

Structural and mechanical properties of CeO₂ reinforced Al matrix nanocomposites

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Abstract

In this study, Al-CeO₂ nanocomposites containing various concentrations of reinforcement were fabricated by ball milling and microwave sintering process. A comparison of structural and mechanical properties of the developed nanocomposites is presented to elucidate the effect of reinforcement. Different characterizing tools such as X-ray diffractometer (XRD), field emission scanning electron microscope (FESEM), surface profilometer, microhardness tester and universal compression testing machines were employed. XRD and SEM/EDX analyses reveal the presence and uniform distribution of CeO₂ nanoparticles into the Al matrix. A significant increase in microhardness and compressive strength is noticed with increasing concentration of CeO₂ nanoparticles due to a dispersion hardening effect of the reinforcement. Our study indicates that the concentration of reinforcement has a significant influence of the properties of Al-CeO₂ nanocomposites. It is further noticed that Al-2.0 vol.% CeO₂ nanocomposite demonstrates the best performance as compared to pure Al and other developed nanocomposites. Copyright © 2018 VBRI Press.

Keywords: Aluminum, CeO₂ nanoparticles, microwave sintering, surface profilometry, mechanical behavior.

Introduction

Aluminum metal matrix composites reinforced with nano size particles such as SiC, CeO₂, and Y₂O₃ are considered one of the important classes of metal-matrix composite (MMC). Due to their promising properties, these composites are most widely applied in defense, automotive, aerospace and electrical industries [1-3]. The field of MMC is constantly growing to develop novel composites having improved physical, chemical, structural, mechanical, thermal and electrochemical properties to meet the growing requirement of the engineering devices [4,5].

As per the literature, Al has already been successfully reinforced with ceramic oxide nanoparticles such as TiO₂, Al₂O₃ and SiO₂ to study the mechanical behavior of the composite materials [6-8]. And the results obtained so far are very encouraging which leads to the further study of with the new systems. Rare earth oxides are natural choices as the addition of rare earth elements (REEs) as reinforcement elements enhances various properties of aluminum. Adding a small amount of rare earth to the Al metal matrix can greatly improve the performance of the Al matrix properties [9]. Though, the rare earth elements possess some toxic characteristics but using them in nano-length scale is feasible as they can be used in lower amounts to gain the improvement in properties (typically < 2% by volume). [10]. Among the available rare earth oxides, CeO₂ has been studied by many of the researchers because of its remarkable properties such as high Vickers hardness, oxygen ion conductivity, large

surface area, high chemical stability, high specific capacitance, and non-toxicity [11]. Also CeO₂, one of the most important rare-earth metal oxides, has been in focus of intensive research during recent years due to its wide range of industrial applications [12].

The reinforcement size and amount can also contribute significantly in the properties of the final product. However, in order to be fabricating AMMCs effectively the reinforcement choice and its cost, along with its application can also contribute in the manufacturing process [13-15]. Several ceramic particles have been fabricated successfully in the near past using different methods such as casting and powder metallurgy (PM), which involve mixing the powders, compacting followed by microwave sintering and extrusion [16,17]. Among these stages, sintering is the most important step due to its ability to improve good microstructural properties that enhance the final product [18,6,7].

In this study, we have successfully synthesized aluminum-cerium oxide (Al-CeO₂) nanocomposites using ball milling, cold compaction followed by microwave sintering [19] and investigated their microstructural, surface and mechanical behavior.

Experimental

Materials

Commercially available, pure Al powder (Alfa Aesar, 10µm, 99.5% purity) and CeO₂ nanopowder (Alfa Aesar, 45-55nm mean particle size) were used as the precursor materials.

