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Preparation and Characterization of Al-Cu-Al_2O_3/Gr Nano Compound by Metallic Powder Method and Study of the Effect of the Rolling Process on Physical Properties as Suitable Materials for Heat Sink

Fathei. Nouh, T. El-Bitar, Hossam M. Yehia, Omayma El-Kady

***Abstract** Al-Cu-Al_2O_3 / Gr Nano compound was successfully prepared by metallic powder method, 6 samples were prepared with different weight ratios of graphene, 10% copper and 2.5% Al_2O_3 and proportions of 0.5, 1, and 1.5 graphene were prepared, the samples were run in a mill from Ceramic in a ratio of 1:5 powder into balls for 35 hours, which helped to break the aluminum and copper particles and reduce their size, which helps in the process of homogeneous mixing of the compound. The samples were compressed at 60 bar pressure and sintered in a vacuum at 550 and 565 degrees Celsius for 60 minutes. Sintering at 550°C proved to be more suitable for the mixture. Two identical sets of samples were prepared. Both SEM were used to investigate the microstructure and components of the sintered nanocomposites. Relative density, hardness, electrical conductivity and thermal conductivity study. The rolling process of the samples was carried out with a percentage of 35% of the sample size successfully at a temperature of 480 degrees Celsius, and this led to an improvement in the density of the samples and hardness and an increase in the diffusion of the reinforces, which led to an improvement in electrical conductivity by 16: 25% and a better improvement in thermal conductivity. Improved up to 1.5% graphene by weight after rolling and 1% graphene before rolling.*

***Keywords** Powder technology, forming, aluminum ingot, metallization, Graphene nano-sheets, Electrical properties, Hardness, hot rolling, Thermal properties*

I. INTRODUCTION

In light of the progress and the increasing need for the requirements of societies, there is more demand to significantly increase the number of working hours for electronic devices, which affects their efficiency due to the high temperatures resulting from the high heat flow, which is produced as a result of present restrictions at work [1]. Computers, laptops, and large movie screens rely on heat sinks to keep internal components from damage caused by high temperatures and get rid of them quickly [1]. Heat sinks keep electronic parts at ambient temperature and prevent them from heating damage, allowing them to operate at peak efficiency. This necessitates an efficient cooling system that can dissipate heat in a small space. They are used very effectively as fins for air conditioning units and engine bodies, and therefore it is required that the heat sinks have a great thermal conductivity efficiency, Many different sorts of effective cooling systems must be supplied in order to quickly and safely drain heat from the working area of electronic components. [2] [3]. The heat sinks are designed in the form of fins, usually produced from aluminum, using a rolling and casting process, and aluminum fins are used to increase the surface area used for heat dissipation. For example, the processor in a computer's control processing unit (CPU). [4]. Heat sink materials are among the most affordable and widely utilized. Heat sink materials must have specific physical features, such as high thermal conductivity and low coefficients of thermal expansion (CTE). The most common materials used for this are copper and aluminium. Aluminum has a high coefficient of thermal expansion, which contributes to its high thermal conductivity (CTE) [5]. Many studies aimed to improve aluminum's mechanical characteristics. especially the dispersants used in engine bodies, which slightly reduced the physical properties, because aluminum alloys were reinforced with materials such as tungsten that have lower physical properties [6] [7]. In this study, ceramic materials such as graphene were reinforced to improve the mechanical and physical properties together, in order to produce a heat sink that has the ability to work in more extreme conditions. Graphene is a two-dimensional carbon strip with outstanding mechanical strength, high thermal and electrical conductivity (5300 W/mK), moderate thermal expansion, and a large specific surface area (SSA-500-1200 m2/) that makes it an effective reinforcing material. [nine] Graphene's influence The effects of nano sheets on copper's physical and mechanical properties were investigated. The tensile strength of graphene-reinforced composite materials was up to 500 times that of the basic materials.

