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Influence of Exfoliated Graphite Nanoplatelets Particles Additions and Sintering Temperature on the Mechanical Properties of Aluminum Matrix Composites

El-Sayed M. Sherif[1,*], *F.H. Latief*[1], *H. Junaedi*[2], *A.A. Almajid*[1,2]

[1] Center of Excellence for Research in Engineering Materials (CEREM), College of Engineering, King Saud University, P. O. Box 800, Riyadh 11421, Kingdom of Saudi Arabia
[2] Department of Mechanical Engineering, College of Engineering, King Saud University, PO BOX 800, Riyadh 11421, Kingdom Saudi Arabia
[*]E-mail: esherif@ksu.edu.sa

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Different Aluminum-exfoliated graphite nanoplatelets particles (xGnPs) composites, namely Al-0.0wt.%xGnP, Al-1.0wt.%xGnP, Al-3.0wt.%xGnP, and Al-5.0wt.%xGnP, were fabricated using powder metallurgy technique. The mixture powders were consolidated at 500 MPa for 5min followed by pressure-less sintering at 400, 500 and 600°C for 5 h. The compression, density, fracture surface, and hardness measurements were performed in order to evaluate the mechanical properties of the different Al-xGnP composites. Results showed that the compressive strength and Vickers hardness increase, while the density decreases as a function of exfoliated graphite nanoplatelets content. The best condition was reached by sintering at 600°C for 5h for all composites.

Keywords: Metal-matrix composites; nanoplatelets particles; fracture toughness; hardness testing; sintering temperature

1. INTRODUCTION

Aluminum and its composites have a great diversity of industrial applications such as aircraft, space and automotive industries because of their low density and good workability, but the use of these alloys is limited due to their relatively low yield stress. Aluminum matrix composites possess many advantages over monolithic materials including higher specific strength, good wear resistance, higher thermal conductivity, lower coefficient of thermal expansion, etc [1-5]. Aluminum matrix composites can be fabricated in the solid state through powder metallurgy (P/M) technique. This method is used due to its great versatility and low cost of production. P/M is a highly evolved method of

manufacturing reliable net shaped components by blending elementalor pre-alloyed powders together. The process of P/M fabrication consists in mixing the hardening particles with the metallic powders followed by consolidation and sintering [6-8].

The process of mixing raw materials is an important first step since it controls the distribution of particles and porosity of the composites both of which, influence the mechanical and tribological behaviors [9]. If increased mechanical properties are desired, very fine particles must be used for reinforcement [10]. Decreases of the reinforcement particle size effectuate an increase in the mechanical strength of the composite [11-13]. The strengthening effect due to the presence of reinforcement particles is the result of elastic interactions between the particles and matrix dislocations, which hinder dislocation motion. However, the efficiency by which reinforcement particles strengthen the matrix depends on their type, size, morphology, volume fraction and overall distribution [14]. Exfoliated graphite nanoplatelets (xGnPs) have recently attracted the attention as a substitute for carbon nanotubes, given the predicted excellent mechanical, structural, thermal and electrical properties of graphite and their similar properties to nanoscale carbon black and carbon nanotubes [15, 16]. Structurally, graphite and carbon nanotubes are made up of the same building blocks [17]. Recently, it has been developed a process that can produce exfoliated graphite nanoplatelets of 4–10 nm in thickness and from 1 to15 μm in diameter [18].

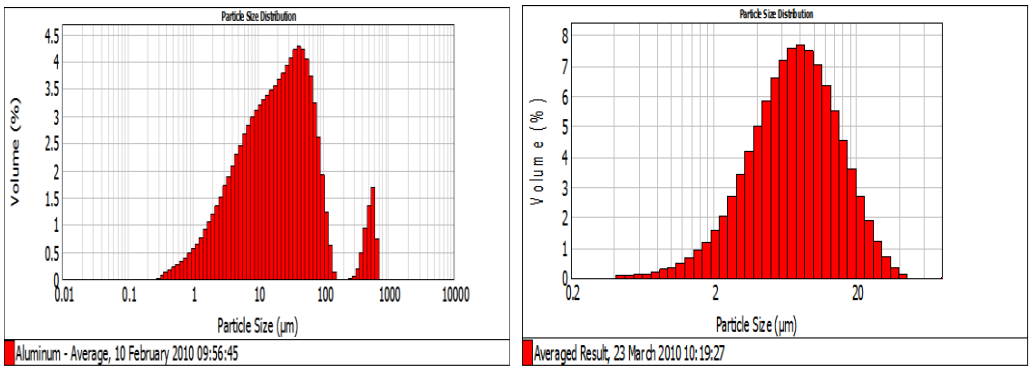
The aim of the present work was to fabricate different Al-xGnP composites using powder metallurgy method. The aim was extended to study the influence of 0, 1.0, 3.0 and 5.0 wt.%xGnP additions and sintering temperature on density, microstructure, compression strength and micro-hardness of aluminum. The best conditions for obtaining the most useful properties of the fabricated composites were also reported.

