Fabrication of Ti(Al, O)-Al_2O_3 Powder Feedstock for Thermal Spraying and Evaluation of the Composite Coating

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Abstract

Ti(Al,O)-Al_2O_3 composite powders were produced by high energy mechanical milling of a mixture of Al and TiO_2 powders followed by a combustion reaction. The powders were subsequently thermally sprayed on H13 steel substrates. Microstructural examination was conducted on the composite powders and thermally sprayed coatings, using X-ray diffractometry (XRD) and scanning electron microscopy (SEM). The performance of the coatings was evaluated in terms of micro-hardness and thermal fatigue. The thermally sprayed coatings performed very well in the preliminary thermal fatigue tests and showed no wetting tendency to molten aluminum.

Keywords: Mechanical milling; Thermal spraying; Ti-Al_2O_3 powder; Coating

1. Introduction

Mechanical milling facilitates the chemical reaction at low temperatures, in some cases at room temperature; by means of substantially increasing the reaction kinetics of the chemical reactions (mostly displacement reactions). This is often referred to as mechanochemical synthesis thanks to the pioneering work by Schaffer and McCormick in the late 1980s. Combining mechanical milling with a chemical reaction, mechanochemical synthesis has been widely used to produce a variety of in-situ metal-matrix composites, through the reaction of Al and certain metal oxides, e.g. CuO, Fe_3O_4 and V_2O_3. The present paper focuses on fabricating a composite powder feedstock using an admixture of Al and TiO_2 as reactants, by mechnochemical synthesis. This feedstock is potentially a good candidate for thermally sprayed coatings because it contains a ceramic phase Al_2O_3 that has excellent high temperature properties, and a metal phase, Ti, which is probably used as a binder enabling good ahhesion to the substrate. Mechanochemical synthesis is accomplished by the process developed in Waikato University [1,2] in combination with a combustion reaction process.

2. Experimental and results

TiO2 (rutile) powder with a particle size ranging from 5 to 50 μm was blended with commercial purity aluminium powder with a mean particle size of ~ 100 μm. The molar ratio of TiO_2:Al was controlled to be 3:4 according to the nominal reaction $3TiO_2 + 4Al \rightarrow 3Ti + 2Al_2O_3$. About 500-gram powder blend was mechanical milled using a Rocklab Split discus mill for up to 6 hours. A process control agent (PCA), isopropyl alcohol, was added at the level of 3 wt% prior to milling. The ball-to-powder mass ratio used was 4.4:1. Metallographic samples were examined with a Hitach S-4100 field emission scanning electron microscope (FE-SEM) equipped with an energy dispersive spectrometer (EDS). Thermal analysis was conducted to evaluate the progress of mechanical alloying by using a simultaneous SDT-2960 analyzer at a heating rate of 10°C/min. X-ray diffraction analysis (XRD) was performed on the as-milled, combustion reacted powder and the thermally sprayed coatings using an X'pert diffractometer with Cu Kα radiation. The operating voltage was 40 kV and the scanning speed was 0.02°/sec. The microhardness of the coating surface was measured with a Leco LM-700 micro-indenter. Preliminary thermal fatigue experiments were performed by dipping the coated coupons into a molten aluminium bath kept at 710°C for 2 minutes and then quenching into water.

A homogeneous mixing in the composite powder was achieved after 6 hours of milling. The phase constituents were examined with X-ray diffraction analysis, showing that up to 4 hours the chemical reaction between Al and TiO_2 did not occur, albeit peak broadening and shift was observed, which suggests the refinement of particle size, internal strain within the particles and the probable occurrence of solid dissolution of Al into TiO_2 (Fig. 1). It should be noted that some new phases, Ti and Al_2O_3 were observed in the 6-h milled powder, as shown in the XRD spectrum (Fig. 1). This was not observed in our previous work, which shows that chemical reaction does not occur even after 60 hours of milling [3]. The possible underlying reason is that the 6h milled powder had been ignited locally and hence a partial combustion reaction occurred, during the powder handling process. This needs to be further clarified in the future work

