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# STUDYING PROPERTIES OF AL-12WT%SI ALLOY REINFORCED WITH $CeO_2$ NANO POWDERS PREPARED BY POWDER METALLURGY

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**ABSTRACT:**

In this paper, Aluminum – 12wt% Silicon alloys reinforced by cerium oxide ($CeO_2$) nano powder with a different percentage (1, 2, and 3 wt.%) were prepared by powder technology method. Wear, porosity, apparent density tests were conducted for composite materials and reference alloy. The results showed that there is a slight change in the density and increases in the porosity of nanocomposite materials compared to reference alloy. The wear rate decreases with the increase in the proportion of the reinforced particles for the reference alloy as well as the wear rate increases with increased applied load.

**KEYWORDS : Al-Si Alloy, Cerium Oxide, Powder Technology, Wear test.**

# دراسة خواص سبيكة Al-12%Si المقواة بمسحوق نانوي من$CeO_2$ والمحضرة بطريقة ميتالورجيا المساحيق

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**الخلاصة:**

في هذا البحث تم تحضير مواد متراكبة ذات اساس من سبيكة المنيوم – 12% سليكون ومقواة بمسحوق نانوي من مادة سيراميكية اوكسيد السيريوم ($CeO_2$) بنسب مختلفة (1، 2، 3%) بطريقة تكنولوجيا المساحيق. تم اجراء اختبارات البلى، المسامية وكذلك الكثافة الظاهرية على المواد المتراكبة والسبيكة الاساس. اظهرت النتائج ان هنالك تغيير طفيف في الكثافة وزيادة في المسامية للمواد المتراكبة النانوية مقارنة بالسبيكة غير المقواة . اما معدل البلى فيقل مع زيادة نسبة الدقائق المقواة بالنسبة الى السبيكة غير المقواة وكذلك يزداد معدل البلى مع زيادة الحمل المسلط .

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## INTRODUCTION:

Metal Matrix Composites are being increasingly used in aerospace and automobile industries owing to their enhanced properties such as tensile strength, elastic modulus, hardness at room and elevated temperatures and wear resistance (Veeresh2011 and Dwivedi2011). Composite materials based on light metals like Aluminium, Magnesium and Zinc find applications in many industries due to their low density (Shabani2012, Srivastava2011 and Cavaliere2006). Different reinforcing materials like SiC, $TiB_2$, $Al_2O_3$, $B_4C$, zircon sand, SiCrFe and CrFeC, and cerium oxide have been used to reinforce the metal-based matrices in an attempt to improve their mechanical and wear properties (Poddar2009 and Hamid2008). Aluminium alloy-based particulate-reinforced composites with adequate strength and wear resistance have a large potential for a number of engineering applications (Madhu2012 and Son2003). Powder metallurgy is an important processing technique for MMCs, which can eliminate the segregation of the reinforcement that is typical of the ingot metallurgy process (Smagorinski1998 and O'donell2001). Wear is the progressive loss of material due to relative motion between a surface and the contacting substance or substances (Peter1997). The wear damage may be in the form of micro-cracks or localized plastic deformation (Sanchez2006) .

Many studies were concentrated on improvement of Al-Si alloy properties (Haitham2010), studied the mechanical properties of modifier Al-12% Si with different wt% Sb, reinforced by ceramic particles ($Y_2O_3$) with different wt% using vortex technique. He showed that the addition of Antimony leads to the microstructure refinement and change the silicon shape in the alloy from the flake – like or lamellar – like to fibrous – like In addition to the increasing the hardness when Sb is up to 0.3%, after that the hardness will decrease, as well as the addition of ceramic particles increase the hardness and decrease the wear rate. While (Prabhu2010) studied the effect of heat treatment on strength and abrasive wear behavior of Al 6061/SiC composite. He showed that with using composites better micro hardness, tensile strength and good wear resistance were obtained compared with Al matrix alloy. (Lee1992) Characterized wear behavior of aluminum matrix composites by the dry spindle wear test under various conditions (volume fractions of reinforcements, sliding distances and speeds). They found that wear resistance of composites was improved due to the presence of reinforcements, but with no noticeable improvements observed in the wear resistance with more than 20% addition of reinforcements.

