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Short Communication

Pre-review study of the aluminum/alumina master alloy made through pressure infiltration

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ABSTRACT

The work is focused to prepare Al based master-alloy pellet with 50 vol.% of Al_2O_3 for subsequent manufacturing of aluminum matrix composites with desired amount of reinforcement (5–20%). Since master-alloy is manufactured via pressure infiltration and proper interface between particles and melt is required for uniform particle distribution within the melt, fundamental correlation between parameters of pressure infiltration and quality of the Al/Al_2O_3 interface is revealed in this study. Standard observation techniques as 3-D computed tomography (CT), scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDS), transmission electron microscopy (TEM) and X-ray diffraction (XRD) are used for structural characterization. Drop test was used to estimate effect of time, temperature, annealing of Al_2O_3 and its type on the wettability of Al_2O_3 with Al. Differential scanning calorimetry (DSC) and thermogravimetry (TG) were used to study changes within the Al_2O_3 prior infiltration. Stir casting was used to prepare the final composite and dynamical mechanical analysis (DMA) was used to estimate the Young's modulus of as-cast composite. The proper infiltration parameters was defined in this work and it were shown that the infiltration temperature and pressure have direct correlation on the interface between particle and aluminum.

1. Introduction

Metal matrix composites are a group of advanced materials due to their specific material properties which are controlled by sort and amount of the reinforcement particles. The matrix can be selected on the basis of oxidation and corrosion resistance or other properties. Nowadays researchers all over the world are focusing mainly on aluminum because of its unique combination of good corrosion resistance, low density and excellent mechanical properties [1]. AMCs reinforced with particles or whiskers are widely used for high performance application such as automotive, military, aerospace and electricity industries [2]. Researchers have done experiments on Al and its alloy based MMCs using wide variety of dispersoids such as Al_2O_3, SiC, TiB_2, B_4C, SiO_2, TiC, ZrO_2, TiO_2, WC, BN, W, graphite, mica, illite-clay and shell-char [3].

However, the good interface between the particles and melt is needed to transfer the load during the operation of such materials [4–14] and to assure their uniform distribution within the melt. Stiffening and strengthening rely on load transfer across the interface. Toughness is influenced by crack deflection at interface and ductility is affected by the relaxation of peak stress near the interface [2]. Experimental values of properties differ from the modeled due to lack of relation between the particle and the matrix, which negatively affects the load transfer during operation [4–7,9].

Metal matrix composites can be obtained by different technologies. Many researchers prepared aluminum composite materials by stir casting technology to improve mechanical properties. Stir casting technology is one of the possibility to manufacture particle composites relatively inexpensive [2,15].

The method of particle introduction to the matrix melt is a very important aspect of the casting process. There are a number of techniques for introducing and mixing the particles. However, some of these methods have several disadvantages. Gas injection of particles for example will introduce a quantity of gas into the melt, which negatively influences mechanical properties. The gas–liquid interface becomes 'decorated' with particles. The gas bubbles, which are significantly lighter and try to rise through the molten composite, are coated with the silicon carbide, which is heavier than the molten composite. As a result, the bubbles are stabilized and remain suspended in the composite eventually leading to porosity. Some methods are not very effective in dispersing the particles and some, such as ultrasonic technique are very expensive, and difficult to upscale the method to production level [16].

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A novel idea in this study is to manufacture aluminum matrix composites with desired amount of reinforcement using master alloy pellet with high content of Al_2O_3 particles. Pre-review study of the master alloy manufacturing is therefore necessary.

The master-alloy pellet with high amount of ceramic particles is produced through the pressure infiltration and afterwards it is dissoluted in aluminum melt. The work is focused to reveal the effect of the infiltration parameters on the quality of the master alloy and on the interface quality between aluminum and Al_2O_3. Also, dissolution experiment and DMA measurement of as-cast composite is accompanied.

2. Experimental approach

2.1. Infiltration

Master alloy pellets were produced by infiltrating loose ceramic particles with pure aluminum via pressure infiltration technique under various temperatures, pressures and times. Prior the infiltration, Al_2O_3 was annealed at 950 °C for 2 h to increase their wettability due to dehydroxylation of the powders and changes within the powder surface [7]. The commercial alumina powder AMDRY 6060 with particle size 45 μm was used for this study. DSC and TG were used to study changes within the Al_2O_3 prior infiltration to confirm earlier statement in [7]. Amount of Al_2O_3 prior infiltration was estimated by 3-D computed tomography as well as calculated from the microstructure to about 48 ± 4 vol.%. Ceramic content was constant for each experiment. The loose Al_2O_3 were filled in high porous graphite crucible, which was immersed in the Al melt after preheating to adjusted temperature of infiltration. Subsequently, various infiltration pressures were applied to find out the effect of infiltration pressure on the quality of infiltration. On cooling, infiltrated pellets were lifted out of the melt under applied pressure and were released after temperature decrease below melting point of aluminum.

