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Fabrication of Carbon Nanotube Reinforced Al_2O_3/Cr_2O_3 Nanocomposites by Coprecipitation Process

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Abstract

In this research, the effect of multi-walled carbon nanotubes (MWCNTs) on the alumina/chromia (Al_2O_3/Cr_2O_3) nanocomposites has been investigated. Al_2O_3/Cr_2O_3-MWCNTs nanocomposites with variable contents of Cr_2O_3 and MWCNTs were fabricated using coprecipitation process and followed by spark plasma sintering. XRD analysis revealed a good crystallinity of sintered nanocomposites samples and there was only one phase presence of Al_2O_3-Cr_2O_3 solid solution. Density, Vickers microhardness, fracture toughness and fracture strength have been measured in the sintered samples. The results show that the relative density, microhardness and fracture strength of nanocomposites are significantly improved at low contents of Cr_2O_3 and MWCNTs. The increase of MWCNT's content in the nanocomposites has adversely affected due to increasing the tangle and interaction of MWCNTs with each other, which leads to agglomeration in the nanocomposites. Increasing of Cr_2O_3 content in nanocomposites increases formation of Al_2O_3-Cr_2O_3 solid solution that actually requires the high sintering temperature to achieve good densification. The fracture toughness of Al_2O_3/Cr_2O_3-MWCNTs nanocomposites was enhanced by increasing the carbon nanotube content.

Keywords: *Al_2O_3/Cr_2O_3 nanocomposite, MWCNTs, coprecipitation process and spark plasma sintering (SPS).*

1. Introduction

The field of the nanocomposite materials has taken a lot of interesting and close observation of engineers and scientists in recent years [1].

Nanocomposite materials can be considered of solid structures with nanometer scale dimensional repeat distances between the various phases that constitute the structure. The most common methods for processing nanocomposites are mechanical alloying, coprecipitation process, sol-gel synthesis and by thermal spray synthesis [1,2]. The increasing attention in the solid solutions of ceramic oxide materials is mostly related to their thermal and chemical stability and their structural characteristics [3].

Alumina/chromia (Al_2O_3/Cr_2O_3) is a simple binary system, as shown in Fig. 1. [3,4]. Al_2O_3 and Cr_2O_3 are the stable sesquioxides having the same corundum crystal structure (hexagonal system with the Al^{3+} and Cr^{3+} ions taking two thirds of the octahedral interstitial sites). At high temperature, they form complete substitutional solid solution without consistence of any eutectic over the entire composition range [3,5]. Al_2O_3–Cr_2O_3 system is great interest due to of its high refractoriness and chemical stability at high temperatures. Also, this system has superior mechanical properties and good thermal shock resistance [4,6]. Densification behavior, strength, modulus of rupture and hardness exhibited irregular dependency reliance on each other [5,6]. However, low fracture toughness of ceramics still cannot match the command of many practical fields, which greatly limits its applications [7].

Recent researches are devoted to the possible incorporation of carbon nanotubes (CNTs) in ceramic matrix composites in order to improve their performance [8,9]. CNTs have attracted a great attention as potential strengthening and toughening materials in industrial applications due to their particular structure, low density, high strength and toughness and excellent corrosion resistance [7,9].

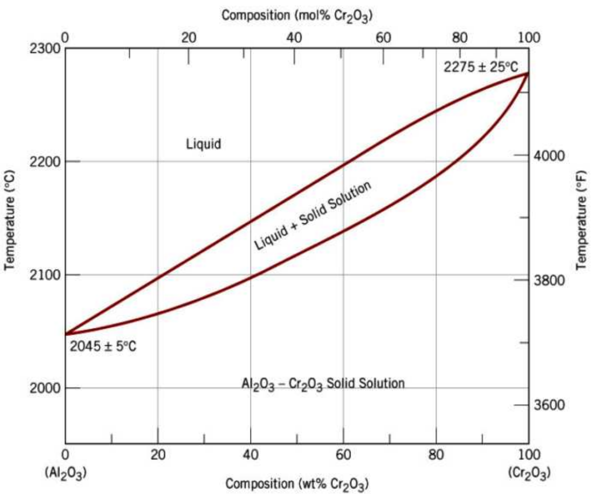

Fig. 1. Phase diagram of Al_2O_3–Cr_2O_3 system [3].