2. EXPERIMENTAL DETAILS

A 99.0% aluminum powder supplied by Riedel-De Haen Ag Seelze-Hannover, Germany was used in this study. Whereas, the xGnPs that act as reinforcement were supplied by Asbury Graphite Mills, USA, with label of Asbury 3772. The particle size distribution of Al powder and xGnPs was measured by particle size analyzer as demonstrated in Fig. 1. After measurement, the median particle size of Al powder and xGnPs were 20μm and 7.5μm, respectively. The morphology of the Al powder and xGnPs as starting materials is shown in Fig. 2. In addition, the thickness of xGnPs used in this work was approximately 5-10 nm as seen in Fig. 2b. The density of aluminum powder was 2.7 g/cm^3 and the density of xGnPs was nearly 2.2 g/cm^3.

Aluminum-exfoliated graphite nanoplatelets composites were fabricated as follows: First, the xGnPs were dispersed in acetone using a disperser machine at speed of 2000 rpm for 30 min. Second, the Al powder was slowly added into solution with the xGnP particles content of 0, 1, 3 and 5 by weight percent. The mixing process was continuously performed for 1 h to obtain a homogenous mixture. The mixture was then filtered and dried at 80°C for overnight to generate a dried mixture powders. The mixture powders were green compacted at pressure of 500 MPa for 5 min to produce a disc-shaped specimen with the ratio of 1:1 between diameter and height. Afterward, the disc specimens

were sintered at temperatures of 400, 500 and 600°C for 5 h in a pressureless furnace. The prepared composites were observed by a computer controlled optical microscopy (OM, Olympus, Model BX51M, Japanese made) to recognize the distribution of xGnP particles within aluminum.



A **B**

Figure 1. Particle size distribution of (a) as-received Al powder and (b) exfoliated graphite nanoplatelets.

The experimental density was measured according to the Archimedes principle (Bouyancy method), by using a balance with an accuracy of 0.1 mg. Meanwhile, the theoretical density of composites was measured by the rule of mixture using the theoretical density 2.7 g/cm^3 for Al powder and 2.0 g/cm^3 for xGnP particles. The phase of compacted composites was determined using Philips automatic X-ray diffractometer with Cu target Kα radiation. Compression test was carried out under room temperature using an Instron Universal Testing Machine with a strain rate of 10^{-3} s^{-1}. At least three compressive specimens were tested for each composition. The hardness of composites was tested by a micro-indenter (Vickers hardness) under a load of 200 g and a dwell time of 15 s. The measurement was performed for 5 times from random locations on the central region of polished cross-section and they were then averaged. The morphology and fracture surface were observed by means of a JEOL (model JSM-6610LV) scanning electron microscope.

