International Journal of Recent Technology and Engineering (IJRTE)
ISSN: 2277-3878, Volume-8 Issue-3S3, November 2019

Silver Particles on the Thermal and Electrical Characteristics of ECAs

Muhammad Zulkarnain, Muhammad Sobron Yamin Lubis, Silvi Ariyanti and Mariatti Jaafar

***Abstract*: This study presents information on the thermal and electrical characteristics of silver (Ag) in range size of 2–3.5 µm and 80 nm in diameter. The present method demonstrates the thermal conductivity analysis and electrical resistivity influence by various particle content of Ag in Epoxy matrix for both micro and nano-sized. Furthermore, new technique of thermal properties and electrical resistivity observation is proposed by hybrid-sized analysis to characterize the effect of Ag size. The proposed hybrid-sized technique uses micro- and nano-sized particle ratios to generate the composite. The thermal and electrical resistivity characteristics of the epoxy composite-filled micro-, nano-, and hybrid-sized Ag particle are correlated with their morphology. The thermal conductivity of the electrically conductive adhesive sample is affected by Ag particle size. The micro-sized Ag particle is better filler than the nano-size Ag particle in increasing performance of the thermal conductivity in the matrix epoxy. The results of the electrical resistivity of micro- and nano-particles demonstrated similar characteristics that transition within insulator into conduction occurred at 6 vol%. While hybrid-sized systems shown decreasing in thermal conductivity performance when decreasing number micro-sized ratio. Other observation in hybrid-sized presented that the better performance of electrical conductivity has shown at 50:50 weight ratio.**

Index Terms*: *thermal conductivity; electrically conductive adhesives (ECAs); particle contact resistance; particle structure

I. INTRODUCTION

Currently, conventional interconnecting in electronic device such solder has been abandoned and it has been replaced by alternative electrically conductive adhesives (ECAs) material. ECAs has been dominant and widely used in interconnecting material such as die attach adhesives, flip-chip interconnection, and surface-mount electronic component due to propose some advantages such as good electrical conductivity, low cost, fine pitch interconnect extendibility, and environment friendliness [1,2]. Due to ECAs exhibit in high-performance conductive adhesives, it induce to replace free lead solder that apply as substrates and as electrical transport in electronic devise [3-6]. Although electrical conductivity is the main purpose of most studies, heat releasing material is highly desired for electronic devices, because integrated electronic devices exhibit a fundamental maximum operating temperature. The device material must reliably and rapidly decrease the operating temperature. The thermal conductivities of ECAs must be known and can substantially improve the electronic performance system.

Performance of thermal conductivity can be increased by adding various conductor particles that embedded in epoxy resin such as silver (Ag), graphite, copper (Cu), aluminum oxide (Al_2O_3), aluminum (Al), aluminum (Al), boron nitride (BN), zinc oxide (ZnO), and diamond [7]. The thermal conductivity has resulted in eight variations, in which the graphite filler showed high thermal conductivity. Furthermore, flaky-shaped fillers tend to generate particle networks in contrast with spherical-shaped fillers. The sharp-corner-shaped fillers propose contact surface and it generate high thermal conductivity. Other possibility in high thermal conductivity has been investigated in BN/polymer composite [8]. BN successful present high performance of thermal conductivity by adding amphiphilic agent that can improve particle distribution and orientation. The finally, this agent ($C_{14}H_6O_8$ and $C_{27}H_{27}N_3O_2$) produce higher thermal conductivity in BN/PVB composite compare to the PVB composite with pristine BN only.

Based on morphological analysis, particle dispersion brings paramount parameter in improving both electrical and thermal conductivity due to improving particles networks inside composite. The particle shape and dimensions cause different treatment and strategy to be mixing with matrix resin that could produce conducting network. Kim *et al* (2012) propose suing graphene nano-sheet particles to generate particles networks in the epoxy and coated by Al(OH)3 in Hummers method [9]. This agent helps particles to well distribute in the epoxy and these phenomenons cause the synergistic effect on the thermal conductivity. This positive phenomenon has been reported by previous researcher [7]. The thermal conductivity results was 6.8 times higher compare to pure polymer phenolic formaldehyde resin (PF) that occurred at 30 wt.% BN and 30 wt.% T-ZnO.