The present work focused on evaluating and prediction the effects of the addition of various amounts of cerium oxide nano powder on wear properties of Al-Si alloy. Density and porosity are also determined in this work.

## EXPERIMENTAL WORK:

### Powders Preparation

The materials used for preparation the metal matrix nanocomposites (MMNCs) are Aluminum with average particle size 50µm and purity 99.99%, Silicon with average particle size 75µm and purity 99.98% (supplied by METCO Co. Ltd), and 75-125nm of $CeO_2$ (supplied by MTI Co. Ltd) depended on using sieving vibration to determine the practical size of powders. The powders were mixed with a percent (Al-12wt%Si) as shown in Fig. (1). Nano powder ($CeO_2$) has been added as listed in Table (1). Electric rolling mixer (ball mill) with velocity rotation 750 rpm and 15 mm diameter of alumina balls was used for mixing these powders together in order to obtain good particles distribution for 30 min (dry mixing), the percent weight of alumina ball to powders was 1:20. After that, they used manual mixing for 15min at room temperature to ensure the quality and volume homogenous of powders mixing.

**Compact Powders**

After mixing process, the powders were compacted in a cylindrical die made from tool steel (diameter =1 cm and height = 6 cm), by using a manual hydraulic press. The powders compacted by using cold press method (uniaxial) with compact pressure (175 MPa) for 30 seconds to obtained good bonding between its powders.

**Sintering of Compacts**

The sintering process for green compacts was carried out by put the compacts in a container and inserts then into the electrical furnace (0 to 1200 °C ± 2°C). The furnace contacted to digital numeric and also a gas regulator. Argon inert gas is used to prevent powder oxidation during the sintering process, and therefore, to get good diffusion bonding between powder particles. The furnace was heated to 473 °C at heating rate 15 °C/min, with pumped of gas at flow rate (1 milliliter/min), this temperature calculated according to the equation (Jartych1995):

$$T_s (K) = (T_m+273)\ (0.7 \text{ to } 0.9) \qquad (1)$$

Where:
Ts: sintering temperature.
Tm: melting temperature of Aluminum = 660 °C.

After the temperature of furnace reached to 473 °C, the samples were kept it in the furnace for 60 min, to ensure that all samples reached to the required temperature. After that, the temperature of the furnace was reduced to 150 °C at cooling rate 20 °C/min and argon gas were closed, then the furnace was switch off and the samples kept it in the furnace at room temperature. Fig. (2) show the steps of the sintering process.

**Tests of Samples:**
**Density& Porosity Testing**

Apparent density and porosity were calculated after the sintering process for samples according to ASTM B962-15 standard which is based on Archimedes principle. The Apparent density of material is given by equation (1) (Singer1979), and the porosity is given by equation (2) respectively (ASTM1986):

$$\rho = [w_1 / (w_1 - w_2)] * \rho_{Ethanol} \qquad (2)$$

$$P = [(w_3-w_1) / (w_3-w_2) * \rho_{Ethanol}] \qquad (3)$$

Where:

$\rho$: apparent density (gm/cm$^3$).
$w_1$: weight of dry sample (gm)
$w_2$: weight of suspension sample in ethanol (gm)
$w_3$: weight of wet sample (gm)

**P**: porosity %

**Wear Test:**

(Pin-On-Disk) wear apparatus under dry conditions was used in this work. The samples were prepared with dimension (diameter 1 cm and length 2 cm). Wear test carried out under the constant parameters in which rotational speed of the disk (500 rpm), the distance from the center of the sample to the center of the disk ($\boldsymbol{r}$ =7cm) and the hardness of the steel disk (35 HRC). Applied load using in the wear test are a static load 10 N, 15 N respectively. Wear rate was calculated by the following relationship (Eyre1976):

$$\textbf{Wear rate } (\boldsymbol{Wr}) = \frac{\Delta \boldsymbol{w}}{\boldsymbol{2\pi r n t}} \tag{4}$$

Where:

$\boldsymbol{Wr}$ : wear rate (gm/cm).

$\Delta\boldsymbol{w}$: weight loss (gram).

$\boldsymbol{r}$: The distance from the center of the sample to the center of the disk: $\boldsymbol{r}$: =7 cm.