Synthesis of Al-CeO₂ nanocomposites

Different concentrations of CeO₂ nanoparticles (0.5, 1.0, 1.5 and 2 vol.%) were intimately mixed with pure Al using a ball mill employing a rotation speed of 200 RPM for 30 min. At this stage no balls were used to prevent particle size reduction. After blending, the mixed powders were then compacted into cylindrical pellets using a hydraulic press. A uniaxial compression pressure of 30 MPa for two minutes under ambient conditions was applied. The green compacted pellets were then subjected to sintering process. Sintering is the most important step in the synthesis of the composites as its process variables such as sintering temperature and soaking time plays an important role in the physical and mechanical properties of the final product. The microwave sintering process was carried out in a microwave furnace (VB Ceramic Consultants, Chennai, India), which has a silicon carbide ceramic crucible with alumina insulation as inside lining of the furnace. The samples were placed in the central cavity and sintered in a microwave furnace (multimode cavity) at 2.45 GHz. SiC microwave susceptor was used to assist the heating and sintering of the composite materials. The sintering process was carried out at 550°C implying a heating rate of approximately 30°C/min. The pellets were soaked for 30min at the sintering temperature and then the sintered samples were slowly cooled in the furnace to room temperature.

Characterization

X-ray diffraction studies of the microwave sintered composites were carried out using an automated Shimadzu diffractometer. The samples were exposed to Cu K α radiation ($\lambda = 1.54056\text{\AA}$) in the scanning range 20-90° at a scanning rate of 2 θ /m. The microstructural investigations of the sintered composites were carried out using field emission scanning electron microscope (FESEM, Jeol Neoscope JSM 6000). Surface roughness was measured using a surface profilometer (Leica DCM8, Switzerland). The microhardness values of the samples, were measured using a Vickers's microhardness tester (MKV-h21) applying a load of 100 gf for 10 sec) and the reported values are the average of at least five successive indentations for each sample. Universal testing machine-Lloyd 50KN at a strain rate 10⁻⁴/sec was used to measure the compressive properties of the cylindrical pellets with a diameter of 10 mm and a height of 3 mm. All the tests were carried out at room temperature.

Results and discussion

X-ray diffraction analysis

Fig. 1 shows the X-ray diffraction spectra of the microwave sintered Al-CeO₂ nanocomposites containing 0.5, 1.0, 1.5 and 2.0 vol.% CeO₂ nanoparticles. It can be noticed that the developed Al-CeO₂ nanocomposites have a crystalline structure. The reinforcement (CeO₂ nanoparticles) was identified in

expanded XRD peaks (inset). There was no impurity observed from the vials during milling. In addition, there is no observation of any diffraction peaks showing the formation of any oxide phase, although the blended procedure was carried out at room temperature in the absence of any protective medium.

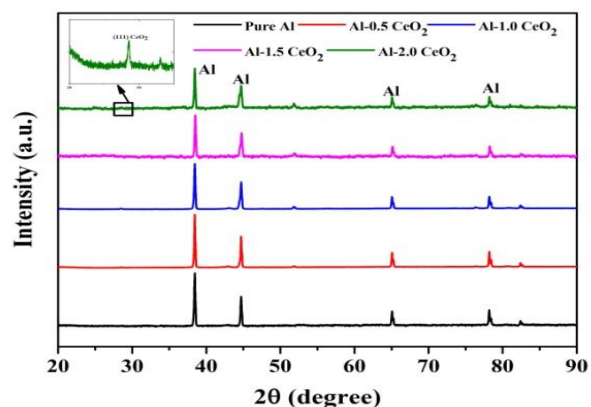


Fig. 1. XRD spectra of Al-CeO₂ nanocomposites.

Fig. 1 reveals the existence of CeO₂ particles (inset) within the Al matrix which confirms the formation of a composite structure. It can also be noticed that Al is the major phase in matrix material. Similar behavior has also been reported for Al-SiO₂ composite system [20].

SEM and EDX analysis

Field emission scanning electron microscope (FE-SEM) was used to analyze the morphology of the developed composites. The microstructure of the Al-CeO₂ nanocomposites is as shown in **Fig. 2 (a-d)**.

