Synthesis and characterization of AA 6061- Graphene - SiC hybrid nanocomposites processed through microwave sintering

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Abstract: As one of the most essential industrial and engineering materials, Aluminum alloy 6061 have been extensively used in automobile industries and many engineering applications due to its impending properties like low density, good structural rigidity, feasibility to incorporate and enhance the strength by addition of various reinforcing materials. The essential criteria in enhancing the properties without sacrificing the ductility is always challenging in Aluminum and its alloys based composites. In the recent years, enormous research has been carried on ceramic based and carbon based reinforcement materials used in Aluminum metal matrix composites. But the combination of both is never tried so far due to lack of processing methods. The current research work is carried out to process, synthesize and perform the characterization of Al 6061 matrix nanocomposites with Graphene of flake size 10 nm and SiC of particle size 10 µm as reinforcement combinations in various proportions (weight percentage) which are carried out through powder metallurgy (PM) approach. The powders are processed through ultrasonic liquid processing method and the mixtures were ball milled by adding SiC particles followed by uniaxial hot compaction. Thus prepared compacts are sintered (conventional and microwave) and mechanical properties like hardness, density are investigated as a function of Graphene and SiC concentrations (weight fraction). Relevant strengthening mechanism involved in the Al6061 – Graphene -SiC composites in comparison with monolithic Al 6061 alloy were discussed.

Key words: Graphene, SiC, Powder metallurgy (PM), Hardness

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1. Introduction

The research on the composite materials is one of the interesting topics in the material science and metallurgy and it have number of applications in various engineering and structural materials. In satisfying the demands on the materials properties requirement, lot of experimentation and the research has been carried out by known methods of combining various alloy addition, aging methods and heat treatment. Metal matrix composites (MMCs) are the potential candidate for current advanced engineering applications. Further keen attention given has been to improve their properties by incorporating advanced materials with novel processing techniques [1]. But, still the challenges related to metal matrix composites materials facing lot of issues including amalgamation, lack of processing methods, characterization and inhomogeneity for their wide range of applications in various sector [2]. On the other hand incorporation of a second reinforcing (particulate or fiber) phase in composites especially in metal matrix composites has been an effective route to enhance their strength and other physical properties for practical engineering applications [3]. Al 6061 is extensively used for automobile and transport materials; also, for a wide range of engineering applications due to superior physical properties with high strength. Also, Al 6061 posses corrosion resistance properties which makes it suitable candidate for marine applications. Lot of work has been carried out to synthesis Al 6061-SiC composites for the sake of specific engineering applications. But there is not much literatures found on hybrid composites in combination with carbon based additives and ceramic particles so far [4]. It needs advanced processing methods to achieve the isotropic properties in the developed composite material due to unusual behaviour of the materials during high temperature conditions [5]. In the last few years, the research on metal–carbon composites was done for the diverse range of engineering material applications and Fig.1 shows various molecular forms of carbon element in the same physical state (graphitic forms). Also, research shows the considerable improvement in the materials properties [6]. Among all other graphitic forms, Graphene has fascinated by its unique strength and properties in the last few years. Graphene endowed with excellent physical and mechanical properties such as Tensile strength 130 (GPa), elastic modulus (0.5 – 1 TPa) and thermal conductivity (5.3×10^3 $Wm^{-1}K^{-1}$) and has got attention in the field of research [7]. Also with the piolet studies on polymer and ceramic composites reinforced with Graphene proved to be effective reinforcing materials for the composites [8]. Compared to other physical form of carbon materials like graphite, CNTs and fullerenes, Graphene has been predictable to outperform due to its unique properties. Graphene have great potential in developing the nanocomposites when it is incorporated into matrix materials. Also, several processing methods are already used to fabricate ceramic – Graphene composite aiming to uniform dispersion [9]. An attempt is made to synthesis of Al 6061-SiC-Graphene composites by an innovative processing powder in liquid media through in powder metallurgy route (PM). The work is carried out to study the effect of the SiC & Graphene addition in the Al 6061-SiC-Graphene. Ultrasonic dispersion method is carried out to avoid the agglomeration of Graphene and to achieve the homogeneous dispersion in the matrix followed by ball milling by adding SiC. Thus prepared powder mixtures are compacted and sintered. The effects of SiC addition with different weight fractions are discussed.

