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EFFECT OF α-Al_2O_3 NANOPARTICLES ADDITION ON SOME PROPERTIES OF CoCrMo ALLOY FABRICATED BY POWDER METALLURGY ROUTE

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ABSTRACT

CoCrMo alloys are used as biomaterial due to their good corrosion and wear resistance, with reasonable biocompatibility. The present study aims to investigate the effect of (1, 3 and 5) wt% α-Al_2O_3 nanoparticle additions on some properties of pre-alloyed CoCrMo when fabricated by powder metallurgy route. The density, porosity, microhardness and microstructure of the fabricated samples were examined. The results revealed that the addition of α-Al_2O_3 nanoparticle has a distinguished effect on the density of sintered CoCrMo alloy samples, where continues increase in the porosity was happened and reached to its maximum value at the higher addition amount. While the density showed an inverse trend to that of the porosity with the increasing of the nanoparticle addition. The microhardness was decreased as the amount of the nanoparticle addition to CoCrMo alloy was increased. The microstructure observation by SEM and SEM-Mapping is revealed the partial isolation of CoCrMo particles by the nanoparticle additions and it was associated with the formation of passive Cr_2O_3 oxide. In turn the XRD analysis depicted the co-existence of γ-Co and ε-Co phases besides, the Cr_2O_3 oxide. Although the increased porosity with the nanoparticle addition but, this will perhaps, increase the tissue ingrowth when the fabricated nanocomposites were utilized as biomaterial.

Keywords: Al_2O_3 nanoparticles; CoCrMo alloy; powder metallurgy; XRD; microstructure

تاثير اضافة الدقائق النانوية من الالومينا على بعض الخواص لسبيكة كوبالت- كروم- مولبدنيوم المصنعة بطريقة ميتالورجيا المساحيق

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الخلاصة

تستعمل سبائك CoCrMo كمواد احيائية بسبب مقاومتها الجيدة للتاكل والبلى والتوافقية الجيدة. ويهدف البحث الحالي الى تاثير اضافة الدقائق النانوية من الالومينا (1و3 و5)% نسبة وزنية على بعض الخواص لسبيكة كوبالت- كروم- مولبدنيوم المصنعة بطريقة ميتالورجيا المساحيق. وقد تم قياس المسامية, الكثافة , الصلادة وفحص البنية المجهرية للنماذج المصنعة. وقد اظهرت النتائج ان اضافة دقائق الالومينا النانوية لها تاثير واضح على الكثافة للنماذج الملبدة , حيث حصلت زيادة مستمرة في المسامية وقد وصلت الى القيمة القصوى عند اضافة 5% الومينا. بينما ابدت الكثافة سلوك مغاير للمسامية مع زيادة نسبة الالومينا. وقد انخفضت قيمة الصلادة مع زيادة نسبة الالومينا المضافة الى السبيكة. وقد اظهرت الصور المجهرية الماخوذة بالمجهر الماسح مع توزيع العناصربالمجهرالماسح (SEM-Mapping) حصول انعزال جزئي لدقائق سبيكة الكوبالت- كروم- مولبدنيوم باضافة دقائق الالومينا مع تكوين طبقة اوكسيدية واقية من اوكسيد الكروم (Cr_2O_3). وقد اظهرت نتائج التحليل XRD وجود الاطوار γ-Co و ε-Co و Cr_2O_3 وعلى الرغم من زيادة المسامية مع زيادة نسبة الالومينا ولكن هذا سوف يساعد على نمو نسيج الجسم مع المواد المركبة النانوية المصنعة كمواد احيائية.
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لكلمات المفتاحية : دقائق الالومينا النانوية , سبيكة كوبالت- كروم – مولبدنيوم , ميتالورجيا المساحيق , البنية المجهرية

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INTRODUCTION

Because of their good bulk mechanical properties, like as their strength and wear resistance combined with the good corrosion resistance, therefore, CoCrMo alloys (CCMAs) utilized in the industrial applications, such as bearings, engine components, wind and gas turbines and aero-engine, and nuclear systems [Jéferson et al.,2018; Hainol et al.,2017; Eleonora et al.,2013]. Among all implant materials, CCMAs establish the most valuable balance in fatigue, strength, and wear resistance along with resistance to corrosion [Abdull Hamid et al.,2016]. CCMAs were utilized at different positions in human body, to retain and improve the performance of mankind health related activities. The biomedical application were included, fabrication of orthopedic prostheses, shoulder, wear resistant coating, artificial hip and knee joints, besides the fracture fixation devices [David ,2007; Raudhah ,2010; Ferdiansyah Mahyudin &Hendra Hermawan ,2016; Chonlawit ,2018]. Products made from CCMAs are able to manufacture by casting, hot forming and by PMR [Oksiutaa et al.,2009]. In order to further improvement of CCMAs properties several researchers were devoted their works to study the effect of some elemental metallic additions. Gongjun et al.,2016 studied the effect of (Ag) addition on the wear behavior of self-lubricating Co-matrix alloys at high temperature prepared by P/M. In turn Haydar et al., 2016 studied the properties of CoCrMo (F75) when it was doped by yttrium. While Ali et al.,2016 studied the properties of CoCrMo Alloys (F75) when doped by Ge. Other interested researchers' attempts were devoted toward the micron-sized particulate ceramic additions effects on the properties of CCMAs. Rohaya et al.,2016 referred to the microhardness was increased about 25% when 5% bio glass (BG) was added. Oksiutaa et al.,2009 were referred to that 10 wt% of BG remarkably increases the mechanical properties as well as the corrosion resistance of the porous CCMA. Grqadzka et al.,2008 informed that the improvement of composite tribological properties can be realized using different hard, wear resistant particles, such as (e.g. Al_2O_3, B_4C, Si_3N_4, SiO_2) to reinforce the metallic matrix. Peng et al.,2013 learned that the nanoparticles (NPs) have been considered an attractive family of materials over the last decades due to their novel properties that are not present in the bulk. The unique properties of NPs result from the very little sizes and huge specific areas. Tok et al.,2006 stated that the aluminum oxide (alumina, Al_2O_3) is currently one of the most useful oxide ceramics, as it has been used in many fields of engineering such as coatings, heat-resistant materials, abrasive grains, cutting materials and advanced ceramics. This is because alumina is hard, highly resistant towards bases and acids, allows very high temperature applications and has excellent wear resistance. Peyman et al.,2018 were made an interesting review about aluminum oxide nanoparticles (AlNPs). They were explained the importance of AlNPs from the broader its utilization particularly, in biomedical applications. It was utilized for drug delivery, bio-sensing, therapy, immunotherapy, ant-microbial effects …etc. Muna & Jawdat,2019 studied the wear behavior and mechanism of CoCrMo alloy which fabricated by powder metallurgy route compacted under (1000MPa) and sintered at (1200ºC) in the argon atmosphere. Wear test type pin-on-disc was performed under dry sliding condition at four applied loads (10,15,20 and 25) N were applied at fixed sliding speed and time (15 min). The results showed that the wear rate increased by increasing the applied load and a transition from mild wear to severe wear was occurred at (20 N) for (15min). Muna & Jawdat,2019 studied the corrosion behavior and mechanism of Co-Cr-Mo alloy fabricated by powder metallurgy route in Ringer's solution with pH7.4 and temperature of 37ºC by using a potentiostat to obtain the potentiodynamic and cyclic polarization to measure corrosion rate and determine some corrosion parameters of the alloy. The results showed that the alloy is indicated the resistance to the pitting and crevice corrosions. While the optical and SEM images revealed that the corrosion of the tested alloy was due to interparticle. For about two years of search in World Wide Web, the