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It also improves electrical as well as thermal conductivities [8,9]. Graphene's impact on magnesium metal The mechanical properties of matrix nanocomposites were studied, and the results revealed a homogeneous dispersion of multilayer graphene in the matrix [10]. Graphene as a ceramic material is not wet and does not react with aluminum, and the rolling process was used to improve the mechanical properties and increase the spread of graphene sheets, which increases their efficiency, which enables us to produce high-quality heat sinks that can work more efficiently [11]. The goal of this research is to improve the bonding between graphene strips and Al_2O_3 granules with pure aluminium, as well as to investigate the impact of the rolling process on graphene diffusion, microstructure, density, hardness, and electrical and thermal conductivity of aluminium '[12].

II. PROCEDURE

A. Material

Graphene strips with a thickness of (2-10) nanometers were prepared by YSS Company in the United States of America. For the surface cleaning, distilled water and acetone were utilized. The materials used were stirred for an hour in acetone and an hour in distilled water, and aluminum oxide granules with a thickness of 10-15 were used. Nanometers provided by El Nasr Chemicals Company - Egypt, and copper from El Gomhouria Company - Cairo was used. And aluminum powder was used from the Turkish company TCSTH with a purity of 99.9%, and the powders were ground through the Metals Research and Development Center – Cairo.

B. Sample preparation

The surface of the graphene sheets, Al2O3, aluminum and copper granules, was prepared and cleaned of any impurities that might be present as a result of the first manufacturing process of the powders, by stirring the powders in acetone and distilled water for 60 minutes and drying in an electric oven at 70 degrees Celsius for 120 minutes. Samples were weighed from metallic powders of aluminum as base metal, adding 10% copper and 2.5% Al_2O_3 and adding graphene in proportions of 0.5, 1, and 1.5% from graphene strips, and the powders were ground through a mechanical ball mill at a ratio of 1:2 for 35 hours to ensure the distribution of Homogeneous components of the compound reduce the size of the aluminum and copper granules, and hexane liquid was used as a lubricant to prevent the adhesion of the graphene nanometer strips to each other and the internal grinding cylinder body. The graphene sheets and Al_2O_3 granules were coated with silver by 5% by dissolving sulfur nitride in distilled water after adding ammonia liquid until it reached PH = 11 or a little more, then adding graphene sheets and Al_2O_3 granules respectively and adding Formaldehyde liquid to catalyze the reaction and the proportions were added through Table 1. Finally, the powders were filtered off, and then dried in an electric oven at 60 °C for 2 hours.

Table 1: Chemical composition bath of Metallization graphene sheets and Al_2O_3 deposition

Materials	Weight
Powder metal	---
sulfur nitride	3g/l
ammonia liquid	500ml/l
Formaldehyde	200ml/l

Composite powder samples were pressed at 60 bar using universal hydraulic pistons and a 10 mm diameter circular pump. It was sintered in a vacuum oven at a temperature of 550 and 565 degrees Celsius in an oven for 60 minutes with a heating cycle as shown in Figure (1). The samples were heated at a rate of 3°C/min up to 250°C and was held for 15 minutes to ensure the exit of gases and temperature regulation due to the total sample size. The temperatures were raised at a rate of 2 degrees / minute to 550 degrees Celsius and 565 degrees Celsius, and it was verified for a period of 60 minutes, and it was sufficient for the sintering process in those conditions, and a small room was used inside the oven and the samples were placed inside it, and the aim was to regulate the temperature regularly on the sample.

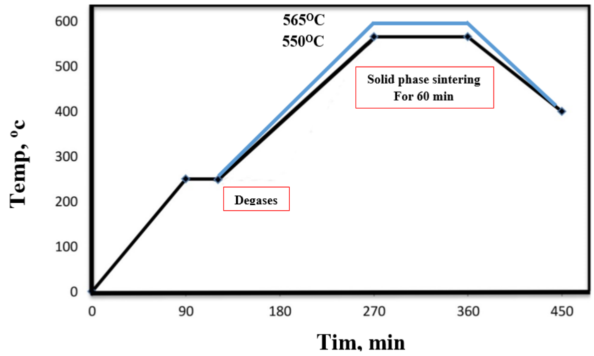

Fig 1. Sintering cycle curve for Al-Cu- Al2O3 /GNs Nano composites at 550 and 565 °C for 60 min