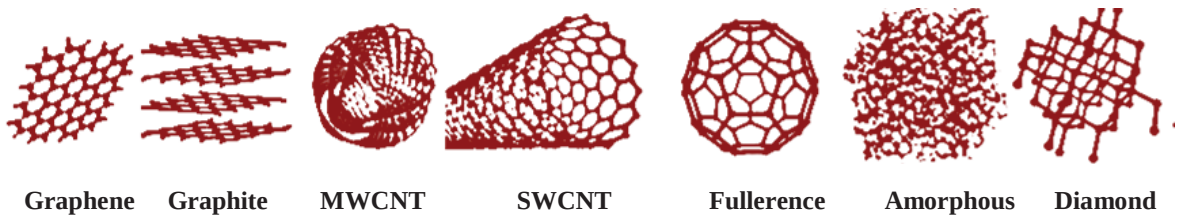

Fig. 1 Structure of allotropes of carbon

2. Experimental

2.1 Materials

Al 6061 powders which are produced through gas atomization method were procured commercially with a density of 2.7 g/cm^3. Its chemical composition is summarized in the table.1. Also, SiC particles with density of 3.20 g/cm^3 and Graphene with average platelets (flake) size >10 µm imported from USA with the density of the as received Graphene is 1.5 to 2.0 g/cm^3 are used in this research work.

2.2 Al 6061-Graphene- SiC blending

AA 6061- Graphene – SiC composites are produced through number of ways. At the first step, dispersion of Graphene is carried out through an ultrasonic dispersion method. SiC with various weight percentage (0.25, 0.5, 0.75 and 1.0 weight %) were added to solvent containing Graphene which is dispersed in acetone. Dispersion (ultra-sonication) is carried out until it turns to complete block solution indicating that no Graphene sediments were left in the beaker (shown Fig. 2a) also, all flakes are individually exfoliated. Further SiC – Graphene mixtures are dried in hot air oven and subjected to ball milling. Ball milling is carried out for 90 minutes with the ball mill ratio 15:1 at 150 rpm rotating speed for all the SiC - Graphene weight fraction precursor. Encapsulation and homogeneous dispersion of Graphene on SiC and homogeneity of mixture are achieved at optimized processing parameters. Al 6061 metal powder is added to ball milled SiC –Graphene precursors and ball milling process is continued with the same milling ratio for another 30 minutes. After this, the precursor (mixture of Al 6061-Graphene- SiC) is dried in hot air oven for 24 hours.

2.3 Compaction and Microwave sintering

AA 6061-Graphene- SiC powder (precursors) mixtures are preheated and compacted in single action C- 12 carbide dies with chromium tool steel die case according to ASTM B 925 – 08. The powder mixtures are preheated at 500 ˚C and compacted at 450 MPa with holding the compaction pressure for 15 min using high temperature lubricants such as boron nitride between the punch and die walls. Thus prepared compacts are (each comprising two sets) subjected to (conventional and microwave) sintering at 610 ˚C in an argon inert gas atmosphere for 30 minutes followed by furnace cooling to attain room temperature. The process followed to synthesis of Al 6061-Graphene-SiC hybrid composites are illustrated in the Fig.2 (a. b, c & d)