observation was the lack to works about nanoparticles addition effects on the properties of CCMAs. This is was one of the motivations to perform the current research. Therefore, the aim was dedicated toward study the effects of nano-alumina (α-type) on some properties of CCMA fabricated by powder metallurgy route.

EXPERIMENTAL PROCEDURES

Spherical shape pre-alloyed CCMA powder with 4.88 gm/cm^3 tap density (As listed in the manufacturer certificate data sheet.) was used. It was supplied by China Jingan Chemicals & Alloy Limited Company. Table(1) shows the chemical composition of as received powder and XRF technique analyses which performed by Unique-Tech company-Turkey using Bruker S8 Tiger XRF spectrometer. Also, (C and S) elements contents were detected by NCS C-S analyses instrument according ASTM E1019 C-S which was made by Metal Quality Control Laboratory/Istanbul-Turkey. Particle size distribution (PSD) of the CCMA was examined also by Uniqe-Tech company-Turkey using Malvern laser particles size analyzer instrument. Nano-Al_2O_3 powder also, exported from the same above denoted Chinese company, with a purity of 99.99% as stated in the manufacturer data sheet. PSD for nano-Al_2O_3 powder was analyzed using NanoBrook 90Plus Particle Size Analyzer instrument / USA. Ethanol was used as dispersant. The test was done at Nano Technology Research Centre-University of Technology- Baghdad. A sensitive accurate electronic balance type OHAUS-model 250g-USA with (0.1 mg) accuracy was used to weight (97 gm) of CCMA powder that was dried at (120 °C) for half an hour before mixing with (3 gm) of stearic acid powder which was added as lubricant to each charge. Mixing and milling of CCMA and stearic acid powders charge combination were carried out using rotating ball mill with 304SS container. Alloy steel balls with (11 mm) in diameter were utilized (with HV 855) value measured by Metkon microhardness tester with 500 gm applied load in Mechanical Engineering College Laboratory-Tikrit University. The mixture charge and balls were introduced with a weight ratio of (1:10) [Nagesha et al.,2013] respectively into the container, and then the charge was milled for (8 hrs.) at (175 rpm). Thereafter, second group (B) of powders mixtures were prepared. The group is consisted of three subgroups (B1, B2 and B3) for which (1, 3 and 5 wt% nano-Al_2O_3) was added, respectively and independently to the main CCMA charge to prepare the required nano-composites. Also 3% stearic acid was added to each nanocomposite charge (B1, B2 and B3). Then, they were milled with the same previously described procedure. Acetone was added to the nano-composite mixtures to prevent the nano-powder segregation in the milling process. Uniaxial hydraulic press type KWP 80 M-Knuth-Germany was used for cold pressing step. UIY8 Intelligent pressure gauge with the accuracy of (0.1 bar = 13.333 Kg) was used to control the applied load. Each sample was double pressed. The applied pressing pressure was 1000 MPa. The green compacts (GCs) were sintered in electric resistance furnace type CARBOLITE-UK in a continuously supplied argon gas stream from the commencement of the sintering process until room temperature. Sintering was performed firstly by heating the GCs to stearic acid de-binding temperature which was 500^{o}C for 2 hrs [Haydar et al.,2018], then heated and soaked for another 2 hrs at 1200^{o}C. The heating rate was fixed at 10^{o}C/min. The furnace switched off and the samples left to slowly cool inside of it. The parallel faces of the cylindrical samples were wet ground using silicon carbide emery papers with (240, 600, 1000, 2000 and 3000) grit then polished using 5μm alumina suspension solution, then washed with distilled water and dried in laboratory oven type LHT/60-UK at 120^{o}C for half an hour. The bulk density and porosity of the sintered compacts were determined by Archimedes' principle according to (ASTM C373-88) and carried out according to the following equations (1) and (2). While theoretical density (ρTh) was calculated according to the equations (3) and (4) [Farouk et al.,2013]:

$$B.D = \frac{W_d}{W_s - W_i} \times \rho_w \quad (1)$$

$$T.P = \frac{T.D. - B.D.}{T.D.} \quad (2)$$

T.P = True porosity

T.D = $\boldsymbol{\rho}$T

$$\boldsymbol{\rho}Th = (\boldsymbol{\rho}Co \times XCo) + (\boldsymbol{\rho}Cr \times XCr) + (\boldsymbol{\rho}Mo \times XMo) + (\boldsymbol{\rho}Si \times XSi) + (\boldsymbol{\rho}Mn \times XMn) \quad (3)$$

Where:
ρTh: Theoretical density (g/cm^3)
ρCo, ρCr, ρMo, ρSi and ρMn: Theoretical density for (Co ,Cr, Mo, Si and Mn) (g/cm^3), respectively.
XCo, XCr, XMo, XSi and XMn: %wt of (Co, Cr, Mo, Si and Mn) respectively.