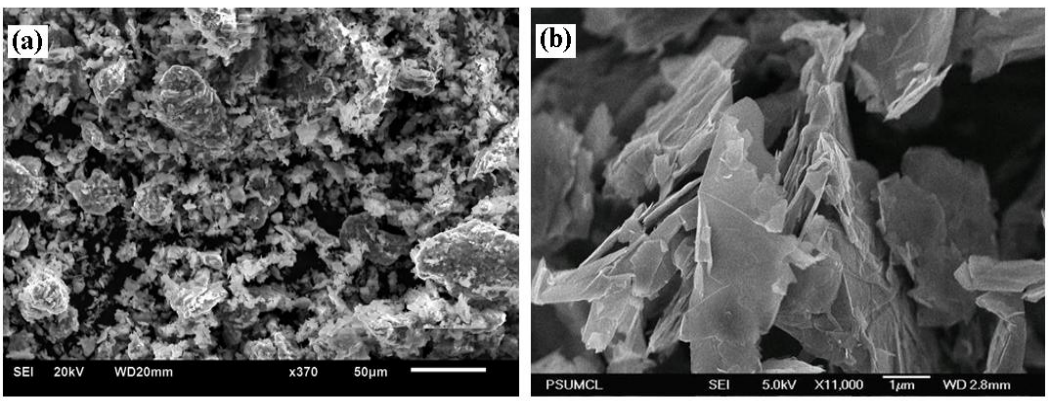


Figure 2. SEM micrographs of (a) Al powder and (b) exfoliated graphite nanoplatelets.

3. RESULTS AND DISCUSSION

3.1. Effect of sintering temperature on the density of composites

The theoretical and average experimental density values of composites are presented in Fig. 3. The theoretical density released the highest density values since the affecting factors such as sintering or other processes or variables- are neglected. Nevertheless, the results indicated that the increase of sintering temperature resulted in decrease of density values of pure Al and composites. The high sintering temperature is able to ease the diffusion of atoms which impacts on the sinterability of composites [19]. In addition, the increase of xGnP particles added into the Al matrix leads to reduction of the density of composites. Since the melting point of xGnP is above 3000°C, it has low tendency to make bonds with pure Al leading to a weak network. Moreover, the sintering process induces the dimensional changes due to the shrinkage, which takes place during sintering itself. The shrinkage occurs due to the density of xGnP particles as reinforcement is smaller than that of Al matrix. The composite with high concentration of xGnP particles exhibited a low density of composites because of the degree of shrinkage is the highest compared to the other composites composition.

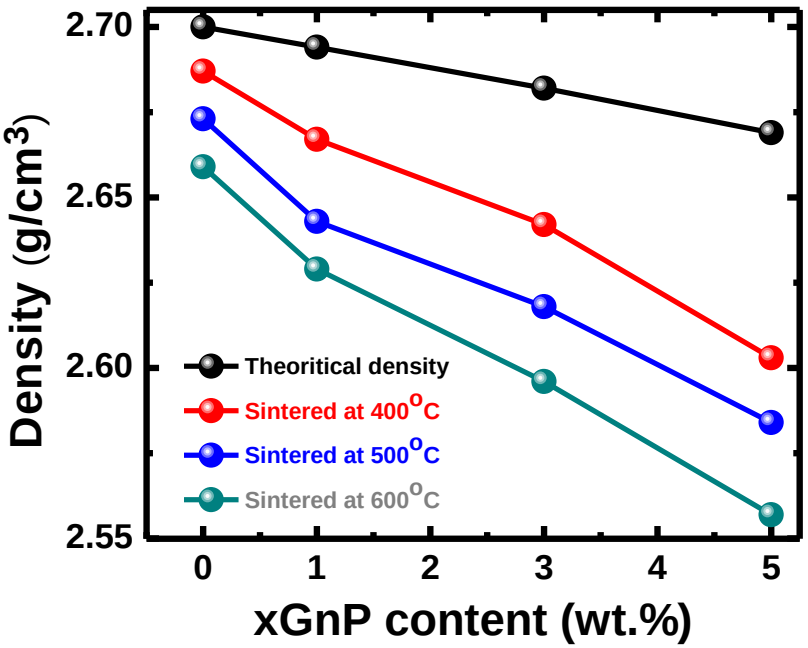


Figure 3. Relative density of composites vs. xGnP contents (relative density = measured sintered density/theoretical density).

3.2. Optical microstructure and X-ray diffraction (XRD) pattern of composites

The optical microstructures of the fabricated composites after sintering at 600°C for 5 h are shown in Fig. 4. The microstructure of pure Al presents a good chemical bonding among the Al particles, where the Al particles are diffused to produce a solid structure as seen in Fig. 4a. The xGnP particles were uniformly distributed within Al matrix as shown in Fig. 4b-d. Furthermore, the Al

powder and xGnP particles were combined to create a good chemical bonding among them under sintering process but some xGnP particles are still not embedded into the composites.

