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Preparation and Addition of Graphene to Enhance the Thermal Conductivity for Recycled Electrical Wires

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Abstract— In current study, effect of graphene on thermal conductivity of as-cast and heat-treated alloys was investigated. The contents of graphene nanoparticles were added in 0.3 and 0.5wt% into 99.5% pure aluminum (scrap wire damaged). The aluminum matrix composites were reinforced by graphene, which prepared using pyrolysis method of asphalt. On the other hand, the aluminum-graphene composites were fabricated by stir-casting technique followed by Thermo-Mechanical Treatment (T81) which including solution heat treatment, cold rolling and artificial aging in a sequential order. Raman spectroscope was applied for graphene to characterize as received of graphene produced by pyrolysis method. Examination of the microstructure of fabricated alloys was done using the optical microscope. Compared with the base alloy wire, the thermal conductivity of heat-treated Al-0.5wt% GNPs wire was 214 W/m.k, which increased by 12% respectively.

Index Terms— scrap wire damaged, graphene, pyrolysis route, stir-casting, thermal conductivity, nanocomposites, Raman Spectroscopy, Optical Microscopy.

1 Introduction

Graphene is the two-dimensional (2D) monolayer structure of carbon in which carbon atoms arranged in a honeycomb crystal lattice such that each atom is linked to three others via (sp^2) bonding **[1].**
Graphene is a single layer of (sp^2) bonded carbon atoms that are stacked in a honeycomb lattice. The atomic structure of graphene leads to exceptional electrical, mechanical, thermal and optical properties. If graphene is stacked perpendicularly to hundreds of layers, it would form three-dimensional (3D) of graphite. When in a ball shape it forms (0D) fullerenes, and when rolled into a tube, graphene forms (1D) carbon nanotubes **[2].** Graphene has excellent electronic, mechanical and thermal properties. It has a very high electronic conductivity at room temperature, and its electron mobility is (2.5×10^5 $cm^2.V^{-1}s^{-1}$). Its thermal conductivity is around (3000 $Wm^{-1}.K^{-1}$). Graphene has a unique two-dimensional (2D) hexagonal structure consists of a single atomic layer of (sp^2) hybridized carbon atoms **[3].** The mechanical strength of graphene is 200 times stronger than steel. It has unique thin nanostructure (10^6 times thinner than human hair) and world's first two-dimensional (2D) material consisting of single layer of hexagonally arranged (sp^2) bonded carbon atoms. Moreover, graphene is chemically inert, highly stretchable, completely flexible, impermeable, high biocompatibility and cost-effective **[4].**

Thermal conductivity of graphene, it can appear by Weidman-Franz. Is dominated by phonons, and the suspended graphene, in room temperature has been proved from (4840 - 5300 W/m.k), when the cooper has (400 W/m.k) and the pyrolytic graphite (2000 W/m.k). Therefore, we are talking about (10 times) better conductivity than cooper. The thermal conductivity of the suspended graphene exceeds (2500 W/m.k) near (350 K^0) and becomes (1400 W/m.k) at about (500 K^0) **[5].**
The thermal conductivity of graphene was measured for the first time by Balandin in (2008) at the California University using optothermal Raman measurement technique. In this method, a Raman spectrometer serves as a thermometer measuring the local temperature rise in graphene in response to the Raman laser heating. Moreover, Balandin also found that the (G) peak of graphene's Raman spectra exhibits strong temperature dependence which enables to observe the local temperature change produced by the laser excitation. The relationship between the amount of power and temperature rise dissipated in graphene, for the sample with given geometry and proper heat sinks, can give the value of the thermal conductivity (K) **[6,7,8].** However, at the moment it is still difficult to determine a well-defined thermal conductivity value of intrinsic graphene since all the experimentally (K) measures scatter between (600 - 5000 $W\ m^{-1}\ K^{-1}$). These differences have been justified in terms of different sample qualities, thickness non-uniformity, strain distribution, defects, grains and temperature of the sample. Generally, two groups of techniques can measure thermal conductivity of graphene: transient and steady-state .The low mass and the strong bonding of the carbon atoms give to graphene and related materials exceptional thermal properties **[9].**
Generally, in a solid material, heat carried via lattice vibrations and electrons. The value of thermal conductivity (ke) relates to electrons, obtainable from the measurement of the electrical conductivity (σ) via the Weidman-Franz law:

$$Ke/\ \sigma T = \pi^2 K^2\ B\ /\ 3e^2 \ \ldots\ldots\ldots\ (1\text{-}1)$$

Where (KB) is the Boltzmann constant and (e) is the electronic charge. Phonons are typically the main heat carriers in carbon materials. Even in graphite, which has metal-like properties **[8].**

