

**PROCESSING AND CHARACTERISATION OF FUNCTIONALLY GRADED MATERIALS
BASED ON FEW-LAYERED GRAPHENE REINFORCED ALUMINUM**

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ozberkozturk@gmail.com*<https://doi.org/10.37904/metal.2022.4427>**Abstract**

Functionally graded materials (FGMs) are advanced engineering materials developed due to their superior properties where traditional composite materials are not sufficient. Nowadays, the development and application of these materials for the potential areas have attracted much more attention. Aluminum (Al) is preferred for physical and mechanical properties such as lightweight, high specific strength, high specific modulus, and low thermal expansion coefficient in these potential applications. Graphene attracts great attention worldwide due to its superior mechanical, electrical and thermal properties. In the current study, few-layered graphene (FLG) produced with high purity electric arc discharge method were used to reinforce the Al matrix using various (in wt%) of 0, 0.1, 0.2, 0.3, 0.5, and 0.7 FLG by mechanically alloying (MA). Composite powders were consolidated by cold pressing with a layer by layer under 450 MPa. Al-FGM composites were designed including six layers and they were subjected to sintering at approximately 590 °C under argon atmosphere. The microstructure of Al-FGM was investigated by optical microscopy and scanning electron microscopy (SEM). It was observed that the FLG placed between the grains and acts as a barrier through the gradation improving the mechanical properties of the Al-FGM. Hardness value of the layer with the highest graphene content was measured as 113 HV. An increase in the Vickers hardness by 18% was observed in the last layer with FLG content of 0.7 wt% compared to the first layer.

Keywords: Aluminum, functionally graded materials, graphene, metal matrix composite, powder metallurgy

1. INTRODUCTION

Functional graded materials (FGMs), which were revolutionary with their discovery, are characterized as an advanced class of materials by differences depending on their position in composition throughout the volume and contribute to changes in material properties with functional requirements [1]. FGMs have advantages over conventional alloy and composite materials. In FGMs the distribution of the reinforced material varies from innermost to outermost compared to other conventional composite materials. Thus, constantly changing properties and controlled non-uniform microstructure are provided in the designed material [2]. FGMs offer possibilities to control the material response to deformation, dynamic loading as well as corrosion and wear. Moreover, FGMs can be used as a high-strength bonding interface to bond two incompatible materials together [3]. Due to these advantages of FGMs; It has potential application areas such as aerospace, automobile, biomedical, defense, electrical/electronics, energy, marine, optoelectronics, and thermo-electronics [4,5]. Today, various methods are used in the production of FGMs according to the requirements of the final product. Vapor deposition, thermal spraying, centrifugal casting, slip casting, tape casting, gel casting, laser deposition, sedimentation, electrochemical methods, and powder metallurgy (P/M) are the production techniques commonly used for FGMs [5,6]. P/M over other techniques, it has some advantages such as availability of good raw material, suitable for mass production, control of chemical composition, production of porous and complex shapes and metal matrix composites, which are very difficult to produce by casting method due to

density difference [7]. In the last few years, among metal-based materials, Al, due to their good thermal and electrical conductivity, high tensile strength-to-weight ratio, high hardness, and ductility properties; is of great interest in structural and functional applications [8,9]. As an alternative to ceramic materials such as B_4C , SiC , Al_2O_3 , different types of C-based reinforcement materials have also been added to the materials with Al-based P/M [10]. Graphene which is one allotrope of the carbon has some attractive features such as high electrical conductivity ($15000 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$) [11], high modulus of elasticity ($\sim 1 \text{ TPa}$), high surface area ($2630 \text{ m}^2/\text{g}$), high thermal conductivity ($5000 \text{ W m}^{-1} \text{ K}^{-1}$) [12], 100-300 times stronger than steel, extremely hard, low mass. Considering these unique properties, graphene emerges as an ideal reinforcement material. In this study, various weight fraction of 0, 0.1, 0.2, 0.3, 0.5, and 0.7 FLG produced by EAD method was reinforced to the Al matrix as reinforcement by MA. It was carried out to design FGM by stacking FLG reinforced Al matrix composite powders, layer by layer. Structural and mechanical properties of FGM were examined. Thus, the effect of graphene content on FGM design was investigated.