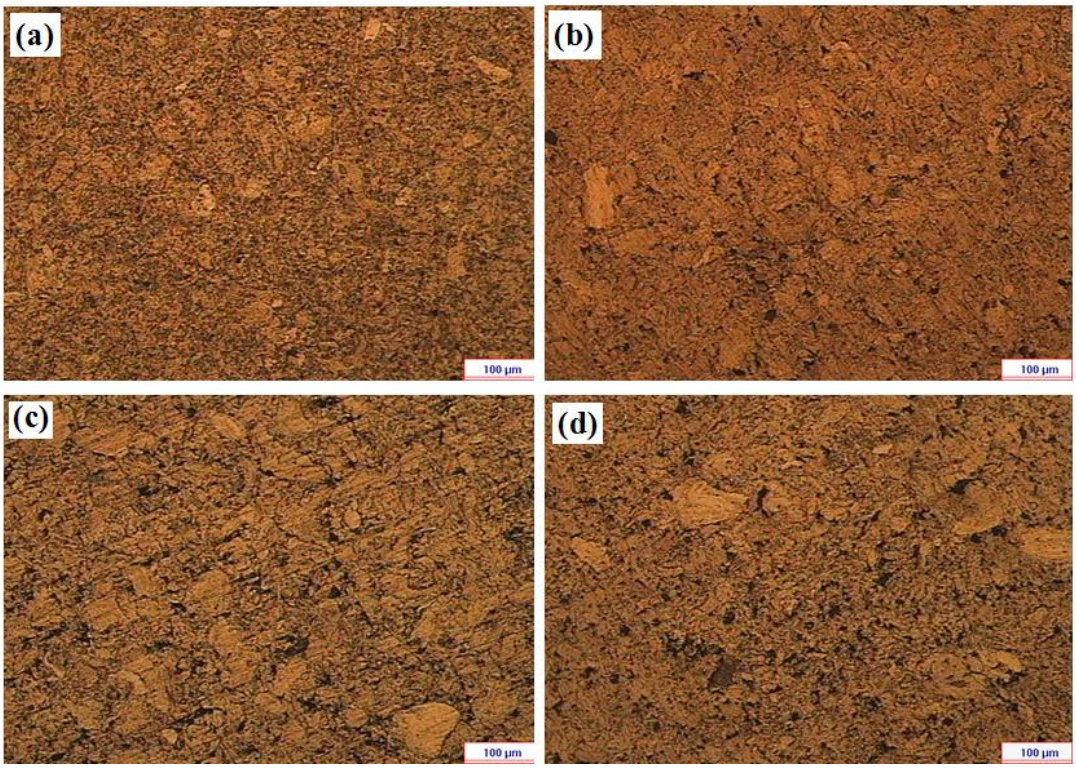


Figure 4. Optical microstructures of (a) pure Al (b) Al-1wt.%xGnP (c) Al-3wt.%xGnP and (d) Al-5wt.%xGnP composites after sintering at 600°C for 5 h.

It is clearly seen from Fig. 4 that the grain size is low at high xGnP concentrations because xGnP particles act as barriers against the tendency of grain growth. At higher sintering temperatures, a denser structure is easily formed due to higher diffusion rates [20]. The sintering temperature thus plays an important role as controlling factor in sintering process [21]. The following equation confirms this phenomenon [22]:

$$D = D_o \exp\left(-Q/RT\right) \quad (1)$$

Where D is diffusion coefficient, D_o is constant, Q is activation energy, R is Boltzmann's constant and T is temperature.

Fig. 5 demonstrates the X-ray diffraction pattern of pure Al and composites. The addition of xGnP particles represents a new peak which referred to the presence of xGnP itself in all composites. The xGnP peak is present at 2θ equal to 26.5° for all composites. The different content of xGnP particles could be recognized from the fluctuation intensity of xGnP peaks which increases with increasing the xGnP content.