$\boldsymbol{n}$: Number of cycles steel disk (500 rpm).

$\boldsymbol{t}$: Time of test = 10 min.

In this test, the flat end of specimen 10 mm in diameter and 20 mm length was fixed in chuck jaws to prevent specimens from rotation during the test. Axial load was applied to the pins against the plane surface of the rotating disc. The specimen's ends were polished with (800, 100) SiC emery paper and cleaned with acetone. The wear test was carried out at room temperatures. Each specimen was weighed before the experiment and after it by a digital balance (type Denver with max 210 gm. management system ISO 9001) having a sensitivity of 0.0001 gm. The duration of the experiment was controlled by a digital timer.

**Scanning Electron Microscope Test.**

Microstructural characterization studies were done to observe the microstructure of sample surface test. This is done by using scanning electron microscope. Characterization is done in etched conditions. Etching was done using the Keller’s reagent (1 volume part of hydrofluoric acid (48%), 1.5 volume part of hydrochloric acid, 2.5 volume parts of nitric acid and 95 volume parts of water). The samples were characterized by (Angstrom) Scanning Electron Microscope (SEM) in Nanotechnology and advanced materials research center / University of Technology.

**Microstructure Examination Test**

The microstructure of Al -12wt%Si and Al-12wt%Si reinforced with $CeO_2$ nano powder were examined by an optical microscope (100W Carl Zeiss Jane, Germany, EP. Type 2) connected to a digital camera. One surface of all specimens was initially grinding using a series of (500, 800, and 1000) waterproof SiC papers with increasing fineness. Finally, polishing was carried out on a disc polisher using diamond pastes of 1 μm particle size with polishing liquid as a cooling lubricant. Polished samples were cleaned with distilled water and alcohol. The prepared samples were etched using Keller’s solution for about 30-50s in order to reveal the microstructure with grain boundaries and finally, the polished specimens were taken for optical microscopy .

## RESULTS & DISCUSSION :-

### Apparent Density and Porosity

Table (2) shows the apparent density, porosity results of Al-12wt% Si with and without wt% $CeO_2$ nano powder additives. Alloy 1 represented the Al-12wt% Si alloy without any additives, apparent density is 2.7260, while the porosity of this alloy is 18%. The results show the addition of wt% $CeO_2$ nano powder to Al-12wt% Si alloy will decrease the apparent density with increased the nano $CeO_2$ addition; conversely, the porosity increased with increases the wt% $CeO_2$ nano powder addition. Fig. (3 and 4) shows the relationship between densities, porosity with alloy Al-12wt%Si (in different additives of wt.% $CeO_2$ nano powder). It is clear that the apparent density of the alloy samples decrease with increased addition of wt.% $CeO_2$ nano powder, while the porosity increased with increases addition of wt.% $CeO_2$ nano powder to matrix alloy (Osama2010 and Fadhil2010), the decreases in density due to pores expansion from the sintering process (different thermal expansion in compacted samples) which leads to the expansion of porous gaseous inside the compacted samples. As a result of these changes will increase the size of the pores in the compacted samples (this will lead to the expansion of most compacted) after sintering process, and this is due to the of diffusion grain, and most gaps gaseous (pores) transformed from closed gaps into open gaps and appeared as a semi-spherical form.

### Wear Rate Results:

From Table (3) and Fig. (5) shows the relationship between wear rates for the Al-12wt%Si alloy (in different additives of $CeO_2$ nano powder). It was clear that the wear rate of all the Al-12wt%Si reinforced by $CeO_2$ nano powder (1, 2, and 3% wt) were been less than the wear rate of the alloy without additives at least with the increase in the volumetric fraction of particles and this is due to the hardness increase with increasing volumetric fraction. Wear rate increase with increase applied load and decrease with increase the $CeO_2$ nanoparticles (Madhu2012 and Haitham2010. The increase in the wear rate with increase the applied load is due to increase the amount of small separate from the material (debris) particles sample (composite material) as a result of increased friction and pressure with a hard disk device and these particles increase the wear of the sample surface because it works stress center on the sample in the areas of presence [21, 22]. It is noted that the increase in load leads to increase the wear rate where the wear of shifting mild wear to wear transition and to the wear severe and this is due to plastic deformation quotient to the tops of bumps surface of the sample, leading to Increase the density of dislocations and thus a strain hardening.

