Synthesis of Nanocomposite Material and Studying some of its Mechanical Properties

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ABSTRACT

This study aims to synthesis a nanocomposite from Al_2O_3 – Cr_2O_3 and studying some of its mechanical properties like microhardness and fracture strength.
X-Ray diffraction patterns shows a good crystallinity of the composite with very sharp and neat peaks. TEM photographs shows the good distribution of the particles which have an average particle size of (3-6 nm). The microhardness is increased with the increasing of sintering temperatures having the height value at 15% chromia while the fracture strength behaves as a bell shape having again the height value at 15% chromia.

Keyword: Nanocomposite, sol-gel, microhardness, fracture strength, TEM.

تصنيع متراكب نانوي ودراسة بعض خواصه الميكانيكية

الخلاصة

يهدف البحث الى تحضير متراكب نانوي من الألومينا والكروميا ودراسة بعض الخواص الميكانيكية مثل الصلادة المجهرية ومتانة الكسر. أظهرت فحوصات حيود الأشعة السينية ببلورية جيدة لهذا المتراكب حيث القمم الحادة والنظيفة. أما صور المجهر الألكتروني الخارق اظهر ان توزيع الحجم الحبيبي جيد ومعدل الحجم الحبيبي(٦-٣)نانومتر. أزدادت قيم الصلادة بزيادة درجة حرارة التلبيد وكانت اعلى صلادة لنسبة ١٥% من الكروميا. اما متانة الكسر ايضا ازدادت مع نسبة الكروميا وايضا نسبة ١٥% هي الأعلى ومن ثم تبدأ بالنزول.

INTRODUCTION

In ceramic materials, the trend is always to prepare finer powder for the ultimate processing and better sintering to achieve dense materials with dense fine – grained microstructure with better properties for various applications [1].

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The fineness can reach up to (1 nm- 100nm), by special processing technique. More in the fineness, more is the surface area. Therefore, the densification occurs very well at lower temperature than that of conventional ceramic system.

Nanocomposite materials are complex of nanophase materials and other materials, which optimize the performance of traditional materials. Various nanocomposites have been synthesized using a wide range of processes such as polymerization, sol-gel, deposition, sputtering, supercritical fluid and laser. Among these works, many strategies were addressed to improve nanocomposites mechanical properties by inclusions fibers, whiskers, or particles. The embedding of inclusions in a host matrix to make composites, which result in material properties not achieved by either phase alone, has been a common practice for many years. Traditionally, composites were reinforced with micron sized inclusions [2].

Grisan et al. showed that the sol-gel process allow synthesis of Al_2O_3, TiO_2, MgO, Fe_2O_3 system and have been characterized by XRD, IR-spectroscopy, differential thermal analysis (DTA), thermogravimetry (TG), and TEM [3].

Zhang et al. studied the synthesize of nano SnO_2 powder by sol-gel method. The obtained SnO_2 particles range from 2.8 to 5.1 nm in size when the gel is sintered at different temperatures. The nanocrystalline have been characterized by means of TG-DSC, FT-IR, XRD, and TEM [4].

Tok et al. prepared agglomerate nano sized Al2O3 particles with a size range of (5 – 30)nm by using the flame spray pyrolysis method. The precursors and the resultant oxide powders were characterized by chemical analysis , XRD and TEM [5].

Panasyuk et al. prepared nanocrystalls of Al_2O_3 with diameter (50- 100)nm, prepared by decomposition of supramolecular structure . The several characterization techniques such as XRD, DTA, and TG were used [6].

Gibot et al. studied highly insensitive/reactive thermite prepared from Cr_2O_3 nanoparticles. Homogeneous composites were prepared by mixing Al nanoparticles with chromium(III) oxide Cr_2O_3 micro and nanoparticles respectively in hexane solution. The morphology and microstructure of the samples were examined by XRD, SEM and TEM techniques. A value of 10nm was thus found for the nano Cr_2O_3 particles [7].

Septawendar et al. studied alumina-zircona nanocomposite that had been synthesized at room temperature and calcined at lower temperature than 1000ºC of about 800˚C by sugar precursor process. In the synthesis process of the nanocomposite powder, sugar was used as a gelling agent. The calcined powder was characterized by XRD, a particle size analysis, and TEM. The TEM results showed that the average grain sizes of the nanocomposite powder were below 50 nm in diameter [8].

Jalilpour et al, (2012) studied effect of aging time and calcination temperature on the cerium oxide nanoparticles synthesis via reverse co-precipitation method. The synthesized nanopowder was investigated by XRD, FT-IR, SEM and TEM. The size distribution of particles is uniform and the average crystallite size is 3 and 6nm. They show that increase in both the aging time and calcination temperature lead to appreciable grain growth in the crystallite size [9].