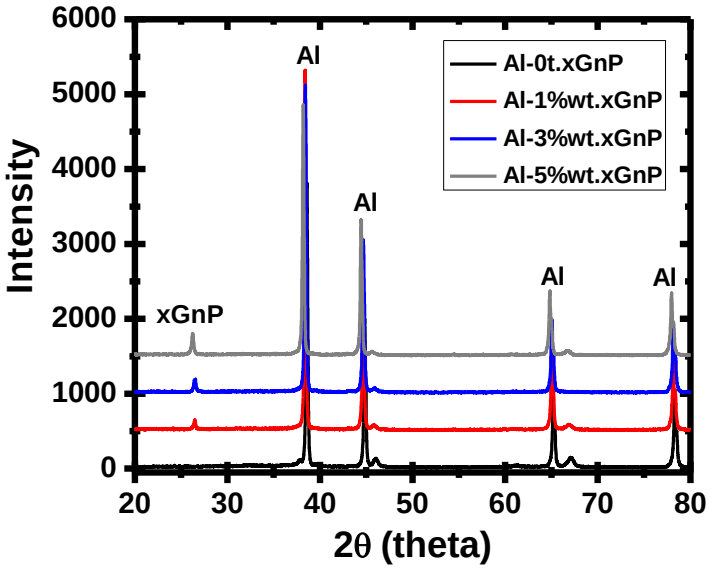


Figure 5. XRD pattern of pure aluminum and aluminum-exfoliated graphite nanoplatelets composites.

3.3. Effect of sintering temperature on the mechanical properties of composites

Fig. 6 presents the effect of sintering temperature and xGnP content on the compressive strength of composites.

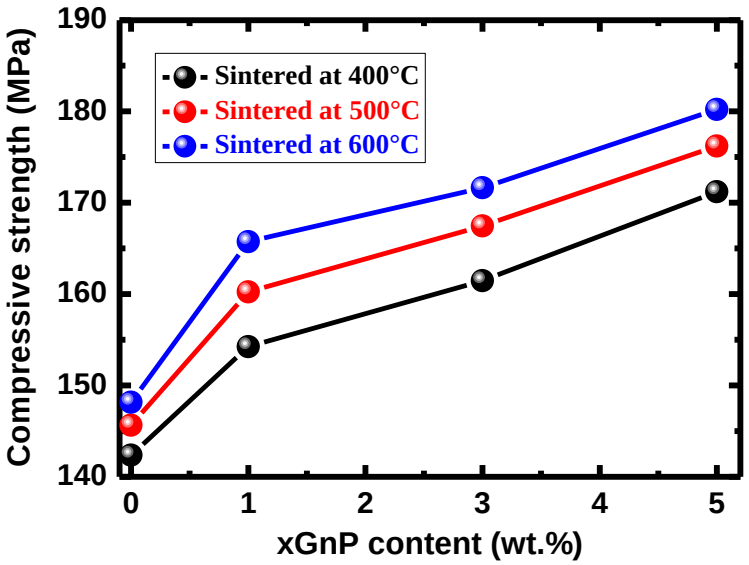


Figure 6. Compressive strength of composites as a function of xGnP contents at various sintering temperatures.

The compressive strength of composite with 1.0 wt.% xGnP particles significantly increased and then showed linearly increment of compressive strength with increasing the amount of xGnP

particles. It can be seen that a relatively high sintering temperature provides better compressive strength. At high temperature, the diffusion of atoms was readily occurred which motivates the formation of a chemical bonding among the available particles inside the structure. The addition of xGnP particles up to 5wt.% into Al powder was effectively increased the compressive strength of composites. If the reinforcement particles are added into matrix, the distance between them decreases. As consequence, the dislocation movement is more difficult because they face more obstacles and the accumulation of dislocation occurs at this stage.

The effect of sintering temperature and xGnP content on the average hardness of composites is shown in Fig. 7. High sintering temperature provides better hardness value because the particles were diffused to make a chemical bonding between matrix and reinforcement. This interfacial phenomenon was greatly affected the properties of composites. On the other hand, the increase of xGnP content resulted in increase of hardness values of composites. However, the interfacial area between Al powder and xGnP particles was larger since the xGnP particle has high specific surface [23].