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Indeed, in carbon material, the strong (sp^2) covalent bond between neighboring carbon atoms results in an efficient heat transfer via lattice vibrations **[7].** The aim of this study is to prepare and add graphene to the base alloy to show its effect on the thermal conductivity of the fabricated alloys using stir-casting technique.

2 Experimental

2.1 Synthesis of graphene by pyrolysis route

Asphalt essentially consists of saturated and unsaturated aromatic also aliphatic compounds with up to an estimated (150) carbon atoms. Generally, asphalt largely consists of 80 % by weight of carbon, up to 10 % by weight of hydrogen, and 6 % by weight of Sulphur, small amounts of oxygen and nitrogen, and traces of metals such as nickel, iron and vanadium **[10].** The graphene precursor is a (plastic roof cement), which is consists mainly of thermal asphalt reaction (TAR) with some impurities such as clay, cellulose and water. This method mainly depends on mixing (70 %) of used asphalt with (30 %) of 99.99 % ethanol. During this route, reaction vessel is a (90 mL) ceramic crucible with an interior crucible (30 mL) which filled with (15 g) of asphalt and inserted in the bigger ceramic crucible and well glass covered as illustrated in Scheme (2-1). For the thermal asphalt reaction (TAR) method, the casserole crucibles were placed in a furnace and heated up to (640- 650 ^{o}C) for (15-20 min.). After that, the furnace is switched off and then the crucibles left in the furnace for (10-15 min.) and then the crucibles removed from the furnace to be cooled down until room temperature. This experiment was done to get a few layered graphene paper (FLG) in order to control the number of graphene layers via this method. This technique is illustrated in the Fig. 1:

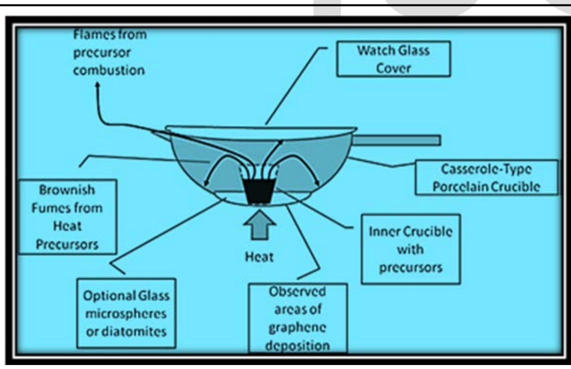

Fig. 1. Graphene deposition technique **[11]**.

The prepared graphene samples during this technique are indicated in Fig. 2:

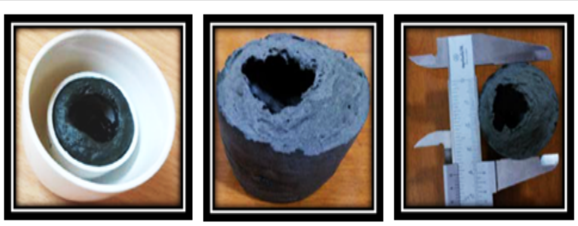

Fig. 2. Prepare of graphene samples.