Many studies have studied the mechanical properties, especially the toughness, of ceramics through incorporating CNTs in ceramic matrix. For example, Echeberria et al. [8] demonstrated that 0.1 wt% of MWCNTs or single walled carbon nanotubes (SWCNTs) added to the zirconia toughened alumina (ZTA) composites, and followed by spark plasma sintering at 1520°C, is enough for obtaining to high hardness and fracture toughness. Ahmad et al. [10] studied the influence of the carbon nanotubes content on the mechanical properties of Al_2O_3 that fabricated by hot-pressing. They reported a 32% increase in fracture toughness and 12% increase in hardness were observed with 2 wt% of CNTs additive, when compared with the monolithic Al_2O_3. Siegel et al. [11] reported a 24% increase in fracture toughness of Al_2O_3 with additions of 10 vol% MWCNTs.

The present work aims to study the effect of various amounts of multi-walled carbon nanotubes on the some mechanical properties of Al_2O_3/Cr_2O_3 nanocomposites. Coprecipitaion process is used to disperse MWCNTs homogeneously in Al_2O_3/Cr_2O_3 solid solution matrix by entrapping the dispersed carbon nanotubes in the gel network. The prepared nanocomposites samples are densified using a new sintering process such as spark plasma sintering. Density, Vickers microhardness, fracture toughness and fracture strength of the Al_2O_3/Cr_2O_3-MWCNTs nanocomposites as a function of the MWCNTs and Cr_2O_3 contents are investigated.

2. Experimental Work

Aluminum nitrate ($Al(NO_3)_3.9H_2O$, >98%, Alfa Aesar, UK) and chromium nitrate ($Cr(NO_3)_3.9H_2O$, 99%, Alfa Aesar, UK) were used as precursors for preparing of alumina/chromia (Al_2O_3/Cr_2O_3) nanocomposites. Multi-walled carbon nanotubes (MWCNTs) having dimensions of about (20-30) nm in outer diameter, (0.5-10) µm in length and density of 1.85 g/cm^3, (provided from Sigma–Aldrich, UK), was used as reinforcement material. Ceramic nano-composites of Al_2O_3 with two weight percentages of Cr_2O_3 (10 and 20) wt% were prepared using coprecipitation process. In this process, aluminum nitrate and chromium nitrate were separately dissolved in solution of (distilled water/ethanol) (1/1) wt% with a molar concentration of 0.5 M for each one. Appropriate amount of chromium nitrate solution have been mixed with aluminum nitrate solution for obtaining two solutions with different percentages of Cr_2O_3 in Al_2O_3 (Al_2O_3 -10wt% and -20wt% Cr_2O_3). The solutions were stirring at 250 rpm in a magnetic stirrer for 1 hour at 120°C. The MWCNT was dispersed in small amount of ethanol solution and then added to the alumina/chromia solutions during stirring process. The weight fraction of MWCNTs was controlled as (0, 1.5, 3 and 4.5) wt% from the total weight of the samples as shown in table (1). The mixed solutions were ultrasonicated for 20 min to avoid agglomeration and more homogenization of carbon nanotubes in alumina/chromia solutions. Addition of ammonium hydroxide drops to the total mixed solutions until alumina/chromia-MWCNTs gel was formed. The prepared gels have been filtered, and calcined at 500°C for 4 hours in an electrical furnace. The calcined gels were ground using mortar and pestle. The samples sintering were carried out by spark plasma sintering (SPS) technique. The sintering temperature was 1500°C at a heating rate of 100°C/min, the applied pressure is 80 MPa and the holding time is 5 min. The dimensions of sintered samples were 20 mm in diameter and 5 mm in thickness. A flow diagram for the experimental work is shown in Fig. 2.

Characterizations of the sintered specimens were evaluated using X-ray diffraction (XRD), scanning electron microscope (SEM). Apparent density of the sintered specimens was determined using Archimedes method, and the relative density was calculated by dividing the apparent density by the theoretical density.

In this work, 3.94 g/cm^3, 5.21 g/cm^3 and 1.85 g/cm^3 were assumed for the theoretical density of Al_2O_3, Cr_2O_3 and MWCNTs respectively.

