



Influence of Cr content and initial Cr particle size on the dielectric properties of plasma-sprayed Cr/Al₂O₃ coatings



Liang Zhou^{a,c,d,*}, Yanli Dong^{b,**}, Zhenjun Wang^a, Yuxin Wang^a, Dongpeng Hua^a

^a School of Materials Science and Engineering, Chang'an University, Xi'an 710064, China

^b State Key Laboratory for Mechanical Behavior of Materials, Xi'an Jiaotong University, Xi'an 710049, China

^c State Key Laboratory of Solid Lubrication, Lanzhou Institute of Chemical Physics, Chinese Academy of Science, Lanzhou 730000, China

^d Centre for Advanced Coating Technologies, University of Toronto, Toronto, M5S 3G8, Canada

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ABSTRACT

Cr/Al₂O₃ coatings were deposited by low-power plasma spray process with dry-crushed powders. The coatings exhibited a microstructure formed by fully molten splat-like metallic lamellae and partially molten or un-molten submicron Al₂O₃ particles, surround by a fully molten alumina matrix. The content and size of metallic Cr in the coatings played a dominant role in the formation of metallic clusters. Complex permittivity measurement showed that the coatings with higher Cr content and larger Cr particle size exhibited higher ϵ' and ϵ'' values due to the enhanced interfacial polarization and conductance loss. Furthermore, strong frequency dependency was observed for the coatings with 15 wt% Cr addition and 50–74 μm Cr particle size.

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1. Introduction

Plasma spraying is a generic coating technique whereby droplets of molten or partially molten materials are deposited onto a substrate to build up a coating [1]. In the process of plasma spraying, the high temperature plasma jet created by the plasma arc expands, heats the feedstock particles, and accelerates them toward the substrate, where they impact, deform, and solidify [2]. The characteristics of high temperature and energy density make spray a wide range of refractory materials (e.g. Al₂O₃, SiC and ZrO₂) possible [3]. Besides, it is also an attractive technique because of its high achievable deposition efficiency, cost effectiveness, and very specific coating properties. Over the years, plasma spraying has gained wide popularity to produce coatings to guard against corrosion, abrasion and high temperature [4,5]. Typical industrial examples of plasma spraying include defense, biomedical, marine, nuclear, chemical, automotive, aeronautical and mining [6].

Recently, with the rapidly expanding of communication devices (e.g. mobile telephones, local area network systems and radar systems),

microwave absorbing materials, being able to absorb the incident radiation, have been critically needed for thin thickness, broad bandwidth, light weight, strong microwave absorption and multifunctional properties [7]. Plasma-sprayed microwave absorbing coatings have attracted considerable interest due to their extensive applications in civil, commercial and military fields. In general, the coating materials used for spraying are classified as ferromagnetic, polymers and cermet materials [8–10]. However, the applications of carbonaceous materials (e.g. carbon nanotubes, carbon fiber and carbon black) in high temperature environments are restricted because of their weak oxidation resistance. Besides, the ferromagnetic based coatings, including magnetic metallic fillers and ferrites, will be invalid when the applied temperature is above the Curie point [11]. Therefore, plasma-sprayed cermet coatings with appreciate microstructure has a potential application in microwave absorbing field.

Cermets possess the properties of both ceramics and metals, including a fraction of hard ceramic with high temperature resistance and another fraction of ductile metal with special stiffness [12]. Alumina is an attractive ceramic material by virtue of its low density (3.97 g/cm³), high hardness and melting point (2054 °C). Meanwhile, Al₂O₃-based composite coatings have attracted much attention in the past decades, and some researchers have developed cermet coating using Cr as absorber [13,14]. These research results encouragingly showed that plasma-sprayed Cr/Al₂O₃ coatings presented controlling dielectric and microwave absorbing properties. So far, the researches on the plasma-sprayed cermet coating are

* Correspondence to: L. Zhou, School of Materials Science and Engineering, Chang'an University, Xi'an 710064, China

** Correspondence to: Y. Dong, State Key Laboratory for Mechanical Behavior of Materials, Xi'an Jiaotong University, Xi'an 710049, China.

E-mail addresses: zhouliang@chd.edu.cn (L. Zhou), dongyanli86@mail.xjtu.edu.cn (Y. Dong).

mainly focused on the dielectric properties by tuning the coating composition [15–17], few studies have been carried out on the comprehensive relationships among composition, microstructure and complex permittivity of such coatings. According to the report of Li et al. [18], the initial particle size in the feedstock powders has an important effect on the microstructure of thermal-sprayed cermet coatings. Furthermore, based on the other reports [19,20], the particle size strongly influences the microstructure and dielectric properties of conductor/insulator composites.

In the present study, dry-crushed feedstock powders were prepared with alumina and chromium as raw materials. Cr/Al₂O₃ coatings were fabricated by low-power plasma spray system with an internal fed torch. The phase composition and microstructure have been tested and analyzed. By tuning the Cr content and initial particle size contained in feedstock powders, the dielectric properties of plasma-sprayed Cr/Al₂O₃ coatings in the frequency range of 8.2–12.4 GHz were reported and the possible mechanisms were discussed.

