

# In-Situ Formation Fe<sub>2</sub>Al<sub>5</sub> Intermetallic-Reinforced Aluminum Matrix Composite by Annealing Treatment of Cold-Sprayed Precursor Deposit

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**Abstract.** Aluminum matrix composite (AMC) reinforced with in-situ Fe<sub>2</sub>Al<sub>5</sub> intermetallics particles was prepared through annealing treatment of the cold-sprayed Al/Fe composite precursor with an Al/Fe weight ratio of 85:15. The deformation characteristics of Al and Fe particles in cold spraying process and the effect of heat treatment temperature on the in-situ Fe<sub>2</sub>Al<sub>5</sub> intermetallics formation and the morphology evolution were investigated. The results showed that the cold-sprayed Al/Fe composite deposit had a dense microstructure and its composition was nearly same as that of original powder mixture, and soft Al particles exhibited the significantly intensive deformation compared to Fe particles, which caused the elongation of Al grains. The Fe<sub>2</sub>Al<sub>5</sub> intermetallics were formed and uniformly dispersed in the Al matrix after annealing at 500°C. The content of Fe<sub>2</sub>Al<sub>5</sub> phase increased with raising annealing temperature and Fe particles in Al/Fe composite were fully consumed by diffusion alloying reaction with Al matrix above 550°C and simultaneously the fragmentation of Fe<sub>2</sub>Al<sub>5</sub> intermetallics particles occurred.

**Keywords.** Aluminum matrix composites, Fe<sub>2</sub>Al<sub>5</sub> intermetallics, in-situ formation, cold spray, annealing treatment

## 1. Introduction

Because of their attractive high specific strength and good wear resistance, Al-based matrix composites (AMCs) have been widely interested [1-2]. Generally, SiC, TiC and Al<sub>2</sub>O<sub>3</sub> etc. ceramic particles were used as reinforcement for AMCs [3-5]. However, ceramic particle-reinforced AMCs had some drawbacks for commercial applications, including high production costs, difficulty to machinability and incompatibility with the environment [2]. Because of its high hardness, high stability and good compatibility with aluminium matrix, in-situ intermetallic particles are expected to as reinforcement to

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fabrication of AMCs [6]. Many processes or technologies can be used to produce the AMCs, for example sinter, hot pressing, casting [7-9], thermal spraying (TS) [10-12], etc.

Cold spraying (CS) technology has been attracting interest in fabrication dense and no oxidation composite coating and deposit. In this process, particles in a solid state occur intensive plastic deformation when impacting upon substrate and /or the pre-deposited particles and consequently form a coating without the deleterious problems inherent to conventional TS, for example oxidation, phase transformation, decomposition, grain growth and etc [13]. A few papers showed that the dense Ti/Al [14-15] and Ni/Al [16] composites can be produced by CS of powder mixture and post-spray treatment would occur Al-based intermetallic phase formation.

Therefore, the present work was to fabricate in-situ  $Fe_2Al_5$  intermetallics-reinforced AMC and study annealing temperature effect on in-situ formation of  $Fe_2Al_5$  intermetallics and structure evolution of cold-sprayed AMCs.

## 2. Materials and Procedures

The elemental powder were Al (99.5 wt.%,  $-48 \mu\text{m}$ ) as a matrix and Fe (99.8 wt.%,  $\sim 30 \mu\text{m}$ ) powders as reinforcement. The powder mixture composition ratio of Al and Fe was 85:15 wt.%, namely 94.2:5.8 in the volume fraction. The mixing process was performed in planet style ball mill with the mass ratio of ball-to-powder about 2:1 at speed of 150 rpm for 2 hrs. Fe powders were angular and Al powders were spherical in figures 1(a) and 1(b), respectively.

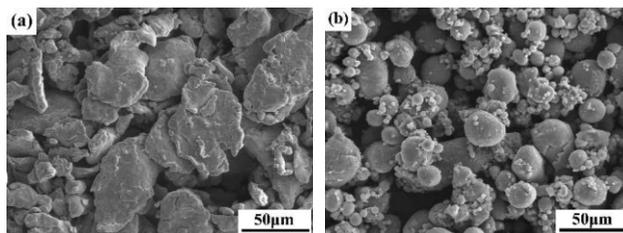


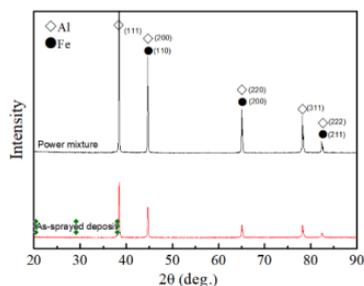
Figure 1. SEM images of the Fe (a) and Al (b) powders.

