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Stress Analysis on Hybrid Nanoparticle GNP/Ag for Flexible Semiconductor Packaging Devices

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ABSTRACT

This research investigates the stress analysis of hybrid nanoparticle GNP/Ag conductive inks under various curing conditions, specifically varying time and temperature settings. The study addresses the challenges of reliably quantifying the adhesive strength of printed conductive inks for flexible semiconductor packaging devices, aiming to ensure the reliability and long-term sustainability of printed electronics under various environmental conditions. To formulate the GNP hybrid conductive ink, GNP powder was mixed with ethanol and sonicated. Subsequently, Ag and SA were added to the GNP/ethanol mixture, followed by sonication for one hour with each addition. The resulting GNP/Ag/SA mixture was heated on a hotplate and stir using a magnetic stirrer until the ethanol evaporated and then cured in a furnace for one hour. The mixture was then ground into a fine powder and combined with 1-butanol and terpineol to form a paste. This paste was printed in overlapping layers on two copper substrates and cured at 240°C, 250°C, and 260°C for 4, 5, and 6 hours respectively. Testing was conducted in accordance with ASTM D1002 Standard Test Method to determine the shear strength of adhesives using the lap shear test. The findings indicated that increased curing temperatures and duration negatively impact the shear strength of the conductive ink, highlighting the correlation between curing conditions and adhesion properties. Future research should focus on integrating additives or modifying the ink composition to enhance adhesion qualities during high-temperature curing.

1. Introduction

Flexible electronics have significantly transformed the semiconductor packaging devices industry, presenting fresh opportunities for application in wearable technologies and is considered one of the most emerging technologies [1,2]. Integrating hybrid nanoparticles, specifically Graphene Nanoplatelets (GNP) and Silver (Ag), into semiconductor packaging materials has become a potential

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method to improve electrical and mechanical properties. This occurrence prompts substantial research in the area of conductive ink production, with a particular emphasis on strengthening adhesion, improving thermal conductivity, and optimising electrical and mechanical performance [3, 4]. Die-attach adhesives are required for the production of internal components in various electronic devices. An essential element of these devices is the conductive ink utilised to form electrical pathways on flexible substrates.

The significant challenge in printed conductive inks for flexible semiconductor packaging devices involves the necessity for part testing and qualifying methods that take into account both electronic performance and functioning in severe environmental conditions, such as high temperatures and humidity, as well as vibration and shock [1-3]. Nevertheless, there are only a few techniques accessible for accurately quantifying the adherence of printed inks to ensure sufficient system performance. Printed systems that have integrated functionality necessitate the deposition of varied layers, including additional structural elements, onto electronic characteristics [1]. However, existing methods are not adequately suitable for assessing the interfacial strengths of the printed inks due to the fact that the inks are applied and cured in a manner similar to a coating, but subsequently serve as the underlying layer for successive layers. Coating adhesion tests such as scotch tape testing and cross-hatch scratch testing can be used to assess the ink's ability to adhere to the deposition substrate but these tests only offer qualitative indicators of adhesion, making it challenging to establish accurate comparisons [4].

Hybrid nanoparticle conductive inks, specifically including graphene nanoplatelets (GNP) and silver (Ag), have attracted considerable interest owing to their distinctive blend of electrical conductivity and mechanical flexibility [2,5,6]. These hybrid inks utilise the exceptional electrical properties of silver and the outstanding mechanical strength and flexibility of graphene, making them well-suited for applications involving flexible semiconductor packing. In conductive ink formulation, the conductive material is the most essential component. The choice of conductive materials depends on the physical characteristics of the pattern to be printed, such as its ability to adhere to the substrate, and the desired physiochemical characteristics of the inks, such as their compatibility with the printing process [2]. This covers limits in nozzle size, aggregation, stability, rheology, electrical and mechanical properties from nanoparticle printing [7-12]. The efficacy of these conductive inks relies significantly on their capacity to endure mechanical strains, which are common in flexible electronic devices. The incorporation of GNP and Ag nanoparticles improves the mechanical strength and electrical conductivity of the ink, hence enhancing the overall performance of the semiconductor devices [13].