All the procedures for the preparation of sintered compacts were performed at Workshop and Metallurgy Laboratory/ Technical College of Kirkuk/ Northern Technical University. Vickers micro-hardness for sintered compacts (SCs) was measured with the same above method used for milling balls. Optical microscopy using invert microscope type OPTIKA-Italy, SEM-EDS and SEM-Mapping using TESCAN instrument at Production Engineering and Metallurgy/University of Technology/Baghdad. Also, XRD was performed by lab ***X***-XRD-6000 Shimadzu-Japan in Ibn-Al Haitham College of Education for Pure Science/Baghdad University. The above techniques were used in the characterization step of the used powders and phase identification analysis.

RESULTS AND DISCUSSION

The optical microscope image of CCMA powder before the milling process displays in the Figure (1) indicates the spherical shape of the powder particles with size approximately less than 50 µm. On other hand the SEM-EDS analysis of the CCMA powder shown in the Figure (2) indicates the well agreement with its result and manufacturer analysis that was referred in the Table 1. With respect to nano-Al_2O_3 powder, that was observed by SEM as depicts in the Figure (3). It reveals the approximately spherical size and the powder particles existence in cluster like arrangement. This figure also explains the particle size of the nano-powder being relatively, less than 100 nm. PSD of the CCMA result shown in Figure(4) illustrates the value of d (0.5) is 47.983 µm and the size distribution has a narrow span. While, PSD of the nano-Al_2O_3 powder shown in the Figure (5) reveals the effective powder particles diameter is 27.2 nm. Figure(6) views the XRD analysis diagram for the utilized Al_2O_3 nano-particles. The diagram is matched with (α-Al_2O_3 card number 00-010-0425) as explained in the attached table to the diagram. The milling process effect was mainly the change the spherical shape of the CCMA powder to semi disc-like shape with some what distorted powder particles surface grains. This is indicated when comprising between the SEM image of the powder before and after milling shown in Figure(7). The nano-Al_2O_3 addition amounts were based on several previous studies performed to fabricate nano-composites. The comprehensive study of Evgenii with Co-workers,2010 mentioned to the addition of different types of nanoparticles which included WC, ZrO_2, Al_2O_3, and Si_3N_4 to Fe and Co powder to fabricate Fe- and Co-based alloys by powder technology. The nano additions amount does not exceed 6 wt%. The applied compacting pressure was determined according to the higher measured green density which was clarified in else where [Jawdat &Muna ,2019]. Wilson et al.,2011 applied compacting pressure reached to 1000 MPa to cold press the water atomized spherical

CoCrMo powders to fabricate the test samples. In turn Barbara et al. 1999 referred to the application of the cold compaction pressure as much as 1235 MPa to cold compact the utilized CoCrMo powders mixtures. The value of density of the sintered CCMA was decreased by increasing the α-AL_2O_3 nano-particle addition as explained in the Figure(8). The relative density was decreased with the values (9.46, 12.13 and 15.74)% when the nanoparticle addition was increased with amounts (1, 3 and 5) wt% respectively. This is really attributed to two reasons. The first is the lower density of Al_2O_3 arranged between (3.99) gm/cm^3 [Pertti ,1996]. The second is the increasing of the calculated total porosity of the sintered samples by increasing Al_2O_3 addition, as illustrated in the Figure (9). The 5 wt% Al_2O_3 made the density decreases from 7.16 to 6.03 gm/cm^3 which mean 15.782 % decreases. In turn the porosity increased with the increase of nano alumina addition amount can be due to several reasons. The first is that the isolation function of the nanoparticles that detached CCMA powder particles from each other was increased with the nanoparticle percentage increease as shown in Figure(10). This action partialy prevented the necking formation between the CCMA particles. The alumina nanoparticles that equipid resnable volume fraction between the CCMA as well as the high surface area that the nanoparticles have were reflected as the formation of a high fraction of nano sized pores between them. The second is based on the fact that the nano addition being ceramic material hence, the nanoparticles were packed mechanicaly with each other without diffusion occurance between them at 1200^oC sintering temperature. Accordingly pores between them will remain and the only factor may minimize its amount will depend on the value of the used cold compacting pressure. The change of the alumina nanoparticles distribution from point view of the increase of the area fraction that it equipys was measured by (Image-J) software program. Figure (11) shows the increase of the area percentage from (16.857 to 39.669)% when the nano addition percentage was increased from (1 to 5) wt%. The proccessed samples microstructure by Image-J program anfurtunatly was unable to distingushe the pores from chromum oxide and distributed alumina nanoparticle. But it gave a general idea and aproximate estimation of the variation in the microstructure with the variation of the nanoaddition amount. Also researchers such as Rohaya et al. , 2016 referred to the increase of the porosity of CoCrMo alloy with the addition of bio-glass powder when the samples were fabricated by powder metallurgy raoute. Abdul Raheem et al.2017 also observed the increase of porosity of when α-alumina with (1.439 μm) particle size addition was increased at 650 MPa compaction pressure. Evgenii et al. [20] stated that increase of α-Al_2O_3 nanoparticle form (0.92 to 3.3) wt% tend to increase the porosity% from (5.8 to 16.1%) for cobalt samples prepared by hot-pressing. The addition of α-AL_2O_3 nano-particles contributed to decreasing the sintered CCMA microhardness, as shown in the Figure (12). This decrease is related to the increased porosity amount of the composite material group-B (CCMA + nano α-AL_2O_3), as mentioned formerly. The same case was observed by Gopalu et al.,2017. They observed the hardness drop of the Fe + 0.5% C alloy samples fabricated by PMR when 0.5% nano Al_2O_3 was added. Also, they interpreted that the hardness drop is due to increase the porosity when nano α-AL_2O_3 was added. Also Evgenii et al.,2010 reported that the addition of 3.3 wt% nano-Al_2O_3 powder to fine Co powder led to the decrease of the Brinell hardness from 105 to 97 when the composite was fabricated by hot pressing method. From the observation of the Figure (12), some of the answers one may ask about the variation of density and microhadness of the composite materials formed from the independidtly addition of α-Al_2O_3 nanoparticle to the base CCMA compactes. Figure(13-A) illustrates how α-Al_2O_3 addition greatly contributed to increase the porosity of the materail which also reflected in as roughning appearance of the topography at the microstructure scale, although application of approximatly high compacting pressure (1000 MPa). It is difficult to explain the situation of hardness dropness. But, the logical thought here can be that the action of nano-Al_2O_3 was, such as a fluid bed yielded itself up to