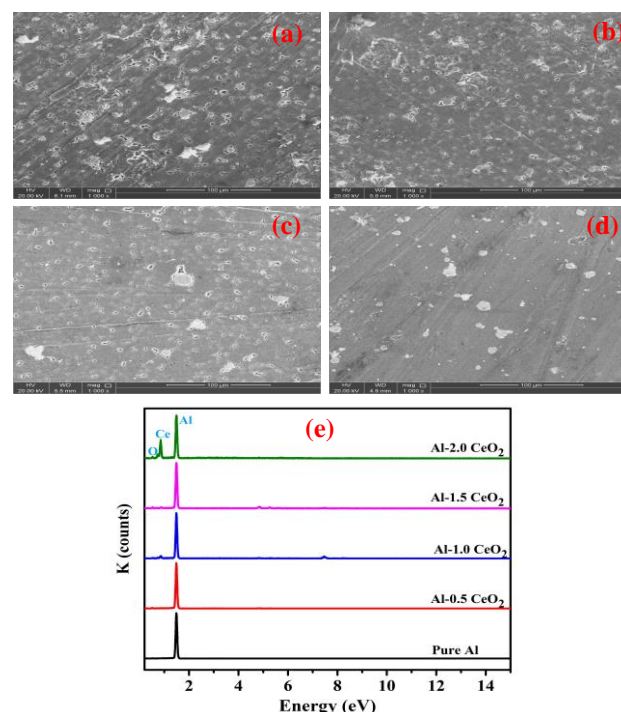


Fig. 2. (a-e) SEM/EDX images of Al-CeO₂ nanocomposites.

The dark region in the microscopic images contains mainly Al matrix and white region shows the CeO₂ particles. The images also demonstrate proper interfacial bonding without any cracking between the Al matrix and CeO₂ reinforcements. The uniform distribution of the CeO₂ nanoparticles (white) in the Al matrix (dark grey) can be noticed. However, agglomeration of the reinforcement can also be observed. The EDX analysis was conducted in order to determine the chemical composition of these two phases. It is noted that aluminum, cerium and oxygen are dominant in the matrix phase (see Fig. 2(e)).

Surface profilometry analysis

Surface morphology is examined by three-dimensional profilometer. The surface roughness parameter results of the developed Al-CeO₂ nanocomposites are furthermore reflected by surface images and profiles from a 3D profiler. Fig. 3(a-d) shows the surface topography of Al-CeO₂ (0.5, 1.0, 1.5 and 2.0 vol.%) nanocomposites. The average surface roughness of the composites was found to be 0.260 μm , which is relatively less compared to the unreinforced aluminum (0.395 μm).

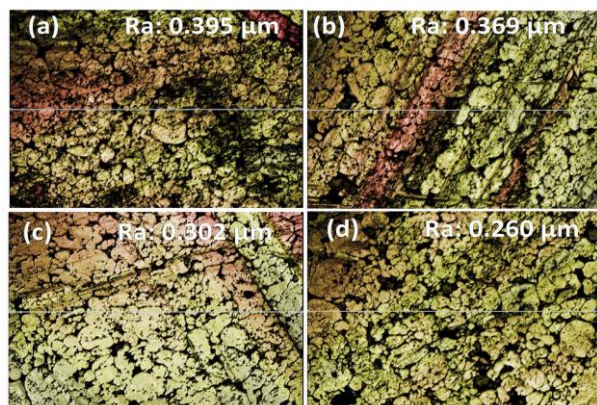


Fig. 3. Surface profiles of Al-CeO₂ nanocomposites.

Microhardness

The variation in microhardness of samples with different concentration of reinforcement (CeO₂) is presented in Fig. 4.

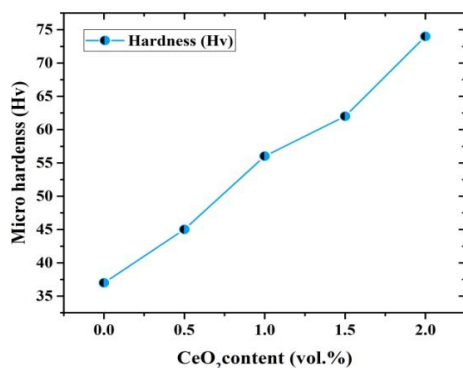


Fig. 4. Variation of microhardness with CeO₂ content in Al-CeO₂ nanocomposites.

