Fabrication of Pure Aluminium Reinforced Graphene Composite for Heat Sink Applications

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Abstract. Technology is growing fast and as a result of this, the performance of electronic devices is rapidly increasing. These devices generate heat as current flow through them and this heat energy has to be safely managed and dissipated which is achieved with the help of a heat sink. Amongst the heat sink materials, graphene reinforced metal matrix composite is emerging as an alternative promising high thermally conducting material and an effective material for proper heat management. In the current work, Metal matrix composites based on pure aluminium reinforced with graphene powder are fabricated using stir casting method. Thermal and mechanical characteristics studies were performed on the stir cast composite specimen. Certain parameters like argon for stirring, processing temperature (820°C), stirring time (5-10 sec) and graphene content (10 volume% of pure aluminium added) were considered for the experiment. The enhancement in heat conduction of the test samples were confirmed by the wear testing method. High rate of heat conduction and comparatively low wear rate were observed in aluminium-graphene composite rather than pure aluminium as received sample. The test samples were analysed using optical microscope, SEM and EDS. Thus, this paper present insight in to the fabrication of Aluminium-graphene composite by stir casting method. In addition, thermal and mechanical characterizations of the fabricated samples are also examined.

Keywords. Heat Sink, aluminium-graphene composite, stir casting.

1. Introduction
Today due to technical developments there is great need of materials which have high efficiency in working, light weight and more durable. Thermal management material has been of significance in the electronic devices. The development of electronic devices with higher calculating speeds within a more compact size leads to more heat generation per device. In order to meet thermal dissipation requirements for electronic packaging applications, materials with high thermal conductivity and low thermal expansion must be developed. Carbon/metal composites are currently used in several applications [1].

The need for high thermal conducting materials in heat sink applications has led to the development of Aluminium matrix composite. Graphene as a reinforcement is used in many applications because of many factors like low density, good mechanical properties and thermal properties, etc. Graphene is a single layer structure and cause conduction by n bonded electrons which are free to move throughout graphene sheet and through lattice vibration through the σ bonds between the carbon atoms. Thermal conduction is due to vibrating atoms in sp2 hybridized σ bonds which has compact bonding to have good conduction through thermal vibration. Today’s technological development is concentrating on devices with small size and better efficiency. Therefore, their wider use in components requiring good thermal management is quite possible in near future.
Conventional aluminium used for heat sink has a thermal conductivity of around 235 W/mK. Aluminium-Graphene composite in which graphene powder is reinforced enhances the thermal conduction. Thermal transport in graphene is a thriving area of research, thanks to graphene’s extraordinary heat conductivity properties and its potential for use in thermal management applications. The measured thermal conductivity of graphene is in the range of 3000-5000 W/mK at room temperature. The thermal conductivity of graphene increases logarithmically and researchers proposed that this is due to the stable bonding pattern as well as being a 2D material. As graphene is considerably more resistant to tearing and is also lightweight and flexible, its conductivity has some attractive real-world applications [3]. Also, besides good thermal conductivity, Aluminium-Graphene composite can be used in aerospace and automobile industries due to their low density, high strength and good corrosion resistance. Thus, this paper present insight into the fabrication of Aluminium-Graphene composite by stir casting method for heat sink applications.

### Table 1. Properties of Aluminium and Graphene

| Properties                  | Aluminium | Graphene  |
|-----------------------------|-----------|-----------|
| Density (g/cm³)             | 2.7       | 0.2-0.5   |
| Thermal Conductivity (W/mK) | 237       | 5300      |
| Thermal expansion coefficient (µm/mK) | 23.1     | -0.8-0.7 |
| Young’s modulus (GPa)       | 70        | 1000      |
| Tensile strength (MPa)      | 90        |           |

### 2. Experimental

#### 2.1. Casting of specimen

For the casting process, pure Aluminium with graphene equal to 10 vol% of Aluminium is added. Graphene added is provided by Graphene Development cell, Tata steel. The composition of Graphene is 91-94% Carbon, 4-5% Oxygen, 0.1-0.3% Sulfur, 0-1% Hydrogen. Graphene powder is in the form of grey flakes. Induction furnace need to be used. Processing temperature should be kept at 820°C with stirring time 5 to 10 sec, stirring speed 400 rpm and holding time after addition of graphene to be 10 minutes [4],[6].