## MICROSTRUCTURE :

Microstructural characterization studies were done to observe the microstructure of sample surface test. This is done by using an optical microscope and SEM. Characterization is done in etched conditions. Figs. from (6-9) show the microstructure of Al-12wt%Si alloy without and with additives $CeO_2$ nanoparticles after the sintering process. It is noticed clear that different in microstructure in shape and size of Si-phase and eutectic Al-Si between nanocomposites (MMNC) reinforced with nano powder $CeO_2$. The microstructures of Al-12wt%Si alloy consist of Al matrix and Si phase and different pores dispersed in the matrix. A little agglomeration was occurred leading to a formation of large particles; this is due to insufficient mixing time for powders. Figs. from (10-12) show the scanning electron microscope of nanocomposite materials. It is clear that nanoparticles are dispersion and distributed uniformly in the Al-12%Si matrix. The microstructure evaluation also shows that, for a given series of composites, the size of the particles in the nanocomposites increases as the content increases. A small agglomeration of particles in the Al matrix has been noticed and this is mainly due to non-homogeneity involved in mixing process carried out before sintering as shown in Fig. (11).

## CONCLUSIONS :-

1. The addition of Cerium oxide as a nanoparticles reinforcement to the Al-12wt%Si alloy was contributed to decreasing the wear rate for all additions percentage of $CeO_2$ nanoparticles.
2. Wear rate of the base alloy and nanocomposites increases with increasing applied load.
3. Apparent density decreases slightly with increasing the $CeO_2$ nanoparticles reinforcement that added to the Al-12wt%Si alloy.
4. Increasing the weight percentage of Cerium oxide nanoparticles led to increasing the porosity for nanocomposites as compared to the base alloy.

**Table (1) The weight percent of composition samples.**

| No. of sample | Al% | Si% | $CeO_2$% |
|---|---|---|---|
| 1 | 88 | 12 | - |
| 2 | 87 | 12 | 1 |
| 3 | 86 | 12 | 2 |
| 4 | 85 | 12 | 3 |

**Table (2) The apparent density, porosity of samples.**

| **Samples** | **Apparent Density gm./cm$^3$** | **Porosity %** |
|---|---|---|
| Al-12%Si | 2.726012 | 18 |
| Al-12%Si-1%$Ce_2O$ | 2.720406 | 22.4 |
| Al-12%Si-2%$Ce_2O$ | 2.710619 | 24.2 |

| | | |
|---|---|---|
| Al-12%Si-3%$Ce_2O$ | 2.694933 | 26.5 |

**Table (3) The wear rate of samples, at P=10N, P=15N.**

| Samples | Wr ($10^{-7}$) (gm/cm) at P=10N | Wr ($10^{-7}$) (gm/cm) at P=15N |
|---|---|---|
| Al-12%Si | 5.82 | 7.16 |
| Al-12%Si-1%$CeO_2$ | 4.45 | 6.17 |
| Al-12%Si-2% $CeO_2$ | 3.59 | 5.53 |
| Al-12%Si-3% $CeO_2$ | 3.17 | 5.06 |