2. MATERIALS AND METHODS

FLG reinforced Al based FGM was produced from the Al (-325 mesh; 99.5%, Alfa Aesar), and FLG (surface area, $153 \text{ m}^2/\text{g}$) as starting powders. High purity FLG were synthesized by EAD method using an originally designed stainless steel reactor in our laboratory [13]. The FLG were reinforced to the Al matrix using various weight fraction of 0, 0.1, 0.2, 0.3, 0.5 and 0.7 by MA by MA which was carried out via planetary ball milling with 7:1 ball to powder ratio at 500 rpm for 5 h in Ar atmosphere. These FLG reinforced Al matrix composite powders were formed by stacking in six layers using a uniaxial manual press at 450 MPa. Then green bulk FGM which were subjected binder removal at 420°C for 2 hours were sintered at 590°C with a $5^\circ\text{C}/\text{min}$ heating and cooling rate for 3 h under Ar atmosphere. **Figure 1** shows schematic illustration of the design of functionally graded FLG reinforced Al.

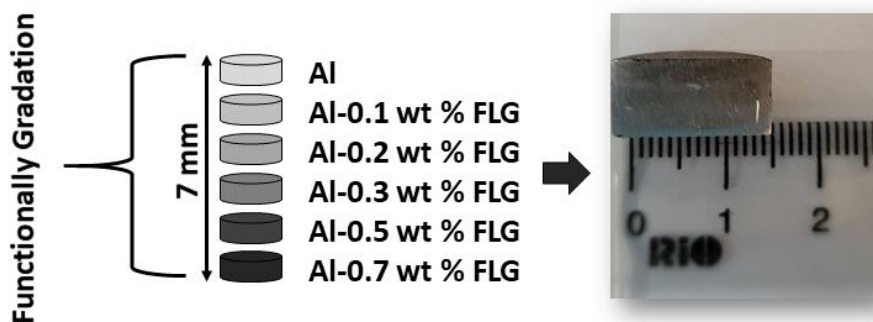


Figure 1 Schematic illustration of the design of functionally graded FLG reinforced Al

Thermal analysis of Al and 0.7 wt% FLG reinforced Al matrix powders were carried out using by a Linseis PT1600 differential scanning calorimeter (DSC) up to 700°C with a heating rate of $10^\circ\text{C}/\text{min}$ under nitrogen (N_2) atmosphere. XRD analysis for FLG reinforced Al matrix composite powders obtained by MA was performed with Bruker AXS/Discovery D8 X-Ray Diffractometer with $CuK\alpha$ radiation. SEM analyses of Al as starting powder, FLG which is synthesized by EAD and 0.7 wt% FLG reinforced Al matrix composite powders by MA and were performed with Carl Zeiss/Gemini 300 scanning electron microscope. The optical microscope which is Nikon Eclipse was used to investigate microstructure of FLG reinforced Al matrix. The microhardness values of the FLG reinforced Al matrix composites were determined for 15 seconds with a load of 200 g via a Qness Q10M microhardness tester. By taking five measurements separately from each of the six layers and these were averaged for each layer region. The density of the sample was measured based on the Archimedes' principle.

3. RESULTS AND DISCUSSIONS

Figure 2(a) shows SEM image of Al as starting powders. It is seen that Al consists of many large and small amorphous particles. **Figure 2 (b)** gives information about SEM image of FLG which was synthesized by EAD. According to the SEM images, nanoparticles and nanolayers with particle size between 40 and 60 nm were detected for FLG. There are large clustered agglomerates of nano-sized spherical particles [14-15]. It is considered that graphene nanoparticles tend to agglomerate, due to their very small particle size at the nanoscale.

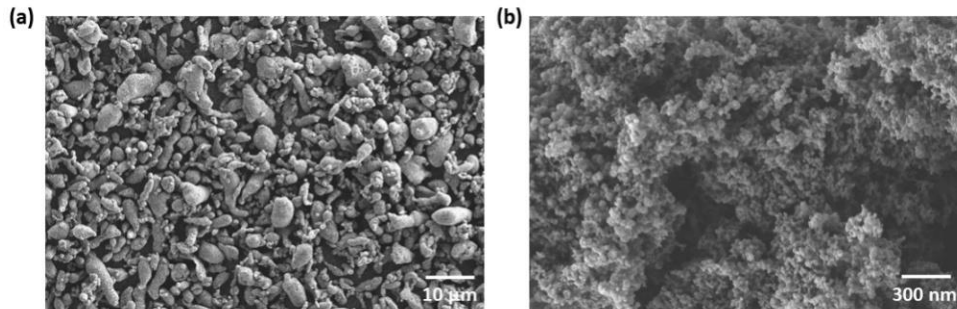


Figure 2 SEM images of **(a)** Al as starting powders, **(b)** FLGs which was synthesized by EAD

Figures 3(a) shows the XRD patterns of Al-based powders obtained with 5 h MA. There are distinctive characteristic peaks of Al in FLG reinforced Al-based composite powders. It is considered that the peak formation of FLG is not seen in XRD pattern, because there is a trace amount of FLG in FLG reinforced Al matrix composite powders [16]. Moreover, it is seen that the FLG are homogeneously distributed in the Al matrix. DSC graph of Al and 0.7 wt% FLG reinforced Al composite powders are shown in **Figure 3(b)**. Endothermic peak formation is observed for both powders. According to the graph, there is no change in the melting temperature of Al with 0.7 wt% FLG reinforcement on the Al matrix powder.