Glass fiber-reinforced polymer (GFRP) composites also have some advantages in electronic device application as substrates to support and link electronic devices [10,11]. Different particle filler sizes embedded onto carbon fiber-reinforced epoxy laminates positively affected thermal conductivity by the through-thickness of GFRP [10]. The prepared composite was a combination between graphene nano-platelets and silver nanoparticles/nanowires onto

Revised Manuscript Received on November 05, 2019.

Muhammad Zulkarnain, Universiti Kuala Lumpur Malaysian Institute of Marine Engineering Technology (UniKL MIMET), 32200 Lumut, Perak, Malaysia.

M.Sobron Lubis, Depertment of Mechanical Engineering, Engineering Faculty, University of Tarumanagara, Jakarta, Indonesia.

Silvi Ariyanti, Depertment of Industrial Engineering, Engineering Faculty, University of Mercubuana, Jakarta, Indonesia.

Mariatti Jaafar, School of Materials and Mineral Resources Engineering, Universiti Sains Malaysia, 14300 Nibong Tebal, Seberang Perai Selatan, Penang, Malaysia

laminated GFRP. The synergistic effect of the physical interactions that could enhance the through-thickness thermal characteristic was investigated. The thermal conductivity remarkably increased in laminates containing both graphene nano-platelets and Ag nanoparticles/nanowires. The thermal conductivity of Al_2O_3-filled GFRP composites was evaluated [11]. Al_2O_3/GFRP composite was effective filler for thermal conductive fabrication (1.07 W/mK) compared with epoxy/ Al_2O_3 composite. Hybrid system also offers some advantages on electrical conductivity. ECAs filled with Ag nano-particle (Ag NP)-decorated graphene positively affect electrical conductivity, which in turn affects curing. Ag-NP with an average size of 9 nm has been dispersed onto the graphene surface by using mercaptopropionic acid. Electrical conductivity increases because of the sintering between small Ag NPs on the graphene surface and Ag flakes, thereby causing the electrical resistivity to be close to that of eutectic Pb based solders and attaining 4.6 9 10^{-5} Ω-cm of 220 °C for solid phase temperature.

Ag-filled epoxies exhibit some advantages when applied as ECA in the interconnection material electronic device component. One probable reason for this is high thermal conductivity, as well as electrical conductivity properties, especially for the attachment of chips to substrates [4,8,12-17]. Both thermal and electrical conductivities greatly affect ECA performance. Thus, investigating the Ag particle size effect is still difficult because of the lack of information on the influence of thermal conductivity of Ag particle on the micro- and nano-sized effects. The nano-sized filler is implemented in his research to observe the aggregated as particles interconnecting among micro-sized fillers in the conductive adhesive. This paper involves the measurement of the thermal and electrical properties and characteristics of epoxy adhesives with different volume fractions of Ag to analyze the effect of micro- (2–3.5 μm of diameter), nano- (80 nm of diameter), and hybrid-sized (80 nm and 2–3.5 μm mixed) Ag particles. A hybrid size technique is introduced in this research to observe particle size's effect in influencing the thermal and electrical properties.

II. EXPERIMENTAL METHOD

A. Materials

The epoxy product used in this investigation was bisphenol-A-(epichlorohydrin), which commercial name is EPON^{TM} Resin 8281 (EPON 8281) that produced by USA Chemical company of Hexion. The curing agent polyetheramine D230 (PEA; density = 0.946 g/mL at 298 K), which was used for EPON 8281, was manufactured by BASF Corporation. The condition mixture between EPON 8281 and PEA was fixed at 100:32. The Ag particles used in this study were purchased from Sigma–Aldrich, Inc. The sheet data from the supplier shows that the average particle sizes were 80 nm and 2–3.5 μm, while 10.49 g/cm^3 as density. The lubrication is implemented by Chloroform in 99.0%–99.4% of purity for particle distributions in the epoxy resin with a mixing ratio of 1:1 by weight, which was sonicated in an ultrasonic bath (NeyALTRAsonik) for 10 min.