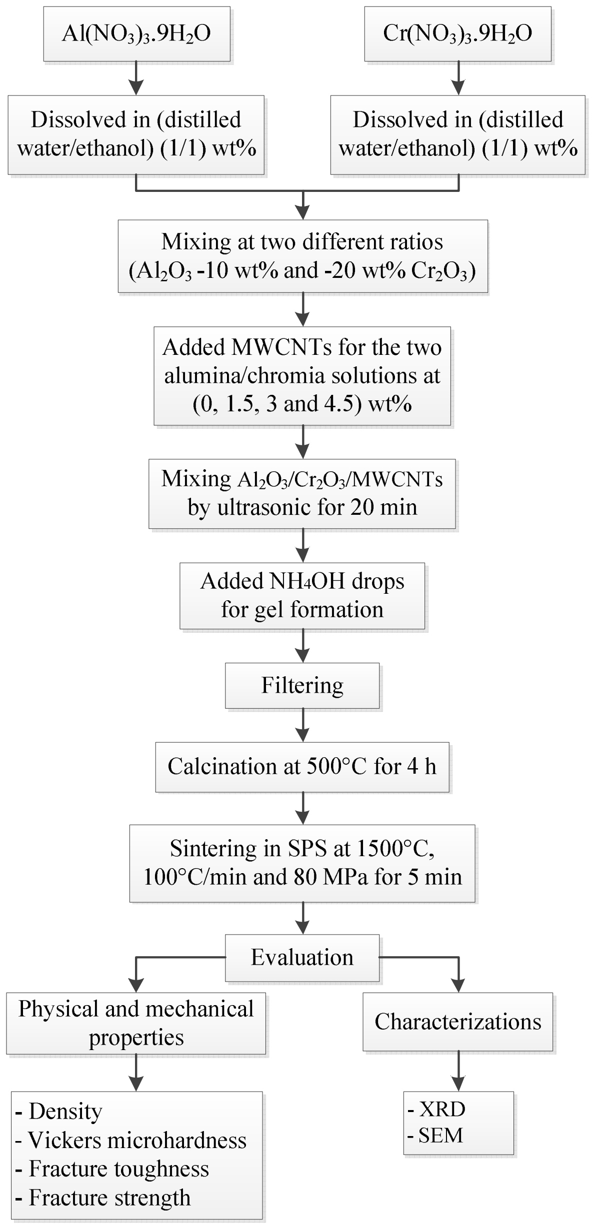

Fig. 2. Flow chart for the experimental part of this work.

The hardness was measured using a Vickers microhardness testing machine (Mitutoyo HM 122) at load of 9.81 N for 15 sec. Indentation diagonal lengths and crack lengths extending from the corners of the Vickers indentations were determined using optical microscopy for fracture toughness (K_{1C}) measurements. The fracture toughness is calculated using Shetty et al. method as in equation (1) [12].

$$K_{IC} = 0.0319\ \frac{P}{d\ .\ l^{1/2}} \qquad \dots (1)$$

Where: K_{1C} = fracture toughness (MPa.m$^{0.5}$), P = applied load (N), d = Vickers indent diameter (m) and l = crack length (m).

The fracture strength is determined using the diametrical compression disc test and the equation (2) was applied to calculate the strength of the samples [13]:

$$\sigma_s = \frac{2\ F}{\pi\ D\ T} \qquad \dots (2)$$

Where: σ_s = fracture strength, F = applied load (N), D = diameter of the disc (mm) and T= thickness of the disc (mm).

Table 1,
Preparation of nanocomposite samples.

Sample Composition	MWCNTs Additions
Al_2O_3-10wt% Cr_2O_3	(0, 1.5, 3 and 4.5) wt%
Al_2O_3-20wt% Cr_2O_3	(0, 1.5, 3 and 4.5) wt%

3. Results and Discussion

3.1. X-Ray Diffraction (XRD)

X-ray diffraction patterns for sintered Al_2O_3-10 wt% Cr_2O_3 nanocomposite with different MWCNTs content (0 and 3) wt% are chosen in this test as shown in Fig. 3-a and -b respectively. It has been shown that all peaks were matched of α-Al_2O_3 with small shifting, corresponding to JCPDS card No 43-1484. As known, the addition of Cr_2O_3 in the Al_2O_3, the distances between a-Al_2O_3 planes increase and there is a shift of all the peaks conforming to the pure alumina which indicates the formation of solid solution [3-5].

From these figures also, it is clear that the XRD pattern for the nanocomposite with 0 wt% of MWCNT exhibit almost similar pattern to that with 3 wt% of MWCNT and there are no new phases appearing. This indicates the carbon nanotubes are stable at elevated temperature during the sintering process and did not reacted with Al_2O_3 and Cr_2O_3. Fig. 3-b also reveals only the typical crystalline peaks from alumina and no peaks from carbon nanotubes were observed. The diminishing of MWCNTs peaks are possibly due to the high crystallinity of Al_2O_3/Cr_2O_3 solid solution phase and low contents of CNTs in the nanocomposites.

