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Enhancing the Mechanical and Electrical Properties of Adhesive Coating Materials by Cu@Sn Core Shell Nanomaterials Mixed with Hybrid Polymeric Composite Material

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ABSTRACT

A new adhesive consisting of Cu@Sn core shell nanomaterials prepared through a series of chemical deposition reactions to preserve the copper nanoparticles from oxidation, where they were coated with tin. The ultimate goal of this process obtaining certain mechanical, physical and thermal properties. This mixture of Cu@Sn core shell nanomaterials was added at a weight ratio of 15 % to a polymeric composite material consisting of polyvinylpyrrolidone (PVP) and polyvinyl alcohol (PVA) in equal proportions of the two polymer materials (PVP and PVA). The mechanical, optical and thermal properties tests of the new adhesive bonding layer, the result which was characterized by a shear stress approximately (τ) 8 Mpa and with electrical conductivity (σac) 15 Ω cm^{-1} for Cu@Sn core shell nanomaterials. After mixing with polyvinylpyrrolidone (PVP) and polyvinyl alcohol (PVA), the electrical conductivity was found to be 1.8×10^{-3} Ω cm^{-1}. In light of these results, the new adhesive material promises wide applications in the electronic, automotive, aerospace and medical devices industries due to the attractive properties of the material in addition to its friendliness. Prog. Color Colorants Coat. 18 (2025), 189-199© Institute for Color Science and Technology.

1. Introduction

The creation of the adhesive involved using a mixture of materials to achieve the desired characteristics. One important component, in the production process was the Cu@Sn core shell, which was formed following a method. Additionally, during the blending phase Polyvinyl Pyrrolidone (PVP) and Polyvinyl Alcohol (PVA) were added to ensure dispersion and bonding within the adhesive. Incorporating these materials played a role in improving both the electrical properties of the metallic nanocomposite adhesive. By incorporating the Cu@Sn core shell, a strong foundation for the adhesive was established while also ensuring conductivity. The inclusion of PVP and PVA helped boost the adhesion strength and flexibility of the adhesive making it suitable, for industrial applications [1, 2].

Some micromechanical theories have made predictions, about the properties of materials but the unpredictable nature of each system and the distinct qualities of its components pose challenges. Certain theories aid in the advancement of materials and reinforcements. Factors like filler characteristics, interphase development and matrix dispersion play a role, in predicting the properties of polymer based nanocomposites. To enhance the performance of materials researchers, need to grasp these aspects and apply suitable modeling techniques [3]. Epoxy glues have the potential to substitute fasteners, in industries such, as automotive, aerospace, electronics, construction, sports and packaging [4]. The selection of terials for coating implants includes options such as metal oxides

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and nitrides [5].

The rapid growth of the electronics industry has led to a demand, for epoxy based conductive adhesives to cool down high performance devices [6]. If heat is not effectively dissipated at or above the rate it is produced the components of the device will continue to heat up leading to reduced reliability and performance. The concept of failure factor as defined by the US Department of Defense relates to the ratio of failure rates at temperatures compared to expected temperatures [7]. Overheating can result in fractures and deformations in the microstructure ultimately causing system failures concerns regarding user safety and health [8] as losses in electronics assembly processes [9]. Conventional thermal management methods may not be sufficient for cooling heat chips; therefore, efficient and reliable electronic devices necessitate suitable connecting materials with excellent thermal properties [10]. The failure rate escalates significantly beyond 75 degrees Celsius according to a graph provided. Epoxy based conductive adhesive (TCA) can effectively link components together. Dissipate device heat. These adhesives are capable of bonding chips with substrates, metal elements, polymer composites and concrete structures. Tasks that other methods may not achieve satisfactorily. Epoxy glue undergoes polymerization and crosslinking processes that offer advantages in microstructure such as diverse curing techniques, low curing thresholds and strong weight resistance capabilities [11]. A visual representation in Figure 1 illustrates how failure rates, for devices increase with rising temperatures [12].

Numerous studies have focused on developing polymer nanocomposites, with metal nanoparticles to control their electrical properties. This involves selecting combinations of polymers and metal nanoparticles well as managing factors like particle size, concentration and distribution within the polymer matrix. Controlled manufacturing processes are employed in creating high performance capacitors conductive inks, electronic devices, sensors and other applications [13]. In epoxy amine systems the introduction of carbon nanoparticles influences dynamic mechanical and dielectric properties based on filler content and curing conditions. The design of pathways within the structure enhances electrical conductivity at specific concentrations of carbon black [14].

