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Powder injection moulding of an Al-AlN metal matrix composite

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1. Introduction

Particle reinforced aluminium metal matrix composite materials have attracted much attention due to their unique combinations of properties such as stiffness, wear resistance, damping capacity and tailored coefficient of thermal expansion, while maintaining the light weight, ductile properties of aluminium [1–11]. There have been significant advances in developing high performance aluminium matrix composites. However, there remain major technological challenges to making these materials affordable for large-scale commercial applications. Factors such as shaping, machining and joining are problematic [1,2]. Casting is a traditional net shape forming technique for aluminium. However, when casting aluminium metal matrix composite, problems such as poor material homogeneity, deleterious matrix/reinforcement interface reaction and volume fraction limitation of reinforcement are encountered. These problems can be overcome by adopting a powder metallurgy route. Existing techniques such as extruding, powder forging and press/sinter powder metallurgy can produce relatively simple shaped parts from composite materials [4,5,8-10]. However, these processes have limitations such as restrictions in shape and form, and fast tool wear.

The powder injection moulding (PIM) process allows complex, net shaped parts to be manufactured out of metal, ceramic or composite materials [12–18]. It is particularly suited to net shape fabrication of small but geometrically complex parts. It involves

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ABSTRACT

A powder injection moulding process for producing fine AIN particle reinforced aluminium matrix composites was developed. The process is a promising low-cost technique for net shape production of complex aluminium metal matrix composite components. Net shape parts of near full density with good strength and ductility were produced. The AIN reinforcement particles exhibited good compatibility and were uniformly distributed throughout the AA6061 matrix. An Al–AIN composite/AI hybrid was also successfully produced. The hybrid parts exhibited good interfacial adhesion and were free of distortion.

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mixing powders and polymer binder together to prepare feedstock. The binder enhances compressibility and flowability of the fine powders so that moulding to the required shape can be achieved efficiently and effectively using conventional plastic injection moulding technology. After the shaping process, the polymer binder is removed and the powders can be sintered to near full density.

AlN is an ideal reinforcement as it has good compatibility with aluminium [19,20]. Due to their high thermal conductivity and low thermal expansion coefficient, AlN reinforced aluminium composites have potential applications in microelectronics for components such as heat dissipation devices or electronic packaging [7]. The authors have recently developed a PIM process for a heat treatable aluminium alloy [21]. This has been adapted for the production of a particle reinforced aluminium metal matrix composite. In this report the microstructure and mechanical properties of the composite are evaluated.

Sintering of aluminium is complicated by the presence of a thermodynamically stable oxide layer. Magnesium is known to react with the oxide and it therefore plays a major role in the sintering of aluminium [22–26]. The atmosphere is also known to be important and nitrogen is widely regarded as necessary [25,27,28]. A key feature in the beneficial use of nitrogen is the formation of aluminium nitride, which can occur because the interior region of the compact becomes essentially oxygen free through an autogenous gettering process [29]. The formation of AlN is thought to reduce the pressure in the closed pore spaces, which unbalances the meniscus forces. This induces pore filling which is therefore an important densifying mechanism in the sintering of aluminium [28]. Tin is also an important sintering activator under nitrogen because it moderates the formation of AlN [30].

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Fig. 1. SEM image showing the morphology of the AlN reinforcement powder. The chart (insert) shows the particle size distribution of the powder.

2. Experimental methods

The feedstock was prepared by pre-mixing AA6061+2wt% Sn+10wt% AlN powders using a Turbula mixer for 30min. The aluminium powder AA6061, supplied by the Aluminium Powder Company Ltd., was rounded in shape and had a D_{10} , D_{50} and D_{90} of 5.6 µm, 13.4 µm and 24.1 µm, respectively. The Sn, supplied by Atlantic Equipment Engineers, USA, was also rounded in shape and had a particle size <43 µm. Reinforcement AlN powders were supplied by Atlantic Equipment Engineers. They are polygonal in shape as shown in Fig. 1. The insert chart shows the bi-model particle size distribution. There are some fine particles less than 1 µm in size. The D_{10} , D_{50} and D_{90} values are 2.20 µm, 8.37 µm and 16.76 µm, respectively. The powders and polymer binder ingredients (palm oil wax, stearic acid and high density polyethylene) were compounded using an industrial planet kneader at 165 °C for 3 h. The powder loading was 82.5 wt% or 61 vol.%. The feedstock was injection moulded into dog bone tensile test bars (Fig. 2) using an Arburg injection moulder.