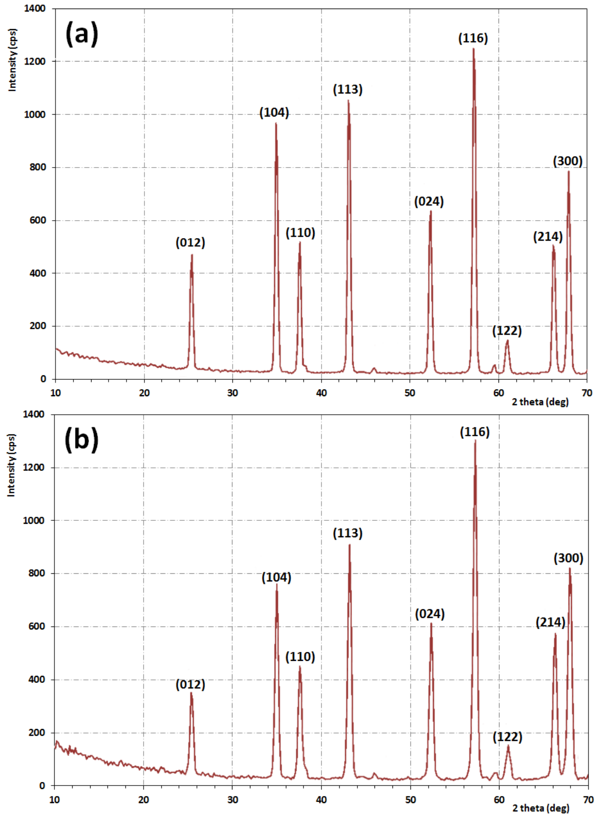

Fig. 3. XRD pattern of the sintered Al_2O_3-10wt% Cr_2O_3 samples with different MWCNTs contents, (a) 0 wt% and (b) 3 wt%.

3.2. Relative and Apparent Density

The effect of various carbon nanotubes content on the relative density of sintered nanocomposites can be observed in Fig. 4. Fig. 4 indicates that the relative density of Al_2O_3-10 wt% Cr_2O_3 samples is slightly increased to reach the maximum value (99.5%) with the addition of 1.5 wt% MWCNTs, and then decreases for further increase in the MWCNTs content. When the content of carbon nanotubes is less, carbon nanotubes may be well adhered and dispersed within the matrix without apparent damage to the microstructure and morphology. However, the increase of MWCNTs content in nanocomposites the relative density decreases. It is reported that the increasing presence of CNTs in a ceramic matrix play a negative role and act same as an impurity, which restrains the flow of matrix through the sintering stage, hence accounts inhibits the densification of the nanocomposite and decrease in the relative density [7,14].

From Fig. 4 also, it is seen that the Al_2O_3-10wt% Cr_2O_3 nanocomposites samples have higher values of relative density, at different carbon nanotube content, than Al_2O_3-20wt% Cr_2O_3. This is because the chromia has a higher melting point (2440°C) than alumina (2070°C), therefore with increase the chromia contents in alumina; the final composites will need a higher sintering temperature to achieve good densification.

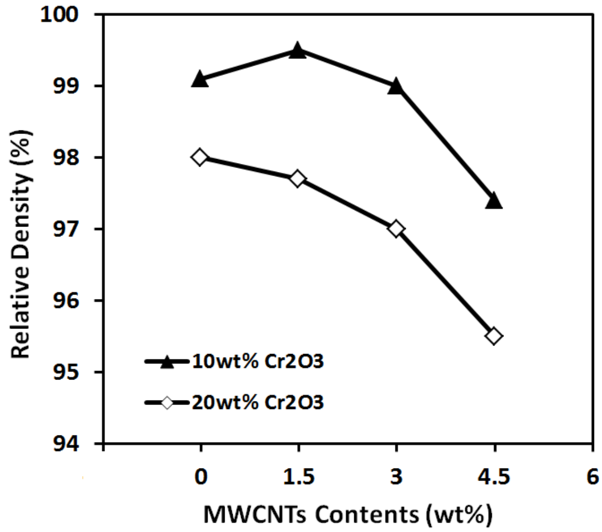

Fig. 4. Effect of MWCNTs addition on the relative density of Al_2O_3/Cr_2O_3 nanocomposites samples.