A sample holder was prepared from pure aluminum free of any reinforcing materials to have a lower strength than the prepared samples, and it was tightly fitted inside by preparing a hole with a diameter of 10.05 mm and pressing the samples into the holes as shown in Figure (2). , The length of the holder was 30 cm and the height was 10 mm the same as the height of the cylindrical samples and the samples were formed between a pair of rollers with a diameter of 20 cm at a speed of 45 rpm, an electric furnace was used to heat the samples and raise the temperatures at 480 degrees. The thickness of the samples was reduced at a rate of 0.2 mm for one cycle, and it is taken into account to reheat the samples after each cycle of the rolling process. The high temperature of the rolls was taken into account by passing warm holders through them just before the rolling process to reduce the heat loss rate of the samples during formation. 3.5 mm of the sample height was safely reduced without cracks or fractures.

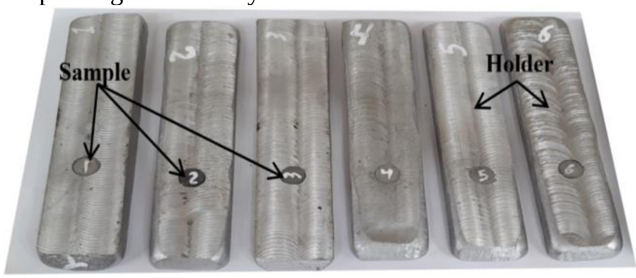

Fig 2. Sample preparation by a pure aluminum holder for the rolling process

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III. ANALYSIS AND CHARA CTERIZATION

Archimedes' principle according to the standard ASTM B962-14 is used to evaluate the density of all the sintered composites. Measurements are performed at room temperature with distilled water as floating liquid.

$$\rho_{Arsh.} = \frac{W_{air}}{W_{air} - W_{water}} \rho_{liquid}, g/cm^3 \qquad (1)$$

The relative density (Rd) is calculated by using the following equation:

$$R_d = \frac{\rho_{Ar}}{\rho_t} \rho_{liquid} \qquad (2)$$

Where ρAr is the Archimedes density, ρt is the theoretical density, and liquid is the liquid density. Theoretical density ρth of the composites were determined by using the following equation:

$$\rho_{th.} = \rho_1 wt.\%_1 + \rho_2 wt.\%_2 + \rho_3 wt.\%_3 + \ldots\ldots \qquad (3)$$

Where (ρ1) and (wt. %1) are the density and the weight percentage of the matrix element, respectively, (ρ2) and (wt. %2) are the density and the weight percentage of the first reinforcement element respectively and (ρ3) and (wt. %3) are the density and the weight percentage of the second reinforcement element respectively ……...

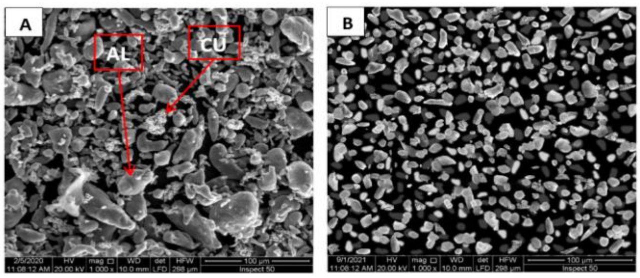
The surface of the samples was prepared by polishing them with silicon carbide paper with a grain size of 800, 1000, 1500 and 2500, respectively. The microstructure and morphologies of the sintered composites are studied by using Quanta FEG250. X-ray energy dispersive spectrometer of the model D8 XPORT is used to analyze the phase composition and crystal structure of the prepared composites. Vickers macro hardness tester model 5030 SKV England is used to measure the hardness of the sintered composites. 5 kg load and 15 sec loading time are the suitable parameters for the test. The average of five readings for each hardness value was calculated. Electrical conductivity at room temperature for all sintered composites is measured by using PHYWE SYSTEME GMBH. 37070 Gottingen, Germany. In order to determine the thermal conductivity values, equation of Weidman- Franz was used.

$$\frac{\lambda}{\sigma T} = \frac{\pi^2 k_B^2}{3e^2} = L = 2.443x10^{-8} w\,\Omega / k^2 \qquad (4)$$