Research indicates that enhancing the properties of pyrrolidone/carboxymethyl cellulose blends can be achieved by incorporating tungsten trioxide (WO_3) nanoparticles. The combination of WO_3 nanoparticles with components establishes an interfacial connection that boosts the performance of nanocomposites [15]. Introducing fillers into polymer matrices is recognized to improve radiation resistance, thermal stability, and overall mechanical strength [16].

The epoxy-based adhesive was used because of its superior mechanical properties and to improve these properties, copper nanoparticles were added to it in different proportions 1, 2, 5, 10, 15 and 20 % and it was found that 15 % was the best in improving the mechanical properties while 20 % was the best in improving the thermal conductivity [1]. Adding nanoparticles such as CuNPs to epoxy-based adhesives is recognized to enhance their characteristics. Understanding the failure processes is crucial and may be obtained using SEM imaging. The addition of additives such as CuNPs to epoxy adhesives may increase the flexibility of joints [2]. In addition, the mechanical strength of polymer composites may be increased by using fillers [17]. Polyvinyl alcohol (PVA) is a notable choice as a polymer matrix for nano-composites. These polymer composites function as radiation shields because of their flexibility and superior mechanical qualities [18]. Various industries heavily rely on adhesives to connect materials together. Adhesives play a role, in applications such as paints, coatings, lightweight structures, high strength components and solutions for preventing corrosion. In the aerospace industry the combination of SiO_2/CNT nanocomposites in PVDF HFP provides properties like icing resistance and superhydrophobicity. Additionally, the use of oxide nanosheets (GONs) along with epoxy based polyaniline (Epoxy PANI) offers benefits in preventing fouling. Polymer nanocomposites used in vehicle components are valued for their efficiency due to their nature. Enhanced mechanical properties. Moreover natural fiber polymer composites are preferred for their qualities like strength, design flexibility, impact resistance, corrosion resistance, noise reduction capabilities, cost effectiveness and biodegradability. These composites find applications in parts such as door panels seat backs, floor mats, dashboards among others due, to their exceptional characteristics [19].

In summary advancing the understanding and enhancement of the characteristics in polymer based materials is crucial for developing nanocomposite adhesives with enhanced functionalities, for various industrial purposes. Also achieving management of synthesis parameters and thorough blending, with polymers are essential for creating the Cu@Sn core shell configuration and incorporating it into an adhesive compound. This strategy aims to boost the conductivity and mechanical durability of the nanocomposite adhesive enhancing its effectiveness in industrial settings [2, 20].

This research focuses on enhancing the strength and conductivity of adhesives aiming to improve bonding capabilities, across industries such as electronics, automotive, aerospace and medical devices. By incorporating metal nanocomposites into adhesives new opportunities arise to boost performance and longevity in applications. Furthermore this study contributes to advancing functionality through exploring synthesis techniques, preparation methods and characterization approaches. The outcomes of this study may lead to enhanced solutions that better cater, to the evolving needs of industries.

2. Experimental

2.1. Materials

The nanocomposite adhesive was crafted using materials. The synthesis process involved creating a Cu@Sn core shell through a technique. PVP and PVA were introduced to the adhesive, for dispersion and bonding purposes. These additions enhanced the properties of the adhesive. The Cu@Sn core shell acted as both a base and a conductor. The inclusion of PVP and PVA boosted strength and flexibility making it suitable for applications. By selecting and preparing these components specific qualities could be customized. A balanced blend was formulated to optimize strength, electrical conductivity and adhesion. Meticulous material selection and processing techniques defined the characteristics of the adhesive. Through an approach that considered each ingredient, a robust industrial adhesive, with electrical properties was developed.

2.2. Preparation of the polymeric composite

Two types of polymeric materials, namely polyphenol-pyrrolidone and polyphenol alcohol were used to find the best polymeric compound that gives the best adhesion properties. These materials were provided from local markets. Table 1 shows the characteristics of these materials [21].

2.2.1. Activation of polyvinyl alcohol (PVA)

10 g of polyvinyl alcohol are taken with 90 mL of distilled water and placed in a circular flask equipped with a nozzle connected to a condenser, and heated in a water bath at a temperature of 90 degrees Celsius for 6 hours continuous hours, after which the polyvinyl alcohol ready for the mixing process. The result shows in Figure 1 represents the working method.