Disc samples of AA6061 + 2 wt% Sn + 10 wt% AlN/AA6061 + 2 wt% Sn hybrid material with dimensions of ϕ 25 mm × 3 mm were prepared by hot compaction in a Simplimet hot mounting machine (Buehler Ltd., USA), more commonly used for the preparation of metallurgical samples. Approximately 2.5 g of feedstock of either AA6061 + 2 wt% Sn or AA6061 + 2 wt% Sn + 10 wt% AlN were loaded into the chamber, which was heated to 165 °C for 5 min under 30 MPa of pressure. The compacts were subsequently cooled to about 80 °C and ejected. The hybrid green part was prepared by compacting the discs of the two different mate-



Fig. 2. Optical image of the green and sintered parts. The sintered part is free of defects and shows proportional shrinkage from the injection moulded green part.

rials together following the same procedure as that described above. As the two materials have different densification kinetics, a different powder loading was used for each half to match the shrinkage from the green to the sintered parts. The powder loading for the AA6061+2Sn was 63 vol.% and the loading for AA6061+2Sn + 10AlN was 61 vol.%.

Solvent debinding was conducted in hexane at 45 °C for 48 h. Thermal debinding and sintering was conducted in a single heating sequence [21]. The furnace was evacuated to less than 5.0×10^{-2} Torr before initiating heating. The samples were heated to 450 °C over 16.5 h and then heated to the sintering temperature at 0.8 °C/min and held for 2 h. The temperatures evaluated for sintering of the tensile test bars were 630 °C, 635 °C, 640 °C and 645 °C, while a sintering temperature of 630 °C was employed for the hybrid material. Backfilling of nitrogen started at 120 °C and nitrogen gas flow was maintained at 11/min for debinding and sintering. Several Mg blocks were placed beside the samples to scavenge oxygen.

Sintered densities were measured using Archimedes method and reported as a percentage of the theoretical density (2.781 g/cm³). Polished and un-etched samples were used for both optical and scanning electron microscopy (SEM). The optical micrographs were taken using an Olympus AX70 optical microscope with a Diagnostics Instruments SPOT digital camera. SEM observations were performed on a PHILIPS XL30 instrument. Tensile testing was conducted on an Instron 4505 machine with extensometer gauge length of 25 mm and crosshead speed of 0.6 mm/min. All tensile specimens were tested without any machining or polishing. Rockwell hardness (HRH) was measured on sand paper polished surfaces with a 3.2 mm steel ball indenter and 60 kg load.

Transmission Electron Microscopy (TEM) specimens were prepared using an xT Nova NanoLab 200 dual focused ion beam (FIB). Thin sections (~100 nm) suitable for TEM examination with typical dimensions of about 5 μ m × 12 μ m were milled using a Ga⁺ beam. The sections were placed on amorphous carbon coated copper grids. TEM was performed using a Philips Tecnai 20 FEG instrument fitted with an EDAX thin-window energy dispersive X-ray (EDS) detector.

3. Results and discussion

Images of the injection moulded tensile test bar (green part) and the sintered part are shown in Fig. 2. The sintered part is free of defects such as blisters or cracks. There is uniform and proportional shrinkage and the part shape is maintained well.

The microstructure of the sintered composite material is shown in Fig. 3. The fine AlN reinforcement particles are uniformly dispersed throughout the Al matrix and the composite is well sintered. Agglomeration is a common problem for particle reinforced composite materials, especially when the particle size is small. However in the present research, a uniform distribution of AlN particles was achieved by ensuring a complimentary match of reinforcement/matrix particle size and homogenous mixing of the polymer binders and powders. It has been shown that the reinforcement particle size should be similar to that of the Al powder to ensure the composite is well sintered and does not exhibit particle clustering [4,8].

The density, hardness and tensile test results for the composite material for various sintering temperatures and heat treatments are summarized in Table 1. The results reported are the average from testing of at least three samples. Results for the non-reinforced AA6061 + 2Sn PIM material are included for comparison. The sintered density increases with increasing sintering temperatures and reaches a maximum of ~97% at 640 °C. This optimal sintering temperature is 10 °C higher than that for non-reinforced AA6061 + 2Sn

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