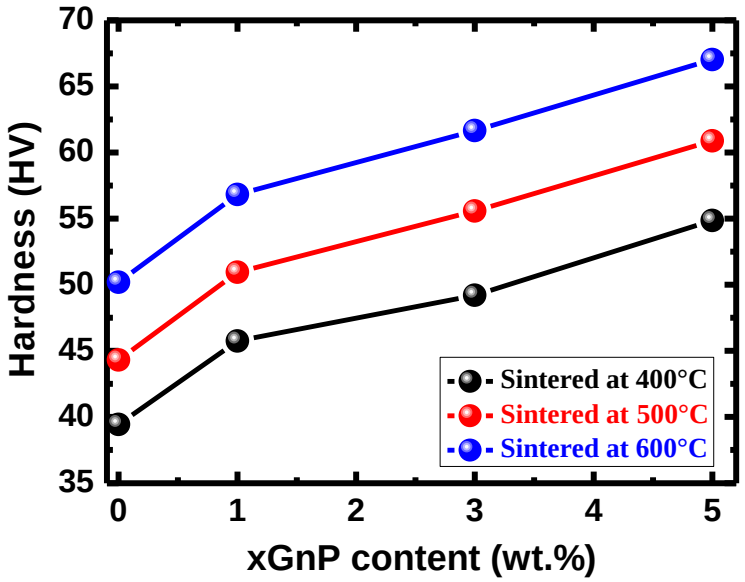


Figure 7. Vickers hardness of composites as a function of xGnP contents at various sintering temperatures.

The achieved results show that the addition of xGnP particles from 0-5wt.% into pure Al increase the mechanical properties of composites in both compressive strength and hardness . It can be explained by following equation [24]:

$$\lambda = \frac{4(1-f)\mathrm{r}}{3f} \qquad (2)$$

Where λ is the distance between the reinforcement particles, f is the xGnP particles volume fraction and r is the xGnP particle radius, assuming that the xGnP particle is spherical. According to

Eq. (2), increasing the amount of xGnP particle leads to a decrease in the distance between the xGnP particles since its median radius is about 3.75 μm. Lowering the distance between the xGnP particles according to Eq. (3) will increase the required tension for dislocations movement between the xGnP particles leading to an increment in mechanical properties of materials.

$$\tau_0 = \frac{Gb}{\lambda} \quad (3)$$

Where, τ_o is the shear stress, G is the shear module, b is the Burger's vector and λ is the distance between particles [23]. Based on Eq. (2) and Eq. (3), it could be understood that the decrease in distance between particles would increase the shear stress of dislocations and as consequence the composite strength would be increased [25, 26]. In addition, the xGnP particles act as obstacles that can prevent the movement of dislocations in pure Al matrix through dispersion strengthening mechanism [24]. Increasing the temperatures causes easier diffusion, which in turn will change the properties of composite, particularly mechanical ones.

When the reinforcement particles is added to composite, the distance between them decreases and then the movement of dislocations is tough because they face more obstacles and dislocations pile up occurs. On the other hand, the improvement on compressive strength of composites can be explained by the following equation [24]:

$$\sigma_c = \sigma_m f_m + \sigma_r f_r \quad (4)$$

Where, σ_c is the strength of composite, σ_m is the strength of matrix, σ_r is the strength of reinforcement, f_m is the volume fraction of matrix and f_r is the volume fraction of reinforcement.

The addition of xGnP particles leads to the higher hardness [27, 28]. This phenomenon can be readily explained by the rule of mixtures, applied to composite materials as shown in the below equation [24]:

$$H_c = H_m f_m + H_r f_r \quad (5)$$

Where, H_c is the hardness of composite, H_m is the hardness of matrix, H_r is the hardness of reinforcement, f_m is the volume fraction of matrix and f_r is the volume fraction of reinforcement.

Fig. 8 shows the fracture surfaces of pure Al and composites after sintering at 600°C for 5h. The dark regimes shown in Fig. 8a represent the pores or voids which were left behind by evacuation of xGnP particles from the surfaces during the polishing process. These pores or voids are responsible for the crack initiation that will surely reduce the strength of material. In general, the xGnP particles are uniformly distributed within the Al matrix as seen in Fig. 8b-d. This uniform distribution of the second phase within the matrix is a characteristic of the in-situ powder metallurgy method [23] and it improves the mechanical properties of composites. But few xGnP clusters were found in the Fig. 8d due to detachment of xGnP particles from the Al matrix.