Stress analysis plays an important role in assessing the effectiveness and reliability of semiconductor packaging materials. It assists in comprehending the mechanical characteristics of these materials, such as bending, stretching, and compression, that they undergo during the process of manufacturing and operation. Nevertheless, the examination of the stress distribution inside the hybrid nanoparticle matrix and its influence on the device's lifespan during operational conditions continues to be a crucial field of research. Current study has concentrated on the quantitative analysis and experimental validation of GNP/Ag hybrid inks under different environmental circumstances. Wahid *et al.,* [14] examined the thermal and mechanical characteristics of GNP/Ag die-attach adhesives, emphasising the important contribution of hybrid nanoparticles in improving heat conductivity and mechanical stability. Similarly, Zotti *et al.,* [15] highlighted the significance of aligning and distributing nanoparticles within the polymer matrix to enhance the electrical and mechanical characteristics of the composites.

The development of advanced manufacturing techniques, such as inkjet printing and screen printing, has facilitated the precise deposition of hybrid nanoparticle inks on flexible substrates.

These techniques allow the production of complicated electrical circuits with specific and detailed characteristics, which are important for reducing the size of flexible semiconductor devices [16]. The reliability and durability of these devices under cyclic loading circumstances depend significantly on the optimisation of curing procedures and the comprehension of stress-strain behaviour in hybrid nanoparticle inks [17]. From past literature, the behavior of graphene nanoplatelets under stress testing was analyzed to understand the performance of conductive ink during fatigue tests. The study found that resistance increased over time when the ink was subjected to cyclic stress.

However, there is a significant gap in understanding the impact of curing time and temperature on the adhesion and conductivity of hybrid GNP/Ag conductive inks, despite these advancements. This gap is significant because optimizing these parameters is essential for enhancing the durability and performance of flexible semiconductor devices in real-world applications. Hence, the objective of this research is to do a stress analysis on GNP/Ag hybrid nanoparticle conductive inks utilised in flexible semiconductor packaging devices. This research endeavours to optimise the formulation and processing conditions in order to improve the adhesion and conductivity of the hybrid inks by analysing the effects of varying curing times and temperatures, as well as the mechanical stresses that are encountered during operation. The results of this study will advance the field of flexible semiconductor packaging by facilitating the development of more efficient and dependable flexible electronic devices.

2. Methodology

2.1 Material Preparation

In this research, the material of GNP hybrid conductive ink consists of various constituent each with a distinct role in improving the overall performance of the ink. Graphene nanoplatelets (GNP) are use as the main filler because of its unique physicochemical features, excellent mechanical strength, and distinct electrical and thermal conductivities [18]. Silver flakes (Ag) are incorporated because of its remarkable oxidation resistance and electrical conductivity that allow it to adhere well to substrates [19]. Silver Acetate (SA) operate as a precursor for silver nanoparticles, improving conductivity and stability, Ethanol serves as a chemical solvent for the purpose of dissolving and uniformly mixing the components, hence assuring a homogeneous formulation of ink [16], 1-butanol and terpineol bind ink to substrate, enhancing flow and film development. Material in preparing the GNP hybrid conductive ink is shown in Table 1.

Table 1
Material of GNP hybrid conductive ink

Type	Material
Filler	Graphene nanoplatelets (GNP) with 25 µm nanoparticle size. Silver flakes (Ag) with 10 µm particle size. Silver Acetate (SA).
Chemical solvent	Ethanol 99.99%.
Organic solvent	I-Butanol 99.9% butyl alcohol. Terpineol ~ 65% α, ~ 10% β, ~ 20% γ substances.

2.2 Formulation of GNP Hybrid Conductive Ink

For the preparation of GNP hybrid conductive ink, 0.5 g of GNP powder (xGnP H-5) with 25µm particle size as the main filler was mixed with 50 ml ethanol and sonicated for 10 minutes. The mixture then was added with 4.292 g of Ag and sonicated for one hour followed by the addition of 0.42 g of SA and sonicated for another one hour as shown in Figure 1.

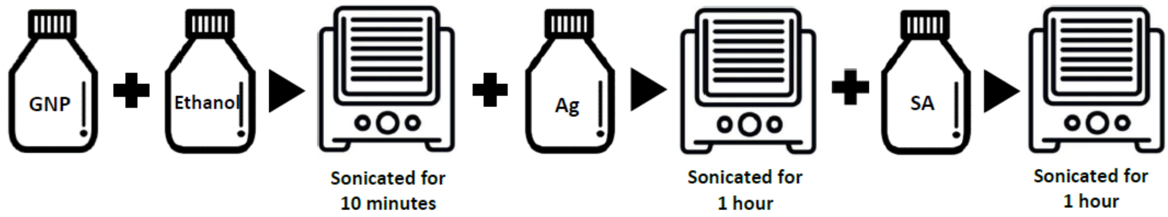


Fig. 1. Filler mixture of GNP hybrid

The solution of GNP/Ag/SA and ethanol was then heated on a hotplate at 70 °C while being stirred at 200 rpm until all the ethanol had evaporated. The GNP/Ag/SA mixture was then put into a porcelain beaker and heated at 250 °C for one (1) hour in a furnace to cure. After the curing process, the GNP/Ag/SA mixture was pound until it produced a fine powder as shown in Figure 2.