B. Preparation of ECA Composite

Single-filler (micro- and nano-sized) conductive adhesives were prepared using 2 vol. % to 8 vol.% fillers. While in the system of hybrid, the total content will be maintained at 6 ol.% by varied amount of micro- and nano-sized. The variation of Silver content are determined by three sets of volume ratios (micro- and nano-sized), as follows: 75:25, 50:50, and 25:75, which micro- positioned on left while nano-sized on right side. The epoxy and Ag filler need stirred for 10 min for homogenizer mixture. Then follow by sonicated for 30 min to confirm particle well disperse. This mixture need to remove the bubble by subsequently vacuumed for approximately 0.5 h without temperature setup. In part curing, this mixture added agent by 32:100 weight ratio of EPON 8281, and the mixture was stirred for another 10 min. Finally, the mixture was allowed to stand at room temperature for 2 h and followed by curing at 100 °C for 1 h and continued by post-curing at 125 °C for 3 h.

C. Characterization

Sample morphology was analyzed on the fracture surface of ECAs by a field emission scanning electron microscopy (FESEM) system (model ZEISS SUPRA^{TM} 35VP) with a Gemini field emission column. Particle size distribution was collected by using 2 equipment's which provide for micron was measured by Malvem model Master Sizer/E version 2.15 while nano-sized was measured by Zeta Sizer Nano Series version 6.0.

Using a Hot Disk TPS-2500, the thermal conductivity was measured at room temperature. Specimens with 1 cm × 1 cm dimensions and 3 mm thickness were prepared for thermal conductivity tests. Instek LCR-817 was used to measure the electrical resistivities by 1 V of voltage transfer without temperature setup. The specimen size was prepared by 2 cm × 2 cm to allow the volume resistivity determination that was calculated by Ohm's Law equations.

$$R_v = \frac{V_t}{I_v} \qquad (1)$$

The volume resistivity of the specimen was expressed as follows:

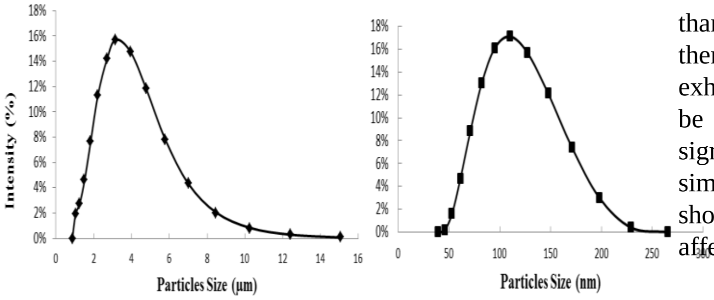
$$\rho_v = \frac{R_v A}{t} \qquad (2)$$

where ρ_v denotes the volume resistivity, R_v is resistivity's volume, while A denote to conductor area, and notation t describe the average thickness of the sample, which is 1.

III. RESULTS AND DISCUSSION

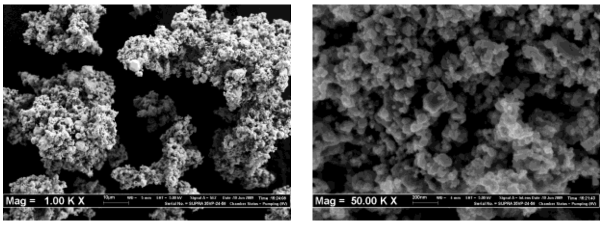
A. Characteristics of the raw materials

Fig. 1 presents the overview of the intensity and size distribution for both micro- and nano-sized Silver particles. As can be seen from the figure below that the average sizes were 3.16 μm and 110.1 nm for micro- and nano-sized, respectively. The results obtained were comparable to the values claimed by the suppliers. In the FESEM analysis, the particle shape was spherical, as shown in Fig. 2, and was comparable with the information in the supplier sheet data.



(a) (b)

Fig. 1. Intensity vs. Particles Size of ; (a) micro-particles Silver and (b) nano-particles Silver



(a) (b)

Fig. 2. The particles shape representative of; (a) micro-particles Silver and (b) nano-particles Silver

B. Thermal conductivity of micro- and nano-sized particles

The thermal conductivity of ECAs was generally characterized by the varied volume fractions of the Ag filler loading. Fig. 3 illustrates the relationship between the experimentally measured thermal conductivity and Ag volume fraction in the epoxy composites for both micro- and nano-sized particles. Both epoxy composites filled with micro- and nano-sized Ag particles showed similar conductivity trends characterized by filler loading. The thermal conductivity of the epoxy composite was increased by adding several Ag particles. Thermal conductivity was dependent on the density, specific heat capacity, and thermal diffusivity of the composites [8,9,18]. The measured data suggested that the through thermal conductivity is strongly influenced by the inclusion of high thermally conductive Ag-fillers. At low Ag contents, the thermal conductivity of the Ag/epoxy composites was slightly lower than the high filler loading of Ag. The difference increases at high filler Ag particle contents. For example, the thermal conductivity of the micro-sized composite was 0.3647 W/mK at 2 vol. % of Ag filler particles, but this increased by almost twice at 0.5082 W/mK at 8 vol. % of Ag filler particles.

