



Effect of intermetallic compounds on the bonding state of kinetic sprayed Al deposit on Cu after heat-treatment



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ABSTRACT

Post heat-treatment has generally been applied to products in the spray coating field as a method of improving mechanical properties. For kinetic spray process, the heat-treatment condition of dissimilar material pair (coating and substrate) should be controlled carefully, since brittle intermetallic compounds can be created at the interface. In this study, Al was kinetic sprayed onto a Cu substrate and was heat-treated under various heat-treatment conditions to investigate the effect of Cu–Al intermetallic compounds on bond strength. When adhesive bond strength was tested, fracture was induced at the lower interface of the CuAl₂ layer in all heat-treated specimens. The bond strength was enhanced at relatively lower temperature heat-treatment (300 and 350 °C) assisted by diffusion bonding between the Al deposit and Cu substrate. However, the bond strength was weakened sharply after heat-treatment at 400 °C due to decreased interaction between the Cu substrate and the diffused Cu elements, which resulted from the formation of a CuAl₂ layer on the side of Al deposit. The bond strength recovered at 450 and 500 °C, since intimate bonding was achieved between the CuAl₂ layer and the lower layer (Cu₄Al₃ or CuAl), which was chemically grown between the CuAl₂ and Cu₉Al₄ layers.

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1. Introduction

Coatings via various processes are often required on a number of components or products in industrial fields [1–6]. These coating layers should retain sufficiently high bond strength to perform properly. For protective coating layers (i.e. wear-resistant, corrosion-resistant coating) fabricated by spray coating processes, it is enough for the deposit to possess bond strength of 30–40 MPa. In particular, kinetic sprayed coatings generally show higher bond strength relative to the case of thermal spray process, assisted by the strong mechanical interlocking and compressive residual stress resulting from severe plastic deformation of the supersonic particles on impact [7–12]. Surely, excessive amount of compressive residual stress can cause the delamination of coating layer, but it is not as hard as to that extent in the case of kinetic spraying [13]. However, for dissimilar materials pair (i.e. Al powder and Cu substrate) in kinetic spray processing, the adhesive strength is much weaker than the cohesive strength due to the lower compressive residual stress at the coating-substrate interface compared to the case of similar materials pair [13]. The weak adhesive strength can cause problems for application involving kinetic spraying technology in industrial fields.

To solve this problem, post heat-treatment of kinetic sprayed products has been used and there are a number of studies related to the heat-treatment of kinetic sprayed deposits [14–17]. The adhesive bond strength can be enhanced by formation of a diffusion bond layer at the coating-substrate interface after heat-treatment. However, for the kinetic sprayed dissimilar materials pair, many researchers have reported that adhesive strength is degraded by lengthy heat-treatment, since brittle intermetallic compounds (IMC) can be created at the adhesive bonding interface [18–23]. Also, it was reported that adhesive strength worsens dramatically, when the thickness of the IMC become greater than 2–3 μm [24,25]. Thus, the use of post heat-treatments to enhance adhesive bond strength should be considered carefully for the case of dissimilar materials pair in kinetic spray process.

Despite its difficulty, there are a limited number of studies related to the correlation between bond strength and microstructural/phase evolution at the interface induced by post heat-treatments of kinetic sprayed dissimilar materials pair. In this study, the Al was deposited on the Cu substrate via kinetic spray technology under appropriate process conditions. Afterwards, the IMC type and thickness was classified in reference to changes in heat-treatment temperature. The mechanical properties (i.e. hardness, elastic modulus and fracture toughness) of each IMC were also evaluated. On the basis of such analysis, the adhesive bond strength was measured, and the effect of the IMC state and

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microstructural evolution on the bond strength was compared and discussed.

2. Experimental procedure

2.1. Feedstock characterization

Gas atomized high purity (99.9%) Al powders (ALE16PB, Kojundo Co. Ltd., Japan) were used as feedstock material. Fig. 1a and b shows the morphology and cross-section of the as-received feedstock powder, which is dense but irregular in shape. The size distribution of the feedstock was measured using laser scattering particle size analyzer (HELOS/KR, Sympatec) ranged in $-8 + 37 \mu\text{m}$ (mean, $20 \mu\text{m}$) (Fig. 1c).

2.2. Kinetic spraying and heat-treatment condition

The Al feedstock powder was deposited on the Cu substrate ($50 \times 70 \times 5 \text{ mm}^3$) via commercially available kinetic spraying system (KINETIC 3000, CGT). A de-Laval converging-diverging MOC type anti-clogging polybenzimidazole (PBI) nozzle was used to prevent clogging of the nozzle from the small, low melting point Al particles. Prior to the deposition process, the Cu substrates were grit-blasted and cleaned with alcohol to reinforce mechanical interlocking between the deposited Al and Cu substrate, which is generally applied method in spray coating field [26]. Detailed kinetic spraying parameters are listed in Table 1. Afterwards, the kinetic sprayed Al coatings were heat-treated at $300 \text{ }^\circ\text{C}$, $350 \text{ }^\circ\text{C}$, $400 \text{ }^\circ\text{C}$, $450 \text{ }^\circ\text{C}$ and $500 \text{ }^\circ\text{C}$ for 1 h in a box furnace under argon atmosphere, in order to protect from oxidation. The heat-treated Al deposits were furnace-cooled after heat treatment. Samples were simply named to distinguish conveniently: HT300, HT350, HT400, HT450 and HT500.

