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A study on thermal shock response of Al-Al₂O₃ micro- and nanocomposites

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Abstract

Metal matrix composites (MMCs) combine metallic properties (ductility and toughness) with ceramic properties (high strength and modulus) leading to greater strength in shear and compression and to high service temperature capabilities. The supercritical applications of these metal matrix nanocomposites necessitates their study with respect to service conditions. The service exposures of structural components encompass thermal as well as humid conditions. Aluminum-alumina system has extensive applications in aerospace and automobile industries. A significant thermal expansion mismatch may result in de-cohesion at the particle/matrix interface and/or a possible matrix cracking, particle fragmentation due to thermal stress. The asreceived aluminium (Loba Chemie, purity > 99.7%, average size~ 22.09μ m) and alumina (Sigma Aldrich, average size~10µm and <50 nm) powders were blended separately. Two sets of nanocomposites and microcomposites containing 1, 3, 5 and 7 vol. % of Al₂O₃ (average size<50nm) and 5, 10, 15, 20 vol. % of Al₂O₃ reinforced in aluminium were fabricated by conventional sintering at 600°C for duration of 60 minutes under argon atmosphere. The micro- and nano-composites were subjected to thermal shock. For one batch of specimens the treatment started from +80°C temperature (60 minutes) to -80°C temperature (60 minutes) (down thermal shock) and for the other batch the treatment was done in the reverse order (up thermal shock). After the thermal shock test mechanical and physical property determination was carried out i.e. hardness, and wear test followed by characterization under scanning electron microscopy for both microand nano-composites. The microstructural evolution during different thermal conditions would project an idea about the interfacial interaction at those conditions.

Keywords: Nanocomposite; Microcomposite; Thermal shock; Interfacial de-cohesion.

1. Introduction

The aerospace and automobile industries' major engine needs is catered by the material engineering industry. Metals are irreplaceable entities among the engine amenities. Advanced engine materials comprise of metal matrix nanocomposites. Nanocomposites comprising of nanoparticulates are the most viable and cost effective. The strengthening due to grain refinement can be delegated to a number of theories such as the Hall-Petch relation, Orowan bowing mechanism, Taylor relationship and several other models [1]. Metal matrix composites (MMCs) combine metallic properties (ductility and toughness) with ceramic properties (high strength and modulus) leading to greater strength in shear and compression and to high service temperature capabilities. The supercritical applications of these metal matrix nanocomposites necessitates their study with respect to service conditions. The service exposures of structural components encompass thermal as well as humid conditions. Composite structures also undergo different loading conditions during their service life, e.g. sports equipment at high loading rate to pressure vessels at low loading rates. Aluminum-alumina composites have been studied extensively fabricated by powder metallurgy. This system has extensive applications in aerospace and automobile industries. Powder metallurgy gives scope for fabricating critical components, and these components are subjected to thermal stress in their service life. Hence, the evaluation of thermal stress in different service conditions requires in-depth understanding. The difference in thermal conductivity of the matrix and reinforcement generates a thermal gradient throughout the composite. Thermal expansion coefficient of metals are substantially greater compared to ceramics which leads to either enhancement or degradation of proximal contact between particle and matrix under the influence of temperature gradient [2]. A significant thermal expansion mismatch may result in de-cohesion at the particle/matrix interface and/or a possible matrix cracking, particle fragmentation due to thermal stress [3].

The Al-Al₂O₃ composites have intricate applications in automobile and aerospace sectors and so their potential can be assessed by accelerated weathering. The immediate transitions of high and low temperature would induce disparities in the matrix-reinforcement interface. These disparities can be evaluated by microstructural study. This would enable applying the composites at safer temperatures in shock environments. The rocket engines requirements give way to aluminium matrix composites, hence cryogenic propellants come in contact with the AMCs, components subjecting to high temperature and cryogenic environment immediate transitions.

There is a paucity of literature in this field hence this investigation would lead to a comprehensive study of the composite with particle size variation. The advancement of science and technology has been rapidly demanding newer material which can endure extreme weathering exposures and excursion. This may necessitate the design of experimental process and procedures to generate data and findings which would lead to the prediction of reliability of mechanical performance of material behaviour in unpredictably harsh and hostile environments. Oguocha et al. [4] have worked on the thermal shock behavior of Al matrix composites. Ma et al. [5] studied the cryogenic properties and fracture behavior of Al composites predicting the rise in tensile strength at cryogenic

temperatures from the room temperature. They have also illustrated different fracture modes and features in cryogenic temperature range. Poza et al. [6] investigated the fracture mechanisms of Al composites at cryogenic and elevated temperatures. The fracture characters at elevated temperatures are dominated by interfacial decohesion rather than the reinforcement particle fracture.

