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Electrical Conductivity and Mechanical Hardness of Al_2O_3-1wt% MWCNT Nanocomposite Synthesized by Ball Milling Method

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Alumina (Al_2O_3) powder is dispersed with 1wt% multi-walled carbon nanotube (MWCNT) by ball milling method and then electrical conductivity and mechanical hardness of the formed nanocomposite was determined. Scanning electron microscopy (SEM), Energy-dispersive X-ray spectroscopy (EDX) and X-Ray diffraction studies were made for the morphological, compositional and constitutional analysis of the formed nanocomposite. Results showed that Al_2O_3-1wt% MWCNT nanocomposite performs better in electrical conductivity and mechanical hardness as compared to Al_2O_3 alone.

Keywords: Al_2O_3, MWCNT, Nanocomposite, Electrical conductivity, Micro-hardness, Nano-hardness

Introduction

Nanocomposites made from CNT have been studied extensively[1,2]. A large number of ceramics-CNT nanocomposites have been prepared and it is found that the mechanical properties are improved[3]. Al_2O_3 is used as structural material but because of its brittleness, the use is restricted. However, if CNTs are dispersed in the matrix of Al_2O_3, the fracture toughness is improved[4]. Number of methods has been used for the preparation of Al_2O_3-CNT nanocomposites but the ball mill method is most suitable[5]. In this study sol gel followed by ball milling method has been used for the preparation of Al_2O_3-1wt% MWCNT nanocomposite. The nanocomposite was characterised using different techniques. The electrical conductivity and micro-hardness values of the formed nanocomposite were determined and compared with the pure alumina powder.

Materials and methods

Materials

Aluminium isopropoxide(AIIP) (~98%), nitric acid, stearic acid, Polyvinyl Alcohol (PVA) and ethanol were purchased from MERCK. The Multi-walled carbonnanotubes (purity > 96%) used in this study were purchased from Ishu international India. MWCNTs were refluxed with nitric acid for 1h at 100 °C for removing impurities.

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Methods

Synthesis of Al_2O_3-MWCNT nanocomposite

Two step process was used for the synthesis of Al_2O_3-1wt% MWCNT nanocomposite. In the first step, sol-gel hydrolysis method was used for the dispersion of MWCNT in alumina powder. AlIP precursor was used for preparation of alumina. Hydrolysis of AlIP gave alumina xerogel (porous) in aqueous medium whereas in the presence of non-aqueous medium alumina aerogel is formed[6]. For the formation of Al_2O_3-1wt% MWCNT nanocomposite, 50 mg/ml of AlIP in ethanol was stirred for 30 min and then mixed with 1wt % MWCNT and stirred for another 1 h at 80 °C. The formed gel was washed with warm distilled water and then dried (3 h) in a furnace at 300 °C. In the second step, a planetary ball mill having stainless steel balls of diameter 12 mm was used. Ball to powder ratio (BPR) was kept 8:1. The dried mixture was ball milled at 300 rpm for 1 h for achieving ultrafine grain of Al_2O_3-1wt% MWCNT nanocomposite. During the ball milling process, stearic acid was used as a process control agent (PCA) for the protection of alumina from agglomeration[5,7].

Sample characterization

X-Ray diffraction pattern was recorded with Shimadzu XRD 6000 using CuK_α radiation (λ=0.1542 nm) at 4.8 kW, 40.0 kV and 120.0 mA. The morphological and EDX characterisation of the nanocomposite was done using FESEM (JSM-6400, JEOL, Japan).

Micro and nano hardness

Pellet of nanocomposite (diameter 1.14cm & thickness- 0.15 cm) was made by using hydraulic press at a pressure of 1 ton for 30 sec. The micro-hardness values (HV) of the prepared nanocomposite were determined by using Vickers micro-hardness tester (VH1202 Wilson@ buehler, U.S) at working load of 20g for dwell time of 10 sec. Nanohardness of the formed nanocomposite was determined by performing nano-indentation tests on Bruker's hysitron nano-indenter equipped with berkovich tip (pyramidal geometry) at a working load of 100µN with holding time of 10 sec before being unloaded.To check the consistency of the results 5 indents at random points were performed on each sample.