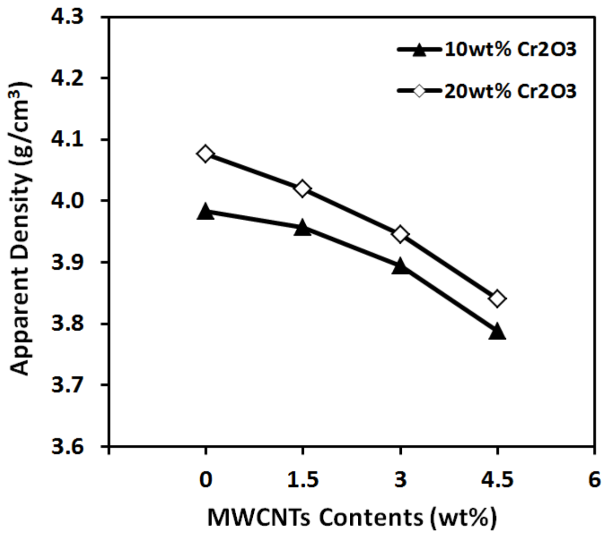

Fig. 5. Effect of MWCNTs addition on the apparent density of Al_2O_3/Cr_2O_3 nanocomposites samples.

Fig. 5 reveals the apparent density versus MWCNTs additive for sintered nanocomposites samples of (Al_2O_3-10 wt% and -20 wt% Cr_2O_3). It has been shown that the apparent density of Al_2O_3/Cr_2O_3-MWCNTs nanocomposite samples is slightly decreased with increasing the carbon nanotubes contents. The apparent density of the Al_2O_3-10 wt% Cr_2O_3 samples is decreased from (3.98 to 3.79) g/cm^3, and from (4.08 to 3.84) g/cm^3 for Al_2O_3-20 wt% Cr_2O_3 nanocomposite samples. As known, the porosity is inversely proportionate with the density of composites. On the other hand, the tangle and interact of carbon nanotubes with each other through van der Walls force, which increases the probability of the CNTs agglomeration in the nanocomposites, and thereby makes it difficult to acquire a homogeneous dispersion of CNTs in the ceramic matrix. The clustering of CNTs may play a negative role like a pore. The pore density extremely influences on the densification of the ceramic composites [14]. Mass transportation and elimination of pores through bulk diffusion are two important factors that determine the final density during the consolidation process of the alumina ceramics. It is believed that the high values of carbon nanotubes into the Al_2O_3/Cr_2O_3 matrix have adversely affected and this resulted in nano-composites with lower density [10].

From Fig. 5 also, it has been shown that the nanocomposite samples from Al_2O_3-10 wt% Cr_2O_3 having higher values of apparent density than Al_2O_3-20 wt% Cr_2O_3, at different carbon nanotube content. This is attributed to increasing the Cr_2O_3 content that has higher specific gravity of (5.22) as compared to Al_2O_3 (3.94) [5].

3.3. Microhardness

The microhardness of a ceramic material is an important mechanical property because it relates how much the material will in elastically deform when a surface load is applied. Because, the indentation diameters of microhardness Vickers tester for sintered samples are very small. The light optical microscope assisted computer program is used for analysis the indentation images and calculates the Vickers microhardness of the sintered samples. The Vickers microhardness values of the sintered Al_2O_3/Cr_2O_3 nanocomposites samples as a function of MWCNT content are shown in Fig. 6. It is clear that samples of Al_2O_3-20 wt% Cr_2O_3 have higher values of microhardness than Al_2O_3-10 wt% Cr_2O_3 at different carbon nanotubes content. This is attributed to effects of the formation of chromium (III) oxide solid solution in aluminium (III) oxide. Since, both the oxides (Al_2O_3 and Cr_2O_3) form complete substitutional solid solution over the whole composition range. The hardness is increased by microstructural modification that caused the compressive stress generated by the substitution of bigger Cr^{3+} ion (0.076 nm) in place of smaller Al^{3+} ion (0.068 nm). The localized compressive stress, especially on the sample surface, helps to the microstructure strengthening of nanocomposites, and then the microhardness increases by this effect [5,15]. Furthermore, it is well known that chromia has hardness slightly higher than alumina. Hence, that there is increasing increment in microhardness of the nanocomposite with increasing content of chromia [16].