The aim of this study is to synthesis a nanocomposite from Al_2O_3 and Cr_2O_3 both of them have nano sized particles and studying their some mechanical properties.

EXPERIMENTAL PART

Aluminum nitrate and chromium nitrate with a molar concentration of 0.5 M were weighed, these powders are dissolved each one in water – ethanol solution in the molar ratio of 50:50 and mixed for homogenization by the magnetic stirrer for 30 min or around until all the powder dissolved at room temperature for each one. Different percentages (2,6,10,15 and 20) of chromium solution nitrate have been mixed with aluminum nitrate by magnetic stirrer for 30 min. PH should be measured at this stage. Furthermore, several concentrations from the surfactant material (glucose) with percentages (3, 5, and 7)% are measured. In view of the experimental work, it was clear that 7% from glucose as surfactant is necessary to avoid agglomeration and grain growth after calcination of prepared gel as shown in table (1). This is concluded from the many experiments have been done. Many drops of ammonia are added to the mixture. The gel formation begins at PH 5 at room temperature. Then the gel is filtered to remove the excess solution . The filtered gel has been dried at temperature 80°C for 6 hours in programmed electrical oven. The gel was crushed and calcination at temperature 600°C was followed at heating rate 5°C/min for 2 hours. The sintering process of the compact samples were carried out in air atmosphere. The sintering temperatures used were 1250, 1450 and 1650°C with heating rate and cooling rate of 5°C /min.

Table (1) Preparation of composite samples.

Sample code	Aluminum nitrate (ml)	Chromium nitrate(ml)	Surfactant (ml)	NH_4OH (ml)	PH Before add	PH After add
D1	98	2	7	8	2.2	5.7
D2	94	6	7	9	2	6.2
D3	90	10	7	9	1.9	5
D4	85	15	7	8.5	1.9	5.5
D5	80	20	7	9	1.8	6

RESULTS AND DISCUSSION

Most of the researches using one of the components of the composites or the reinforced materials in the range of nanoparticle. Our work is too different than those, where both the components are in nanosized particle.

Figure (1) shows a TEM photograph of the prepared composite (15% Cr_2O_3) which appeared the good distribution of the particle which has a mean particle size of (3- 6nm).

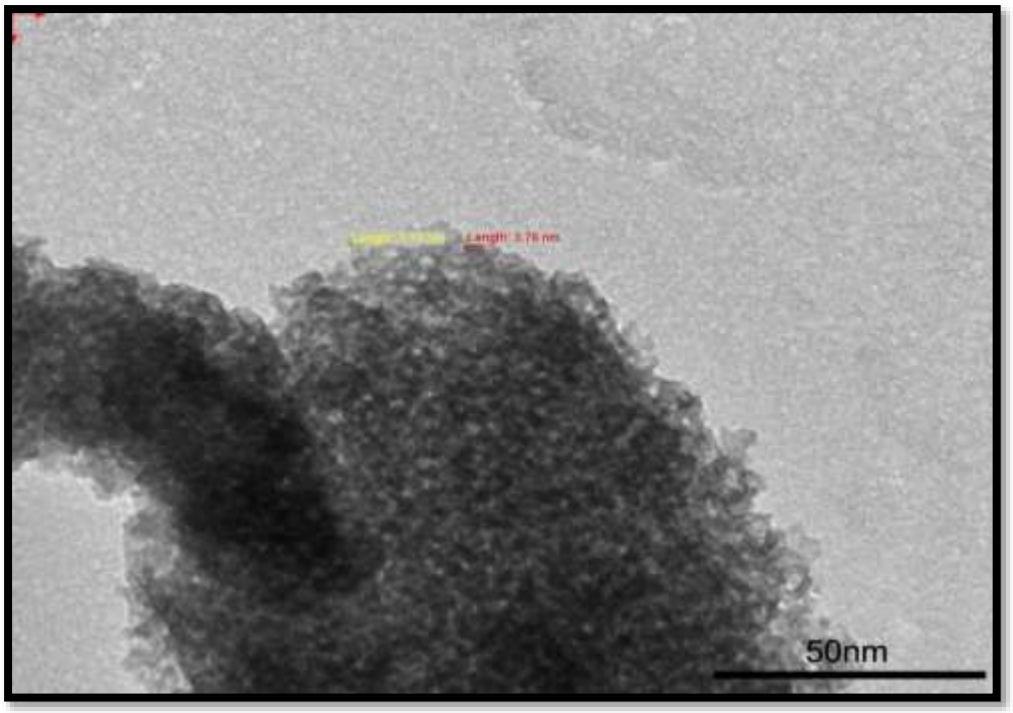

Figure (1) TEM image for prepared nanocomposite powder after calcination at 600℃ for 2 hrs and with 15% Cr_2O_3.