Table 1: Properties of PVP and PVA.

Property	PVP	PVA
Appearance at 25 ℃	White powder	White powder
Molecular Weight (g/mole)	40000	1750
Glass temperature Tg (℃)	163	200
Bulk density (g/cm^3)	0.5	1.19

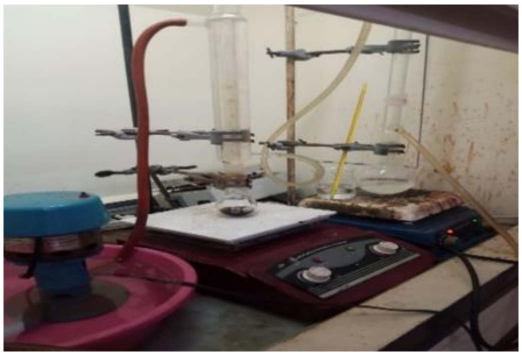

Figure 1: Preparing viscous solutions.

2.2.2. Activation polyvinylpyrrolidone (PVP)

This was done by adding 16 g of (PVP) to 50 mL of distilled water, the mixture stirred by a mixer, at room temperature for 30 minutes, whereby a PVP solution was ready to be used. as in shown Figure 2.

2.3. Preparation of Cu@Sn core shell nano-composite

The process entails following a series of steps. To make sure, all materials, such as copper grains, tin and all the solutions which involved in the deposition reactions needed to form the core and shell, must be secured. The distinctive design of the Cu@Sn core shell recognised for its properties in the setup is quite remarkable. In the synthesis procedure, tin ions are deposited on copper nanoparticles to create the core-shell structure through a deposition reaction. Varying factors, such as temperature, reaction duration and the amounts of materials involved in the reactions can help to achieve the intended core-shell shape. Table 2 illustrated all the materials and quantities which needed to obtain Cu@Sn cor shell. The preparation process achieves in the stages as shown below.

The first stage: the Sodium hypoph-sphite, Hydro quinone, EDTA and Thiourea substances were dissolved in 480 mL of distilled water, while the Methane sulfonic acide substance was dissolved by adding 2.7 mL of glycol and then added to the previous solution with continuous stirring with a magnetic mixer at room temperature until the mixture was well homogeneous.

The second stage: dissolve 12 g of tin chloride with 6 mL of hydraulic acid, and continue stirring until the chloride is completely dissolved.

The third stage: the Cu@Sn core shell process consists of cleaning the copper granules which obtained from local markets with an average grain size of 48 nanometers by placing 12 g of these granules in a cylindrical glass container containing an ethanol solution with 5 % hydrochloric acid. Then the mixture put the container and then place an ultrasonic device which was used to clean the copper granules from contaminants and the oxidized outer layer for half an hour, and the granules were washed six times with deionized water.

Table 2: Materials used in forming the Cu@Sn core shell.

No.	Material name	Mass (mol/L)	M.W	Required quantity (g)
1	Sodium hypoph-sphite	0.2	105.99	1.696
2	Hydro quinone	0.0036	110.11	0.0317
3	EDTA	0.0014	292.25	0.0327
4	Thiourea	0.65	76.12	3.95824
5	Methane sulfonic acide	0.0042	96.11	0.1936

Figure 2: Preparing the polymer polyvinylpyridone.

The fourth stage: Tin chloride dissolved is in the solution prepared in the first stage with continuous stirring using a magnetic stirring device until the mixing is complete and the color of the mixture changes from white to grey. Next, the freshly washed copper granules were dropped into the final mixture, as quickly as possible, with continuous magnetic stirring for 3 hours. Finally as result a Cu@Sn core shell was obtained after filtering the last solution.

2.4. Mixing different compounds

When creating adhesive it's important to mix PVP and PVA. PVP acts as a stabilizer to prevent nanoparticles from clumping. Ensures even homogenies distribution of the nanoparticls in the mixture. While PVA enhances the strength and adhesion properties [22]. By combining PVP and PVA in the Cu@Sn core shell during mixing it improves compatibility and interactions within the mixture coating each nanoparticle uniformly with polymer material. Adjusting factors like temperature, stirring speed and time can help achieve a dispersion of nanoparticles with PVP and PVA [23]. Additionally including PVP and PVA in the formula can improve conductivity by aiding electron transfer between nanoparticles resulting in enhancements in mechanical and electrical performance. The combination of PVP and PVA is essential for producing quality adhesive suitable, for industrial use [24]. Several mixtures have been prepared and tested to obtain the best mechanical, electrical and thermal properties, as illustrated below: Mixing polyvinyl pyrrolidone with polyvinyl alcohol in different proportions (0, 25, 50, 75, 100 %) as one relate to the other.