the motion under the action of the applied prussure and behaved some what as spongy material when the samples was tested to measure its hardness. The inertness of the ceramic nano-particles makes it unable to combine chemically with the CCMA paricles. Hence, the broader surface area of the composite suffered from the problem of yielding to the motion due to the applied load. The pores size in some postions are larger than (20µm) as shown in the Figure(10-A). While the magnified image in part-B from the Figure(13-B)shows the size of the pores at particles boundaries which is somewhat less than (5 µm). Also, it is important to observe the sirrated edges of CCMA particles that appeared in the Figure(13-B) which indicates the exposure of the material to brittle fracture due to detachment of amount of the exsited material from these positions when grinding process was done leaving behined them cavities which are strongly contributed in the drop of the microhardness measured value. It is so important to reveal with more accuratly how the added nano-particles were distributed in the microstructure of the base CCMA, because it aids to undurstand the sitiuation of the microstructure of the examined cromposites. Proceeding from this precept, the elemental distribution of the fabricated composites were examined by SEM-Mapping technique facility. Figure (14) demonstrates the mapped area of group-B2 composite material. The main phenomenon that faces the obsever when looking at the map of elements distribution in this figure is the depletion of cobalt from particles boundaries at the same time of the inverse appearance of chromium element which is increasingly concentrated at the bounderies, while the molybdenum element disrtruibution simulated that of Co, but taking into the conisideration the lowest content of Mo.The migration of Cr element toward the particles, bounderies as shown in the figure has been happen in greater trend than the case of CCMA samples free from nano-particles. The reason of this behaviour comes from the idea that the only varied thing here is the addition of α-Al_2O_3 nano-particles to the alloy. The nano addition is ristricted mainly at particle bounderies and with lower amount within the particles as illustrated in the Figure(14) from the purple colored part from mapping image) and associated with the blue color image which refers to the oxygen distribution. Hence, the α-Al_2O_3 nano-particles played as a porosity source, in which the oxygen gas perhaps trapped and then played as a driving force for the chromium atoms migration to form Cr_2O_3 at these sites %). Bhairav et al., 2010 reveald the formation of Cr_2O_3 when they prepared CoCrMo alloy at temperatures arranged between (1050-1175 ^oC) in a furnace under vacuum (5 Pa). In turn Karsh et al.,2011 also referred to the formation of the oxide when the samples produced from mixed and pressed elemental Co, Cr and Mo were heated between (800-950 ^oC) under argon atmosphere. Perhapes, the mapped area does not represent the over all surface aera of the examined sample. The part-C in the Figure(10) as example covers a larger area than mapped one illustrated in Figure(14). Thus it reveals the difference in the width of the alumina nanoparticle and Cr_2O_3 distribution layer btween CCMA particles from position to other one that is at the same nano addition percentage (5 wt%). Hence the amount of oxygen will be lower at those positions.Figure (15) shows the XRD analysis made for group-B3 which contains relatively a high amount of α-Al_2O_3 nano-addition, which perhaps able to detect it by this analysis more easier than other two subgroups (B1 and B2). The diagram reveals the presence of the three main phases, that is Co-ε, Co-γ and Cr_2O_3. The added nano α-Al_2O_3 was also detected, where its peaks were denoted by numbers (4) and (5). Besides, the only remained peak, which is pointed out by number (6), is identifed by (00-021-0869 ICDD) card that refers to $CoMoO_3$ oxide. This compound is formed due to high cobalt element content in the base alloy which in turn, facilitated the formation of it at high temperature. Manman et al.,2014 proved that the $CoMoO_3$ phase formation occurs when the ratio of Co to Mo in molar equal to (0.7) perhaps, at the position of the formed phase.

CONCLUSIONS

1- The nanocomposites are successfully made from CCMA with different α-Al_2O_3 nanoparticle additions by conventional powder metallurgy route.
2- The hardness drops with the increasing of nanoparticle addition particularly, at 5 wt% α-Al_2O_3 make the choice to be focused in the future on the lower amount of the nanoparticle addition.
3- The porosity increases due to nanoparticle addition will enhance the utilizing of the nanocomposite in biomedical applications, such as bone fixation.
4- Although α-Al_2O_3 nanoparticle partially isolated the CCMA particles from each other but, it does not prevent the formation of Cr_2O_3 passive oxide amongst the particles.