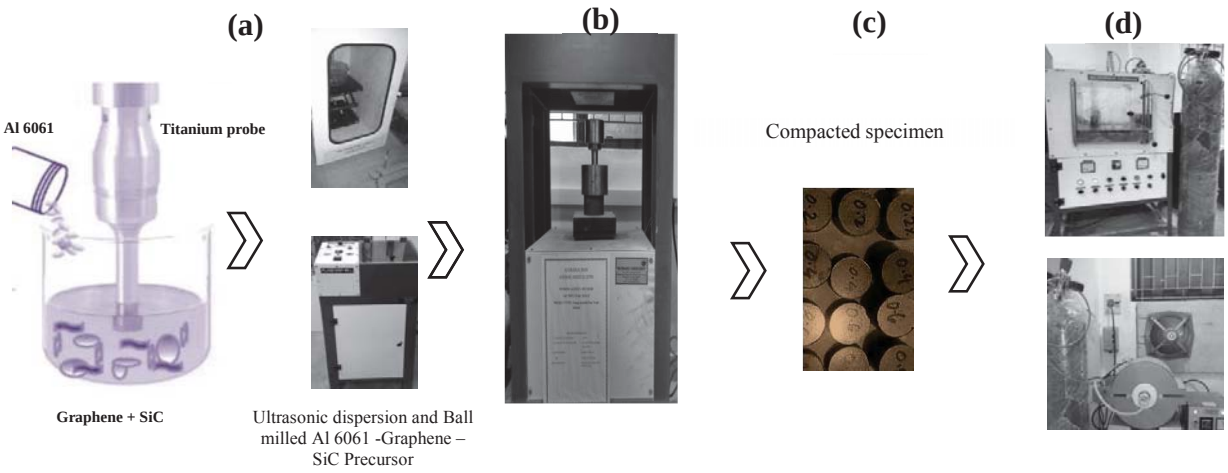

Fig. 2 (a) Powder processing through ultrasonic liquid processor and ball milling (b) Powder compaction in hydraulic press (c) Al 6061 - Graphene –SiC compacts (d) Microwave and conventional (Tubular) sintering with inert gas facility

Table.1 Composition of Al 6061 used as matrix material (wt. %)

Element	Si	Fe	Cu	Mn	Mg	Zn	Ti	Al	Others
(%)	0.81	0.72	0.35	0.15	1.50	0.25	0.15	Bal.	0.05

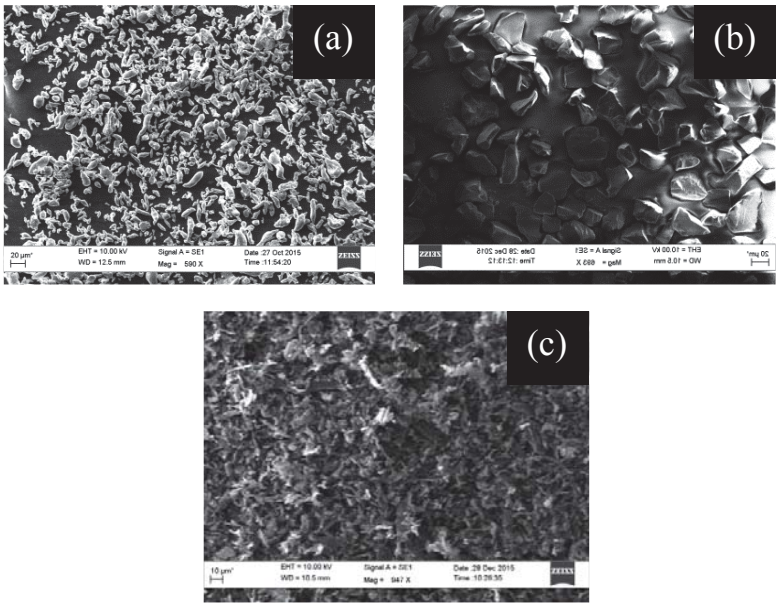

Fig.3 SEM image of (a) Al 6061 Powder morphology (b) SiC particle and (c) Graphene

3. Results and discussions

The morphology of as received Al 6061, SiC and Graphene are observed through scanning electron microscope. Fig.3a &b shows the SEM micrographs of Al 6061 and SiC respectively. Fig.2c shows the flake form, single layered of Graphene morphology in which the layers are stacked upon each other. The portion of the ball milled Graphene-SiC powder mixtures was analyzed under scanning electron microscope (SEM) to confirm the encapsulation. The X-ray diffraction patterns of AA 6061- Graphene – SiC hybrid composite is shown in the fig. 4. It is clear that the Graphene peaks are present at 2θ equal to 26.50˚ diffraction phase of Al 6061. The Aluminum carbide peaks are not detected in any of the microwave sintered hybrid sample of any of the composition. Graphene in aluminum alloy nano particles leads to formation of new peak in the composites, new peaks at 26.50˚ (Related to Graphene) which signifies the presence of Graphene in the developed composite (Crystalline carbon) . But the formation of aluminum carbide may occur below the level of sensitivity of the XRD apparatus used. The Graphene peaks are very low and only one or two peaks appears in the Al 6061 composites which may attribute to low content or sensitivity of measuring head of the Al 6061 matrix material.