The thermal conductivity values determined for the Ag micro-sized particle were considerably higher than those measured for the nano-sized particles (Fig. 3). This condition showed that the thermal conductivity of micro-Ag particle improved from 0.3647 W/mK at the filling load of 2 vol.% Ag particles, which was higher than nano-sized particle (0.2527 W/mK). However, the thermal conductivity of the epoxy filled with the Ag nano-sized particle at 8 vol.% was 0.3658 W/mK, which was almost twice higher than that of pure epoxy. Nevertheless, the thermal conductivity was still much smaller compare to ECA of micro-sized Silver. For comparison, the results of micro-sized Silver at 8 vol.% of Silver particles was 0.5082 W/mK, which was higher by more than twice that of the pure epoxy. Ag particle size affected the thermal conductivity of ECAs, with micro-sized Ag exhibiting high thermal conductivity. This phenomenon could be attributed to the adding more Silver did not show any significant difference but still shown slightly increase trend. A similar phenomenon was reported by previous work, thereby showing that the thermal conductivity of the composites was affected by particle size [19].

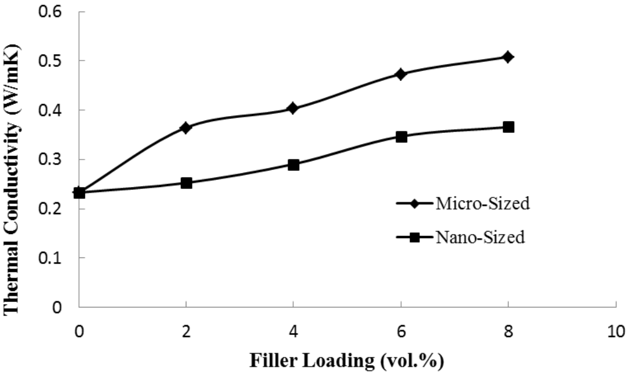


Fig. 3. Thermal conductivity vs. the volume fraction of Silver fillers

Morphological analysis of fracture surface Silver/Epoxy observe by FESEM at containing of 4 and 6 vol.% filler were performed to determine the morphology of the ECAs. Fig. 4 provides the results obtained from FESEM of cross –section sample of Silver/Epoxy conductive adhesive materials for both micro- and nano-sized particles. The cross section of the Ag/epoxy of the micro- and nano-sized Ag was placed on the left and right side, respectively. In the micrograph, the epoxy matrix and Ag particles could be easily distinguished. The Ag particles were the small particles dispersed in the continuous epoxy matrix. From the SEM image, all samples could directly reflect the filler dispersion states in the resin matrix and indirectly verify the above-mentioned thermal conductivity of thermal adhesives results. Fig. 4a shows that many Ag particles were well distributed in the epoxy resin with some contact points (the sample is shown as the red arrow). Some Ag agglomerations (shown as the black circle) could also be clearly seen in certain areas. The avoid area was still dominant at 4 vol. % of filler loading due to the insufficient amount of particle filler. The figure shows a clear separation of both micro- and nano-sized Ag particles in the epoxy matrix (shown as the red circle).

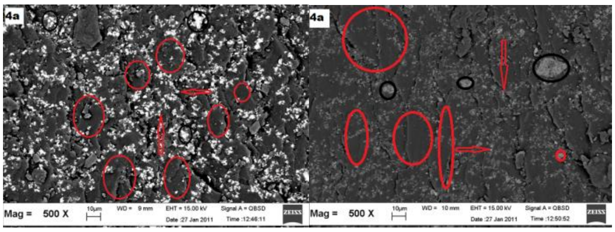


Fig. 4(a). Morphology analysis using SEM images which represent by micro 4 vol. % of Silver content