2.2 Preparation of aluminum-graphene nanocomposites

Fabricated alloy specimens were first prepared by wash and dry electrical scrap wire damaged which used as matrix material to remove remind dirty and then shredded into small wires and weighed and then poured in the crucible for melting. Graphene nanoparticles have been applied as reinforcing phase in molten pure aluminium (99.5%). The fabrication technique that used in this work is stir-casting method, which consists of a gas furnace up to (1000 ^{o}C) where matrix material (scrap wire damaged) is melted and then required quantities of reinforcement material are weighed and added. Initially, aluminum wires were put in graphite crucible inside the furnace for melting up to (750 ^{o}C) and then eliminate the slag caused by the melting process, and then stirrer at 500 rpm was used to achieve homogenously of the mixture. Stirring time was taken as 5 min., after that mixture was reheated and hold at a temperature 950-1000 ^{o}C to make sure mixture was fully liquid. The mixture is poured into a diameter of (9.5 mm) and height of (200 mm) billet by metallic mold, and then cooled from the bottom of mold by using water spray to avoid pore formation. After that, the fabricated rods were cold rolling at room temperature in a 9.5 mm diameter rod form to a 3.5 mm diameter wire. Fig. 3 shows some steps of fabrication technique:

Fig. 3. Steps of the new wire manufacturing.

The alloys in a rod form were drawn from (9.5 mm) to (3.5 mm) diameter wire in (13) step rolling process. Fig. 4 displays the rolling machine and drawing bench that were used for drawing operation.

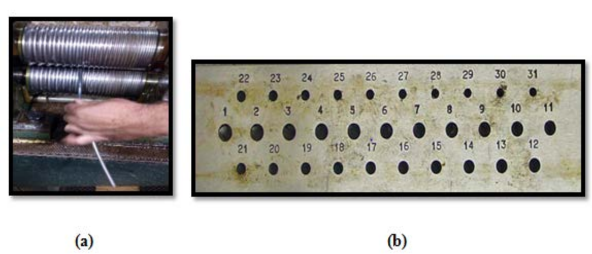

Fig. 4. (a) Rolling machine, (b) Drawing bench.

Rolling machine contains different gaps between each two rolls producing square cross section, while drawing bench contains many holes of different diameter from several (mm) to about (1 mm) that produce circular cross section wire.

TABLE 1
CHEMICAL ANALYSIS OF THE BASE ALLOY (ALUMINUM WIRES).

Alloying element content wt%									
Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti	V	Al
0.107	0.423	0.010	0.005	0.001	0.003	0.008	0.002	0.001	Bal.

TABLE 2
THE WEIGHTING RATIOS OF THE ALLOYS USED IN THIS WORK.

Composition / Alloy code	Graphene wt %	Al (Scrap Wire Damaged wt %)
A	-	100
B	0.3	Rem.
C	0.5	Rem.

The chemical composition of the base alloy (Al wires) show in Table 1.

TABLE 3
THERMAL CONDUCTIVITY OF USED ALLOY IN THIS WORK.

Alloy	Condition	Thermal Conductivity (W/m.k)
A	As cast	191
B	As cast	184
C	As cast	178

The weighting ratios of the alloys used in this work it is included in the Table 2.
The thermal conductivity values of alloy used in this work as shown in the Table 3.
To achieve flat surface for each samples, grinding and polishing were used via grinder and polisher device. Grinding was done using emery papers with various grinding scales 180, 240, 400, 600, 800, 1000, 1500, 2000 and 3000. Polishing was achieved using polishing papers with alumina solution to obtain a mirror surface via polish cloth and alpha alumina (0.5µm and 1µ m) to clean the specimen from dust and dirt, and then washed with distilled water. The etching process of samples was achieved by Killers solution with total volume (12.5 ml) contain 0.3 HNO_3, 0.2 HCl, 0.125 HF, 11.7 H_2O (distilled water) for 10-15 sec and then the specimen was washed with distilled water and dried.

2.3 Thermo-Mechanical Treatment

After the cleaning of the cast samples, homogenizing treatment was done by heating the samples from room temperature at (2°C/min.) up to required homogenizing temperature of 500°C and soaking at 500 °C for (10 hr.) then slow cooling in the furnace, and then heating the samples from (25°C - 500°C) at the rate of 2°C/min. to avoid distortion and cracking and soaking at 500°C for (2 hr.) before quenching in water, and 86.4 % cold rolling. Finally, artificial aging for samples at (170 °C) for (1,2,4,6,8 and 10 hr.). The samples are heated from room temperature with a rate of (60 °C / hr.) and kept at (170 °C) for different aging times. The parameters of thermo-mechanical treatment are listed in Table 4.

TABLE 4
CONDITIONS OF THERMO-MECHANICAL TREATMENT AT 170°C FOR DIFFERENT TIMES.