2.3. Analysis

The cross-section of the as-sprayed and post-heat treated Al coatings were characterized by optical microscope (OM, BX61M OLYMPUS) and scanning electron microscope (SEM, JCM-5700, JEOL). After a heat-treatment, the newly created IMC and diffusion region at the coating-substrate interface were investigated using energy dispersive spectroscopy (EDS) and an electron probe micro analyzer (EPMA, JXA-8100, JEOL).

Based on results of the SEM and EPMA, nanoindentation experiments were performed for samples obtaining thickest IMC (HT500) after heat-treatment. Hardness, elastic modulus and fracture toughness of each phase (Al, IMC and Cu) were evaluated using a Nanoindenter-XP (Agilent, Oak Ridge, TN, USA) with a Berkovich indenter under a constant strain rate of 0.05 s^{-1} , a holding time of 1 s, load limit of 5 mN and a total of 15 indentation tests.

In the case of the adhesive strength measurement, the kinetic sprayed specimens were cut into $10 \times 10 \text{ mm}$ squares and the Al deposit was polished till it was approximately $200 \mu\text{m}$ to increase the accuracy of the relative adhesive strength measurements. Subsequently, strong aluminum-epoxy test studs ($\sim 85 \text{ MPa}$) (P/N 901106, Quad Group) were attached to the fine-polished Al coating surface and then these bonding test specimens were thermally cured at $200 \text{ }^\circ\text{C}$ for 90 min in a box furnace. The stud pull-out coating adherence test [27] was carried out using a Romulus Bond Strength Tester (Quad Group). Afterwards, the fracture surfaces of the bonding test specimens were analyzed by X-ray diffractometer (XRD, D/MAX-2500/PC, Rigaku) under Cu K α radiation ($\lambda = 1.54 \text{ \AA}$) with a 2θ range from 20° to 80° at 40 kV and 100 mA using a scan speed of 0.02° to confirm where fracture from the bond strength measurements had initiated.

3. Results and discussion

Fig. 2 shows newly created IMCs of each post-heat treated specimen. Original interface between Al deposit and Cu substrate is marked with

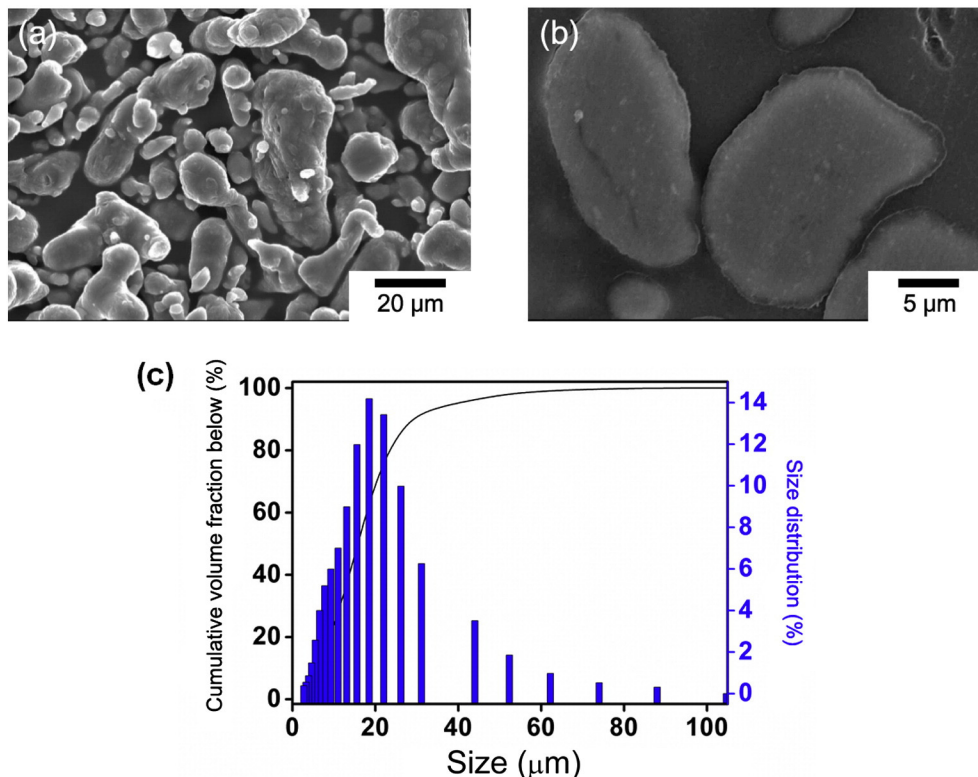


Fig. 1. SEM images (a and b) and powder size distributions (c) of as-received Al feedstock.

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