Electrical conductivity measurements

The electrical conductivities of Al_2O_3 and Al_2O_3-1wt% MWCNT nanocomposite were measured by AC impedance spectroscopic method using HIOKI 3532-50 LCR Hi TESTER at different temperatures and at a frequency of 100 kHz.

Results & Discussion

X-ray diffraction pattern of Al_2O_3-1wt% MWCNT nanocomposite sintered at 600 °C for 1h and EDX analysis of Al_2O_3-1 wt % nanocomposite is shown in Fig.1.The XRD pattern of Al_2O_3-1wt% MWCNT nanocomposite sintered at 600 °C for 1 h (Figure.1) did not show the presence of CNT as the concentration of MWCNT was very low as compared to alumina powder. X-ray diffraction pattern (Fig.1) shows less crystalline character of Al_2O_3-1wt% MWCNT nanocomposite at sintering temperature of 600 ºC. The amorphous nature of alumina may be due to lower sintering temperature ($\leq$ 800 ºC) [6].

EDX analysis shows the compositional constituent of O, Al, C and Pt for the Al_2O_3-1wt% MWCNT nanocomposite at 33.9, 32.7, 17.3 and 16.2 weight % respectively which shows a weight ratio of 1.8 for Al to C. EDX technique shows the purity of fabricated nanocomposite. The individual CNT has very high electrical conductivity value and thus nanocomposite formed by addition of MWCNT increased the electrical conductivity value. Figure.2a shows the

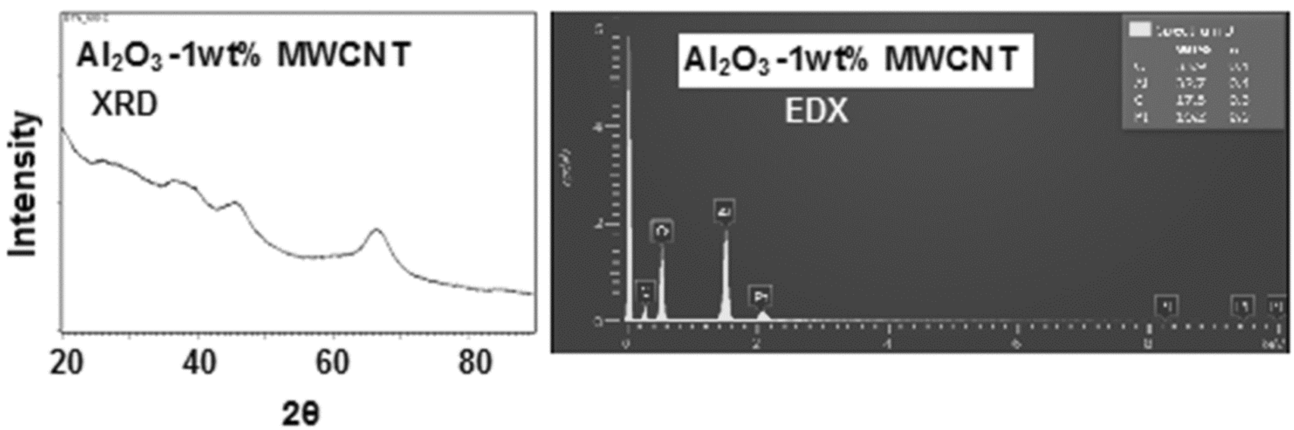

Fig. 1 — XRD pattern and EDX spectra of Al_2O_3-1 wt % MWCNT nanocomposite sintered at 600 °C for 1h

