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## Thermal and mechanical properties of Al/Al<sub>2</sub>O<sub>3</sub> composites at elevated temperatures

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### ABSTRACT

The objective of this study is to determine the effects of phase volume content, processing conditions, the size of aluminum particles, and porosity, on the physical and thermo-mechanical properties of an alumina reinforced aluminum composite in 25-450 °C temperature range. Composites with 0%, 5%, 10%, 20% and 25% volume contents of alumina are manufactured using powder metallurgy technique. Two types of composites with different aluminum powders were manufactured: composite A - 99.97% pure Al with 7–15 µm particles, and composite B – 97.5% pure Al powder with 3–4.5 µm particles. For both composites A and B, alumina powder with <10 µm particle size was used. The composite samples were characterized for their porosity, relative density and distribution of the alumina phase. The elastic properties (Young's modulus and shear modulus) of the composites were determined using resonant ultrasound spectroscopy (RUS) as a function of temperature while the coefficient of thermal expansion (CTE) was determined at various temperatures using thermo mechanical analyzer (TMA). It was found that the composite with smaller aluminum (composites B) particles had higher values of the relative density and thus higher elastic moduli, and lower values of the CTE than the composites with larger aluminum particles (composites A). In all cases, increasing the volume contents of the alumina increases the elastic moduli and reduces the CTE of the composites. Increasing the testing temperature from 25 to 450 °C, significantly reduces the elastic moduli of the composites, while the CTE the composites only slightly changes with temperatures.

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### 1. Introduction

Ceramic particles are often used as reinforcements in metal matrix composites to increase their strength, hardness, stiffness, chemical stability, thermal stability and thermal resistivity. Ceramic particle-reinforced metal matrix composites (CPMMCs) provide a good combination of strength attained from ceramic reinforcements and toughness due to the underlying metal matrix [1]. Often they are the only choice in applications that involve high temperatures, such as thermal barrier coatings, turbine engines, and piston rod, due to their good resistance to elevated temperatures.

Aluminum is a widely used matrix material in CPMMCs primarily because of its low weight, low cost and ease of fabrication. The preferred reinforcement in CPMMCs should have high modulus, low density, good wettability, proper shape with a certain aspect ratio to minimize stress concentration, and thermal expansion

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coefficient comparable to that of the metal matrix to minimize the development of internal stresses due to temperature changes. Often the reinforcements in CPMMCs are either oxides or carbides and of them the most widely used are alumina ( $Al_2O_3$ ), silicon carbide (SiC) and graphite in various configurations such as discontinuous particles or continuous agglomerates [2]. Because CPMMCs are designed for high temperature applications (up to  $450-500\,^{\circ}\text{C}$ ), it is essential to understand the thermal and elastic properties of CPMMCs at elevated temperatures.

In this study, we examine thermo-mechanical properties of Al/Al<sub>2</sub>O<sub>3</sub> composites at elevated temperatures. Al-Al<sub>2</sub>O<sub>3</sub> composites are fabricated using powder metallurgy technique because this technique has proven to be favorable for distributing Al<sub>2</sub>O<sub>3</sub> particles uniformly in Al matrix and for its ability to fabricate the material into practically any shape [3,4]. The disadvantage of using powder metallurgy method is that one cannot produce fully dense composite samples [3,5,6]. Rahimian et al. [6,7] used powder metallurgy technique to fabricate Al-Al<sub>2</sub>O<sub>3</sub> composites and determined the effect of parameters such as Al<sub>2</sub>O<sub>3</sub> particle size, concentration, sintering temperature and sintering time on the relative density, and room temperature mechanical and physical properties of these

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composites. Using finer alumina particles and sintering temperature of  $600\,^{\circ}\text{C}$  composites with relative density ranging from 96.5 to 99% were manufactured. Banjuraizah et al. [8] and Schaffer and Hall [9] found that nitrogen is the best atmosphere for sintering aluminum to produce samples with high relative density, followed by argon. However, the use of nitrogen forms aluminum nitride (AlN) as an additional phase; and hence inert argon gas is used in our study to sinter the composite pallets. The wetting properties, interfacial energy and bonding strength of Al–Al<sub>2</sub>0<sub>3</sub> composite system is studied by Ksiazek et al. [10], Kou et al. [11] and Zhang et al. [12]. The main problem limiting the wetting of aluminum by alumina is the oxidation of aluminum. Hence, inert atmosphere is maintained throughout the sintering process to reduce the oxidation of aluminum.

In this work, Al-Al<sub>2</sub>O<sub>3</sub> composite samples were fabricated with 0, 5, 10, 15, 20 and 25 vol% of Al<sub>2</sub>O<sub>3</sub> through blending, cold compacting, and sintering processes. The structural (phase composition, microstructure, relative density and porosity), mechanical (elastic and shear moduli) and thermal (CTE) properties of the composites are then characterized in 25-450°C temperature range. The effect of alumina particle size on the relative density of composites has been widely reported in the literature [6,7,13], however very limited work has been done to determine the effect of aluminum particle size on the thermo-mechanical properties of composites, and hence in our study we used two aluminum powders with different particle sizes. Though the mechanical properties of Al-Al<sub>2</sub>O<sub>3</sub> composite system using conventional methods (e.g. tensile test and three point bending test) are well documented in literature, most of the studies are done at room temperature [14-16]. The significance of our work is that a nondestructive testing method like RUS is used to measure the mechanical properties of Al-Al<sub>2</sub>O<sub>3</sub> composites at various temperatures. The main advantages of using RUS over these conventional methods are it requires low amplitude loading, which makes a reliable determination of the linear elastic moduli because of a significantly low stress and strain values involved and also it gives all components of the elastic stiffness tensor in one run of the experiment [17]. Finally, the CTE of Al-Al<sub>2</sub>O<sub>3</sub> composite system and their dependency on temperature are also

This manuscript is organized as follows: Section 2 discusses fabrication of the  $Al-Al_2O_3$  composites, density calculations and microstructural studies of the composite systems followed by the characterization of the thermal and mechanical properties in Section 3. Conclusion and a brief recapitulation of the results are given in Section 4.