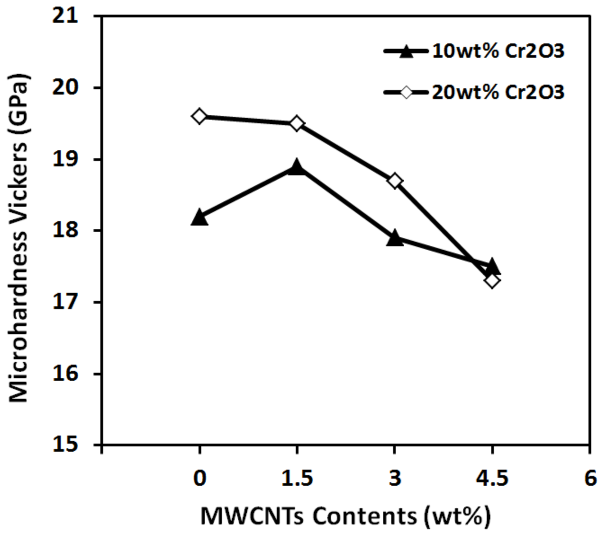

Fig. 6. Vickers microhardness of the Al_2O_3/Cr_2O_3 nanocomposite samples as a function of the MWCNTs content.

The microhardness values of Al_2O_3/Cr_2O_3 nano-composites also affected with the carbon nanotube contents. Fig. 6 also shows that the microhardness values of the Al_2O_3-20 wt% Cr_2O_3 nanocomposite samples decreases gradually with increasing the carbon nanotube contents. While the microhardness value of Al_2O_3-10%Cr_2O_3 samples increases with 1.5 wt% of MWCNTs addition, then it decreases gradually with increasing the carbon nanotube content. The increase in the carbon nanotubes content above the optimal value impedes the densification of the nanocomposite, and hence considerations for the decrease in the microhardness. Because the hardness values are highly correlated with relative density and porosity, thereby reducing the number of defects in a sample is a common way of increasing its microhardness [14,17].

3.4. Fracture Toughness

Fracture toughness of sintered nanocomposites is calculated according to the Shetty et al. crack-length method. The effect of carbon nanotube addition on fracture toughness of the sintered nanocomposites samples of (Al_2O_3-10 wt% and -20 wt% Cr_2O_3) is shown in Fig. 7. From this figure, it is obvious that by increasing the MWCNTs content, the fracture toughness of Al_2O_3-10 wt% Cr_2O_3 and Al_2O_3-20 wt% Cr_2O_3 is increased. This increasing in fracture toughness is attributed to increasing the presence of the MWCNTs secondary phase and solid solution of Al_2O_3/Cr_2O_3 in the microstructure of such samples.

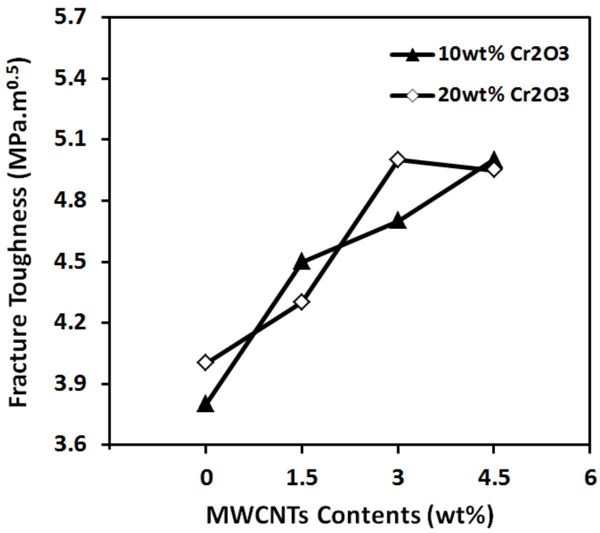

Fig. 7. Fracture toughness of the Al_2O_3/Cr_2O_3 nanocomposites as a function of MWCNTs content.

The increases in quantity of carbon nanotubes in the nanocomposites may make in an easier convey of the stress and thus could account for the considerable increase in the fracture toughness [14]. Furthermore, the residual pores located between the agglomerated multi-walled carbon nanotubes in the nanocomposites, especially at high CNTs content, could function as crack arrestors, resulting to the increase of observed fracture toughness [18].

3.5. Fracture Strength

Fig. 8 shows the effect of carbon nanotubes addition on the fracture strength of the sintered nanocomposites samples of (Al_2O_3-10 wt% and -20 wt% Cr_2O_3). It is found that the addition of carbon nanotube is highly improving in the fracture strength of Al_2O_3-10wt% Cr_2O_3 than Al_2O_3-20wt% Cr_2O_3 nanocomposite samples. In the Al_2O_3-10wt% Cr_2O_3 nanocomposite samples, the fracture strength increases rapidly from 88.3 MPa to 106.8 MPa when addition of 1.5 wt% of MWCNT, then it decreases gradually with increasing the MWCNTs content. A sharp increase in the fracture strength can be related to enhancement in the relative density of nanocomposites. The increase in the mechanical properties at low carbon nanotube additions can be associated with the well dispersed of MWCNTs within the Al_2O_3/Cr_2O_3 matrix and strong interfacial connection between MWCNTs and the matrix. The improvement in relative density with fewer pores, which leads to increasing the particles bonding and consequent strength enhanced [10].