#### 2.2. Sample preparation

Sample planning was prepared in order to determine the number of samples for experimentation. Samples were cut from the rod using abrasive wheel cutter with proper cooling arrangement so as to avoid excessive heat generation which might result in any structural changes in the material. Cutting was done to get samples with precise dimensions required for different tests. For hardness and microstructural characterization, the diameter to height ratio was kept less than or equal to unity for maintaining accuracy in the measurements. For wear sample of diameter 10mm and height of 15mm were selected and hole was drill at height of 3mm on pure aluminium and composite of Graphene reinforced pure aluminium. The machined samples for tensile testing were solutionized to lower the residual stresses generated, solutionizing of the machined samples are done. Solutionizing is done in a muffle furnace at a temperature of 500°C for 2 hours. Solutionized samples are then water quenched.

#### 2.3. Metallography

For the microstructural analysis, first belt grinding on the specimen was done followed by polishing on emery papers with grit size 400, 600, 800, 1000 and 1200 respectively. Then the final polishing was done on lapping machine on velvet cloth with brasso powder. Kellers reagent was used as etchant to reveal the microstructure. Microstructures were observed under image analyzer (Make: Conation Technologies, Model - SuXma-Met I). Scanning Electron Microscope (SEM) using FE-SEM (make – Carl Zeiss, model - Sigma HV) was also used for further investigation of microstructures.
2.4. Wear test
The wear test of the specimen was carried out on Pin-on-disc machine as per ASTM G99. Sample diameter and height should be 10 mm and 15 mm respectively. Hole was drilled on sample at 3 mm height from bottom surface to measure the temperature rise with time in the samples. Stationary pin is allowed to slide a distance of 500 mm against a counter surface of cast iron with a hardness of 526 HV. The sliding distance need to be 2 m/s. A normal load of 4 kg need to be applied. The test need to carry out in an ambient condition of 25°C. The loss in weight of sample need to measured using the digital weighing balance. A comparative study of wear rate of pure Aluminium and Aluminium reinforced with Graphene need to be done. The temperature rises of the aluminium sample and the aluminium graphene sample is compared to see the heat transfer difference.

2.5. Tensile test
The tensile test of the specimen was performed using the Universal tensile testing machine as per ASTM E8, IS1608. Initially, a strain rate of 2 mm/min need to applied for performing the test. Specimen prepared should have a gage length of 30 mm with distance between the shoulders to be 36 mm and a diameter of 6 mm. Tensile strength will be noted by taking three readings.

![Figure 1. Tensile test sample as per ASTM E8](image)

2.6. Area Fraction Analysis
Area Fraction of graphene in aluminium matrix was calculated by using Image J software.

2.7. Macrostructural analysis
For macrostructural analysis of samples, a solution of 50 gm NaOH and 500 ml H2O is prepared. This prepared solution is heated on a heating plate between 60-70°C. Then the samples are immersed into the solution for 5-15 min. After 15 minutes, samples are removed from the solution and rinsed in flowing water. Black smut present on the surface is removed by dipping the samples in conc. HNO3 solution for few seconds. The samples are then analyzed.

3. Results and Discussion

3.1. Comparison of thermal transfer through samples
The thermal transfer through samples were analysed by placing the thermocouple at the height of 3 mm from the wear surface. The temperature was noted for each sample for the comparing the heat transfer through the sample. Figure 2 shows the comparison of the temperature rise with time on the same height of sample by keeping all parameters same for every sample during wear. It is observed from the Figure 2 that the temperature rises in case Al-Gr sample was slightly more as compared to that of the Al sample. Thus, this result indicates that adding Gr nanoparticle increases the thermal conductivity of Pure Al sample. Thus, providing the composite material with high thermal conductivity and light weight material.
3.2. Wear behaviour of fabricated samples

The average of wear rate of samples showed that the wear property is similar in both Pure Al and Al-Gr but with slight rise for Al-Gr samples. The wear resistance of metallic materials, in particular aluminium and aluminium alloys, are key features that are considered in the designing the materials. Here, a slight increase in the wear rate of Al-Gr samples is observed due to weak interfacial bonding between aluminium matrix and graphene. Figure 3 shows the comparison of average wear rate of Al and Al-Gr samples.

3.3. Microstructural analysis

The morphology of the microstructure of all specimen was observed under optical microscope after surface preparation and etching them in Keller’s reagent. The microstructure shows white phase as Al matrix and black phase of Gr. The region surrounding the graphene oxide powder shows the refined...
grain structures with micro cracks surrounding it due to stress generation. At some location Gr thin black phase at the grain boundary region was seen.

**Figure 4.** Microstructural images of the Al-Gr composite: a) shows grain refined region around Gr particles b) shows the presence of black graphene at the grain boundary region.