### 2. Fabrication and structural characterization of $Al-Al_20_3$ composite pallets

### 2.1. Sample preparation

Powder metallurgy technique is used for manufacturing the composite pallets. 99.5% pure aluminum powder of -100 + 325 mesh size (Alfa Aesar, MA) as shown in Fig. 1a and 99.7% pure alumina (Sigma–Aldrich, MO) with average particle size of  $10\,\mu m$  as shown in Fig. 1b were used in this study to process composites labeled as composite system A. The aluminum particles appeared to be mostly spherical and varied in size approximately between  $1\,\mu m$  and  $50\,\mu m$  in diameter whereas the alumina particles were about  $10\,\mu m$  and were polygonal in shape. The above Al–Al<sub>2</sub>O<sub>3</sub> composites are labeled as A-XX, where XX is the nominal volume percentage of Al<sub>2</sub>O<sub>3</sub>. Pure aluminum powder was examined using X-ray diffraction (XRD) which showed that the starting aluminum powder contained 2.3 vol% of alumina (Table 1). The Al–Al<sub>2</sub>O<sub>3</sub> composite system B – further labeled as B-XX, where XX is the nominal

volume content of  $Al_2O_3$  – were processed using 97.5% pure aluminum powder with smaller particle size, i.e. 3–4.5  $\mu m$  (Alfa Aesar, MA) as shown in Fig. 1c, while using the same alumina powder as that of the composite system A (Fig. 1b). Results of XRD analysis (Table 1) showed that the aluminum powder used for the composite system B contained about 3.4 vol% of aluminum oxide. The purpose of manufacturing and studying Al–Al $_2O_3$  composites with 2 different sizes of aluminum particles is to examine the effects of particle size on the overall density and thermo-mechanical properties of the composites.

The composite specimens are fabricated with different volume contents of alumina: 0, 5, 10, 15, 20, and 25 vol% for both composite systems, A and B. To achieve a specific volume content of a composite sample, proper amounts of Al and Al<sub>2</sub>O<sub>3</sub> powders were mixed, ball milled, and put in a cylindrical die of 25 mm diameter and a hydraulic press was used to cold press the powders for 30 min at room temperature. During the mixing, 35 wt% ethanol was added to reduce the possibility of formation of agglomerates [18]. Three different compacting pressures viz. 212, 425 and 502 MPa were used for the composite system A, while for the composite system B compacting pressures of 502 and 580 MPa were considered. The cold pressed pallets were further sintered in a quartz furnace (MTI, CA) at 600 °C. The samples were heated from room temperature to 600 °C at a rate of 5 °C/min, and sintered at that temperature for 2h, and then cooled back naturally to room temperature. During the entire sintering process an inert atmosphere was maintained in the quartz tube by flowing an Argon gas to prevent the oxidation of aluminum.

#### 2.2. Phase composition and microstructural characterization

The microstructure and composition of the processed composites were characterized by scanning electron microscopy, SEM, (JSM-7500F, JEOL, CA); X-ray diffraction, XRD, (Bruker-AXS D8 Advanced Bragg-Brentano X-ray Powder Diffractometer, Bruker, WI) and particle imaging analysis software (Imagel, NIH, USA).

XRD was used to perform qualitative (to identify the phases present in the composite) and semi quantitative analyses (to identify the vol% of each phase in the composite). The sample surface was scanned from  $2\theta = 20^{\circ}$  to  $2\theta = 70^{\circ}$  with a step size of  $0.015^{\circ}$  at a rate of  $0.0375^{\circ}/s$ ; and LynxEye detector was used to record the XRD patterns. XRD patterns in Fig. 2 show that except aluminum and alumina, no other phases were present in the composites manufactured. As the volume content of alumina increases the height of aluminum peaks decreases whereas height of alumina peaks increases. The split peaks observed in Fig. 2 are because of an overlapping of two peaks belonging to kalpha1 and kalpha2 wavelengths.

Upon identifying the phases present in the composite using XRD patterns, the amount of each phase present in the composite was determined using the XRD analysis software EVA (Bruker-AXS Diffrac EVA, Bruker, WI). Table 1 shows a comparison of the measured volume content of alumina in the composites using XRD analysis software EVA, to the nominal volume content of alumina in the composites. The volume content of alumina was found to be a few percent higher than the nominal one in all the processed samples because staring aluminum powders already contained 2.3 or 3.4 vol% of alumina, as discussed earlier. However, it is possible that some of the aluminum additionally oxidized during the manufacturing process.

All processed samples were cut and prepared for SEM by polishing surfaces with 0.1 µm diamond suspension particles. SEM images were taken using Backscattered (BS) and secondary electron (SE) detectors. Fig. 3a and b shows selected but typical BS and SE images of the composite systems A and B reinforced with 10 vol% Al<sub>2</sub>O<sub>3</sub> respectively, while Fig. 3c and d show BS and SE images with

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