Table(I): Chemical composition analysis of the CCMA powder used as compared with the manufacturer specification.

A: Manufacturer specification B: XRF analysis result C: C-S analysis

Element	Co	Cr	Mo	Si	Mn	O	C	S
A	Bal.	28.52	5.95	-	-	0.035	-	-
B	62.97	29.2	6.612	0.38	0.319	-	-	-
C	-	-	-	-	-	-	0.036	0.0071

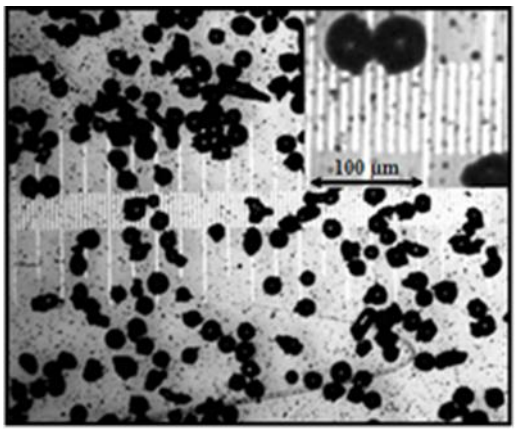


Fig.(1): Optical micrograph for CCMA powder

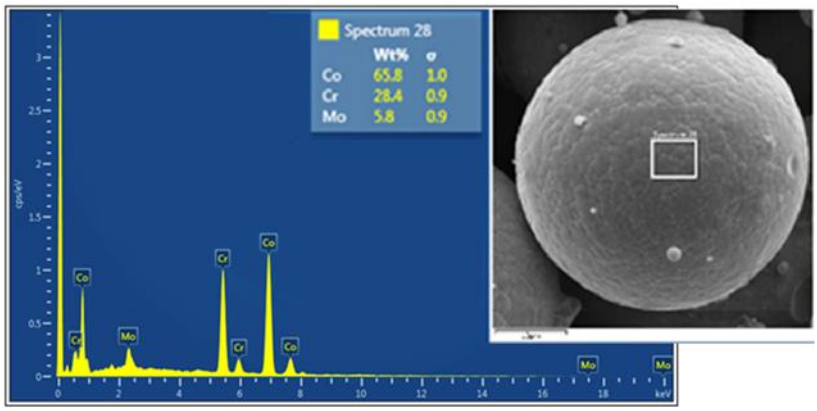


Fig.(2): SEM - EDS analysis for CCMA alloy

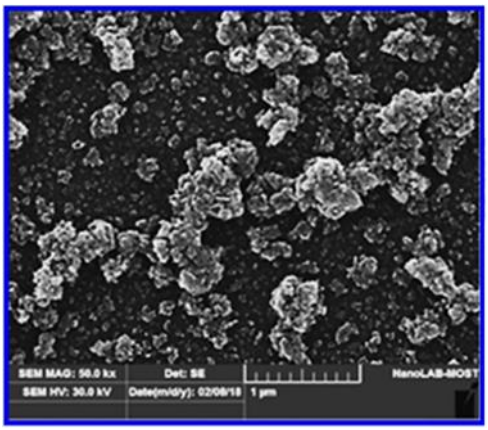


Fig.(3): SEM image for α-Al_2O_3 nano-powders

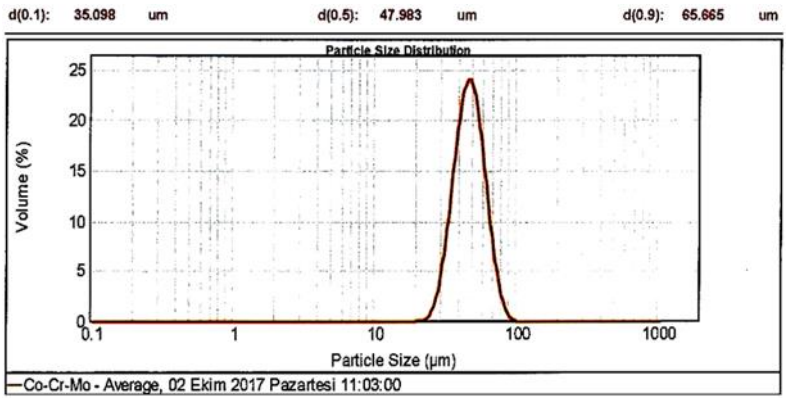


Fig.(4): Particle size distribution result diagram for CCMA alloy powder.

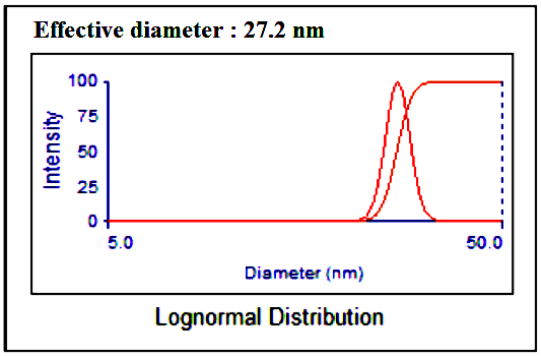
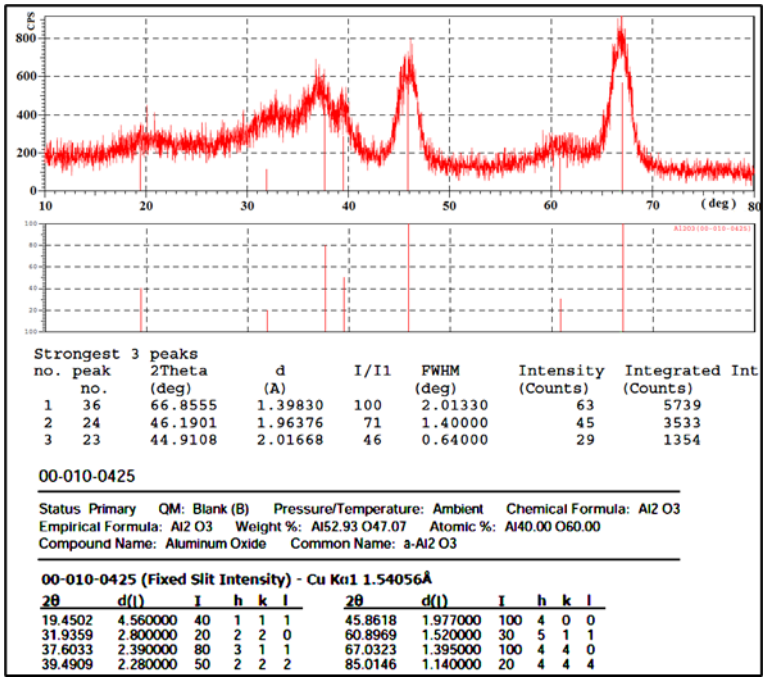


