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Abstract

Title Controlling of Feedstock Powder Material upon Fabrication of AlN Coating in Reactive Atmospheric Plasma Spray Process

## (800 words)

Aluminum nitride (AIN) has emerged as an important material for several applications due to its outstanding electrical and mechanical properties. Plasma spraying is a versatile technique for producing abradable and protective ceramic coatings. However, it was difficult to fabricate AIN coating by conventional plasma spray processes. It is due to the thermal decomposition of AIN powder during spraying without a stable melting phase (Molten or semi-molten phase is required for plasma spray deposition). Reactive plasma spraying (RPS) is a promising technology for *in situ* formation of AIN coating. Recently AIN coating was fabricated through plasma nitriding of aluminum (AI) powder in radio frequency (RF) plasma spray system. However, the coating contains brittle agglomerates and it was difficult to obtain dense AIN coatings by RF spray system due to its low particle velocity. Furthermore, the RF system was carried out in vacuum ambient.

This study investigated the feasibility of reactive plasma spraying in the atmospheric plasma spray (APS) system. It is atmospheric process with high particle velocity (useful to fabricate dense coatings). Generally, the parameters controlling the RPS process are divided into process and reaction related parameters. This study focused on the reaction related parameters and investigated the effect of feedstock powder material, the reaction time and the nitriding species on the fabrication process and coating microstructure. The feasibility of plasma nitriding of Al, alumina (Al<sub>2</sub>O<sub>3</sub>) powders and the effect of ammonium chloride (NH<sub>4</sub>Cl) and AlN addition were investigated. Moreover, the nitriding mechanism of the particles in the atmospheric ambient, the formation process of AlN coating and the effect of feedstock characteristics, process parameters were investigated for each feedstock powder.

It was possible to fabricate AIN based coatings through the reactive plasma spraying in the atmospheric ambient of APS system. The following results were obtained:

- Cubic-AlN (c-AlN) based coating was fabricated through plasma nitriding of Al powder in N<sub>2</sub>/H<sub>2</sub> plasma. The formation of c-AlN phase in APS process is directly related to the rapid solidification phenomena of plasma spray process. The Al particles melted, interact with the plasma and formed aluminum oxynitride (AlON) phase. The AlON is easily nitride in N<sub>2</sub>/H<sub>2</sub> plasma to form c-AlN phase (same cubic symmetry). Controlling the reaction time and feedstock particle size enhanced the nitriding conversion. In order to fabricate thick AlN coatings from Al powder, improving the nitriding of the large particles during flight and/or after deposition is required.
- The NH<sub>4</sub>Cl additive promoted the in-flight nitriding conversion of Al particles. Addition of NH<sub>4</sub>Cl enhanced AlN formation in RPS process through changing the Al reaction pathway to vapor-phase chlorination-nitridation sequences as confirmed by the thermodynamic analysis of the possible intermediate reactions. This changed the nitriding reaction to a mild way, so it is more controlled with no explosive mode and with relatively low heating rates. Furthermore, the evolved gases from NH<sub>4</sub>Cl sublimation prevent the Al particles coalescing after melting. Thick and dense *c*-AlN coating with high hardness was fabricated with a small NH<sub>4</sub>Cl addition (2.5-5 wt.%).
- The AlN additive promoted the nitriding conversion of the Al particles after deposition. High hardness thick AlN coatings were fabricated through plasma spraying of Al/AlN mixtures. The addition of AlN prevented the coalescence of Al particles on the substrate and increased the nitride content in the fabricated coatings gradually. Using 30 to 40 wt% AlN was sufficient to fabricate thick AlN coating (200 μm) with high hardness (1000 Hv).
- Cubic AlN/Al<sub>2</sub>O<sub>3</sub> composite coatings were successfully fabricated through plasma reduction and nitriding of Al<sub>2</sub>O<sub>3</sub> feedstock powder in N<sub>2</sub>/H<sub>2</sub> plasma. The coating consists of c-AlN, AlON, γ-Al<sub>2</sub>O<sub>3</sub>, and remaining α-Al<sub>2</sub>O<sub>3</sub> phases. During spray the Al<sub>2</sub>O<sub>3</sub> particles are quickly melt in the N<sub>2</sub>/H<sub>2</sub> plasma and spheroidized. Then the molten spherical particle reacted with the N<sub>2</sub>/H<sub>2</sub> plasma and formed c-AlON on its surface. Then the particles collide, flatten, and the AlON easily nitride to c-AlN through rapid solidification and plasma irradiation. The nitriding conversion, the coating thickness and composition significantly affected with the flight time and nitriding species.
  Thick and uniform AlN/Al<sub>2</sub>O<sub>3</sub> coating with high nitride content and very small amount of α-Al<sub>2</sub>O<sub>3</sub> was fabricated through RPS of spray-dried fine Al<sub>2</sub>O<sub>3</sub>/AlN mixture. The fine particle size assisted the nitriding conversion (due
- through RPS of spray-dried fine Al<sub>2</sub>O<sub>3</sub>/AlN mixture. The fine particle size assisted the nitriding conversion (due to increasing the surface area) and the AlN additives increased the nitride content in the coating. Furthermore, the nitriding gases hardly affect the phase composition, coating microstructure and hardness in case of fine particles.