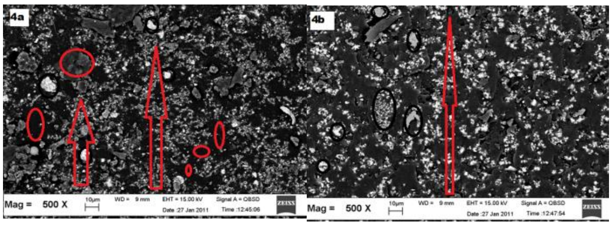


Fig. 4(b). Morphology analysis using SEM images which represent by micro 6 vol. % Silver content

The network began to form as in Fig. 4b of the cross section of the Ag adhesive. It can be seen from the figure that obviously Silver particles were very well disperse in the resin and heat very effective to transport (the sample is shown in the red region) have been formed. The figure shows that a sufficient filler amount to perform networking such particle contact, this perform continuous link formation at obtained at 6 vol.% of Silver content. From this phenomenon, particle structure seemed to contribute to the particles network by presenting a sufficient number of Ag particles. Particle structure can generate electron channels by presenting a dense and contact condition [20]. In Fig. 4b (on the right side), the Silver particles found much agglomerate in epoxy resin it might from high attraction energy each nano-sized particle (the sample is shown as the red arrow) found in the cross section. These agglomerations have negative effect on the formation of heat pathway and are among the causes of the lower thermal conductivity of nano-sized particles than that of the micro-sized particles. The nano-sized Ag particles negatively affect the distribution because of the high energy attraction such as Van-Der Waal make paprticle tend to agglomerate [15,16,19] and decreasing particle network. The particle network resistances exist in the nano-sized environment and it caused affect the thermal conductivity performance. Particle network shown by smaller size is less than the larger particle dispersion [2]. The micro-sized structures reduce the number of contacts by filling the large particle size. Compared with the micro-sized image, the nano-sized structure shows that the particles tend to agglomerate.

C. Thermal conductivity of hybrid-sized systems

In the hybrid system, the mixing ratio between the micro- and nano-sized Ag filler was fixed at 6 vol.% of filler loading. Three ratios were considered, namely, 75:25, 50:50, and 25:75. The thermal conductivity analysis of hybrid-sized system is shown in Fig. 5. The thermal conductivity results from the graph below we can see that varied depending on the particle size combination ratio and was0.4367, 0.4092, and 0.3477 W/mK for volume loading ratios of 75:25, 50:50, and 25:75, respectively. The thermal conductivity decreased with increasing nano-particles in the hybrid system. This phenomenon may be due to the slightly decreasing density of the composites with decreasing Ag particle size. When each particle size reached 25:75 volume ratio of filler loading, the thermal conductivity showed a nearly similar value obtained on nano-sized particles at 6 vol.%. This phenomenon proved that the smaller in particle size leads to a corresponding decrease in thermal conductivity. The results of the correlation analysis shown that thermal conductivity value of the hybrid system decreased by increasing nano-particle content. The decreasing thermal conductivity value was due to the addition of nano-particles in the system.

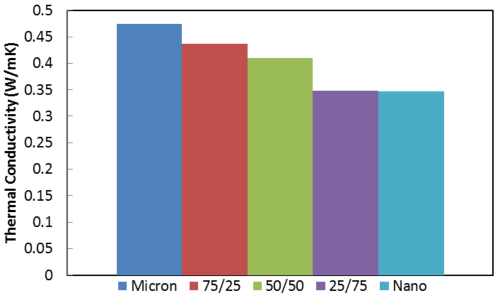


Fig. 5. Thermal conductivity of ECA with 6 vol.% Ag filler loading vs. the ratio of micro- and nano-Ag particles

Fig. 6 shows the morphology particles dispersion of the hybrid system with different behaviors for each set of volume ratio. The sample of several contact points is shown by the red arrow. Some Ag agglomerations are shown by the black circle in certain areas. The avoid area was also clearly seen in the system, and the separation of the Ag particle area is shown by the white circle. Fig. 6a presents that the Silver particles were not well dispersed and tend to agglomerate, as observed on the micrograph. However, the pathway of particle networks of Silver particle was evident in the system, which led to the formation of a heat conduction pathway in the epoxy system. According to micrograph observation, the pathway showed slightly higher size compared with the pathways shown in Figs. 6b and c. Micro-sized particles could reduce the particle contact resistance. In this case, the particle seems to form the effective heat pathways in the epoxy matrix when micro-sized particles dominate the system to perform high thermal conductivity.