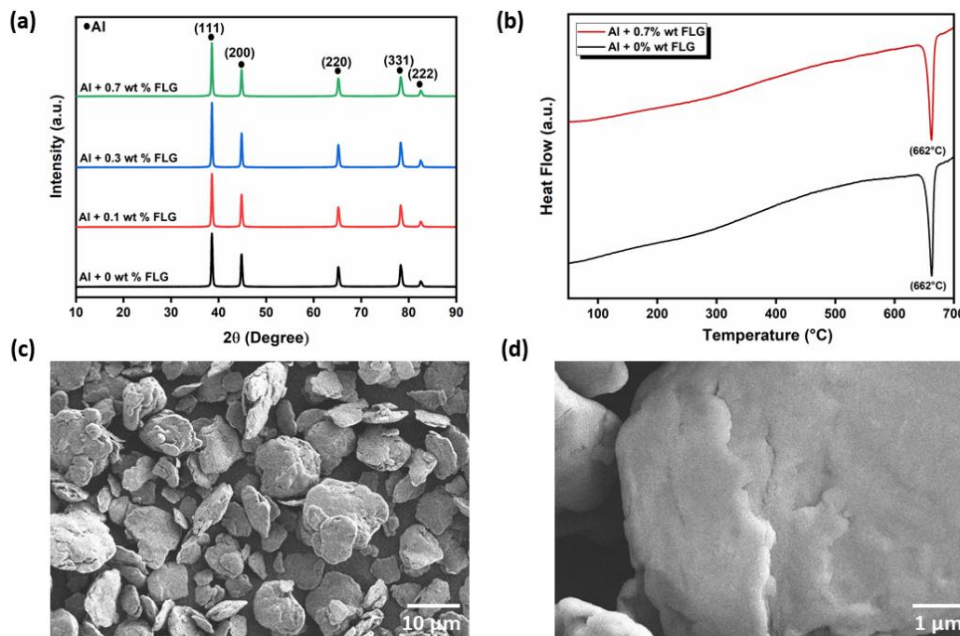


Figure 3 XRD patterns of **(a)** 0, 0.1, 0.3 and 0.7 wt% FLG reinforced Al powders; **(b)** DSC graph of Al and 0.7 wt% FLG reinforced Al composite powders, **(c-d)** SEM images of 0.7 wt%. FLG reinforced Al composite powders at different magnifications

SEM images at different magnifications of 0.7 wt% FLG reinforced Al-based composite powders obtained with 5 hours MA are shown in **Figures 3 (c-d)**. The composite powders have a flat appearance after 5 hours of MA which is different from starting powders. After ball milling as planetary, the powders which was abraded exhibits a flattened shape with repeated severe plastic deformation, fracture and cold welding [9]. It is considered that the FLG were embedded by the Al matrix powders because of residues the FLG are not visible on the surface of the powders. The FLG which were be exposed to agglomerate break down and separate into layers during milling. The FLG which have been subjected to this break, embedded in cold-welded Al particles by the ball milling [16]. The relative density value was obtained as 97.3% for FLG reinforced Al-based FGM. **Figure 4** shows how the FLG are graded on the Al matrix from top to bottom of FLG reinforced Al-based FGM produced by powder metallurgy.

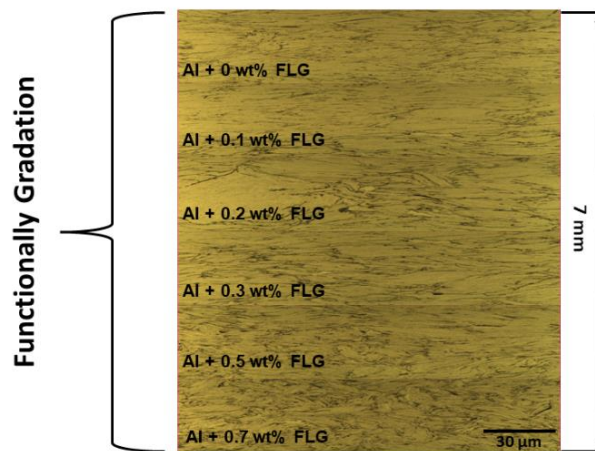


Figure 4 Grading of FLG on Al based from top to bottom by optical images

The optical images of **(a)** 0, **(b)** 0.1, **(c)** 0.2, **(d)** 0.3, **(e)** 0.5 and **(f)** 0.7 wt% FLG reinforced Al-based FGM is shown in **Figure 5**. It is seen that the FLG are homogeneously distributed in the Al matrix. Increasing FLG throughout the layers in the Al matrix which enables FLGs to act as a barrier in the structure. Therefore, it leads to a decrease in grain size [17].