The microhardness of the Al-CeO₂ nanocomposite increases with increasing concentration of CeO₂ nanoparticles. As a comparison, Al-CeO₂ nanocomposite containing the maximum percentage of reinforcement (2.0 vol. %) exhibits the highest hardness. The improvement in hardness can be considered as the effect of hard-ceramic reinforcing nanoparticles of CeO₂ causing an improvement in the load bearing capacity of the composite material and blockade movement of dislocation [18]. Based on the Orowan bowing mechanism, the CeO₂ nanoparticles are very small and hard, which enable them to block or interrupt the Al particles movement, which can be clearly demonstrated in the SEM images leading to an improvement in the microstructure hardness [19].

Compression analysis

Fig. 5 shows the engineering stress-strain curves of the Al metal matrix nanocomposites reinforced with CeO₂ nanoparticles.

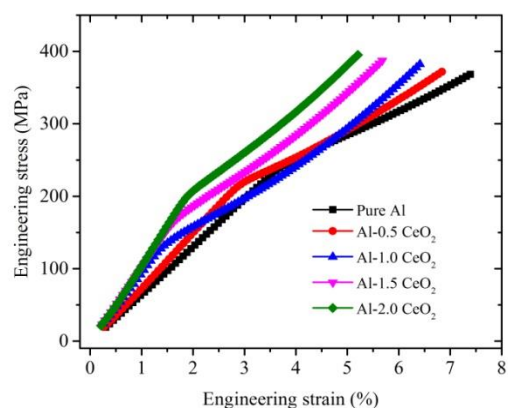


Fig. 5. Engineering stress-strain curves of the Al-CeO₂ nanocomposites under compression loading.

The stress strain curves show that the ultimate compressive strength of the Al-CeO₂ nanocomposite is significantly increased with the addition of CeO₂ reinforcing nanoparticles. The hard CeO₂ nanoparticles act as obstacles in the soft Al matrix and hinder the dislocations movement, hence enhancing the strength of the composites.

It is reported that the type and nature of the reinforcements control the mechanical properties of the composites. A strong interface between the particles and matrix is critical as it transfers and distributes the load from the matrix to the reinforcing particles. Therefore, in our case, it is assumed that the increasing amount of CeO₂ nanoparticles into Al matrix improves the interface which leads to the improvement in compressive strength of the composite material. As a comparison, Al-2.0 vol.% CeO₂ nanocomposite shows the maximum compressive strength of 395 MPa compared to the Al matrix (368 MPa). The decent and improved mechanical properties of Al-CeO₂ nanocomposites may be attractive for some industrial applications.

The strength induced by the dispersion of the CeO₂ nanoparticles in the aluminum matrix can be explained on the basis of Orowan strengthening mechanism. Actually, residual dislocation loops are formed around every CeO₂ particles due to bending of dislocation passing by the hard CeO₂ particles. This loop formation leads to high work hardening rates and helps to strengthen the material. The contribution to yield strength can be expressed as [21, 22]:

$$\sigma_{\text{Orowan}} = \frac{2Gb}{\lambda}$$

Where G and b are the shear modulus of the matrix and Burger's vector of the dislocation, and λ is the distance of dispersed particles.

Conclusion

Al-CeO₂ nanocomposites (0, 0.5, 1.0, 1.5 and 2.0 vol.%) were fabricated through microwave sintering process. The developed Al-CeO₂ nanocomposites demonstrate a well defined crystal structure. SEM/EDX study revealed fine CeO₂ particles are distributed homogeneously in the matrix. The hardness and the compressive strength of Al-CeO₂ nanocomposites increase with increasing amounts of CeO₂ nanoparticles in the aluminum matrix. This improvement in mechanical behavior can be attributed to homogeneous distribution of reinforcement particles and dispersion hardening effect.

Acknowledgements

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