Fig.(5): Particle size distribution result diagram for Al_2O_3 nano-powder



Strongest 3 peaks

no. peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
1 36	66.8555	1.39830	100	2.01330	63	5739
2 24	46.1901	1.96376	71	1.40000	45	3533
3 23	44.9108	2.01668	46	0.64000	29	1354

00-010-0425

Status Primary QM: Blank (B) Pressure/Temperature: Ambient Chemical Formula: Al2 O3
Empirical Formula: Al2 O3 Weight %: Al52.93 O47.07 Atomic %: Al40.00 O60.00
Compound Name: Aluminum Oxide Common Name: a-Al2 O3

00-010-0425 (Fixed Slit Intensity) - Cu Kα1 1.54056Å

2θ	d(I)	I	h	k	l	2θ	d(I)	I	h	k	l
19.4502	4.560000	40	1	1	1	45.8618	1.977000	100	4	0	0
31.9359	2.800000	20	2	2	0	60.8969	1.520000	30	5	1	1
37.6033	2.390000	80	3	1	1	67.0323	1.395000	100	4	4	0
39.4909	2.280000	50	2	2	2	85.0146	1.140000	20	4	4	4

Fig.(6): XRD analysis result for α-Al_2O_3 nano-particles

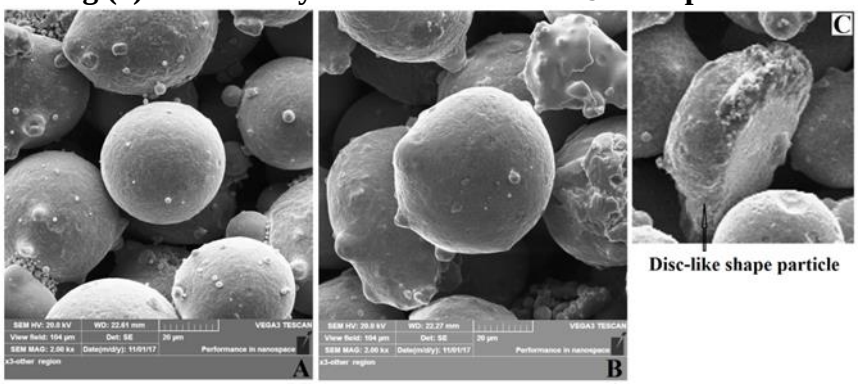


Fig.(7): CCMA powder particles shape, A: before milling , B and C: after milling

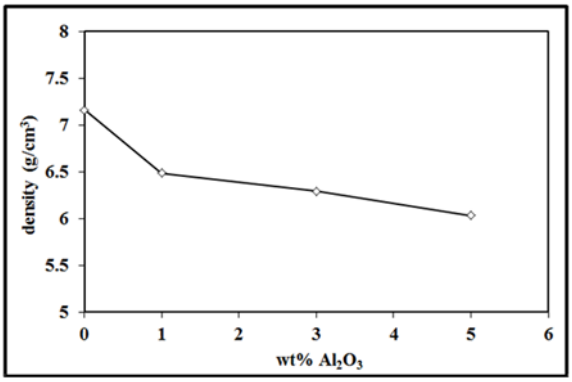


Fig.(8): Variation of density of sintered CCMA at 1200°C for 2 hrs due to α-Al_2O_3

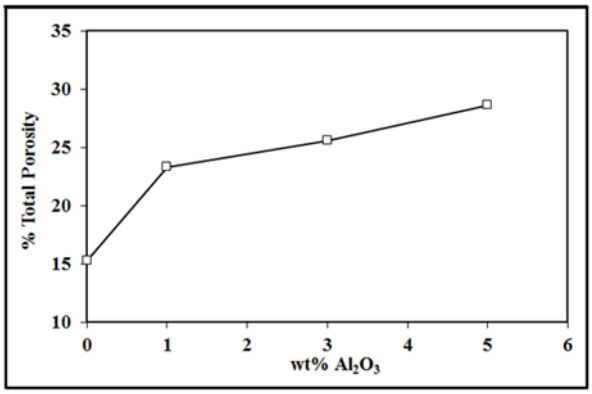


Fig.(9): Variation of total porosity of sintered CCMA at 1200ºC for 2 hrs due to α-Al_2O_3