These four types of mixtures, each type is differ in composition from others, were tested by taking layer of the new adhesive bonding nanocomposite materials and placing between two plats of copper as shown in Figure 3. These samples that were manufactured for shear tests of different adhesives bonding and applying 5 MPa pressure at different temperatures (150, 175, 200, 225 and 250 ℃) and holding time for 10 minutes using a pre-prepared piston for this purpose.

It was found that the best shear stress obtained was when polyvinylpyrrolidone was mixed with polyvinyl alcohol in equal proportions, i.e. 50 % of each. Later, metallic composite materials Cu@Sn and polymeric composite materials were added in proportions of (5, 15 and 25 %).

3. Results and Discussion

3.1. Microstructure study

3.1.1. SEM and EDX

The Scanning Electron Microscope (SEM) is commonly utilized in materials science for examining the structure, shape and composition of materials. It allows us to study the surface features, grain patterns and flaws, in metals and composite materials offering insights for material advancement. Figure 4 shows the SEM test before and after the core shell process, In addition, Figure 5 shows energy dispersive X-ray spectroscopy (EDX) to determine the elemental composition of copper and tin-plated copper.

It is clear from the photographs the Tin composition precipitated and form coating layer surrounding the copper particles with nanocomposite which known as Cu@Sn core shell which it will be responsible for improving the electrical properties of the adhesive bond layer as shows in Figure 4b. Figure 5b shown EDX the intensity of the picks of the nanocomposite material which form on the surface of copper compare with EDX of the pure copper Figure 5a.

Figure 3: Manufactured copper samples to place the adhesive between them.

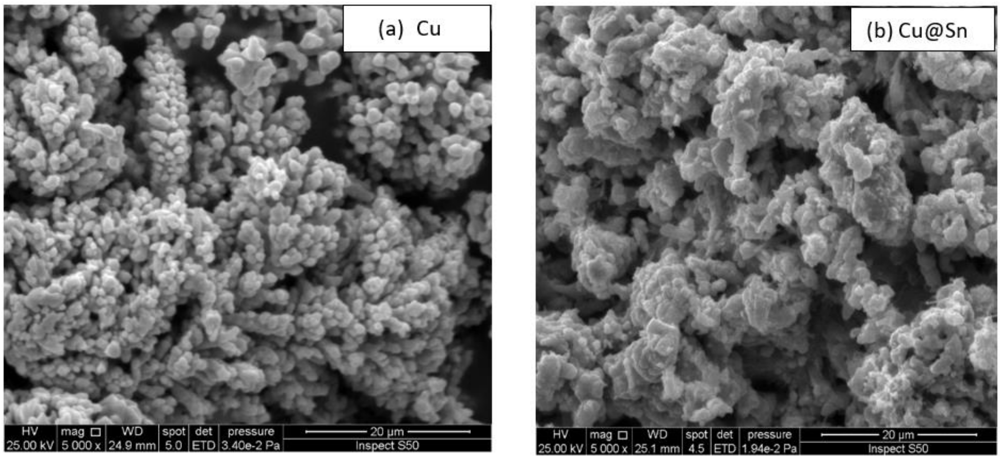

Figure 4: SEM of (a) Cu and (b) Cu@Sn core shell.