SEM images were taken using FE-SEM (make – Carl Zeiss, model - Sigma HV). The images revealed the Graphene phase clearly it was seen that there was not strong interfacial bonding between the graphene and the Al matrix. EDX analysis shows the presence of carbon, Elemental mapping was done to have clear overview of the carbon in sample, this showed the presence of Graphene.

**Figure 5.** SEM image showing Gr particle in Al matrix and EDX analysis of the region
3.4. Macrostructural analysis

The samples were analyzed using the macrostructural analysis technique. During the analysis, Al-Gr sample showed porosity in the samples. It was due to the addition of the Gr particles and weak interfacial bonding between Gr and Al matrix during casting process. Lower wettability between the Gr and Al is the cause of weak interfacial bonding. Due to light weight of Gr during adding the Gr in molten Al caused the problem of complete mixing of Gr with Al matrix and also caused the generation of porosity.

![Figure 6. samples after Macrostructural analysis a) Pure Al sample b) Al-Gr sample](image)

3.5. Area fraction analysis

Area fraction analysis was carried out using image J software. From this Area fraction the weight % of Graphene present in the sample was calculated. Average Area fraction of Gr from the different SEM images analyzed on Image J was 0.06076. Further by assuming Area fraction and Volume fraction to be similar the weight % of the graphene present in the sample was estimated and it was 1.183 weight %. But during the addition of Graphene, it was 10 volume% that is 1.85 weight %. Thus it is observed that there is decrease in the actual Gr content in the sample than that was added. This decrease observed is 36%. This decrease is due to combustion of the Gr in presence of atmospheric oxygen at high temperature when Gr came in contact with the hot molten Al.

3.6. Tensile property comparison

Mechanical properties are the major factor in designing the material. In order to check the mechanical property of the sample tensile testing was performed. The tensile test of the specimen was performed using the Universal tensile testing machine as per ASTM E8, IS1608. The results of test are shown in Table 1. There is a reduction of 4.95% in the ultimate tensile strength of Al-Gr material as compared to pure Al. The weak interfacial bonding between the Gr and the Al matrix, the generation of stress region around the graphene particle and the micro porosity in the sample of Al-Gr were the reason for the decrease in the tensile strength of the Al-Gr material compared to pure Al material.

![Table 2. Comparison table showing the tensile test results of Pure Al and Al-Gr composite](image)

| SAMPLE | SAMPLE NO. | UTS (MPa) | Y.S.(MPa) | %ELONGATION | %REDUCTION IN AREA |
|--------|------------|-----------|-----------|-------------|-------------------|
| Al     | 1.         | 131.608   | 113.991   | 6.667       | 5.523             |
2. 94.080  82.452  5.417  2.973
3. 64.268  51.625  4.583  2.968
AVG 96.652  82.689  5.555  3.821

|     | 1.   | 2.   | 3.   | AVG  |
|-----|------|------|------|------|
| Al+Gr |      |      |      |      |
| 1.   | 115.127 | 105.707 | 6.375 | 2.958 |
| 2.   | 88.961  | 78.495 | 1.042 | 3.284 |
| 3.   | 71.504  | 58.032 | 2.083 | 3.915 |
| AVG  | 91.864  | 80.744 | 3.166 | 3.385 |

**Figure 7.** Graphical representation of comparing UTS of each sample and there average.

4. **Conclusion**

From the above results and discussion, the following conclusions can be drawn from this work:

1. In wear analysis, it is seen that temperature rise of pin is higher for Al-Graphene composite than pure Aluminium sample. Due to high heat transfer the temperature has reached higher level in shorter duration for the composite sample. This proves that the thermal conductivity of the pure aluminium has increased due to addition of graphene in reinforcement.

2. The analysis of microstructure showed the Gr distribution in Al matrix along with a stressed region in the vicinity of Gr particles. Graphene was present superficially in matrix without strong bonding between the Gr and Al due to the low wettability of Gr thus decreasing the mechanical property.

3. Light weight material composite can be prepared by adding the Gr as reinforcement in the Al matrix. However, low density of Gr was causing the problem for fabricating the composite and thus homogenous distribution of Gr in whole matrix is difficult task to be achieved.

4. Combustion of the Gr as soon as it was added in the hot metal in presence of Atmospheric oxygen because the reduction in Gr content by 36% than that was added. This is because graphene starts to burn at 350°C due to large interlayer spacing of graphene sheets. So, it gets
combusted as soon as it was added into liquid metal in presence of oxygen. Carbon in graphene reacts to give CO and CO₂. So, an inert atmosphere can be created to avoid graphene combustion.

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