2. Experimental

The as-received aluminium (Loba Chemie, purity > 99.7%, average size~22.09 μ m) and alumina (Sigma Aldrich, average size~10µm and <50 nm) powders were mixed and blended separately using agate mortar for 60 minutes to ensure homogeneous mixing. Two sets of nanocomposites containing 1, 3, 5 and 7 vol. % of Al₂O₃ (average size<50nm) and aluminium were fabricated by mixing the matrix and reinforcement powders followed by conventional sintering. Another two sets of microcomposites consisting of 5, 10, 15, 20 vol.% of alumina reinforced in aluminium were also synthesized to compare the microstructure and mechanical properties with nanocomposites. The temperature for sintering was maintained at 600°C for duration of 60 minutes under argon atmosphere at a heating rate of 6°C/minute. The densification of all the specimens has been estimated using Archimedes method. Sintered specimens were characterized by scanning electron microscope (JEOL 6480 LV). The micro hardness of all the specimens was determined by Vickers hardness tester (Leco LV 700) applying a load of 0.3 kgf and a dwell time of 5 sec and wear resistance were also measured for the micro and nanocomposites. The micro- and nano-composites were subjected to thermal shock. For one batch of specimens (micro- and nano-composites) the treatment started from +80°C temperature (60 minutes) to -80°C temperature (60 minutes) (down thermal shock) and for the other batch the treatment was done in the reverse order (up thermal shock). The composites were characterized using scanning electron microscopy and mechanical properties such as micohardness and wear resistance were measured before after thermal shock and treatment.

3. Results and Discussion

3.1. Microstructural evolution

The microstructures obtained after sintering for micro- and nano-composites show good distribution of alumina in the aluminium matrix. The alumina nanoparticles have intimately mixed with the matrix and are distributed almost uniformly aluminium. The grain boundary pinning is also effective as seen from the micrographs. The physical integrity of aluminium-alumina seems to be appreciable as no third phase forms in this system. The thermally shocked nanocomposites show decohesion of nanoparticle from the matrix and defect generation in the matrix (Fig. 1 & Fig. 2).



Fig. 1: Al-3 vol.% Al₂O₃ nanocomposites before (right) and after (left) down thermal shock



Fig. 2 : Al-3% Al₂O₃ nanocomposite subjected to up thermal shock showing defect generated in the matrix.

The thermal shock treatment has induced differential expansion and contraction of matrix and reinforcement. Down thermal shock consists of treatment at +80°C temperature followed by immediate treatment at -80°C temperature. At +80°C temperature the aluminium matrix expands and exerts a compressive force on alumina so that physical integrity of aluminium and alumina gets improved. The thermal shock at -80°C induces contraction of copper and alumina leads to interfacial de-cohesion of alumina from matrix due to higher contraction of copper than alumina. The reverse phenomenon takes place during up-thermal shock.



Fig. 3: Al-15 vol.% Al₂O₃ microcomposites before (right) and after(left) down thermal shock



Fig. 4 : Al- 15% Al₂O₃ microcomposites subjected to down thermal shock.

Alumina nanoparticles possess high surface area and as a result the interfacial decohesion and physical integrity induced by thermal shocks have a higher magnitude in case of nanocomposites. The microcomposites also exhibit decohesion and dis-integration in the microstructure (Fig. 3 & Fig. 4).

3.2. Microhardness

Thr microhardness values of nanocomposites before and after down and up thermal shock have been plotted against composition of nanocomposites(Fig. 5 & Fig. 6). The hardness values of nanocomposites after thermal shock show a decrease in microhardness values when compared to the hardness values before treatment. The defect generated in the matrix induced by thermal shock leads to microstructural disintegrity, which results in fall of microhardness.

The accelerated damage of nanocomposites due to subjection of thermal shock renders low micohardness.



Fig. 5: Microhardness of Al-Al₂O₃ nanocomposites before and after thermal shock.

The microhardness values and trend before and after thermal shock for microcomposites behaves in the same manner as nanocomposites. The reason being again disintegration of microstructure.



Fig. 6: Microhardness of Al-Al₂O₃ microcomposites before and after thermal shock.

3.3. Wear resistance

Fig. 7 & Fig. 8 illustrate the wear resistance of 1vol% reinforced Al-Al2O3 nanocomposite and 5 vol% reinforced Al-Al₂O₃ microcomposite. The wear resistance of nanocomposites not subjected to any thermal

shock is higher than the thermal shock subjected nanocomposites due to particle pull out, de-cohesion of alumina from the matrix. The microstructural integrity has been degraded such that the wear indenter while sliding over the surface encounters pulled put particles and disintegrated matrix, with weak interface. This results in lower wear resistance of thermal shock induced nanocomposites. The down thermal shock treated nanocomposites undergo effective gripping of alumina with aluminium as aluminium expand more than alumina at $+80^{\circ}$ C.



Fig. 7: Wear depth of 1% Al-Al₂O₃ nanocomposite before thermal shock and after thermal shock.



Fig. 8: Wear depth of 5% Al-Al₂O₃ microcomposite before thermal shock and after thermal shock.

4. Conclusion

Al-Al₂O₃ (5, 10, 15, 20 vol.%) micro- and (1, 3, 5, 7 vol.%) nano-composites have been fabricated by conventional sintering. The thermal shock induced nano- and micro-composites exhibit microstructural disntegrity showing particle pull out, defect in the matrix. The microhardness values of thermal shock treated nano- and micro-composites are lower as compared to that of non-treated corresponding specimens. The wear resistance of thermal shocked nano- and micro-composites reduces than the composites before treatment. The up and down thermal shock can accelerate weathering of micro- and nano-composites. The micro-phenonmenon taking place during thermal shock treatment can lead to major changes in mechanical behaviour of the composites.

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