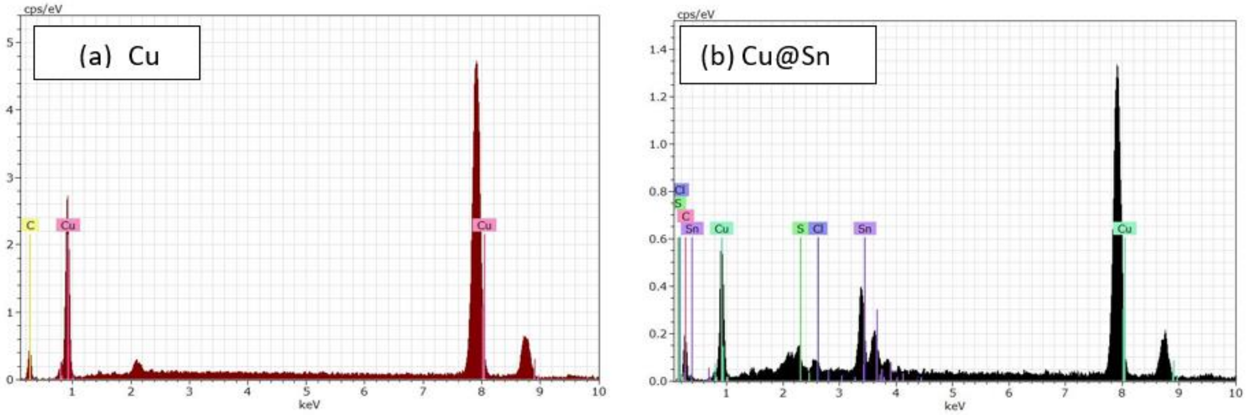

Figure 5: EDX of (a) Cu and (b) Cu@Sn core shell.

3.1.2. Scan Probe Microscopy (SPM)

Scan Probe Microscopy is a method of studying the characteristics of materials on a small scale. This technique involves using scanning microscopy to generate three images of a specimen. The use of scan probe microscopy offers insights, into the surface and structural features of materials at high levels of detail. This technology finds applications in areas, like nanotechnology and biomaterial research.

Table 3 shows the grain size analysis of the copper nanomaterial before and after coating with tin film from scanning probe microscopy analysis, which shows that the grain size of the copper particles was 48 nm and the thickness of the tin film was 5.215 nm.

Table 3: Grain size of the nanomaterial before and after coating.

Function	Cu	Cu@Sn core shell
Avg. Size nm^2	7973.59	9709.25
Avg. Height nm	48.21	53.32
Avg. Diameter nm	100.76	111.19
Max. Size nm^2	136051.72	300129.72
Min. Size nm^2	137.32	137.82

3.2. FTIR

Fourier transform infrared spectroscopy (FTIR) is one of the most widely used analyses in identifying components and bonds in organic and inorganic materials and is a subset of spectroscopy. The PVP and PVA test was carried out to determine the type of material tested by comparing it with the standard specifications and also to know the degree of polymerisation and Figure 6 shows the test of both materials.

3.3. Mechanical study of adhesives

First, PVP & PVA were mixed without adding Cu@Sn to reach the best mixing ratios between the two polymers in different proportions (0, 25, 50, 75, 100 %) one relative to the other. By placing the material between two shear test pieces that were made according to the results, it appears clear from The tables shown below.

Table 4 shows the test results of the samples that were tested for the adhesive in them for different mixing ratios between PVP and PVA.