Figures (2-4) show the XRD pattern of sintered samples of nanocomposite using a 15% of chromia for different sintering temperatures (1250,1450, and 1650°C) for one hour sintering time. Most of the peaks matched of α- Al_2O_3 , corresponding to JCPDS (ASTM) card No 43-1484. There are no peaks from any other phases of Al_2O_3 observed, this leads to obtaining solid solution of Al_2O_3-Cr_2O_3. Furthermore, the diffraction peaks of sintered samples show sharp peak intensities and clean profiles, indicating that the obtained nano composite samples Al_2O_3-Cr_2O_3 after sintering process have high crystallinity. As shown in fig.(1) the highest intensity peak was for (104) plane when the sintering temperature was 1250°C, but at increasing temperature to 1450 and 1650°C the maximum intensity peak of (116) plane, showing that a slight shifting in the diffraction angle leading a slight decrease in the d-spacingof the materials.

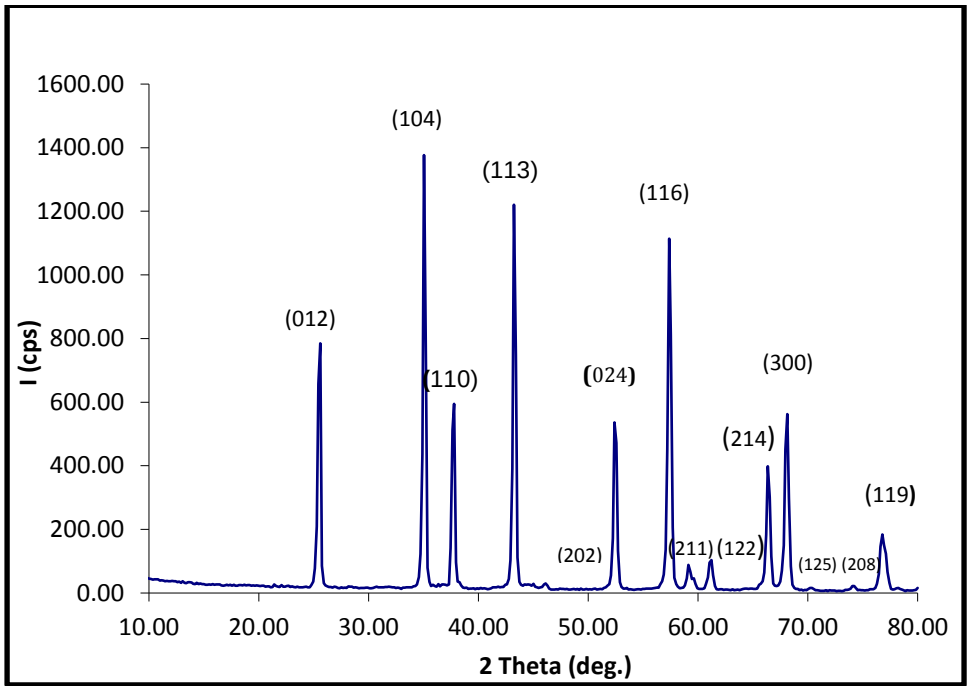

Figure (2) XRD patterns for sintered sample of alumina -15% chromia at 1250℃ for 1hr.