**Fig. (1) The electric rolling mixers.**

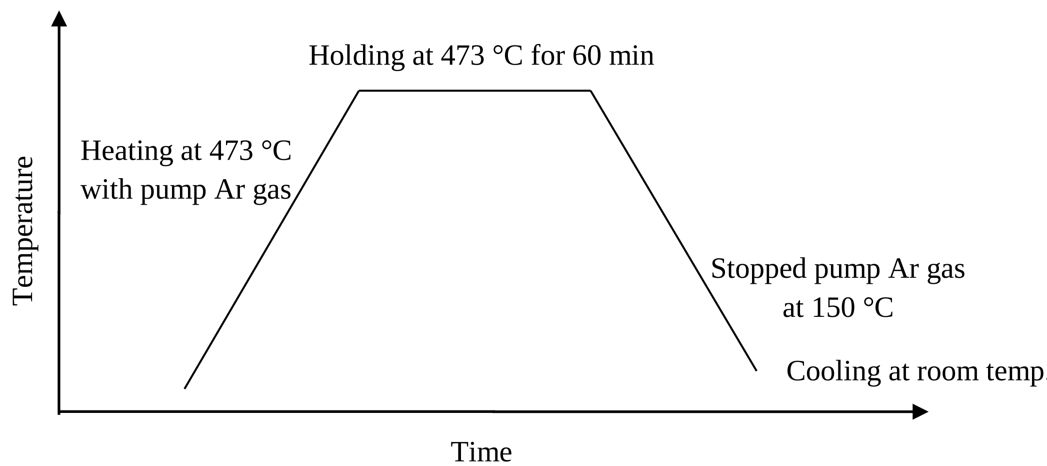



**Fig. (2) The procedure of sintering process.**

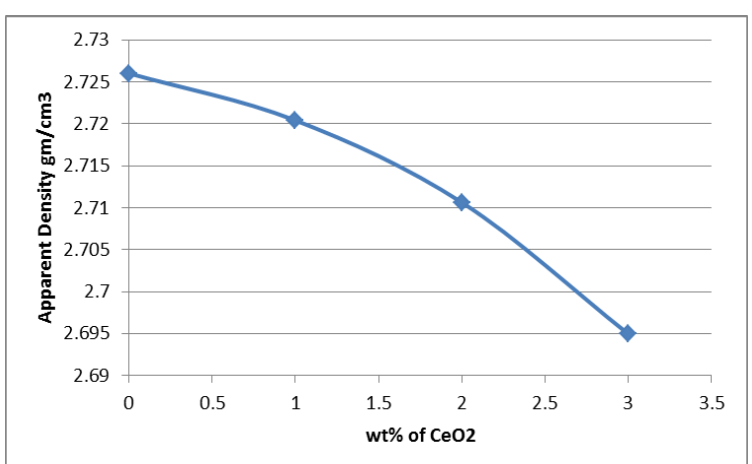





**Fig. (3) Effect of the wt%$CeO_2$ nano powder additions on the apparent density of the Al-12wt%Si alloy**

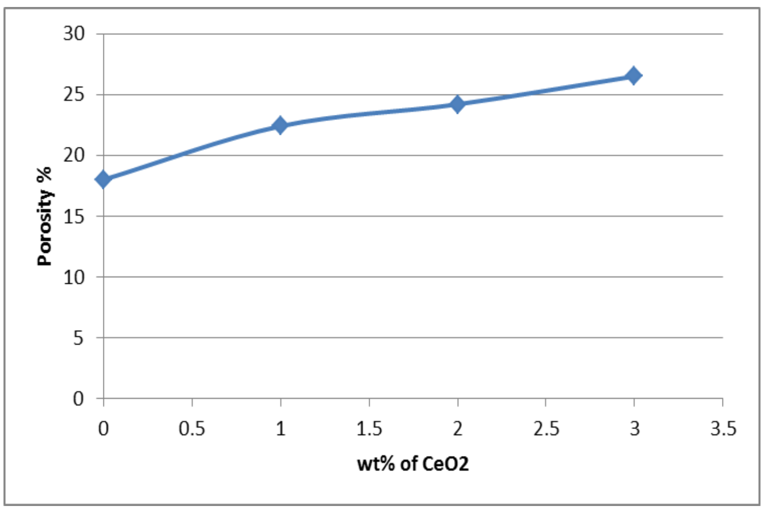



**Fig. (4) Effect of the wt%$CeO_2$ nano powder additions on the porosity of the Al-12wt%Si alloy**

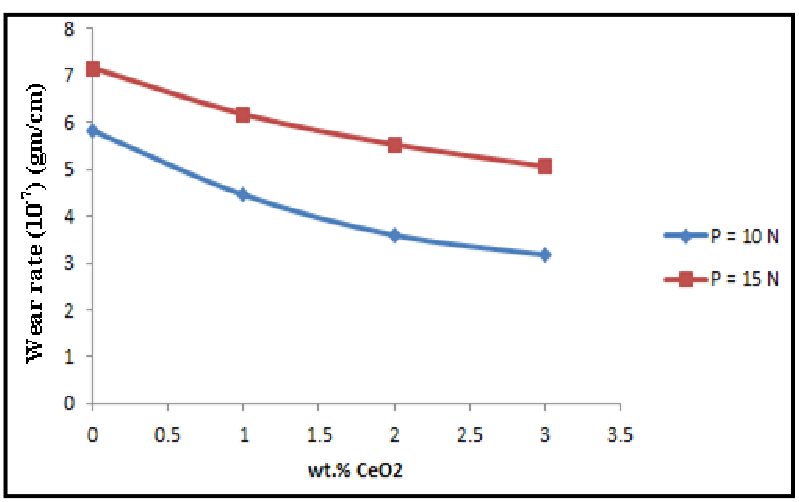