Where, λ is thermal conductivity (W/mK), σ is electrical conductivity Ω.m -1, T is absolute temperature in degree Kelvin (293 k), KB is Boltzmann constant, and L is Lorentz number

IV. RESULTS AND DISCUSSION

A. Powder Characterization



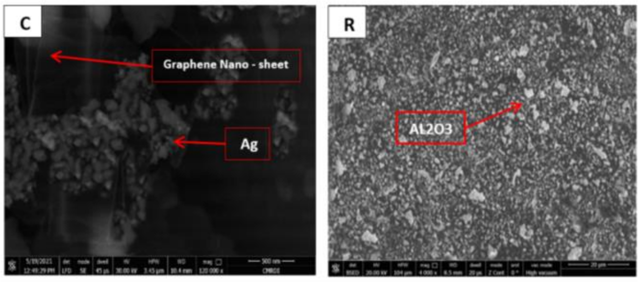


Fig 3, (A) SEM micrograph of (B) Al-Cu-Al2O3/Gr mixture, (C) silver-coated graphene sheets, (D) softened Al-Cu-Al2O3/Gr mixture, (R) Al2O3 powder

Micrograph of Al-Cu-Al_2O_3/Gr powder and graphene plates coated with 5% silver with high rudder as shown in Figure 3. And the picture (a) shows the aluminum and compound particles. They are semi-circular and irregular. The copper granules appear in their tree shape. We see sheets of graphene and Al_2O_3 on the aluminum granules, and they are small in size. And the picture (b) shows graphite slices coated with silver, and we see in the picture (c) the particles of the mixture after the grinding process for 35 hours and it affected the particles significantly and the size of the particles was reduced, which helps in improving the product significantly, and we note that none of the powder particles stick together as a result Effective lubrication process during the line resulting from the use of hexane liquid.

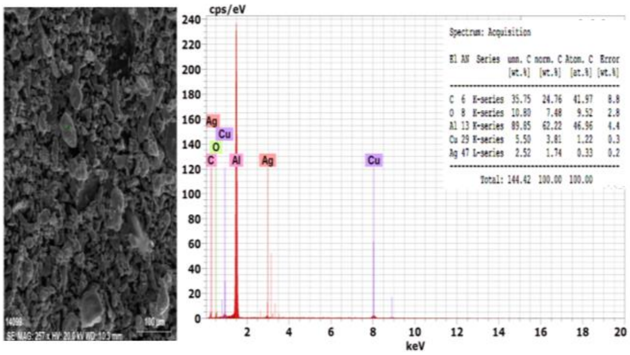
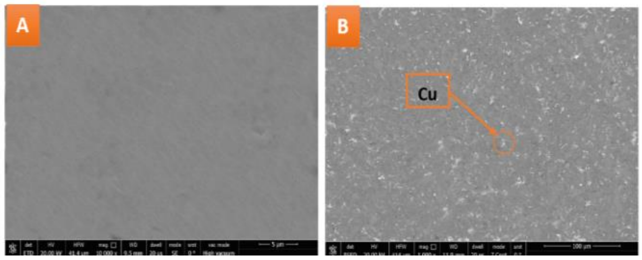


Fig 4. Shows the EDAX analysis of the elements of the Al-Cu- Al_2O_3/Gr compound after mixing

It is noted from the analysis that the sample includes aluminum and copper, as it shows a button of carbon, and this is evidence of the presence of graphene, as it is clear that there is a button of oxygen resulting from Al_2O_3, and this confirms that none of the components of the compound is affected by the mixing processes.