Alloy Code	Condition
B_1,C_1	Homog. at 500 °C for 10 hr. + R.T+ S.H.T at 500 °C for 2 hr. + W.Q+86.4% C.R+Aging at 170°C for 1hr.
B_2,C_2	Homog. at 500 °C for 10 hr. + R.T+ S.H.T at 500 °C for 2 hr. + W.Q+86.4% C.R+Aging at 170°C for 2hr.
B_3,C_3	Homog. at 500 °C for 10 hr. + R.T+ S.H.T at 500 °C for 2 hr. + W.Q+86.4% C.R+Aging at 170°C for 4hr.
$B_4$$C_4$	Homog. at 500 °C for 10 hr. + R.T+ S.H.T at 500 °C for 2 hr. + W.Q+86.4% C.R+Aging at 170°C for 6hr.
B_5,C_5	Homog. at 500 °C for 10 hr. + R.T+ S.H.T at 500 °C for 2 hr. + W.Q+86.4% C.R+Aging at 170°C for 8hr.
B_6,C_6	Homog. at 500 °C for 10 hr. + R.T+ S.H.T at 500 °C for 2 hr. + W.Q+86.4% C.R+Aging at 170°C for 10hr.

S.H.T: Solution Heat Treatment, W.Q: Water Quench, C.R: Cold Rolling, A.A: Artificial Aging.

3 RESULT AND DISCUSSION

3.1 Raman spectroscopy

This technique is a powerful and widely used tool for the characterization of graphene samples due to its key role in the field of carbon material researches and also in the structural characterization of graphitic materials. Raman spectroscopy is a simple method, not requires more sample preparation, a non - destructive method, can be used to identify the layers number of graphene and can determine if the structure of graphene is ideal. The Raman spectra of graphene display simple structure of the G and D band that gives important information about thickness of graphene layer, band shape and intensity.
Fig. 4 illustrates the Raman spectrum of the prepared graphene. The spectrum behavior is very similar to that of the reported results for few layers of graphene **[12]**, which reveals the successful synthesis of high quality graphene.

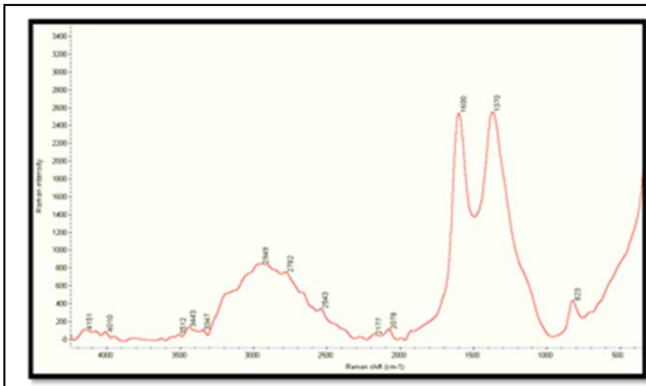

Fig. 4. Raman spectrum of as received graphene nanoparti cles.

In this work, a broad G and D bands are observed in Raman spectra of the prepared sample, which contain (30% ethanol + 70% asphalt) and centred at 1600 cm-1 and at 1370 cm-1 respectively. The G band is appears at around 1600 cm-1 in the graphene spectrum. The position and intensity of G band is very sensitive to the number of sample layers; which allows accurate determination of graphene layer thickness.

3.2 Characterization by Optical Microscopy (OM)

The characterization of microstructure is one of the main means of evaluating alloys to identify the effects of various fabrication and heat treatments. Examination of the microstructure of fabricated alloys at each step in the as cast and aging treatments are done using the optical microscope after grinding and polishing.

Fig. 5 shows grain morphology of base alloy and its composites in as aged state. Fig. 5-a shows the area of chilled surface of the casting rod. The structure of base alloy have bright area of α-Al phase based solid solution. The microstructure is homogeneous and contains traces of pores. Depending on the chemical composites of base alloy in experimental work, there are several impurities in aluminum matrix may be form some phases and intermetallic compounds and they subject from segregation.