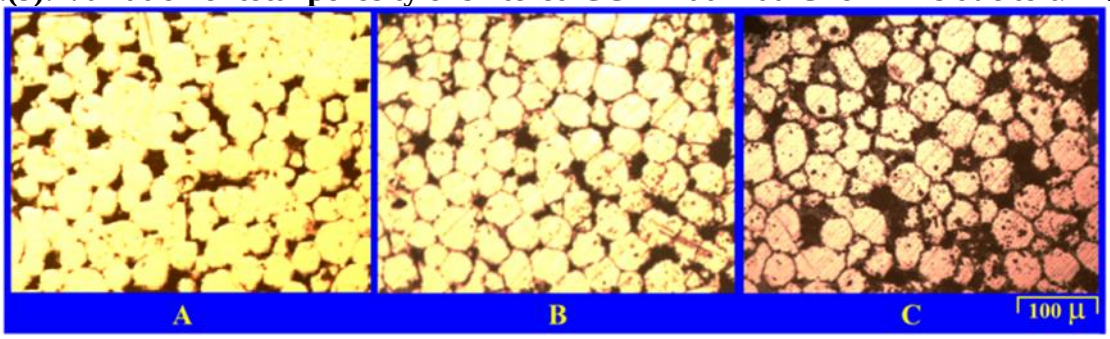


Fig.(10): Variation of the microstructure of CCMA with different α-Al_2O_3 nano addition, A: 1 wt%, B: 3 wt% and C: 5 wt%

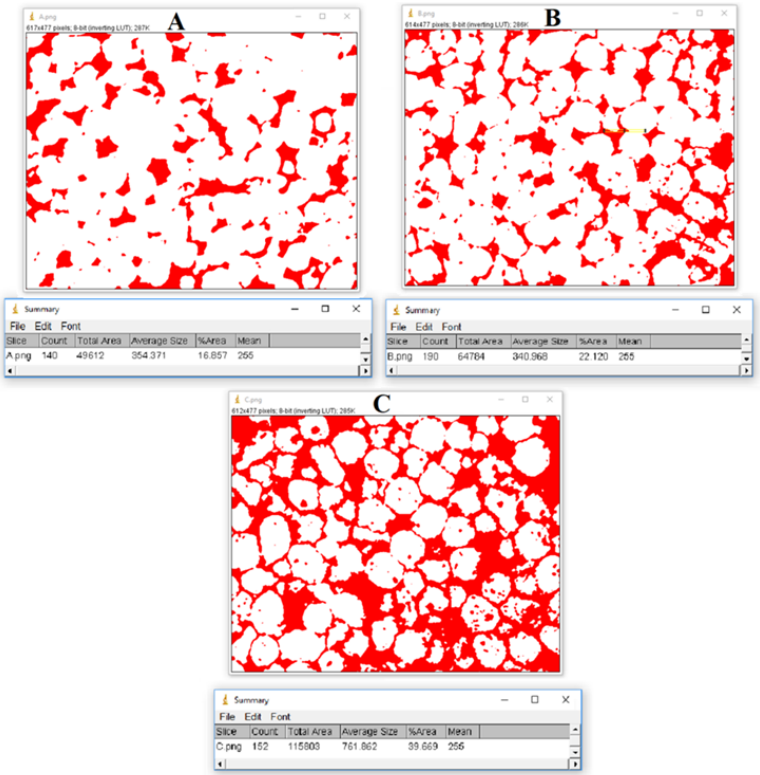


Fig.(11): Variation of area percentage of porosity, Cr_2O_3 and Al_2O_3 nano addition all together at CCMA particles boundaries with A: 1 wt%, B: 3 wt% and C: 5 wt% Al_2O_3 nano addition

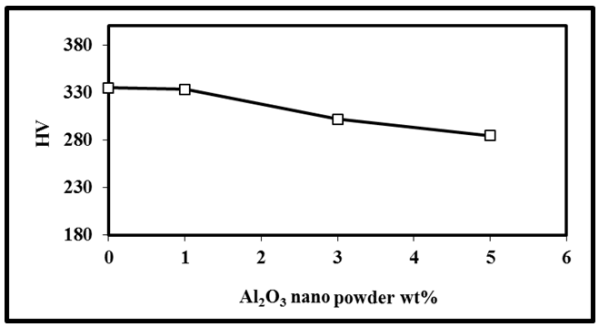


Fig.(12): Effect of α-Al2O3 addition on the microhardness of sintered CCMA

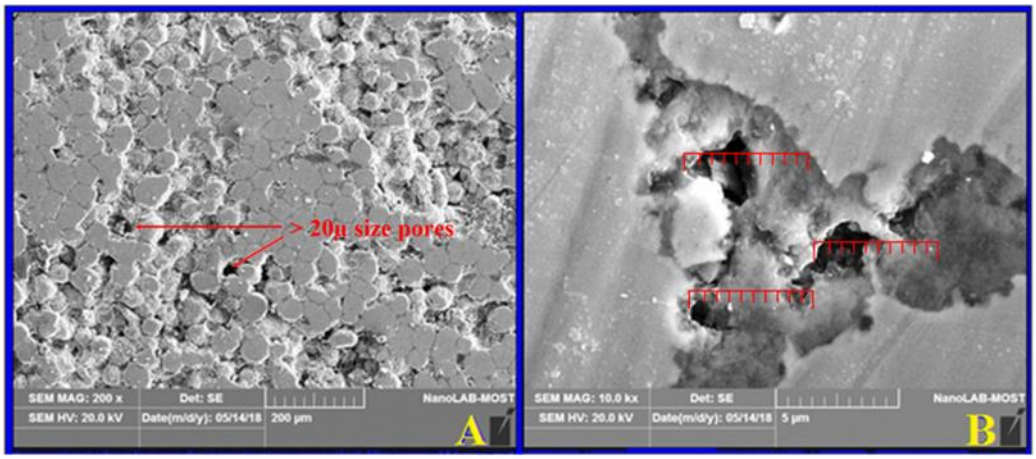


Fig.(13): Effect of 5 wt%Al_2O_3 nano-particle additions on the microstructure of sintered CCMA