B. Microstructure



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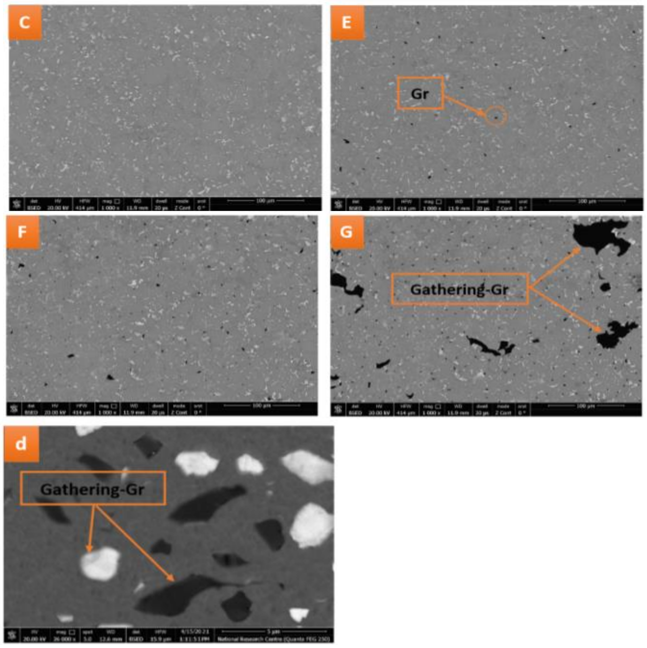

Fig 5 shows the SEM microstructure of the Al-Cu-Al_2O_3/Gr composites, photo (a) aluminum, and photo (b) aluminum-copper-reinforced, photo (c) The Al-Cu composite was reinforced with Al_2O_3, photo (d) was added Composite graphene

Figure 5 SEM shows the microstructure of the sintered samples of Al-Cu- Al_2O_3 / Gr sintered at a temperature of 565 ° C in a completely evacuated atmosphere, and the first image shows the shape of the aluminum in a dark gray balloon, and it is noticed that there are no voids in the sample before the reinforcement process, and gray areas appear Light represents copper and appears to be well and uniformly distributed as a result of grinding processes using a lubricant for long periods, as it was observed that Al_2O_3 particles began to appear in the picture (C) well distributed and the graphene distribution appears completely homogeneous, which means the grinding process using the lubricant liquid is perfectly suitable for the distribution of all Elements of the sample, and some concentrations of graphene began to be observed at the sample of 1.5% despite the good distribution due to the low density of graphene and consequently covering a large amount of it parts of the sample, which leads to accumulation in some areas as shown in the picture (S). In addition, good adhesion was observed between graphene sheets and copper sheets with aluminum as shown in picture (H).

C.Density Measurement

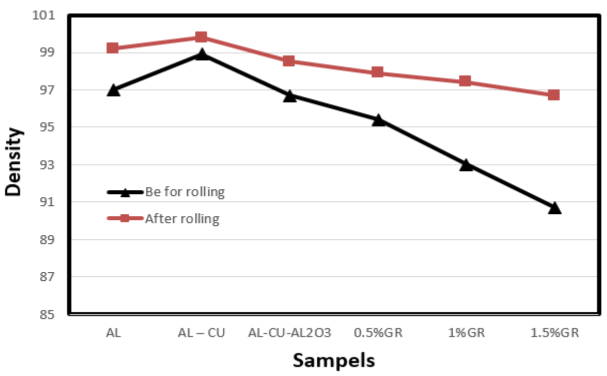

Fig 6, shows the density of samples sintered at 550°C before and after the rolling process

Figure (8) shows the density of the sintered samples at a temperature of 550 degrees Celsius, and it appears from the figure that an excellent density of the samples was obtained as a result of the good adhesion of the matrix particles. The density is improved by adding copper element due to its higher density than aluminum, and the density gradually decreases after adding graphene due to the low density of that Element. The density decreases with the increase in the proportion of graphene in the samples and this is logical. The rolling process led to a significant improvement in the densities, eliminating any voids that might be present inside the samples, and led to the cracking of the large-sized matrix particles and turning them into a fine structure, and the high temperatures during the rolling process helped to Removing the voids inside the samples, and the sliding of the graphene strips increased the bonding between them and the aluminum, which improved the densification process significantly.