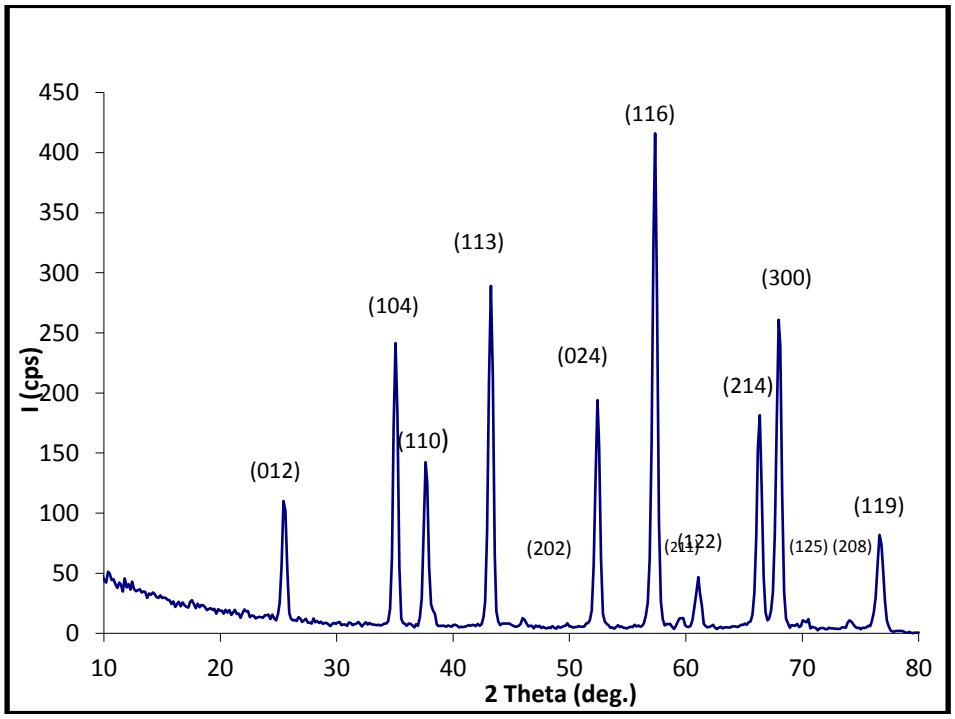

Figure (3) XRD patterns for sintered sample of alumina – 15% chromia at 1450℃ for 1hr.

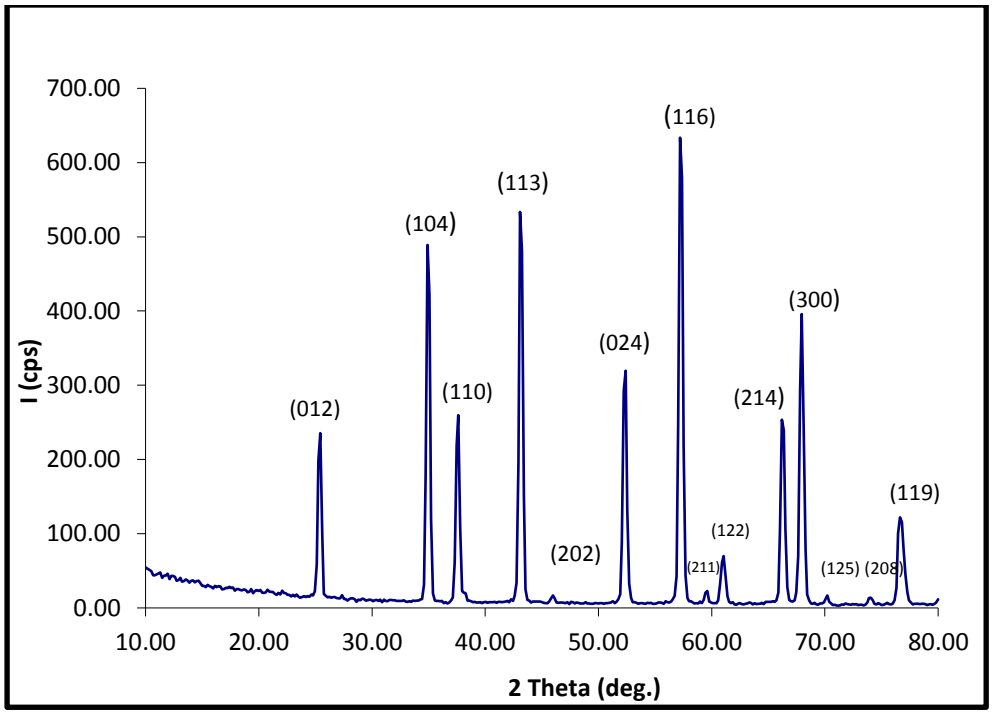

Figure (4) XRD patterns for sintered sample of alumina – 15% chromia at 1650℃ for 1hr.

Figure (5) presents the effect of sintering temperature on the Vickers microhardness of the composite at different percentages of Cr_2O_3. It is cleared from the figure increasing of sintering temperature the hardness is increased and sharply increased after 1450℃. 15% Cr_2O_3 has the highest value of hardness that may be due to the reduction in porosity at this temperature, where the particles of Cr_2O_3 filled most of the pores and have good densification.

It was difficult to calculate the fracture toughness of the samples by using indentation technique because there is not any crack shown on the indent, by using 1Kg as a load which the identical load for microhardness.