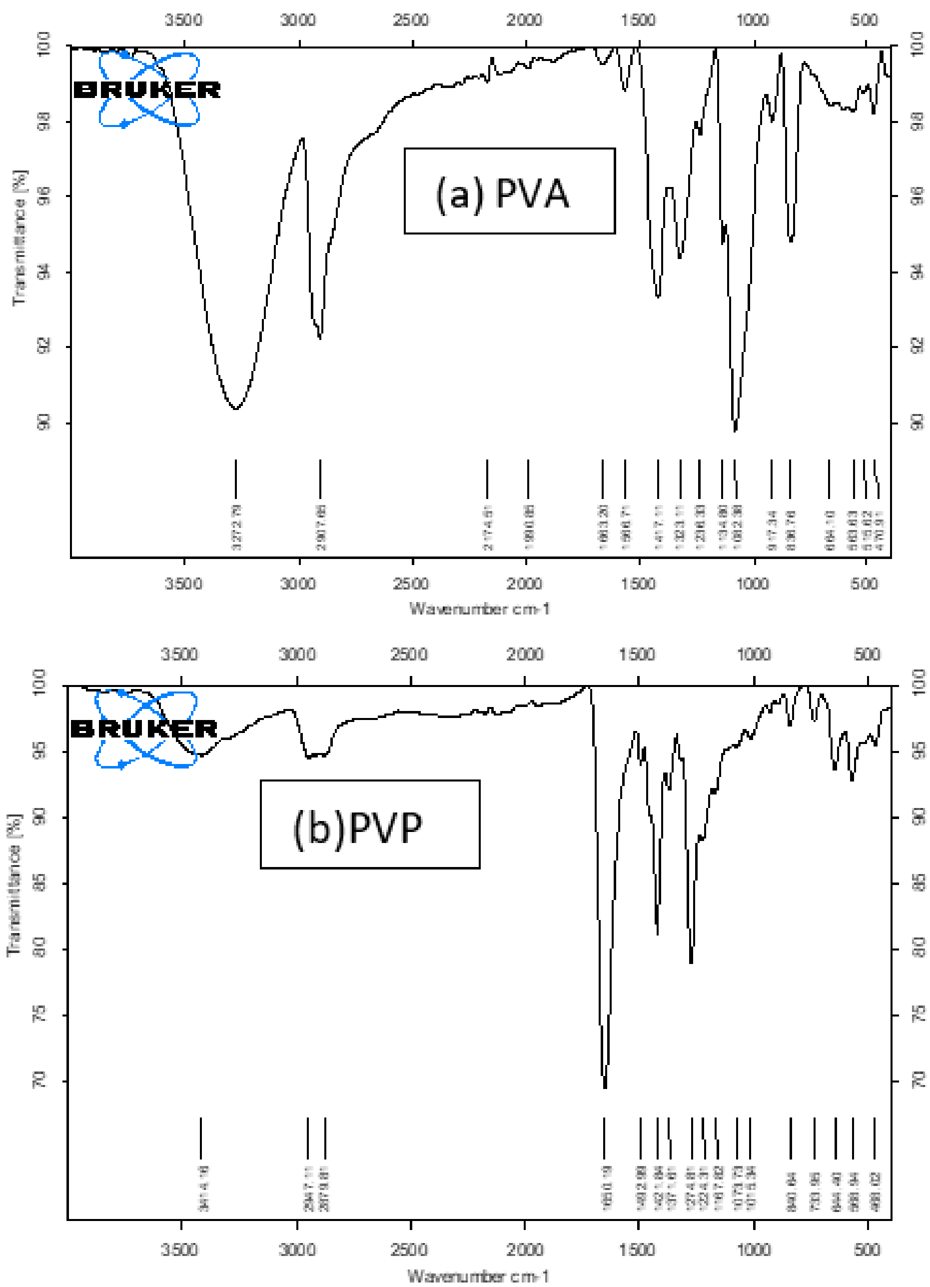

Figure 6: FTIR of (a) PVA and (b) PVP.

Table 4: Shear stress values for samples according to PVA & PVP mixing ratios.

NO.	T (°C)	P (Map)	t (min)	Shear Stress Map for 100 % (PVP)	Shear Stress Map for 25 % (PVA)	Shear Stress Map for 50 % (PVA)	Shear Stress Map for 75 % (PVA)	Shear Stress Map for 100 % (PVA)
1	150	5	10	2.08	3.24	3.4875	1.5	1.15
2	200	5	10	1.75	5.685	5.4685	6.14	1.385
3	225	5	10	1.28	3.155	6.65	2.362	1.122

By comparing the results in the above table, it is clear to us that the best addition ratio of PVP to PVA was at 50 % for both of them, which gave the best shear stress. Then by chosen the best percentage of adding the nanometallic compound Cu@Sn core shell and indicate the extent of its effect on each of the mechanical, electrical and thermal properties, as shown in Table 5.

By comparing the results in the table above, it is clear that the best percentage of the metal nanomaterial was 15 %, which gave the best shear stress. The results illustrated in Table 6 shown a comparison between some of the results reached (50 % PVP@PVA+15 % CU@Sn) and the results extracted from the study [25], where it clearly appears to us that the prepared adhesive material gave better mechanical properties.

3.4. Electrical conductivity

The electrical conductivity test was carried out using the RLC apparatus shown in Figure 7 for the adhesive layer which was fabricated and placed between two pieces of copper designed to fit the test apparatus with an adhesive area equal to that used in shear tests, under different bonding conditions in terms of applied pressure, temperature and time.

Figure 7: RLC device.

Table 5: Effect of adding Cu@Sn core shell to the polymer (PVP+PVA).