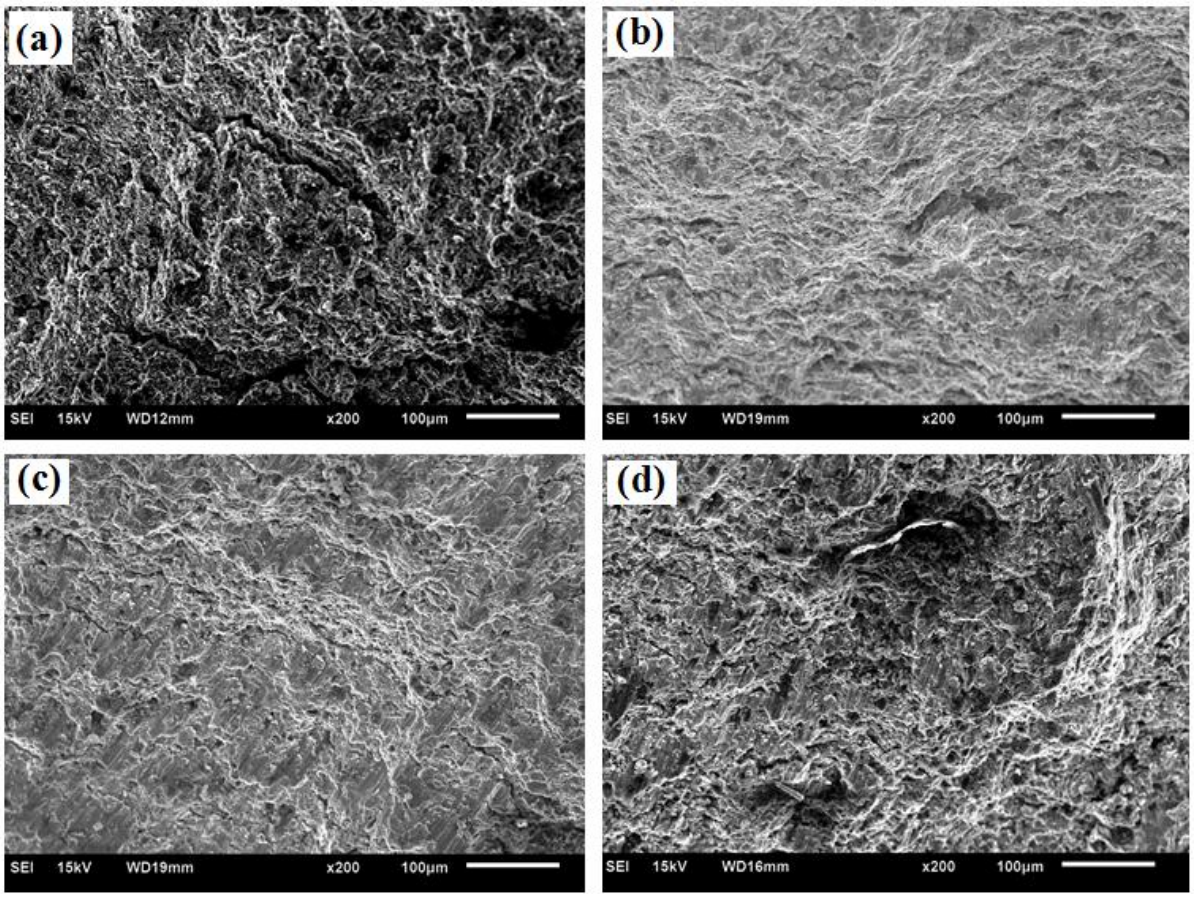


Figure 8. SEM fracture surfaces of (a) pure Al (b) Al-1wt.%xGnP (c) Al-3wt.%xGnP and (d) Al-5wt.%xGnP composites.

4. CONCLUSIONS

Fabrication of Al-0.0wt.%xGnP, Al-1.0wt.%xGnP, Al-3.0wt.%xGnP, and Al-5.0wt.%xGnP were fabricated at different sintering temperatures using powder metallurgy method was carried out. The influence of xGnP additions and sintering temperature on the density, microstructure, compression strength and micro-hardness of pure aluminum were reported. The relative density of Al-xGnP composites was found to decrease with increasing the amount of xGnP due to shrinkage during the sintering process. On the other hand, the compressive strength and hardness of the composites increased with increasing the amount of xGnP particles and high sintering temperature. Results together indicated that the presence of xGnP particles and the increase of its content within the composite as reinforcement as well as increasing the sintering process represent the effective ways to enhance the mechanical properties of Al-xGnP composites.

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