From table.2, it is observed that the maximum measured apparent density (ρa) of Graphene under dry condition is found (0.088±0.0020 g/cm^3) to be lesser compare to other allotropes of carbon such as CNT and graphite also it's governed by the flow rate. Tap density after specified number of taps (300 taps) obtained by relation (1) is appeared to be higher compared to Graphite is attributed to alignment of sheets on a single plane themselves by sliding on each other via providing more packing fraction and it be subject. Also, it depend on the aspect ratio and surface morphology. Apparent density of the Graphene and Al6061 is measured according to ASTM B703-10 using Arnold density meter and tap density is carried out according to ASTM B527-14 by tapping apparatus giving 250 taps under dry conditions. Vickers hardness values are measured for all Al 6061-Graphene- SiC composition samples using 200 gf

$$\rho t \text{ (Tap density)} = \frac{Mt}{Vt} \text{ (g/cm}^3\text{)} \qquad \textbf{(1)}$$

(*Where, M_t = mass of powder, V_t = volume of tapped powder*)

Table 2 Measured Apparent and Tap density values

(g/cm^3)	Apparent Density (ρ_a)		Tap Density (ρ_t)
	Max	Min	
Graphene	0.088±0.002	0.071±0.002	0.1288±0.02
AA 6061	1.118±0.05	0.95±0.05	3.68±0.05
SiC	2.600±0.06	1.9±0.04	3.83 ± 0.03

after fine polishing of the samples. The mean values of at least five measurements from different areas on the composites sample were taken for analyses. The comparison has been done for the composite which are sintered by two methods (conventional and microwave) and the same measured values are projected in Fig.5. Silicon carbide (SiC) possess a high dielectric properties which are very much suitable candidate and have very high affinity to absorb the microwave compare to other type of ceramic particles even at its critical temperature[10]. In AA 6061-Graphene – SiC composites during solid state sintering, material transport mechanism take place through diffusion mechanism that occurs due to vacancy concentration gradient which is depends on chemical potential gradient and the temperature. For the effective grain, large number of high energized movement of atoms should be there at the vacancies but this movement is inhibited by Graphene by creating barrier against grain growth by wrapping. So, densification increases due to increasing in the diffusion rate and it is dependent on sintering temperature and it can ratify by the Arrhenius equation shown in equation no 2. Fig.5 shows the hardness values of Graphene reinforced composites which are superior to the Al 6061 monolithic compacts. It indicates that, addition of SiC through encapsulation with the Graphene at lower concentrations (< 1 wt. %) homogeneous dispersion in the matrix is achieved. The increase in the hardness also expected to be the formation of aluminum carbide in the composite but XRD data reveals that no such peaks to confirm Aluminum carbide formation [11]. This is attributed to usage of SiC encapsulation and microwave sintering with no defects in the development of composites. Further, increase in the hardness is due to better densification, reduced pores which are sintered using microwave sintering method also yields the better densification.

$$N = N_{\circ}\, e^{-q/RT} \qquad (2)$$

(Where, N- Number of vacancy sites, $N_{\circ}$-Total number of lattice sites, q - Activation energy, RT- Average kinetic energy)