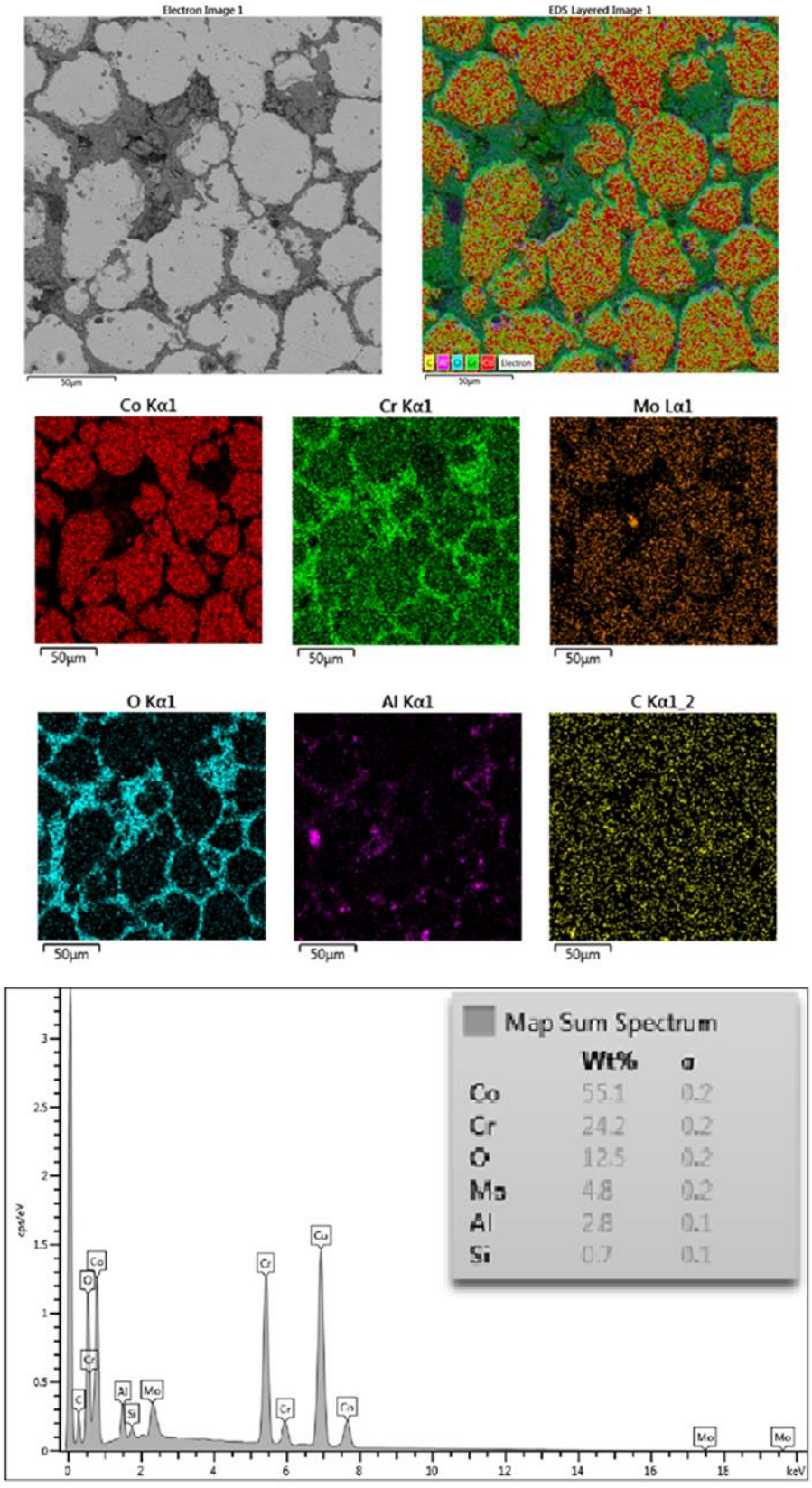


Fig.(14): SEM-Mapping profile and map sum spectrum for sintered group-B2

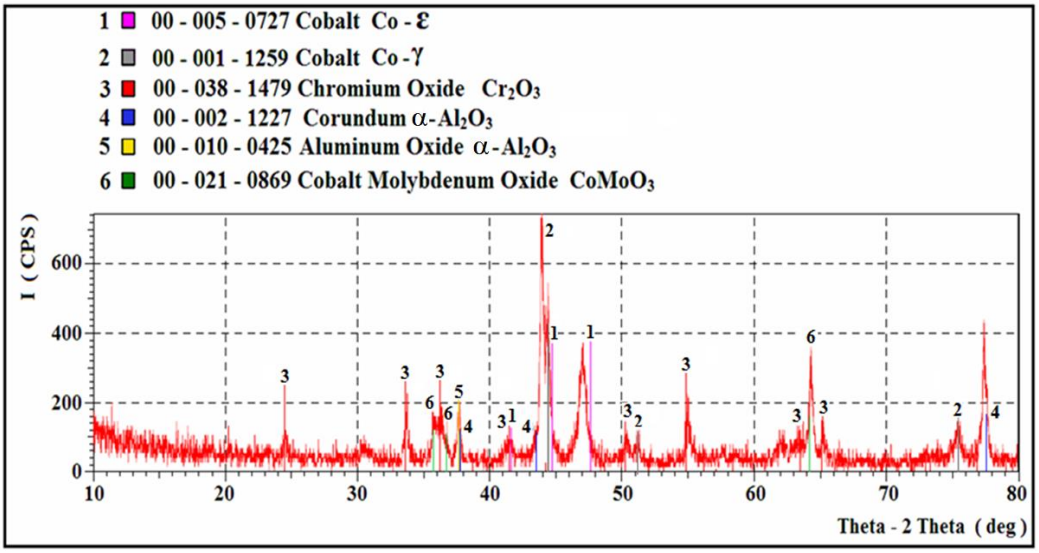


Fig.(15): XRD analysis result for group-B3 sintered sample at 1200 ºC for 2 hrs

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