A cold spraying system installed in our lab was used to prepare Al/Fe composite deposit. Nitrogen was used as both driving gas and powder carrier gas with a pressure of 2 MPa and 2.2 MPa, respectively. Driving gas temperature was about  $200^\circ\text{C}$  and standoff distance was 20 mm. Substrate was Al plate with the thickness about 5 mm and sandblasted before deposition. In order to form intermetallic-reinforced aluminum composites, the as-sprayed Al/Fe deposit was annealed at  $500^\circ\text{C}$  and  $550^\circ\text{C}$  for 5 hours to form in-situ intermetallic compounds by solid-state diffusion. All samples were furnace heated and cooled. The microstructure and phase structure of Al/Fe deposits were examined by scan electron microscopy (SEM) and X-ray diffraction diffractometer (XRD), respectively

### 3. Results and Discussion

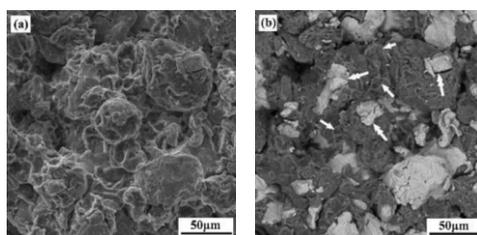
#### 3.1. Microstructure of Al/Fe Composite Deposits

The Al/Fe composite deposit had the same phase components as the powder mixture with only Fe and Al phases. There were no Fe-Al intermetallic phases, Al or Fe oxides existed in the cold-sprayed Al/Fe composite (in figure 2), which was ascribed to the low deposition temperature of cold spray process.



**Figure 2.** XRD pattern of the as-sprayed Al/Fe deposit.

Figure 3 shows typical surface morphologies of the cold-sprayed Al/Fe composite deposit, in secondary electron (SE) and backscattered electron (BSE) mode, respectively. The as-sprayed Al/Fe composite showed rough topography, which was formed by successive impacting particles (namely ‘splat’) piled up (in figure 3(a)). White Fe particle in contrast embedded in the surface of dark Al particles due to its relatively high hardness and high impacting velocity, as marked by white double arrows in figure 3(b). However, many impacting craters resulted from the rebounded Al or Fe particles can be evidently found on the surface of dark Al particles due to its low hardness, as marked by white arrows in figure 3(b). Hence, Al particles experienced more intensive deformation compared to Fe particles.

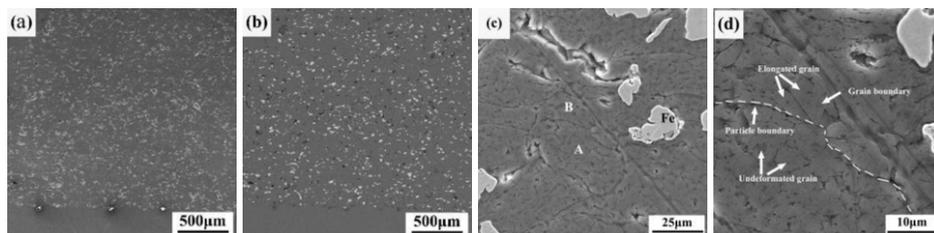


**Figure 3.** Surface morphology of the as-sprayed Al/Fe composites deposit in the SE (a) and BSE mode (b).

The cross sectional microstructure of the as-sprayed Al/Fe composite deposit in SE and BSE mode was shown in figures 4(a) and 4(b), respectively. Al/Fe deposit had the dense microstructure (in figure 4(a)) and its porosity was only  $1.2 \pm 0.44$ , determined by the image analyzing method. Due to be softer than Fe particles, Al particles intensively deformed to make up a dense matrix, in which white Fe particles embedded (in figure 4(b)). Due to different hardness, particle size, particle morphology and etc, Fe particle and Al particle would exhibit different deformation behavior and subsequently different deposition efficiency. The real volume fraction of Fe in as-sprayed Al/Fe composite was

$6.3 \pm 0.21$ , which was nearly same as the nominal volume fraction of Fe in Al85Fe15 composite (about 5.8%).

The as-sprayed Al/Fe composite in the high magnification were given in figure 4(c) and 4(d). The Al/Fe composite was etched using Kroll's reagent (3ml HF: 6ml  $HNO_3$ : 100ml  $H_2O$ ) in order to clearly displayed the boundary characteristics between particles and particles. The Al/Fe composite presented a dense microstructure and Fe particles were embedded on Al matrix (in figure 4(c)). In addition, Al particles exhibited intensive deformation compared to Fe particles. As a result, the Al particles boundaries were nearly indistinguishable. The distinct deformation behavior of Al and Fe particles would be ascribed to their discrepancy in density, hardness and particle size as well. It was noticeable that some Al particles have been deformed into long thin splats (as marked by B in figure 4(c)) and other particles do not experienced significantly deformation (as marked by A in figure 4(c)). At higher magnification (in figure 4(d)), Al particles were in intimate contact at the interface (as indicated by white broken lines), which would be conducive to solid state diffusion between Al and Al or Fe particles during post spray annealing treatment. Our previous research [17] found that this intensive deformation can accelerate the interface diffusion during annealing treatment. In addition, elongated grains mostly appeared close to particle boundary and undeformed grains were mainly in the particle interior, as indicated by white arrows in figure 4(d). This phenomenon also testified that the impacting particle deformation mainly occurred in the particle boundary.