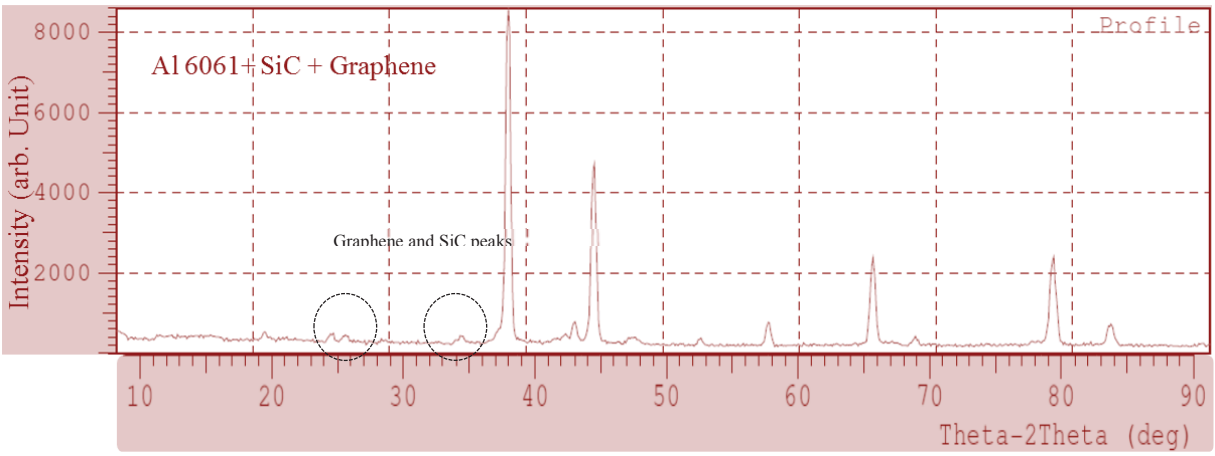

Fig.4 X – ray diffraction analysis of Al 6061-SiC- Graphene composite after microwave sintering

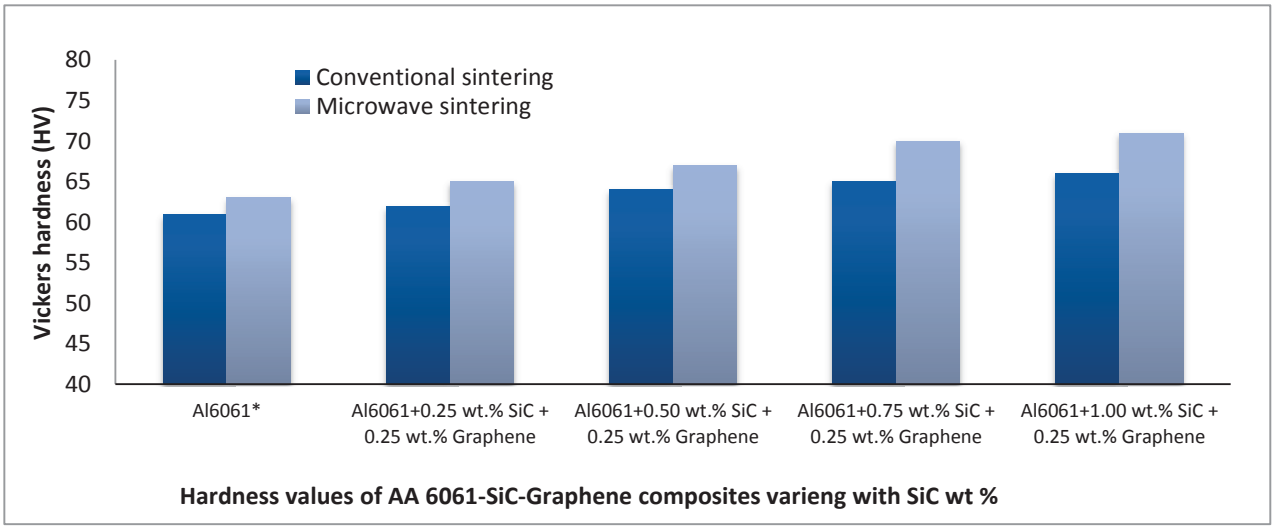

Fig.5 Hardness with varying SiC content in AA 6061- Graphene – SiC

4. Conclusions

In this study, AA 6061-Graphene- SiC composites were processed by ultrasonic liquid processing of Al 6061 particles and Graphene followed by SiC (Varying the SiC concentrations) by ball milling successfully. The precursor are preheated and compacted in the liquid-solid semi state. Finally, the compacts are sintered in conventional and microwave sintering method successfully. XRD analysis has revealed the existence of all major alloying elements of Al 6061 including Graphene (peak at 26.50˚) in the developed composite and no carbide formation at the surface. The micro hardness values are seen better yielded in microwave sintering method compared to conventional method. It can be concluded that, addition of SiC and encapsulation in combination with microwave sintering resulted in the increase hardness values and densities for the developed composite.

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