**Fig. (5) Effect of the wt%$CeO_2$ nano powder additions on the wear rate of the Al-12wt%Si alloy**

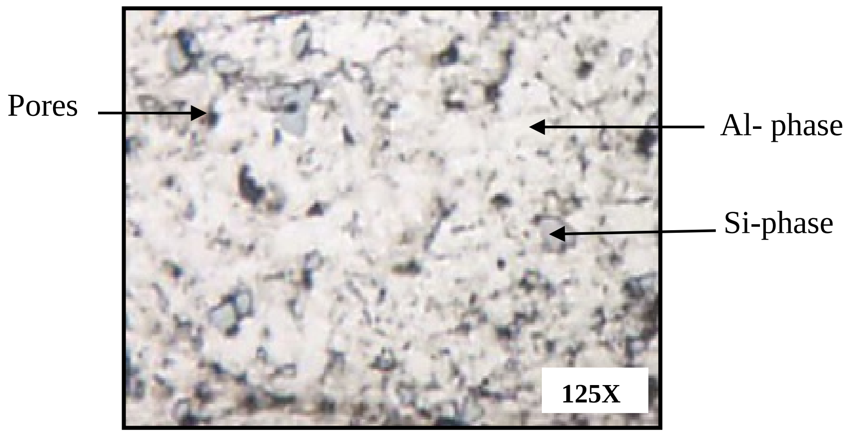



**Fig. (6) Microstructure of Al-12wt%Si.**

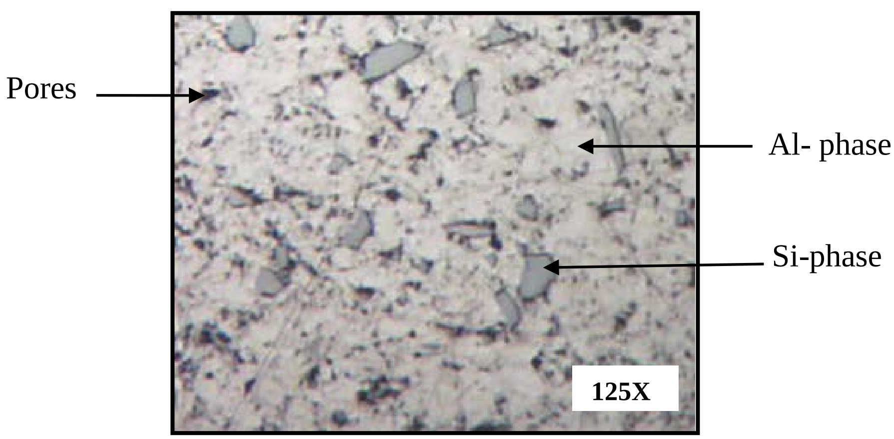



**Figure (7) Microstructure of Al-12wt%Si reinforced by 1wt% $CeO_2$ nanoparticles.**

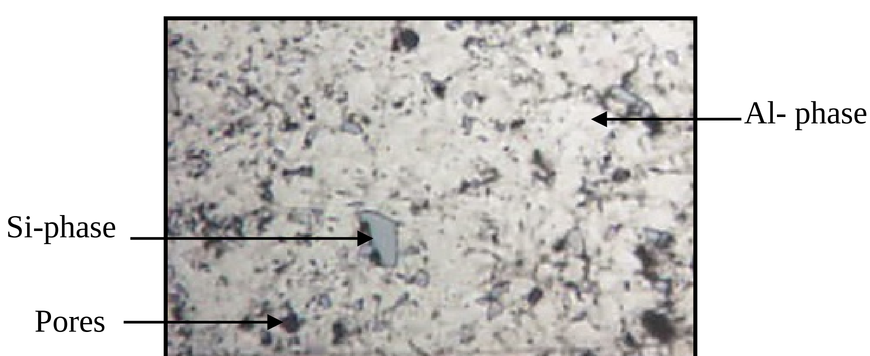


**Figure (8) Microstructure of Al-12wt%Si reinforced by 2wt% $CeO_2$ nanoparticles.**

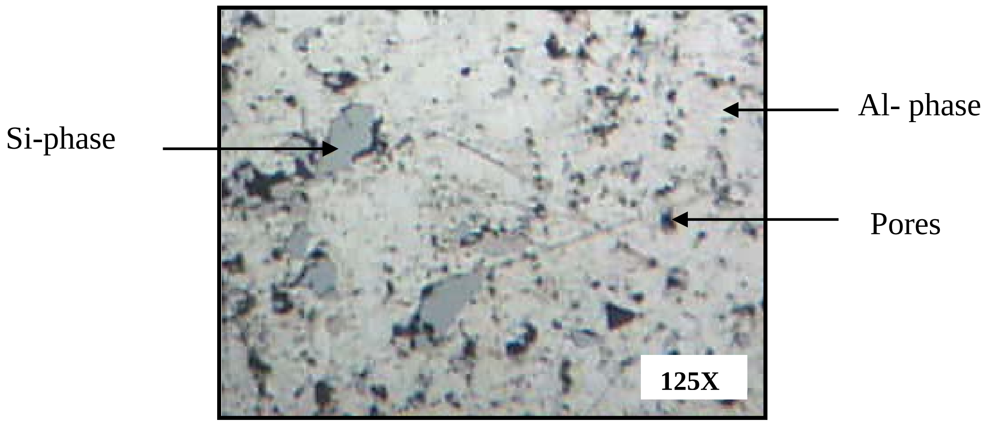