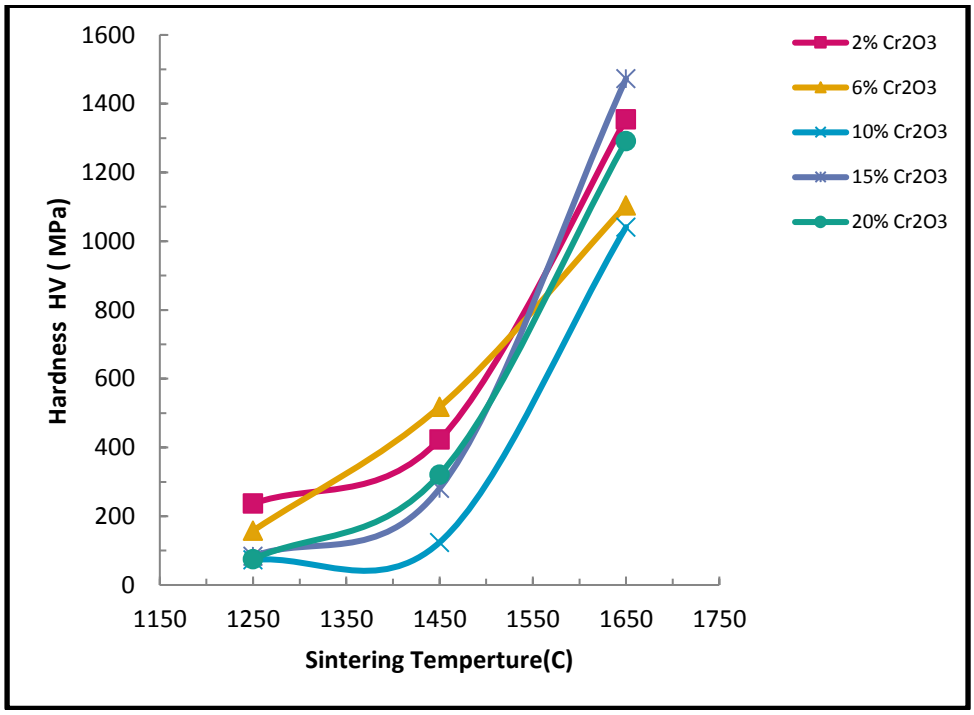

Figure (5) Vickers microhardeness for the sintered Al_2O_3- %Cr_2O_3 compacts versus sintering temperature.

Figure (6) shows the effect of Cr_2O_3 percentages on the fracture strength of the composite. A bell shape has been found from this relation having the maximum strength at 15% Cr_2O_3 which is the same behavior of the effect on microhardness.

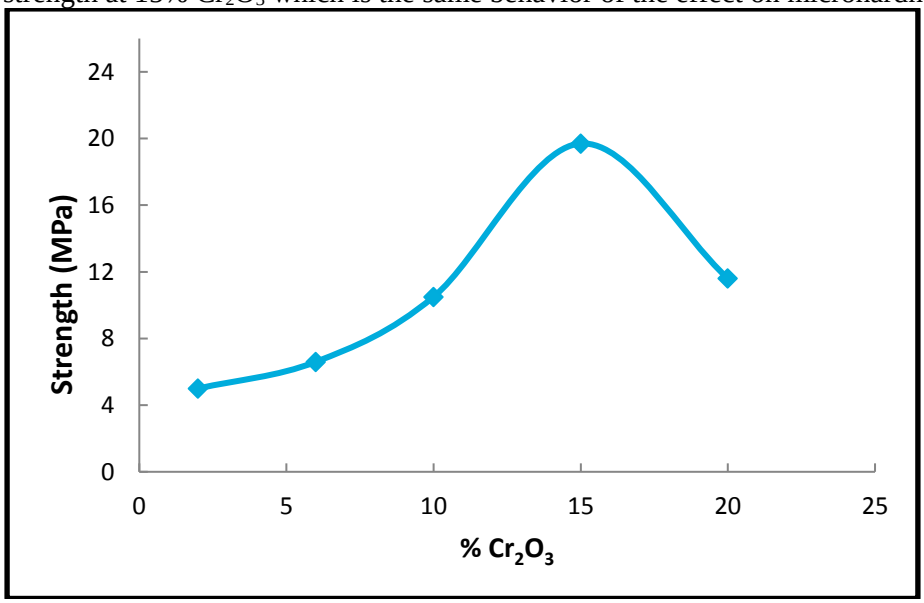

Figure (6) The Tensile strength versus percentages of chromia at 1650℃ for 1 hour.

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