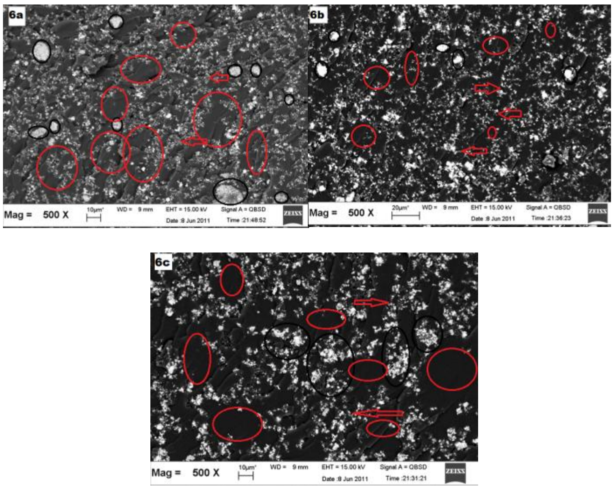
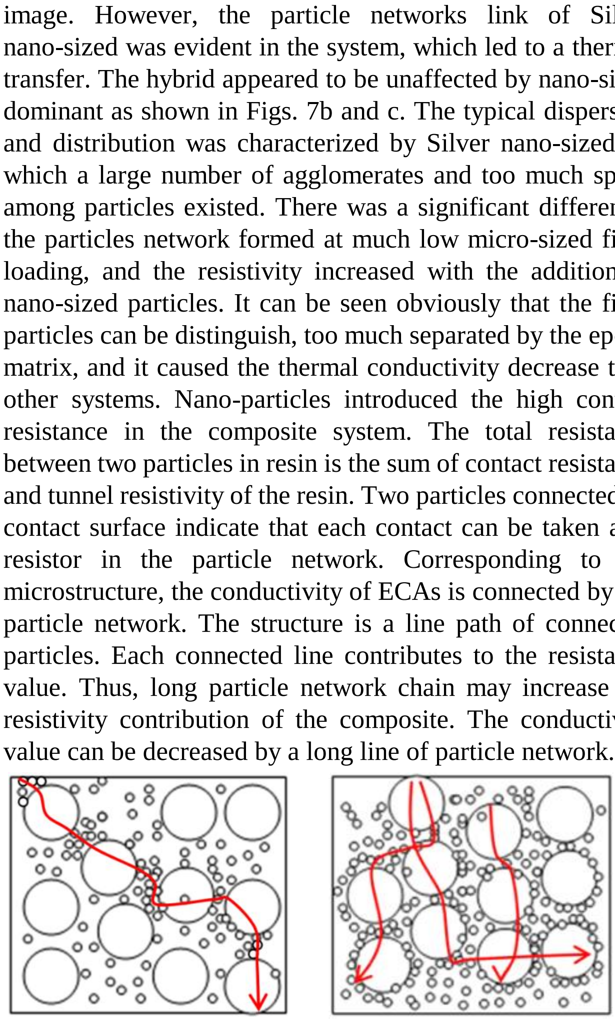


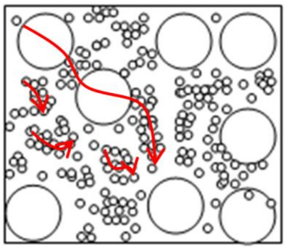
Fig. 6. Morphology analysis of the cross-section of micro- and nano-Ag (6 vol. %) at different ratios: (a) 75:25, (b) 50: 50, and (c) 25: 75. Magnification, 500X.

Fig. 6b illustrates that the Silver particles were well dispersed and tend to homogeneity. The distribution and dispersion were shown significant improving than those in Fig. 6a. This result was verified by founding from the FESEM images of the composite with Fig. 6a. The thermal conductivity decreased due to the decreased micro-sized particle ratio used. The thermal path performance could be evenly spread in the entire ECA, and additional nano-sized particles decreased the thermal conductivity. Particle contact resistance may have enhanced the particle structure system and promoted decreased electron percolation channels and thus reduced thermal conductance. Adding the number of nano-sized particles in the particle structure increased the number of contact points between the particles. This phenomenon can be explained further in the case of the 25/75 ratio. The typical distribution was dominated by the nano-sized Ag particle where a large number of nano-sized was distributed. Consequently, the thermal conductivity value was lower than that of other systems. The addition of nano-sized Ag particles decreased the thermal conductivity due to the agglomeration tendency and increased particle contact resistance. According to morphological analysis, a negative hybrid effect on the particle dispersion was observed when nano-sized Ag particles were dominant.

