as a function of K_{max} . Solid lines denote reference values taken from [8].

Figure 4. Crack growth rate da/dN as a function of effective stress intensity factor K_{eff} . Solid lines denote reference values taken from [8], black line represents the baseline data of a cold-rolled sheet.

Figure 5. R-curves of the Al- and the Cu-based cold sprayed DMMC. Pre-crack length ~ 1.8 mm. The dotted line is the construction line of ASTM E1820 standard. The symbols with center dots define the values of fracture toughness.

Typical crack growth resistance curves used to estimate the fracture toughness are presented in Fig. 5. These curves relate J -integral, i.e., the elastic energy release rate (energy released by a unit area of a newly formed crack) caused by a growing crack to the actual crack extension from the original fatigue pre-crack, Δa . Rising resistance curves were observed for all deposit types. The influence of the clad diamond was significantly higher in the Al-based coatings. For Cu coatings, the R -curves had a rather unusual shape with a decreased slope up to 0.2 mm crack extension, whereas at higher Δa , a rapid increase of the J -integral was observed, suggesting a possible change of the fracture mechanism. The fracture toughness K_{IC} corresponding to the intersection of the construction line with the R -curves is provided in Table 2. Increased K_{IC} was observed

for the deposits with clad diamonds, with the increase being more pronounced for the Al-based deposits.

Micromorphology of the fracture surfaces is shown in Figs. 6 and 7 for slowly growing fatigue cracks ($da/dN \sim 10^{-9}$ m) and in Fig. 8 for a static fracture. The detailed fatigue and static fractures in Figs. 7 and 8 were relatively similar as the dominant mechanism was particle decohesion in both failure modes. This failure mode corresponds well with the steep crack growth curves observed in Figs. 3 and 4. However, a small part of the fracture surface was formed by trans-particle fracture. In the static fracture region, the trans-particle fracture was ductile (indicated by arrows in Fig. 8). In the fatigue region, the trans-particle fracture was controlled by the fine microstructure of the individual deposits (indicated by arrows in Fig. 7).

Figure 6. Fatigue fracture surface of cold sprayed deposits fractured at $da/dN = 10^{-9}$ m. SEM micrograph, backscattered electrons, shadow mode. Crack grows from the bottom to the top.

Figure 7. Details of fracture surface of cold sprayed deposits fractured at $da/dN = 10^{-9}$ m. Arrows show signs of trans-particle fatigue cracking. SEM micrograph, backscattered electrons, shadow mode.

Figure 8. Details of static fracture surface of cold sprayed deposits from fracture toughness test. Arrows show trans-particle fracture with signs of plasticity. SEM micrograph, backscattered electrons, shadow mode.

Conclusions

Cold sprayed diamond-based metal matrix composite coatings were investigated in this study in terms of their mechanical (static and fatigue) properties. It was shown that the effect of the clad diamond additions on the final deposit properties depends on the matrix material:

- The impact of the diamond particles onto the lighter Al matrix invoked a peening effect, leading to a significant increase of compressive residuals stress and probably also better particle bonding. As a result, a significant improvement of the coating properties was achieved, such as superior compressive stress-strain curve, higher ductility and fracture mechanical characteristics.
- The impact of the diamond particles into the heavier Cu matrix invoked an opposite effect, i.e., lower peening in the vicinity of the diamond particles. Small increase of compressive residual stress was also observed, but was not connected to any significant property improvement. The most significant change was observed in the stress-strain behavior, where the elastic modulus in compression was retained, whereas its tensile counterpart decreased significantly.

The particle-to-matrix density ratio, therefore, seems to be an important characteristic of the cold sprayed DMMC coatings and as such requires further attention.

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