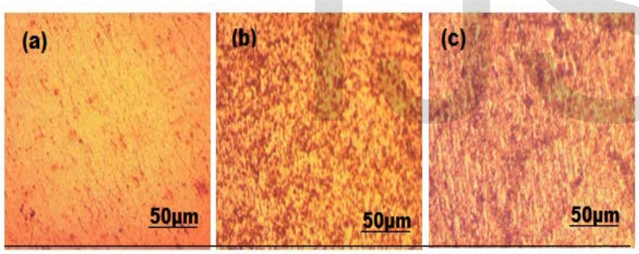

Fig. 5. Optical images showing grain morphology of (a) base alloy, (b) Al-0.3wt%GNPs as aged and (c)Al-0.5wt%GNPs as aged at 400X.

Figure 5-b depicts the optical micrographs for the Al- 0.3% GNPs samples after heat treatment, where we observe the fragmentation of graphene agglomeration after leaving the grain boundaries and are distributed homogeneously over the entire sample area. The optical photographs of Al-0.5% GNPs composite after heat treatment are shown in Fig. 5-c The distribution of graphene agglomeration in composites is relatively homogeneous when the content of graphene particles in the aluminum-graphene composites ranges from (0.3%-1.0%) **[13]**.

3.3 Thermal Conductivity

Thermal conductivity test was achieved for treated and untreated samples to show the influence of reinforcements and artificial aging on thermal conductivity of fabricated alloys. This test was done using Hot Disk device (Thermal constant analyzer type TPS 500) in University of Technology- material engineering department- heat transfer laboratory.

The results of the thermal conductivity (K) of each sample are shown in Table 5.

TABLE 5
THERMAL CONDUCTIVITY OF THE TREATED AND UNTREATED SAMPLES.

B	Code name	B	B1	B2	B3	B4	B5	B6
	Condition	As cast	1hr.	2hr.	4hr.	6hr.	8hr.	10hr.
	Thermal Conductivity (W/m.k)	184	200	203	203	206	208	207
C	Code name	C	C1	C2	C3	C4	C5	C6
	Condition	As cast	1hr.	2hr.	4hr.	6hr.	8hr.	10hr.
	Thermal Conductivity (W/m.k)	178	207	205	211	210	214	212

3.4 Effect of graphene on thermal conductivity of as-cast Aluminum wires

The thermal conductivity of as cast and heat-treated alloys was tested to show the role of graphene particles on thermal conductivity of fabricated alloys.

Fig. 6 shows the thermal conductivity of aluminum wires, including pure aluminum wire, Al-0.3wt% GNPs wire and Al-0.5wt% GNPs wire, which were produced by stir casting method. The thermal conductivity of B alloy (Al-0.3wt% GNPs wire) and C alloy (Al-0.5wt% GNPs wire) were (184, 178 w/m.k) which were decreased by 3.66%, 6.80% consecutively, comparing to the thermal conductivity for base alloy wires 191 w/m.k.

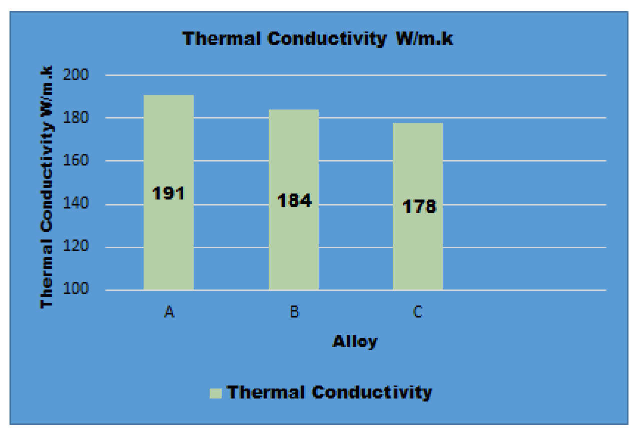

Fig. 6. Thermal conductivity of as cast aluminum wires.

Experimentally measured values at 20 °C indicate that reinforcement addition to base alloy reduced the thermal conductivity of Al-C matrix from 191 W/m.k to (184,178W/m.k) of B,C alloys respectively, which is may be attributed to the existence of graphene agglomeration and **[14]** porosity which increases with increasing the reinforcement volume ratio. The thermal conductivity of as cast alloy decreases as the reinforcement content increases.