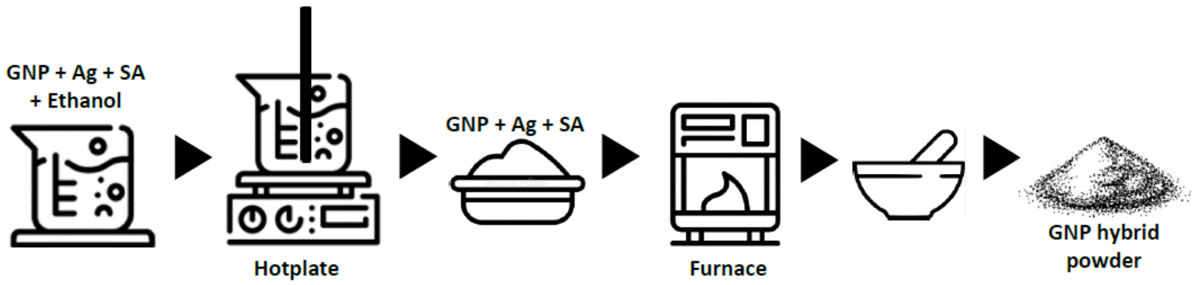


Fig. 2. Formulation preparation for GNP hybrid powder

For the preparation of the GNP hybrid paste, 45 drops of butanol and 45 drops of terpineol were added to the GNP hybrid powder. Before placed in the Thinky mixer for the mixing process, the mixture of GNP hybrid powder with the organic solvent as binder were weighted to ensure the right setting configured in the mixer machine. The process is illustrated in Figure 3.

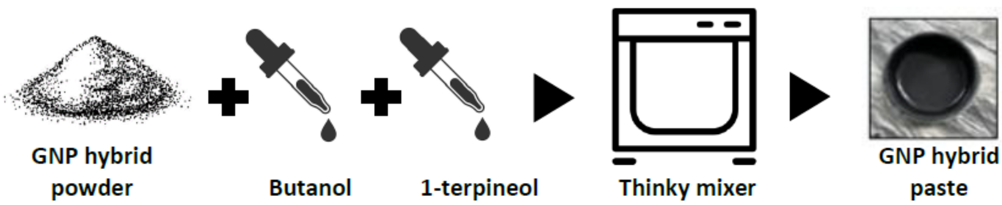


Fig. 3. Formulation preparation for GNP hybrid paste

2.3 Sample Preparation

For the sample's preparation, copper substrates were cut base on the required size. The dimensions of the substrate are 20 mm (*l*) and 5 mm (*w*) as shown in Figure 4. By preventing oxidation of the copper substrates and enhance adhesion between the coating and substrate, sandpaper was used to scratch the surface. Copper substrate samples were prepared to suit the requirements of the shear test. A rectangular shape was chosen for the shear test to facilitate the testing of two substrate samples with bonded interfaces of printed conductive ink. This shape is compatible with the clamping mechanism of the shear test machine.

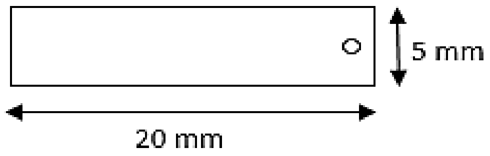
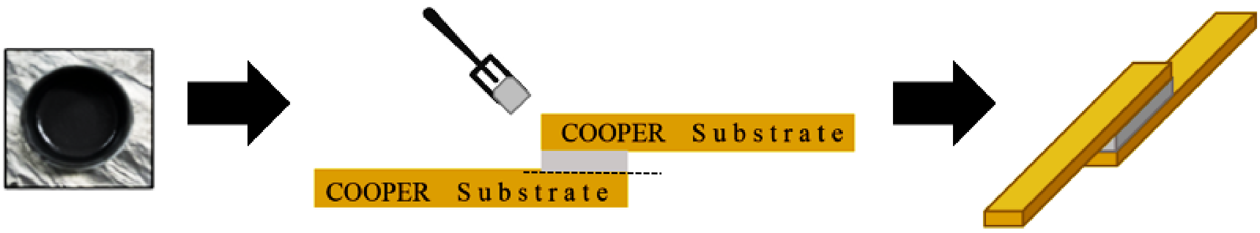