2.2. Methods of characterization

To reveal effect of infiltration temperature and pressure on the quality of Al/Al_2O_3 interface, temperature during infiltration was set to 750 °C, 850 °C and 950 °C and pressure to 3 MPa, 5 MPa and 7 MPa for each temperature. Lower pressures were also investigated at infiltration temperature of 950 °C. For one combination of the infiltration temperature and pressure (850 °C/5 MPa), effect of the infiltration time was studied to find out relation between infiltration time and Al_2O_3/Al interface.

Contact angle between molten aluminum and Al_2O_3 was measured using the sessile drop test under vacuum (6.6×10^{-2} Pa) at temperature 750 °C and 1250 °C. Drop test was focused to reveal effect of time, temperature, effect of Al_2O_3 annealing and ceramic type (loose powder and sintered Al_2O_3 was used for comparison) on the wettability of Al_2O_3 with Al.

The quality of Al/Al_2O_3 interface was revealed by SEM, EDS and TEM. To study the changes in Al_2O_3 with respect to annealing temperature prior to infiltration, X-ray measurement was used. Dissolution experiment of the master alloy pellet in pure aluminum 1050 was performed at temperature of 900 °C without stirring the melt. Dissolution of the master alloy in graphite crucible using mechanical axial flow impeller was performed at 760 °C. The speed of the stirrer was 12 Hz (720 rpm) and the stirring time 10 min. A cooper crucible was used to cast aluminum containing Al_2O_3 particles.

Young's modulus was determined by DMA (Q600 TA Instruments machine) at room temperature using 3-point bending tests. For this test, two samples with dimensions of 2.5 × 4 × 55 mm were used. Young's modulus was measured 4 times on each side. The frequency of 1 Hz, preload force of 0.5 N and dynamic force of 7 N were set up.

3. Results and discussion

3.1. Pre-annealing of Al_2O_3 powder

After heat treatment of Al_2O_3 particles at temperature 950 °C/120 min/air, the needle-like microparticles on the surface of Al_2O_3 transform to a globular as seen in Fig. 1, what probably results decrease of Al_2O_3 surface tension. In study by Hossein-Zadeh et al. [17] the microstructure of composite reinforced with heat treated particles (1000 °C/20 min/argon atmosphere) Al_2O_3 showed good distribution of particles and very low agglomeration of alumina in contrast with Al-nonheat treated Al_2O_3 composite. The morphology of initial particles were modified and a change from irregular to spherical shape was noticed [17].

As mentioned in [4,7], heating of Al_2O_3 on air at 900 °C leads to dehydroxylation and weight loss (Fig. 2), which results into increase of Al_2O_3 surface energy. Moreover, improved wetting after heat-treatment is caused by formation of oxygen deficient surface containing some AlO in a spinel-type structure [7]. On the other hand, Shen et al. [18] measure, that annealing of (0001) α-Al_2O_3 substrate does not have a significant effect on the wettability of the Al–Al_2O_3 system, since the surface structural transformation is reversible. Therefore, the weight loss shown in Fig. 2 probably shows only release of physically bounded water from Al_2O_3.

Except dehydroxylation, TG and DSC measurement do not indicate any phase transformation, which is confirmed also by XRD (Fig. 1d). Since, no peaks broadening or intensity variation on X-ray pattern which should indicate a decline in the crystalline size of particles, were observed, the above mentioned suggestions is relevant [19].

3.2. Sessile drop test

Changes of contact angle with respect to time, temperature, effect of annealing and ceramic type are shown in Fig. 3. Contact angle between loose Al_2O_3 particles and aluminum was above 90° at both tested temperatures 950 °C and 1250 °C. We can therefore conclude that in general, annealing of the particles itself has no measurable effect on wetting (contact angle). Several factors like the oxidation of Al, oxygen partial pressure, surface roughness of the ceramic particles, transformation in surface characteristic, change in surface energy and melt temperature can influence the wetting behavior of ceramic-liquid system. High contact angle at 950 °C is caused by oxidation of the Al droplet and inhomogeneity (roughness) of the ceramic surface. The loose ceramic particles are oriented irregularly and therefore in our case the roughness of the surface is responsible for poor wettability also at higher temperatures. Contact angle of sintered Al_2O_3 at 950 °C was also above 90°. Decrease of contact angle was observed only in case of commercial sintered Al_2O_3 ceramic at 1250 °C, which correspond with previous studies [18,20]. Incorporation of the aluminum melt between the ceramic particles was not observed. This indicates that pressureless infiltration of the loose ceramic particles by Al is not possible even after annealing and therefore, increased pressure has to be applied to prepare Al master alloy with approximately 50% of Al_2O_3.

3.3. Infiltration – effect of infiltration parameters on the aluminum/alumina master alloy structure

3.3.1. Macrostructure

The macrostructure of master alloy in relation to infiltration temperature and pressure is shown in Fig. 4. In general, at lower