D.Hardness Measurement

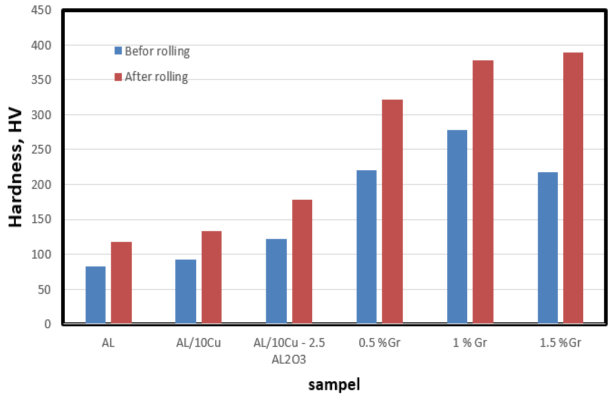

Fig. 7 hardness measurements of the nanocomposites before and after rolling

Figure (7) shows the hardness of the sintered samples at a temperature of 550 degrees Celsius for an hour in a vacuum of oxygen before and after the rolling process, and the results show a significant improvement in the hardness as a result of large grinding operations for 35 hours, which led to the crushing of the particles and reducing their size and thus obtaining stronger samples. The addition of reinforcement materials led to the continuation of the improvement process, and this is a natural result of its mechanical properties being superior to that of aluminum. The addition of the graphene element also led to a better improvement of the percentage of up to 1% by weight of graphene, and then began to decrease due to the presence of some aggregations of graphene, and the rolling process led to the emergence of a fine structure and improved better distribution of the matrix components, and to cracking Al_2O_3 particles and increasing their distribution within the matrix More, and the occurrence of slips of the aggregated graphene strips especially in the sample by 1.5% graphene by weight, which led to an improvement in the hardness of 1.5% graphene by weight.

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E. Electrical Conductivity Measurements

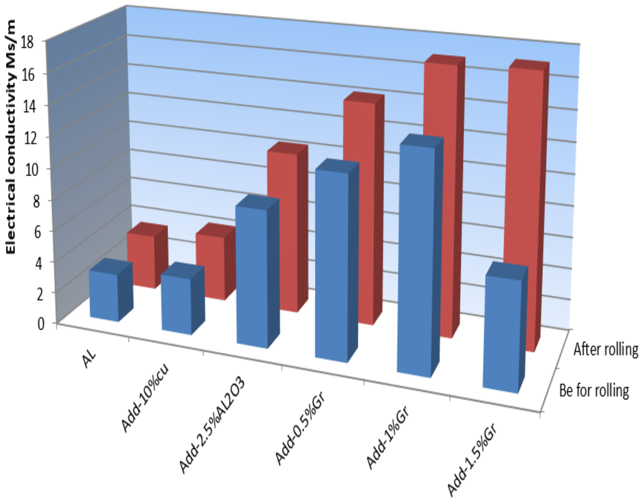

Fig 8, Electrical conductivity of Al-Cu-Al2O3/Gr nanocomposites before and after the rolling process.

Figure (8) represents the effect of the reinforces and the rolling process on the electrical conductivity of the Al-Cu-Al_2O_3/Gr composite. It is clear that the electrical conductivity gradually increases by increasing the graphene content up to 1 wt. %. The results showed that adding 10% by weight of copper improved the electrical conductivity from 3.1 Ms/m to 3.6 Ms/m due to the higher electrical conductivity of copper.

The electrical conductivity of the compound was not affected as a result of adding Al_2O_3 due to the low amount added which is 2.5%, and the silver plating process, which has a high electrical conductivity, also helped prevent the effect of Al_2O_3 on the other hand, adding 1 by weight. % Gr leads to an increase in the electrical conductivity of the Al-Cu- Al_2O_3/Gr Nano composite to 13.62 Ms/m. Improvement of the electrical conductivity of the Al-Cu- Al_2O_3/Gr composite as a result of reinforcement with 1 wt.

The ratio of Gr may be attributed to the high electrical conductivity of Gr. He-she may also be related to the homogeneous distribution of Al-Cu- Al_2O_3/Gr in the aluminum matrix and the good adhesion between the Al_2O_3 and graphene layers with aluminum due to a good coating and sintering process. The process of coating Gr and Al_2O_3 with silver, copper metal, and aluminum enhances the inter-bonding between them, so there are no pores formed. In addition, good sintering ability, suitable sintering temperature and time gives opportunity for all components to react and diffuse with each other. The concentration and dimensions of Gr represented by the surface area and thickness have a great influence on the electrical conductivity of Al-Cu- Al_2O_3/Gr nanocomposites. Gr benefits from large surface area and thin thickness, which are nano-sized.