Fig. 4. Dimension of the cooper substrate

2.4 Printing Process

The preparation of printing process involves applying conductive ink onto copper substrates with a thickness of 0.05 mm. The process entails bonding two copper substrates together to bridge their interface and then applying the printed conductive ink onto this assembly. The hybrid paste was printed on copper substrates as shown in Figure 5. The paste was printed on the cooper substrate and this process was repeated on each sample. The conductive ink paste applied on the copper substrate was precisely measured with dimensions of 5.0 mm in length (*l*), 5.0 mm in width (*w*), and 0.1 mm in thickness (t) on two overlapping substrates as shown in Figure 5.



(a)

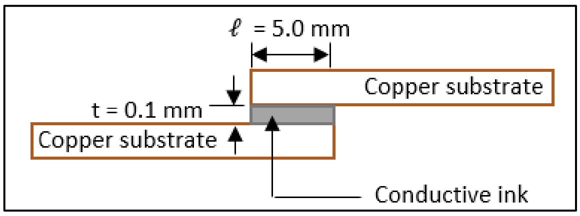


Fig. 5. (a) Printing process for lap shear test sample (b) Conductive ink paste on two overlapping substrates

2.5 Curing Process

The curing method is a subsequent treatment carried out following the preparation of the GNP hybrid conductive ink sample. The curing process is crucial as it enhances the bond between the filler particles and the binder, hence strengthening the bond between them. The main purpose of the curing time experiment is to find the ideal curing time and temperature for filler loading, as well as to investigate the effect of different curing durations on the hybrid conductive ink. For best conductivity, GNP must undergo a curing procedure to reduce porosity between particles and form a seamless conductive track [20]. The parameters for samples for the adhesion test are shown in Table 2.

The curing period ranges from four (4) to six (6) hours, with each sample interval spanning 1 hour. This duration enables a comprehensive examination of the influence of the duration of curing on the properties of the hybrid conductive ink, comprising any potential changes that may occur within this timeframe. Furthermore, commencing the observation period at four (4) hours sets a benchmark for comparison, while prolonging the duration to six (6) hours enables the detection of any further modifications or trends that may emerge during extended curing durations.

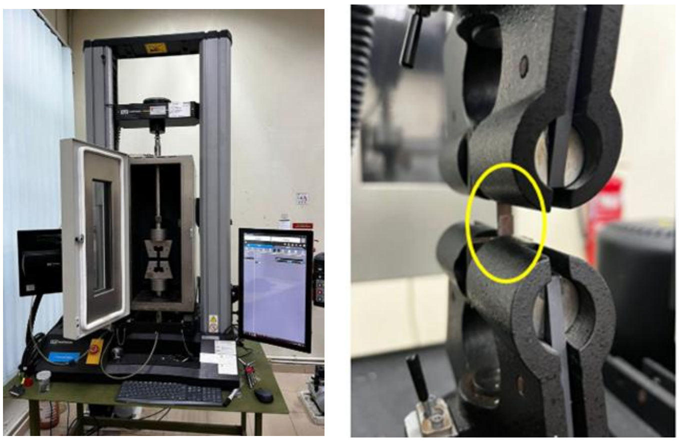
The hybrid conductive ink was cured at temperatures of 240°C, 250°C, and 260°C. The baseline curing temperature of 250°C was set based on research conducted by [20]. In this study, the temperature has been adjusted to 240°C, which is 10°C lower than the previous setup, and 260°C, which is 10°C higher than the initial baseline setting. The objective of this adjustment was to examine the impact of temperature changes on the curing durations of hybrid conductive ink.