3.4 Effect of graphene on thermal conductivity of heat-treated Aluminum wires

In order to improve the thermal conductivity of the fabricated alloy wires produced by stir casting method, artificial aging treatment at 170°C for different times was performed later. Fig. 7 and Fig. 8 show the changing curve of thermal conductivity of aluminum alloys wire versus aging time after solution heat treatment. It can be seen that the thermal conductivity will increase with aging time, and destined to stabilize eventually. After solution heat treatment at 500 °C for 2hr. and isothermal aging temperature at 170 °C for 8hr., the Al-0.3wt% GNPs and Al-0.5wt% GNPs alloy wires reach the optimal performance. The thermal conductivity of Al-0.3wt% GNPs wire and Al-0.5wt% GNPs wire reach 208 W/m.k, 214 W/m.k respectively.

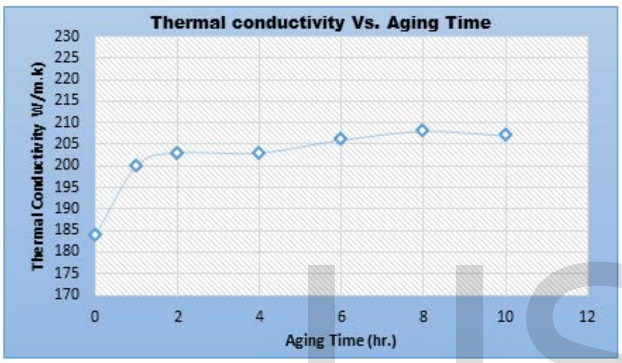

Fig. 7. Thermal conductivity of B alloy wires (Al-0.3% graphene) at different aging time.

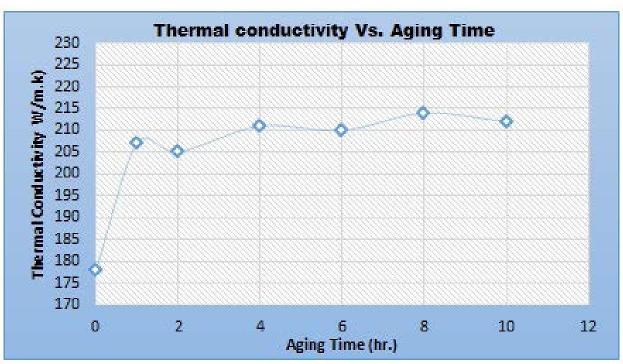

Fig. 8. Thermal conductivity of C alloy wires (Al-0.5% graphene) at different aging time.

Fig. 7 shows the relationship between thermal conductivity and aging time of the fabricated alloy wires after thermo-mechanical treatment T81, which included solution heat treatment at 500°C for 2hr.+ 86.4% cold rolling + aging at 170°C for different times in a sequential order, where we note the optimal comprehensive performance of aluminum wires is after (1hr.) of aging (peak of thermal conductivity) being observed after aging at 170°C for 1 hr. by the peaks (200, 207 W/m.k) of the (B,C) alloy wires, respectively. The thermal stability of the alloy wires was high for most aging stages as the alloys stabilized thermally after 4hr. of aging.

Fig. 9 show Thermal conductivity of fabricated alloy wires (B,C) at different times.

Results obtained showed a specific trend relating thermal conductivity with aging time. It was found that as aging time in fabricated alloy samples increased, thermal conductivity increased as well.

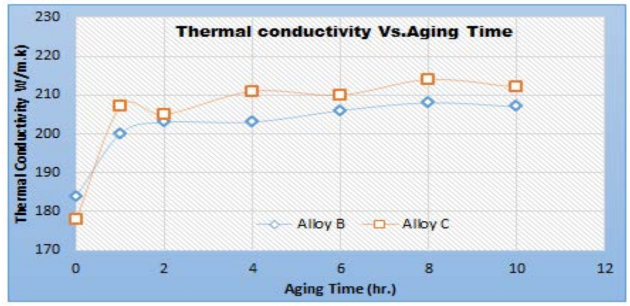

Fig. 9. Thermal conductivity of fabricated alloy wires (B,C) at different times.