The volume loading ratios of 75:25, 50:50, and 25:75 promoted the effect of formation on the thermally conductive pathway, as proven by the three models of micrograph scanning of the hybrid-sized composite. As shown in Fig. 7a, the Silver particles were poor in dispersed and distributed by shown in agglomeration, as observed during micrograph image. However, the particle networks link of Silver nano-sized was evident in the system, which led to a thermal transfer. The hybrid appeared to be unaffected by nano-sized dominant as shown in Figs. 7b and c. The typical dispersion and distribution was characterized by Silver nano-sized, in which a large number of agglomerates and too much space among particles existed. There was a significant difference, the particles network formed at much low micro-sized filler loading, and the resistivity increased with the addition of nano-sized particles. It can be seen obviously that the filler particles can be distinguish, too much separated by the epoxy matrix, and it caused the thermal conductivity decrease than other systems. Nano-particles introduced the high contact resistance in the composite system. The total resistance between two particles in resin is the sum of contact resistance and tunnel resistivity of the resin. Two particles connected by contact surface indicate that each contact can be taken as a resistor in the particle network. Corresponding to the microstructure, the conductivity of ECAs is connected by the particle network. The structure is a line path of connected particles. Each connected line contributes to the resistance value. Thus, long particle network chain may increase the resistivity contribution of the composite. The conductivity value can be decreased by a long line of particle network.



75/25 50/50



25/75

Fig. 7. Schematic iillustration of different ratio of micro to nano particles distribution in hybrid polymer matrix about the formation of thermally conductive pathways

D. Electrical resistivity

For electrical resistivity analysis, Fig. 8 presents the electrical resistivity results of the epoxy composites filled with Ag micro and nano-particles. The characteristics of electrical resistivity decreased as Ag particles content increased and extremely decreased until it exceeded the critical volume (V_c) due to the adhesive conductive channels, which started to form the particle network. In this phenomenon, both epoxy composites filled with Ag micro and nano-particles showed similar resistivity trends (Fig. 8). The electrical resistivity of the ECAs started to decrease at 6 vol.%. The condition below the critical volume might be conductive fillers still isolated by the matrix that form the discontinuous particle network in the composite. Good particle network was observed at high filler loadings ($V_f \geq 6$ vol.%), which were denoted by decreased electrical resistivity. However, the electrical resistivity of nano-particles was slightly higher than that of the Ag micro at given filler loading. This phenomenon proved that the electrical conductivity of micro-particles was slightly higher than that of the nano-particle. A similar phenomenon shows that nano-particles negatively affect the electrical conductivity of composite [21].

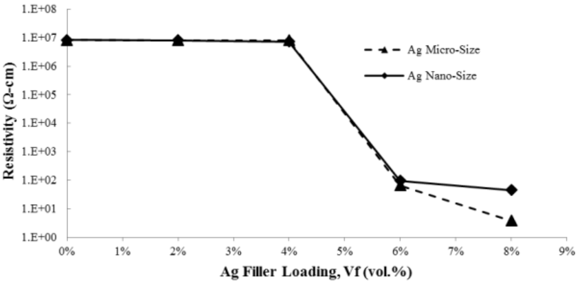


Fig. 8. Resistivity of Ag nano-micro and nano-particle filled epoxy composites versus Ag volume fraction respectively.

The electrical resistivity of the hybrid-sized system is shown in Fig. 9. In this case, the Ag size effect was analyzed in contributing the electrical resistivity. High resistivity occurred in the samples when the composite consisted of high nano-particle ratio (i.e., 25:75). The decrease of resistivity was observed at 25:75, when nano-particle ratio was dominant in the composite. The agglomeration was most probably attributed to the decreased electrical resistivity due to nano-particles contributing to high surface energy attraction (Lee *et al*., 2005). However, when the ratio fraction of nano-particles reached 50:50 mixtures, the mixture of nano- and micro-particles effectively started to fulfill the connection between micro-particles. High nano-particles contents could reduce the electrical conductivity because they clustered and increased the gaps between the micro-particles. This phenomenon has also been reported by previous works showing that the electrical conductivity of the hybrid system composite is increased at decreased nano-particle content [14,15]. This phenomenon was proven by micrographs showing that nano-particles could be filling into the interstices of the micron-sized particles considering their low sintering temperature.