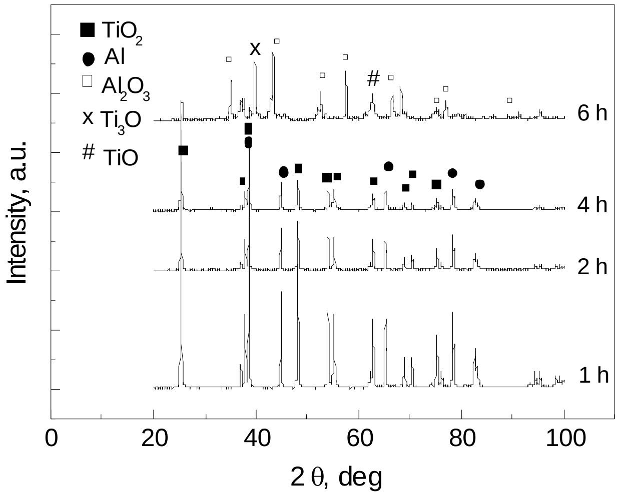

Fig. 1. XRD spectra of the mechanical milled composite powders with different milling time, showing the formation of oxides such as Al_2O_3, Ti_3O and TiO.

Fig. 2 shows an example of the combustion reacted powder, which was subjected to 4 h milling prior to combustion. The average particle size of the as-reacted powder was less than 20μm, which meets the requirement for the particle size of the thermal spray feedstock. The EDS analysis suggested that two types of particles have been obtained: Al_2O_3 and Ti (Al,O).

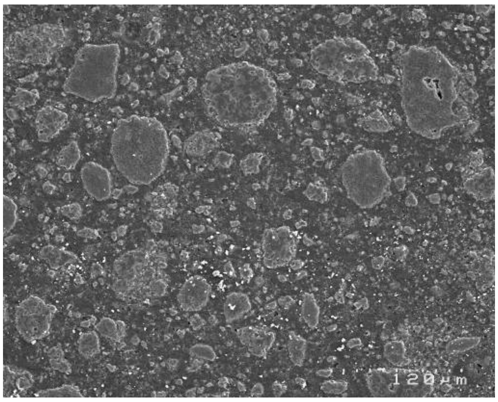

Fig. 2. SEM micrograph of the combustion reacted composite powder. The EDS results show that the white particles were Al_2O_3, while the grey matrix was Ti(Al,O)

The feedstock was applied to deposit a layer of coating using an HVAF equipment. Figs. 3(a) and 3(b) show a cross section micrograph and a top view of the thermally sprayed coating on a H13 steel coupon. The average coating thickness was about 30 μm. The lamellar structure was interspersed with voids. The XRD and EDS analyses confirmed that the coating consisted of Al_2O_3 inclusion and Ti(Al,O) matrix.

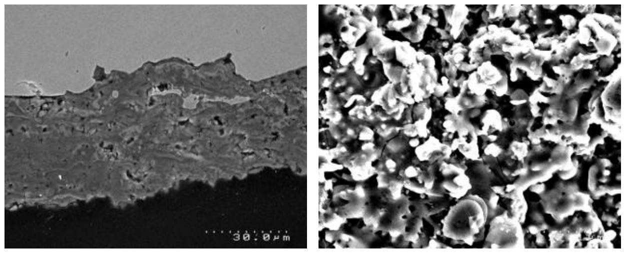

Fig. 3. SEM micrographs of the coating: (a) cross sectional micrograph and (b) top view.

A preliminary thermal fatigue test was conducted. It was found that the uncoated coupon started to suffer aluminium sticking and surface spallation after 18 heating-quenching cycles. In contrast, the coated coupons did not show any sticking of aluminium. Fig. 4 shows macroscopic views of two such coupons: one without a coating and the other with a thermally sprayed coating. It can be clearly seen that, after 43 heating-quenching cycles, the uncoated coupon surface was full of aluminium spots and the spallation was observed. On the other hand, apart from a few aluminium spots on the coating surface, the coating was still intact with the coupon and no spallation was observed.

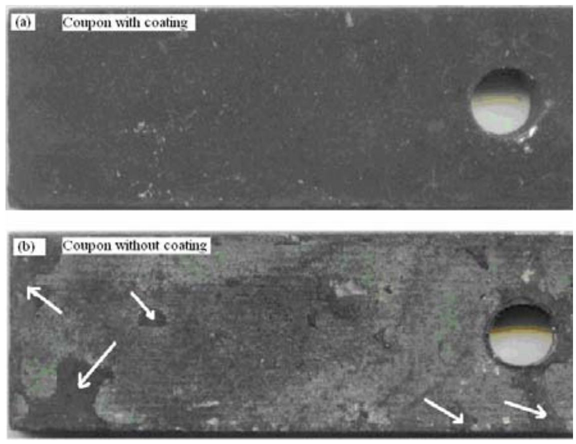

Fig. 4. Macrographs of the uncoated and coated coupons after 43 thermal cycles.

3. References

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