The decrease in the fracture strength at higher carbon nanotube content in nanocomposites may be due to the arising difficulties of densification with higher concentrations of CNTs. Furthermore, the generation of thermally induced residual stresses after sintering of nanocomposite samples is another factor that may have contributed to the lowering in the strength. The mismatch in the thermal expansion coefficient between the matrix Al_2O_3/Cr_2O_3 of (~ 8.1×10^{-6}/°C), and MWCNTs (~ 2.7×10^{-6}/°C) could produce residual stresses around the dispersed carbon nanotubes, which could result the micro cracks, which consequently results in the lowering of mechanical properties. Besides, it is accepted that the CNTs existing in the grain boundaries act as spatial barriers to inhibit closing up of grains [14,18].

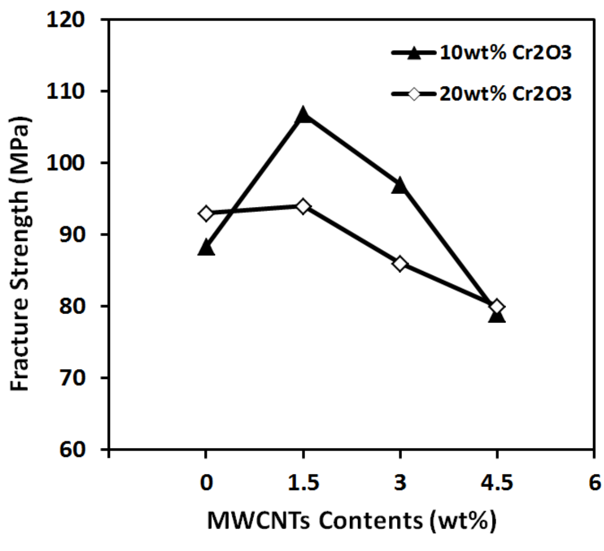

Fig. 8. Effect of MWCNTs addition on the fracture strength of Al_2O_3/Cr_2O_3 nanocomposites samples.

Fig. 9 shows the SEM micrographs of fracture surfaces of Al_2O_3-10 wt% Cr_2O_3 nanocomposite samples with different contents of MWCNTs. Nanocomposite sample without MWCNTs, Fig. 9-a, the SEM micrograph illustrates that the fracture proceeded mostly by intergranular and this can be associated with occurring some of grain growth during the consolidation process. On other hand, it is observed that the addition of MWCNTs to the nanocomposite leads to decrease in the grain size, this clearly shown in Fig. 9-b and -c. The addition of the MWCNTs to the nanocomposites, the fracture surface becomes slightly rough and exhibit two fracture modes (intergranular and transgranular) fracture.