The large surface area allows the passage of a large number of electrons, and the thickness of the nanoparticles facilitates the movement of these electrons during transport, a process that ultimately leads to an increase in the electrical conductivity of the Cu matrix in the presence of Gr. Increasing the proportion of GNs leads to an increase in the surface area, so that the electrical conductivity increases in proportion to the increase of GNs up to 1 wt. %. It is noticed that the electrical range values decrease with the increase in the graphene content. This may be due to the production of some agglomerates of Gr by increasing its percentage, which reaches 1.5%, which led to a decrease in the electrical conductivity to 6.76 Ms/m. The rolling process led to a significant improvement in the microstructure through the appearance of a fine structure, which helps to conduct the best electrical electronics, which improved the electrical conductivity by 16-25%, according to the reinforcement materials. The rolling process also helped in the sliding and spreading of graphene strips, thus getting rid of all agglomerates, and thus improving the electrical conductivity up to 1.5% graphene by weight to 17.18 Ms/m.

F. Thermal Conductivity

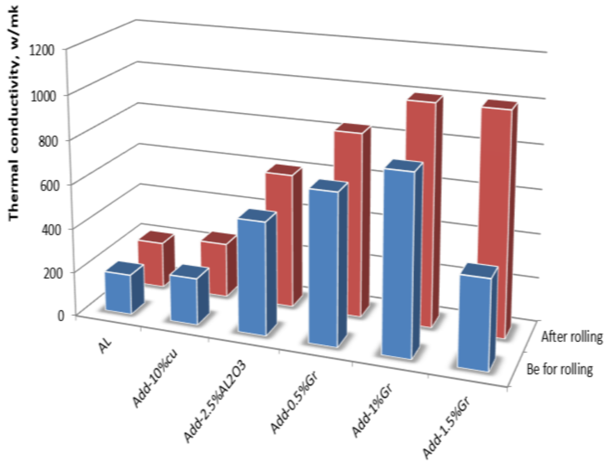

Fig 9, Thermal conductivity of Al-Cu-Al2O3/Gr. nanocomposites

Fig.9, Thermal conductivity of Al-Cu- Al_2O_3/Gr. Nano composites. The effect of adding graphene Nano sheets on the thermal conductivity of the Al-Cu- Al_2O_3/Gr nanocomposite is shown in Figure 9. It is evident that the heat conductivity of the Al-Cu- Al_2O_3/Gr composites was clearly improved as a result of the reinforcement with the graphene Nano sheets up to 1% weight. It gradually decreases due to the presence of agglomerates of graphene strips at 1.5%, which makes the results counterproductive and the presence of a conductive network is necessary to achieve high thermal conductivity values.

To achieve this, good adhesion is required between the different components of the compound. Coating of graphene and Al_2O_3 silver Nano sheets with aluminum and copper increases particle-particle contact and adhesion which creates the conductive pathway network within the composite and improves thermal conductivity.

In addition to good mixing, grinding and sintering process reduces the chance of formation of any pores in which the thermal conductivity of the pores is zero, meaning that there are absolutely no restrictions on the conductive parameters. Thus, the thermal conductivity increases up to 1% by weight of graphene. The rolling process led to the crushing of large grains and the removal of graphene agglomerates, which improved the thermal conductivity significantly, and this appears in the sample reinforced with 1.5% graphene significantly.

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V. CONCLUSION

Powder characterization The figure shows SEM images of Al-Cu- Al_2O_3/GNs powder. The success of the grinding process for 35 hours is suitable for the excellent distribution of the elements. It also shows the success of coating by silver for graphene and Al_2O_3 strips, which was successful in improving the adhesion to aluminum and was positive for thermal and electrical conductivity. Through the hot rolling process, a fine structure appears and a better distribution of graphene at high ratios of 1.5%. The hardness was tested by a range of 40: 45%, and the electrical conductivity ranged between 14: 25%, as well as the thermal conductivity. Through the results, we conclude that the best properties obtained before the hot rolling process at 1% by weight graphene and after hot rolling improved to 1.5% by weight graphene due to the improvement in the distribution of graphene slices. The heating process during rolling also contributed to maintaining the formation without cracks or cracks appearing.

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