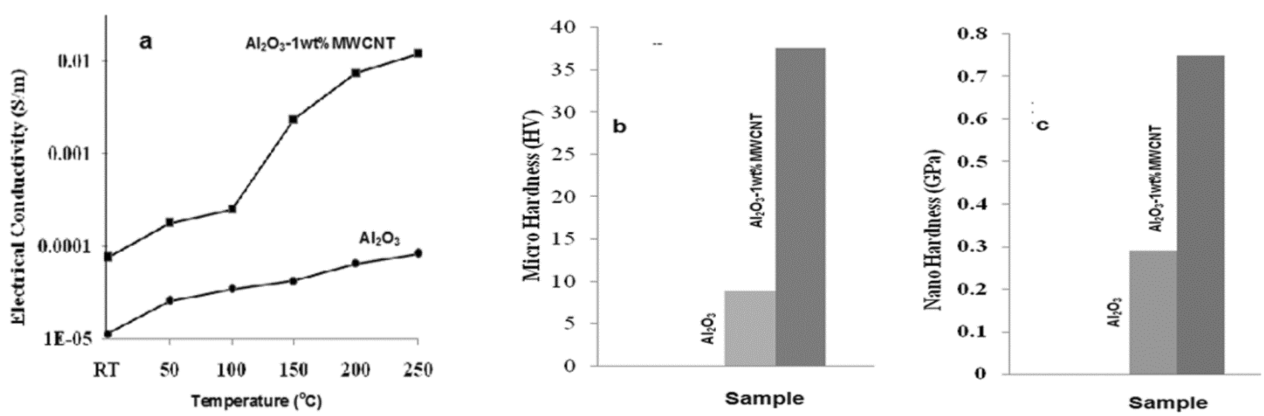

Fig. 2 — (a) Variation of electrical conductivity with temperature (b) Microhardness and (c) Nanohardnes

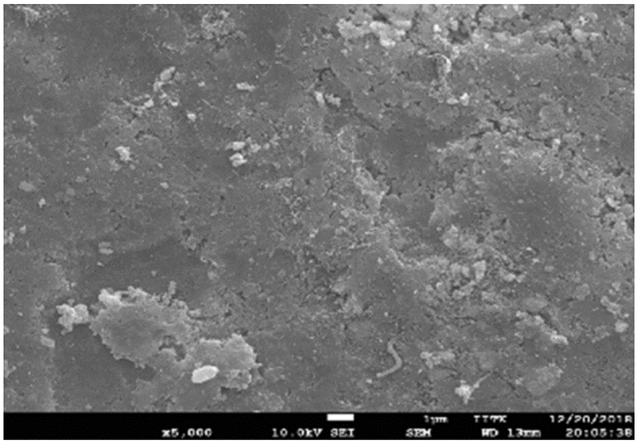

Fig. 3 — SEM of Al_2O_3-1wt % MWCNT nanocomposite

effect of 1wt% MWCNT on electrical conductivity of alumina powder at different temperatures. The electrical conductivity of Al_2O_3-1wt% MWCNT nanocomposite at 250°C was found to be approximately 100 times higher than that of pure alumina powder. This increase in electrical conductivity of the formed nanocomposite may be due to high length to diameter ratio (aspect ratio) of the MWCNT, providing a complex pathway for increase in conductivity. The formed nanocomposite was tested for its mechanical hardness. Before doing micro-structural examination and micro-indentation hardness test, both the samples were ground with abrasive papers of 600 to 1500 grits, followed by polishing with 3 and 0.25µm diamond aerosol and at last vibratory polishing by repeated dispersion of colloidal silica slurry (0.05µm) on napped cloth of the polishing machine. To determine the hardness values of the formed nanocomposite, working load of 20g (10sec) was applied for micro-hardness test and 100µN (10 sec) for nano-hardness test at 5 random points and then taken average of all 5 indentations. Figures 2b and 2c show the microhardness value of 37.55 HV and nanohardness value of 0.75 GPa for Al_2O_3-1wt% MWCNT nanocomposite which are much higher than the corresponding values of alumina powder i.e; 8.92 HV and 0.29 GPa respectively. The increase in mechanical hardness value of the formed nanocomposite is due the strong interfacial bonding between both the powders and adequate densification that confirms efficient load sharing capability.

The SEM image of Al_2O_3-1wt% MWCNT nanocomposite (Figure.3) shows the homogenous mixing of MWCNT in alumina powder. The large length to diameter ratio (aspect ratio) of the MWCNT bridged relatively small grains of Al_2O_3 which improves load carrying capability of the formed nanocomposite and shows the enhanced interfacial property.

Conclusion

Two-step synthesis method was used to synthesise Al_2O_3-1wt% MWCNT nanocomposite. Al_2O_3-1wt % MWCNT nanocomposite has high electrical conductivity, micro-hardness and nano-hardness value of 1.21×10^{-2} S/m, 37.55 HV and 0.75 GPa respectively as compared to pure alumina powder. The large aspect ratio of MWCNT improves load bearing capability of nanocomposite and provide pathway for conductivity.

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