2. Experimental

2.1. Preparation of the powders

Metallic Cr powders (>99.9%) were sifted using a series of sieves with different meshes, and the particle sizes of sieved powders were distributed in different ranges of 0.5–10, 15–30 and 50–74 μm , respectively. Beside, α -Al₂O₃ powders (>99.9%, D50 = 0.8 μm) were used as matrix materials of plasma-sprayed composite coatings. In the preparation process of dry-crushed powders, Al₂O₃ powders, Cr powders with different size ranges and PVA solution were mixed for 1 h by a mechanical stirrer to get the PVA-modified Cr/Al₂O₃ slurry. Then, the Cr/Al₂O₃ slurry was dried for 2 h in the oven with 100 °C to obtain the Cr/Al₂O₃ mixtures. Finally, the Cr/Al₂O₃ mixtures were ground and sieved to get the feedstock powders with an appropriate particle size (about 50–154 μm). The mass fractions of Cr powders with particle size range of 0.5–10 μm were 5, 10 and 15 wt%. The schematic of the feedstock powder preparation procedure is shown in Fig. 1. The composition of the dry-crushed powders and the nomenclature of the samples can be found in Table 1.

2.2. Preparation of the coatings

Low-power plasma spraying equipment with an internally fed powder torch was used to fabricate Cr/Al₂O₃ composite coatings [21]. The coatings were deposited at a power of 9.8 kW and a spray distance of 100 mm. Argon was used as a primary gas operated under a flow rate of 19.2 slpm, while nitrogen was used as a secondary gas at a flow rate of 0.8 slpm. The powder feed rate was 2.5 g/min using nitrogen as a powder feed gas under a flow rate of 3.5 slpm. The coatings were deposited on graphite substrates, and the thickness was controlled to 3.5 mm.

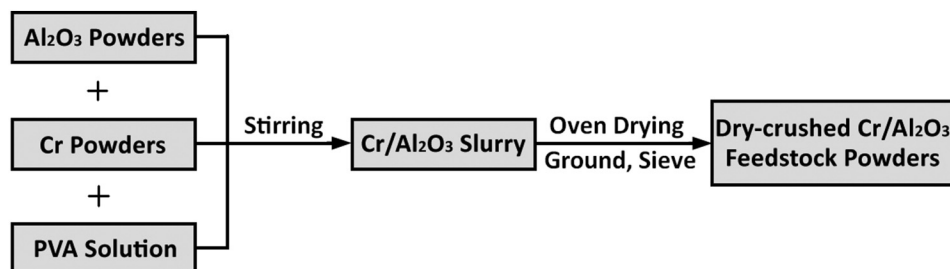


Fig. 1. The schematic of the feedstock preparation procedure.

Table 1

The sample codes and the component of the dry-crushed powders.

Sample codes	Cr content (wt%)			Al ₂ O ₃ content (wt%)
	50–74 μm	15–30 μm	0.5–10 μm	
A1	5			95
A2	10			90
A3	15			85
B3		15		85
C3			15	85

2.3. Characterization

The crystalline structure of the powder and coatings was characterized using X-ray diffraction (XRD) system (D/MAX2500, Tokyo, Japan) with Cu-K α radiation at 40 kV. The scanning electron microscopy (VEGA III SBH, TESCAN, Brno, Czech Republic) was used to examine the morphology of the powders and the cross-sectional microstructures of the coatings. Archimedes method was used to measure the coating porosity based on the procedure suggested by Mancini et al. [22]. The coatings with different Cr content and initial particle sizes were machined into the samples with dimensions of 22.86 mm (length) \times 10.16 mm (width) \times 2.00 mm (thickness). The dielectric properties were measured using the wave-guide method by a vector network analyzer (Agilent Technologies, E8362B: 10 MHz–20 GHz) in 8.2–12.4GHz (X-band).

3. Results and discussion

3.1. Phase composition and morphology of the feedstock powders

Fig. 2(a) and (b) shows morphology of dry-crushed feedstock powders. It can be seen that the particle size ranges from ~52 to ~154 μm with an average size of ~86 μm . The powder shape is irregular due to the crush of dried brittle materials. When observed at higher magnification, it is evidently found that Cr particles are randomly embedded in Al₂O₃ matrix, as seen in Fig. 2(b). Furthermore, the powders are mainly composed of Cr phase and α -Al₂O₃ phase as shown in Fig. 2(c). These results confirm that the dry-crushed process almost has no effect on the size of initial Cr particles, just agglomerates them with Al₂O₃ powders to form a larger composite granule.

3.2. Phase composition and microstructure of the coatings

The XRD patterns of as-sprayed Cr/Al₂O₃ coatings are shown in Fig. 3. It can be observed that all the coatings are consisted of α -Al₂O₃, γ -Al₂O₃ and Cr. The formation of γ -Al₂O₃ is attributed to the high cooling rate (about 10⁶ K/s), suggesting that the deposited spray particles have reached sufficiently melting state prior to impacting on the substrate or previous deposited layers in the process of plasma spraying [23]. Besides, as decreasing the Cr content from 15 to 5 wt%, the peak height

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