Table 2
Parameters for samples for the adhesion test

Sample with rectangular shape	Temperature (°C)	Curing duration (hour)	Bonding area, ℓ, w, t (single copper substrate) (mm)
Sample 1	240	4 hours	5.0, 2.0, 0.05
Sample 2	240	5 hours	5.0, 2.0, 0.05
Sample 3	240	6 hours	5.0, 2.0, 0.05
Sample 4	250	4 hours	5.0, 2.0, 0.05
Sample 5	250	5 hours	5.0, 2.0, 0.05
Sample 6	250	6 hours	5.0, 2.0, 0.05
Sample 7	260	4 hours	5.0, 2.0, 0.05
Sample 8	260	5 hours	5.0, 2.0, 0.05
Sample 9	260	6 hours	5.0, 2.0, 0.05

2.6 Stress Analysis using Lap Shear Testing

Lap shear testing is widely used to determine the adhesive's bonding strength between two overlapping substrates [3]. A Universal Testing Machine (UTM) were used to perform the lap shear tests. Once the shear stress test samples have been prepared, it undergoes testing using a machine that is set up with precise device parameters, particularly a force setting of 10 N. The results are obtained as the machine initiates the application of force to the shear configuration. Figure 6 illustrates the shear test machine and the samples undergoing testing. This testing was performed in accordance with ASTM D1002 Standard Test Method to determine the shear strength of adhesives using the lap shear test.



(a) (b)

Fig. 6. (a) Universal Testing Machine for lap shear testing (b) Sample mounted at UTM

The key parameters measured in this test are the maximum force (F) at which the copper substrate fails and the displacement of the sample at the point of failure, which occurs at a rate of 1mm/min.

3. Results

3.1 Lap Shear Test

The study employs the Lap Shear Test, following the ASTM D1002 Standard Test Method, to evaluate the shear stress characteristics of GNP hybrids at different temperatures and time frames throughout the curing process. The goal is to acquire a thorough comprehension of how these processing parameters impact the mechanical functionality of the GNP hybrid materials. The findings derived from the shear stress tests are essential in improving fundamental knowledge of material behaviour and offer significant data for improving production processes and tailoring the characteristics of GNP hybrids for flexible semiconductor packaging devices. Besides, by given a shear stress, the inks had experienced a breakdown of the polymer network and it is called as an aggregation of metallic particles for Ag. It also negatively affects both materials particle and overall, the integrity of the sample. But, for insufficient stress, it gave an incomplete coverage of the substrate and finally the sample withstand.

Table 3 presents the results of the shear stress strength tests, their relationships and implications for the mechanical behaviour of GNP hybrid materials. Based on the data, when cured at lower temperatures, the hybrid structure exhibits a moderate shear strength, which indicates the early phases of bonding. An increase in shear strength is anticipated as the curing temperature rises. Elevated temperatures enhance molecular mobility and promote the bonding of GNP components, resulting in a more durable adhesive interface throughout the hybrid structure. The duration of the curing process is another important factor in the shear stress test. Reduced curing durations may lead to decreased shear strength, indicating the presence of first bonding stages. Inversely, extended times of curing enable greater bonding and integration of GNPs, which result in improved shear strength [21]. Identifying the optimal curing time is vital, as prolonged durations enable curing agents to react more fully that forging stronger molecular connections and enhancing mechanical performance [22]. The shear stress test findings demonstrate a distinct relationship between the curing temperature and duration. The simultaneous impact of increased temperatures and prolonged curing durations might result in the maximum shear strength, emphasising the intricate interaction between these factors in influencing the mechanical characteristics of the material.

Table 3
Data of shear test

Temperature (°C)	Time (hour)	Sample	Displace-ment at maximum force (mm)	Maximum force (N)	Tensile Displacem-ent at maximum force (mm)	Strain Displacem-ent at maximum force (%)	Cross-sectional area conductive ink (m²)	Young modulus (MPa)	Shear stress (MPa)	Error bar (5%)
240	4	S1	0.15	13.16	0.15	3.05	5.0×10^{-7}	8.63	26.32	1.316
	5	S2	0.09	28.83	0.09	1.85	5.0×10^{-7}	31.168	57.66	2.883
	6	S3	0,14	27.54	0.14	2.88	5.0×10^{-7}	19.125	55.08	2.754
250	4	S4	0.28	24.75	0.28	5.68	5.0×10^{-7}	19.125	49.5	2.475
	5	S5	0.29	26.24	0.29	5.84	5.0×10^{-7}	8.715	52.48	2.624
	6	S6	0.31	30.27	0.31	6.15	5.0×10^{-7}	9.844	60.54	3.027
260	4	S7	0.12	21.29	0.12	2.48	5.0×10^{-7}	17.169	42.58	2.129
	5	S8	0.19	10.7	0.19	3.71	5.0×10^{-7}	5.77	21.4	1.07
	6	S9	0.1	14.51	0.15	2.9	5.0×10^{-7}	9.904	29.02	1.451

3.2 Shear Stress Analysis

Figure 7 illustrates the outcomes of shear stress tests conducted on GNP hybrid samples, subjected to a range of curing conditions. Each sample, labelled S1 through S9, given different curing temperatures of 240°C, 250°C, and 260°C and durations of 4 hours, 5 hours, and 6 hours respectively. This comprehensive testing approach provides useful insights into the complex correlation between curing conditions and the mechanical performance of GNP hybrids.