During the artificial aging treatment for aluminum alloys, the precipitates grow in size. The effect of precipitate size on thermal and electrical conductivity has not been studied extensively. The increased thermal conductivity observed in the aluminum-carbon alloy samples is a result of the precipitates that have formed over time. The precipitates at the thermo-mechanical treatment condition are fully blown and have agglomerated in certain locations, which make the aluminum matrix leaner. This allows the thermal signal going through the aluminum-carbon alloy samples to start flowing faster, and ultimately results in higher thermal conductivity. As aging time increases the precipitates grow bigger, leaving open channels in the aluminum matrix for the thermal signal to travel through it **[15]**.

Fig. 10 shows the optimal performance of fabricated alloy wires. Compared with the base alloy wire, the thermal conductivity of heat-treated Al-0.3wt% GNPs wire and Al-0.5wt% GNPs wire were 208, 214 W/m.k, which were increased by 8.9%, 12% respectively.

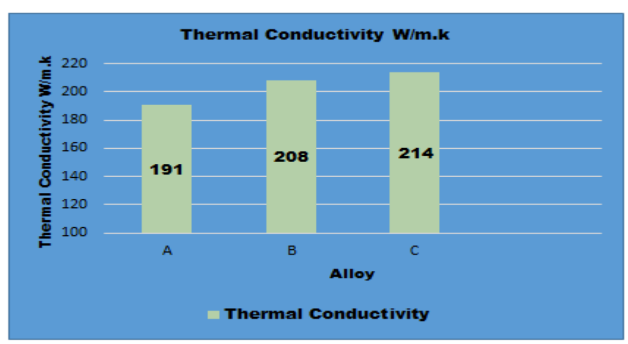

Fig. 10. Thermal conductivity of heat- treated aluminum wires.

Graphene exhibit excellent thermal conductivity 5×103 W/m.k, charge carrier mobility 2×105 cm2 v–1 s–1 and surface area 2600 m2.g-1**[16]**. Due to its exceptional thermal conductivity, graphene is a good candidate for the reinforcement of an aluminum matrix to improvement the thermal conductivty.

There is only study available on bulk GNPs-MMCs composites which show the effect of graphene on thermal conductivity. The thermal conductivity (TC) of the Aluminum-Graphene composite is measured at 250°C by technique of laser flash analysis. The improvement in thermal conductivity with the addition of graphene nanoparticles over pure aluminum was 16% **[17]**. The improvement in thermal conductivity of aged alloys is due to the highly conductive graphene, which when mixed well without any carbide formation or agglomeration may be lead to a highly conductive network of graphene during the aluminum matrix.

Results showed that an addition of graphene particles to the base alloy resulted in reduced coefficient of thermal expansion of composites, this is attributed to that graphene has lower thermal expansion coefficient than that of aluminum matrix. Because of the layered structure of graphene, the heat was absorbed via the graphene particles. Increasing content of graphene led to decrease the (CTE) of composites and shift the maximum temperature of the CTEs, due to the relaxation of compressive stresses in the aluminum matrix **[14]**.

4 CONCLUSION

The conclusions obtained from present study, can be summarized as following:

1. Use of damaged electrical wire scrap as a basic materials for production the base alloy by recycling and then reinforced with graphene reach to the optimum alloy for use in the manufacture of electric power transmission wires.

2. Preparation and produce of graphene was obtained by pyrolysis method of asphalt and ethanol mixture in the percentage of (70%) and (30%) respectively.

3. Characterization of fabricated alloys after aging treatment resulting from adding graphene to aluminum matrix showed homogenous distribution of the reinforcements in the base alloy.

4. Effect of thermo-mechanical treatment on the thermal conductivity of fabricated alloy was investigated.

5. Artificial aging treatment after cold rolling can improve the thermal conductivity of aged alloys.

6. Compared with the base alloy wire, the thermal conductivity of heat-treated Al-0.3wt% GNPs wire and Al-0.5wt% GNPs wire were 208, 214 W/m.k, which were increased by 8.9%, 12% respectively.

7. By the results of this study, it appears that the graphene is a promising material to improve the thermal conductivity of electrical power transmission line.

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