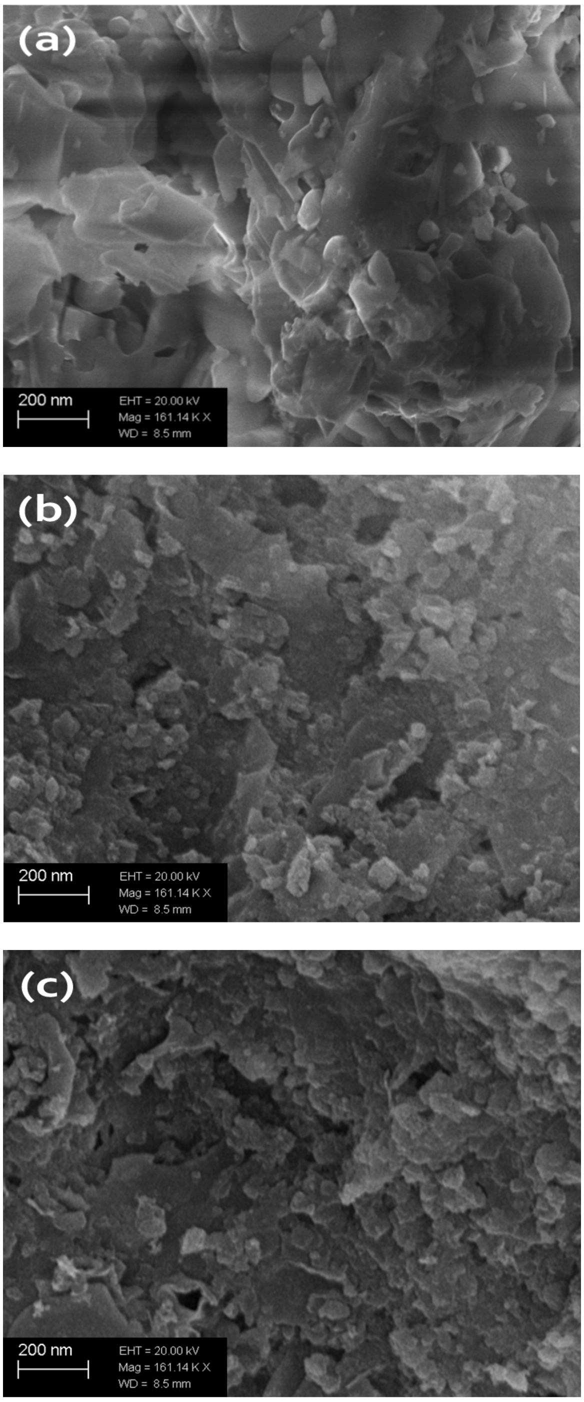

Fig. 9. SEM micrographs of fracture surface of Al_2O_3-10 wt% Cr_2O_3 nanocomposite samples with different MWCNTs content: (a) 0 wt%, (b) 1.5 wt% and (c) 4.5 wt%.

The great irregularity exhibited in the fracture surface of nanocomposites with increasing of MWCNTs addition leads to increase in fracture toughness of the samples. This is because the high deflection angles lead to increment the crack path tortuosity, which crack propagation encounters with a large obstruction [8,19].

4. Conclusions

Al_2O_3/Cr_2O_3-MWCNTs nanocomposites were successfully fabricated using the coprecipitation process and followed by spark plasma sintering technique. MWCNTs were well despaired within the Al_2O_3/Cr_2O_3 nanocomposites samples by coprecipitation process at low carbon nanotube contents. The relative density, microhardness and fracture strength of the Al_2O_3-10wt% Cr_2O_3 nanocomposites were enhanced with addition of 1.5 wt% MWCNTs. The increasing of carbon nanotubes content in the nanocomposites have adversely affected due to increasing the tangle and interaction with each other via van der walls force during the coprecipitation process, which increases the probability of the MWCNTs agglomeration in the nanocomposites. Decreasing in the relative density and fracture strength of Al_2O_3/Cr_2O_3-MWCNTs nanocomposites samples with increasing of the Cr_2O_3 content is attributed to increased formation of Al_2O_3-Cr_2O_3 solid solution that actually absorbs extra heat energy and requires a higher sintering temperature to achieve good densification. Fracture toughness of Al_2O_3/Cr_2O_3 nanocomposites was enhanced by increasing the carbon nanotube content.

5. References

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تصنيع متراكبات نانوية من Al_2O_3/Cr_2O_3 مدعمة بأنابيب الكاربون النانوية بأستخدام عملية الترسيب

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الخلاصة

يتضمن هذا البحث دراسة تاثير اضافة انابيب الكاربون النانوية (MWCNTs) الى متراكبات نانوية من الومينا/كروميا (Al_2O_3/Cr_2O_3). حيث تم تصنيع متراكبات نانوية من الالومينا/كروميا-انابيب الكاربون النانوية مع محتويات مختلفة من Cr_2O_3 و MWCNTs باستخدام عملية الترسيب وتليها عملية التلبيد بالبلازما. كشف تحليل حيود الاشعة السينية ببلورية جيدة لعينات المتراكب النانوي مع تكون طور واحد للمحلول الجامد من Al_2O_3/Cr_2O_3. تم قياس الكثافة، صلادة فيكرز المجهرية، متانة الكسر ومقاومة الكسر للعينات الملبدة. اظهرت النتائج ان الكثافة النظرية، الصلادة المجهرية ومقاومة الكسر تحسنت بشكل ملحوظ عند محتويات منخفضة من Cr_2O_3 و MWCNTs. زيادة محتوى MWCNTs في المتراكب النانوي له تاثير سلبي ويرجع السبب الى زيادة التشابك والتفاعل لانابيب الكاربون النانوية مع بعضها، الامر الذي ادى الى التكتل في المتراكب النانوي. في حين ان زيادة محتوى Cr_2O_3 في المتراكب النانوي ادي الى زيادة تكون طور المحلول الجامد الذي احتاج الى درجات تلبيد عالية لتحقيق التكثيف الجيد. تحسنت متانة الكسر للمتراكبات النانوية مع زيادة محتوى انابيب الكاربون النانوية.