NO.	T (°C)	P (Map)	t (min)	Shear Stress Map for (PVP+PVA) +5 % Cu@Sn	Shear Stress Map for (PVP+PVA) +15 % Cu@Sn	Shear Stress Map for (PVP+PVA) +25 % Cu@Sn
1	150	5	10	2.903	3.356	4.139
2	200	5	10	4.569	4.414	6.376
3	250	5	10	2.179	6.826	1.342

Table 6: A comparison between the results obtained and the results of the source [25].

NO	T (°C)	P (Mpa)	t (min)	Shear stress (Mpa)	Shear stress (Mpa) [25]
1	150	5	10	3.49	-
2	200	5	10	7.44	3.084
3	225	5	10	7.793	3.843
4	250	5	10	3.738	3.174

Table 7: Electrical conductivity values for four samples under different conditions.

Sample	P (Mpa)	T (℃)	t (min)	σac (Ω cm^{-1})
50 % (pvp+pva) + 15 % Cu@Sn	0	200	20	1.11×10^{-6}
50 % (pvp+pva) + 15 % Cu@Sn	5	200	10	3.53×10^{-3}
Cu@Sn	5	200	10	1.8×10^{-3}
Cu@Sn	10	250	30	15

Different methods are used to study the conductivity and electric field behavior. The four point probe measurement is a technique that assesses resistance by passing a current through four probes on the sample and measuring the voltage drop. This method is widely used for conductivity testing. Spectroscopy on the hand examines how materials respond to frequencies of electric fields to reveal dielectric properties [26]. Various factors influence adhesives. The conductivity of composites is determined by the type and amount of nanoparticles. Carbon nanotubes and graphene are known for their ability to conduct electricity due, to their structures and aspect ratios, which enhance properties [19]. Furthermore the surface morphology plays a role in performance enhancement. Nanocomposites with structures or surface modifications show conductivity and interfacial interactions. To optimize the functionality of adhesives it is essential to understand how these components impact their properties [27]. For instance techniques, like four point probe measurement and dielectric spectroscopy can be utilized. By adjusting nanoparticle composition and surface characteristics metallic nanocomposite adhesives can be enhanced [24].

The real ($\varepsilon r'$) and imaginary ($\varepsilon r''$) parts of the permittivity of the material were calculated by using the following formulas (Eqs. 1 and 2):

$$\varepsilon r' = \frac{Cpd}{S\varepsilon 0} \qquad (1)$$

$$\varepsilon r'' = \varepsilon r' * \tan(\delta) \qquad (2)$$

where *Cp* is the capacitance of the sample, *d* is the thickness of the disk, *S* is the surface area of the electrodes, and $\varepsilon_0 = 8.85 \times 10^{-12}$ F/m is the permittivity of free space. The capacitance (*Cp*) and loss factor (tan(δ) or *D*) can be obtained directly from the measurements. The conductivity data were obtained using the following relation (Eq. 3) [27].

$$\sigma ac = \varepsilon_0 \varepsilon r'' \omega. \qquad (3)$$

Four samples were tested under different gluing conditions in terms of applied pressure, temperature and pressure application time. Two samples were selected, two with the polymeric material and two with its addition to study the effect of adding the polymeric material on the electrical conductivity property, where the samples were tested under the influence of a variable frequency (1-10000) KHZ and room temperature and the electrical conductivity was calculated through equation 4 (Table 7).

4. Conclusions

The adhesive was prepared from a material consisting of a polymeric composite material PVP & PVA in equal weight ratios and mixed with a nano-metallic composite material consisting of copper particles coated with tin particles (Cu@Sn core shell) produced by chemical deposition process with a weight ratio of 15 % of the polymeric material. The mechanical and physical tests applied to dozens of samples showed excellent characteristics in the possibility of using the new adhesive in bonding similar and dissimilar materials, as well as in the possibility of using it in bonding electrical components, as the shear stress value of the standard sample reached under conditions (225 ℃, 5 MPa and 10 min) of approximately 7.8 Mpa This is an excellent value for many applications. In addition to the fact that the material has electrical and thermal conductivity properties because its composition included metallic composite materials. In addition, the fact that the material is environmentally friendly is an important matter as the European Union seeks to eliminate the use of lead in electronic and other industries, and ease of use. The meticulous processing and selection of elements significantly influenced the characteristics of the metallic nanocomposite adhesive. A strong adhesive, with improved electrical properties, for industrial applications was effectively developed through a systematic approach carefully considering the contribution of each component.

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