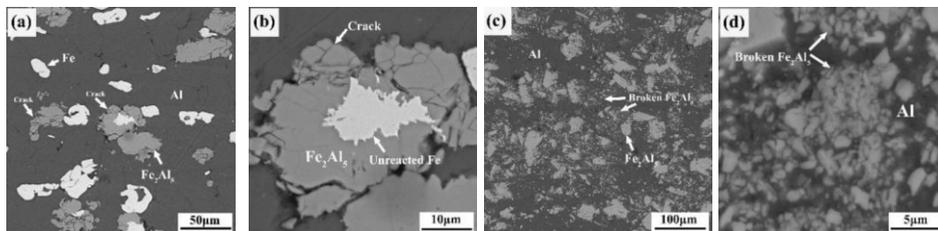


**Figure 4.** Cross section images of the cold-sprayed Al/Fe composite deposit in the SE (a) and BSE mode (b), and (c), (d) in high magnification.

### 3.2. Annealing Effect on Al/Fe composite

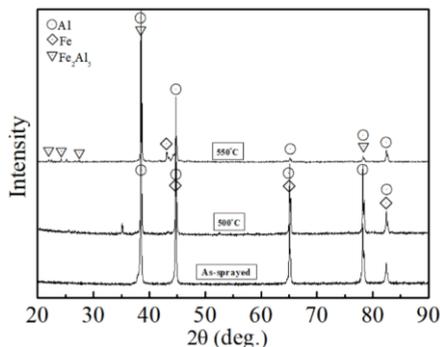
Figure 5 shows SEM images of the annealed Al/Fe composite. Some gray particles appeared beside white Fe and dark Al matrix when annealed at  $500^\circ C$ , as indicated by white arrows in figure 5(a). According to Fe-Al diffusion theory and the previous study [18], these gray particles were “new born”  $Fe_2Al_5$  intermetallics, which had the bigger size comparing to original Fe. This fact indicated that  $Fe_2Al_5$  intermetallics interface grow up toward Al matrix during element diffusion. In addition, some Fe particle did not completely transformed into  $Fe_2Al_5$  intermetallic phase and the unreacted Fe was still present in the inner of particles and some cracks appeared in the outside area of  $Fe_2Al_5$  intermetallic phase, as indicated by white arrow in figure 5(b). However, Fe completely disappeared and transformed into  $Fe_2Al_5$  after annealing at  $550^\circ C$  (in figure 5(c)). It was notable that may small  $Fe_2Al_5$  particles appeared in the annealed composites as indicated by white arrows in figure 5(c). When Fe transform to  $Fe_2Al_5$ , its volume would contract, whereas, the diffusion of Al into Fe structure would cause volume expansion. Such contrary volume change induce different stress state, namely tensile stress in outer surface and compressive stress in inner. Therefore, outer  $Fe_2Al_5$  area is under tension and

inner Fe is under compression. The residual stress in  $Fe_2Al_5$  area was too small to cause obvious crack when the annealing temperature is low (such as  $500^\circ C$ ). However, this tensile stress would significantly increase with raising annealing temperature (such as  $550^\circ C$ ) and finally cause fragmentation of brittle  $Fe_2Al_5$ , as shown in figure 5(d). The detailed fragmentation mechanism of  $Fe_2Al_5$  needs further studies and discussion.



**Figure 5.** SEM images of Al/Fe composite annealed at different temperature: (a) (b)  $500^\circ C$  and (c) (d)  $550^\circ C$ , respectively.

Figure 6 shows the XRD patterns of the annealed Al/Fe composite deposits. It can be seen that the composite annealed at  $500^\circ C$  presents the same phase structure as the as-sprayed deposit consisting of Al and Fe. However,  $Fe_2Al_5$  phase has actually appeared (phase in a gray contrast in the figure 5(a)), which was ascribed to the low content of  $Fe_2Al_5$  phase in the annealed Al/Fe composite. After annealed at  $550^\circ C$ , the peaks of  $Fe_2Al_5$  intermetallic compound can be clearly detected in the coating. This indicated that the content of  $Fe_2Al_5$  phase in annealed deposit greatly increased, as can be testified from figure 5(c).



**Figure 6.** XRD patterns of Al/Fe composite deposits annealed at different temperatures.

#### 4. Conclusions

In-situ  $Fe_2Al_5$  intermetallics-reinforced aluminum matrix composite (AMC) was successfully fabricated by annealing cold-sprayed Al/Fe precursor composite deposit. The as-sprayed Al/Fe composite deposit had a same phase structure and constitute with that of the original powder mixture. The intensive deformation of Al particles caused close interface contact between particle/particle and the remarkable grain elongation in deposited Al particles. In-situ  $Fe_2Al_5$  intermetallic compound appeared in the Al/Fe deposit after annealed at  $500^\circ C$ . All Fe particles completely transformed into  $Fe_2Al_5$

intermetallics by diffusion alloying reaction with Al matrix at 550°C and the fragmentation of Fe<sub>2</sub>Al<sub>5</sub> intermetallics particles simultaneously occurred.

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