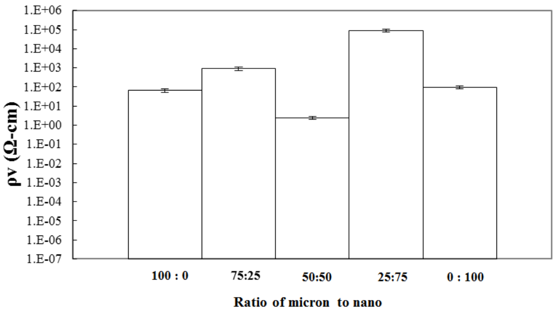


Fig. 9. Resistivity of ECA with 6 vol. % Ag filler loading vs. the ratio of micro- and nano-Ag particles

IV. CONCLUSION

The thermal conductivity characteristics of ECAs by proposing micro- and nano-sized Ag particle effect have been successfully presented. The thermal conductivity of ECAs with micro-sized Ag particle showed positive effects for both volume fraction loading trend and in hybrid-sized system analysis. The ECAs filled with Ag nano-sized particles decreased their thermal conductivity and distribution, which could be attributed to the high resistance between nano-sized particle contacts. FESEM images revealed the distribution of micro-sized fillers in the epoxy matrix, showing that the pathway slightly lowered the particle agglomeration. The nano-sized particles tended to agglomerate in their distribution and increased particle contact resistance. This result decreased electron percolation channels, thereby reducing thermal conductance. Given that the nano-sized Ag particles generated a decreasing effect on thermal conductivity, in the hybrid system observation, a 75:25 volume ratio provided the highest thermal conductivity where micro-sized content was high. The simple addition of nano-particles negatively affected the overall conductivity because of the increased number of contact points. However, a positive effect of nano-particles on the electrical conductivity of the polymer composite required a particular ratio of nano-particles at hybrid system that increases connection particle transport.

AKNOWLEDGEMENT

The authors would like to thank to all people that directly and indirectly involves and support this research especially FRGS grant (203/P.Bahan/6071125) and post-graduate research fund (PGRU) and Universiti Kuala Lumpur Malaysian Institute of Marine Engineering Technology.

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AUTHORS PROFILE

Muhammad Zulkarnain PhD graduated with B.Eng (Hon) in Mechanical Engineering from Syiah Kuala University (UNSYIAH), Indonesia in 2002. He got his M.Eng and PhD degree in Mechanical engineering from Universiti Sains Malaysia (USM), Malaysia in 2007 and 2014 respectively. Currently, He a senior lecturer in Universiti Kuala-Lumpur, Malaysian Institute of Marine Engineering Technology (UniKL-MIMET).

Muhammad Sobron Yamin Lubis, Ph.D graduated with B.Eng in Mechanical Engineering from Islamic University of North Sumatera (UISU), Indonesia in 1991; He got his M.Sc and Ph.D Degree in Mechanical Engineering from Universiti Sains Malaysia (USM), Malaysia in 2000 and 2008 respectively. In 1994-2011 he worked at Muhammadiyah University of North Sumatera as a lecturer in the Mechanical Engineering department and in 2011 until now he has worked as a lecturer and Head of Manufacturing Engineering Section at Tarumanagara University in Indonesia.

Ir. Silvi Ariyanti M.Sc graduated with B.Eng in Industrial Engineering in North Sumatera Islamic University, Indonesia in 1996. She got her M.Sc in Mechanical Engineering from Universiti Sains Malaysia (USM), Malaysia in 2002. In 2002-2011 he worked at North Sumatera Islamic University as a lecturer in the Industrial Engineering department and in 2011 until now he has worked as a lecturer and Secretary of Industrial Engineering department at Mercu Buana University in Indonesia.

Prof. Ir. Dr. Mariatti Jaafar @ Mustapha, School of Materials & Mineral Resources Engineering, 14300 Nibong Tebal, Pulau Pinang. A part as academician and researcher experience more than 10 years involvement, She already supervised postgraduate students at Universiti Sains Malaysia (USM) with coming from different countries around the world. She also excellent in writing high impact Q1 journals which is has been published through higher citation index measurement including SCOPUS, ISI and many of them placed with more than 100 research papers for publications and awards..