**Figure (9) Microstructure of Al-12wt%Si reinforced by 3wt% $CeO_2$ nanoparticles.**

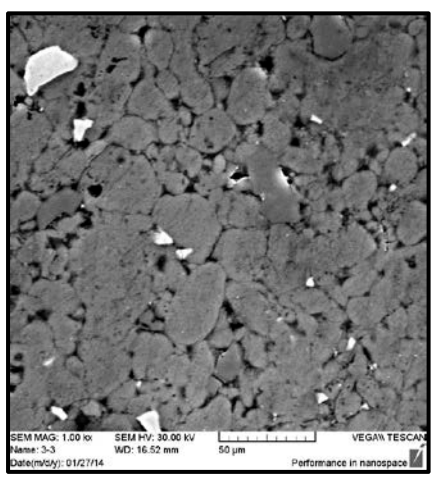
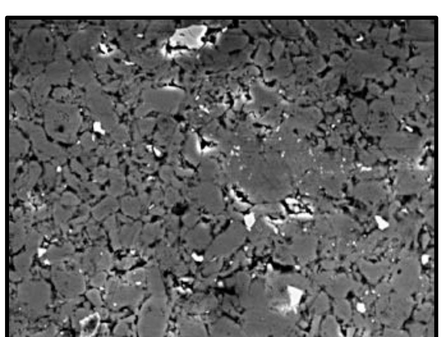



**Figure (10) SEM of MMNC with 1wt% $CeO_2$ nanoparticles.**



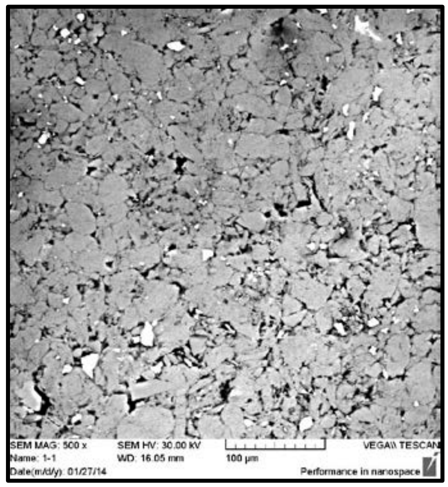



**Figure (12) SEM of MMNC with 3wt% $CeO_2$ nanoparticles.**

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