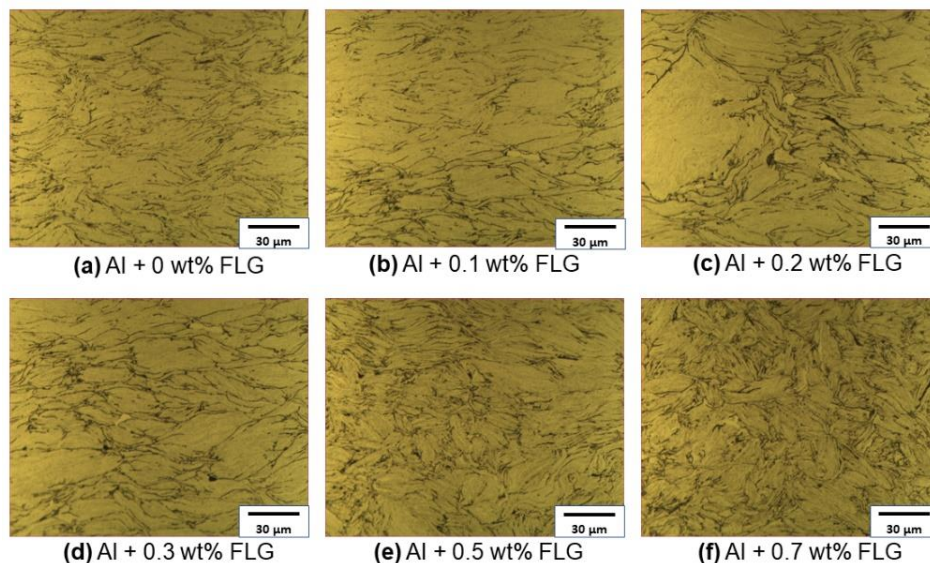


Figure 5 Optical images of **(a)** 0 wt%, **(b)** 0.1 wt%, **(c)** 0.2 wt%, **(d)** 0.3 wt%, **(e)** 0.5 wt% and **(f)** 0.7 wt% FLG reinforced Al based FGM

Figure 6 gives information about microhardness (HV) values for each layer of FGM. In the FLG reinforced Al-based FGM, it was observed that the hardness value increased by 18% from the first layer without FLG to the sixth layer with 0.7 wt% FLG.

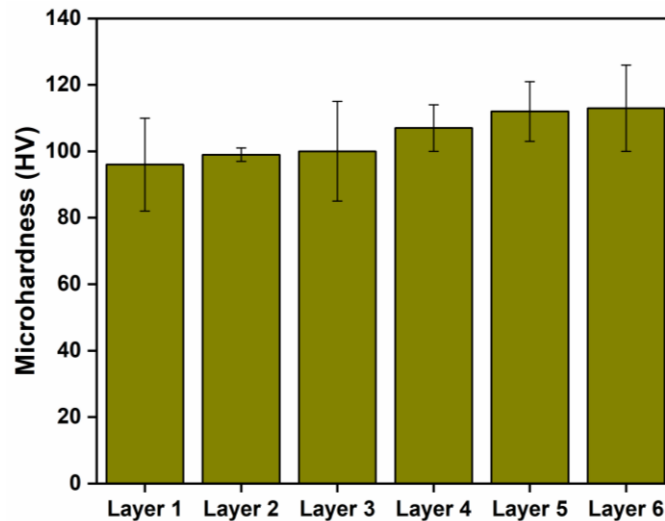


Figure 6 The graph with error bar of microhardness (HV) values for each layer of FGM

4. CONCLUSION

Functionally graded FLG reinforced Al was successfully fabricated by powder metallurgy. The structural properties of FLG reinforced Al matrix composite powders and FLG reinforced Al based FGM were examined. It is considered that FLG are embedded in the Al matrix powders because of plastic deformation due to using ball milling according to the SEM images. It was observed that the FLG were homogeneously dispersed in the Al matrix. It is clearly seen that the FLG increased from bottom to top for the six layers according to the optical images. The increased FLG in the Al matrix relative to the FGM design caused FLG to act as barrier in the structure. An increase in the Vickers hardness by 18% was observed in the last layer with FLG content of 0.7 wt% compared to the first layer. With this work, more detailed studies on the production and characterization of functionally graded materials for graphene reinforced Al or other metal matrix may increase.

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