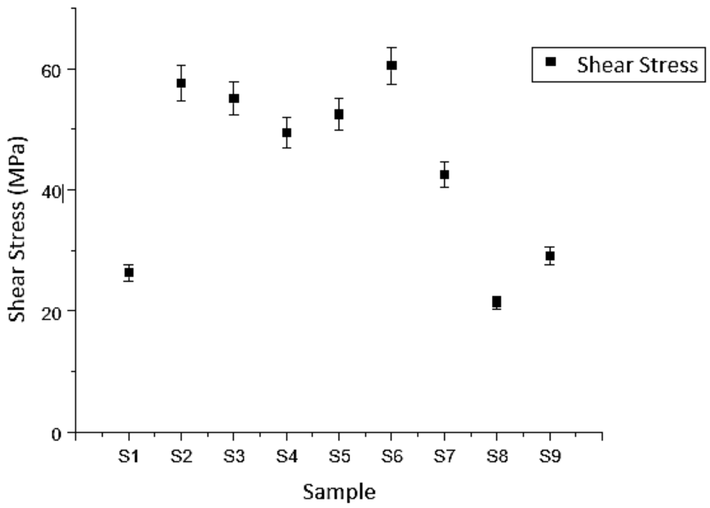


Fig. 7. Shear stress of samples with different temperatures and curing times

The plotted graph provides a clear and visual representation of how various curing conditions influence the shear strength of the samples. This data visualization enables a comprehensive analysis of the synergistic effects of both temperature and time on the bonding strength and overall structural integrity of the GNP hybrid material. Understanding these intricate relationships is paramount for optimizing the curing process to attain the desired mechanical properties, especially in applications where high strength and durability are imperative. Analyzing the stress values at different curing temperatures reveals distinct patterns, consistent with expected trends in polymer and composite curing processes. Generally, an increase in curing temperature aligns with higher stress levels, a phenomenon well- documented in the literature [23].

At a temperature of 240°C, the shear stress measurements exhibit an increasing trend, rising from 26.32 MPa after 4 hours to 57.66 MPa after 5 hours, before slightly decreasing to 55.08 MPa after 6 hours. This pattern suggests that extending the curing duration to 5 hours optimizes the adhesive strength. However, further extending the curing time beyond 5 hours results in a marginal decline in strength. The observed stability at 240°C creates optimal conditions for achieving high shear stress levels, indicating that a 5-hour curing period at this temperature is most effective for maximizing the mechanical performance of the adhesive. When analysing the impact of curing duration on stress, clear differences can be observed. Sample S3 exhibits a decrease in stress compared to sample S2 when exposed to a temperature of 240°C, indicating that prolonged curing can result in reduced material strength [24].

When heated to 250 °C, the shear stress measurements exhibit a consistent increasing trend with extended curing durations. Specifically, after 4 hours of curing, the shear stress is recorded at 49.5 MPa. This value increases to 52.48 MPa after 5 hours, indicating a further enhancement in adhesive strength. The gradual increase in shear stress indicates that extending the curing duration at 250°C significantly improves the adhesive strength of the GNP hybrid conductive ink. The data indicates that, unlike at lower temperatures, prolonged exposure to 250°C does not lead to a deterioration of mechanical properties but rather enhances them. The increment of curing temperature to 260°C introduces additional complexities. A significant decrease in shear stress after 5 hours suggests a sensitivity to elevated temperatures, deviating from the expected pattern, and highlighting the intricate interplay between temperature and cure time.

Comparing shear stress levels across different temperatures during specific curing periods reveals that at 5 hours, the highest shear stress is achieved at 250 °C (52.48 MPa), outperforming the results at 240 °C and 260 °C. This finding underscores the necessity of a finely tuned balance to maximise mechanical robustness [25]. Figure 8 graphically presents the trends in the shear stress tests, providing a visual representation of the relationship between applied force, curing temperature, and duration on the shear stress behaviour of the material.

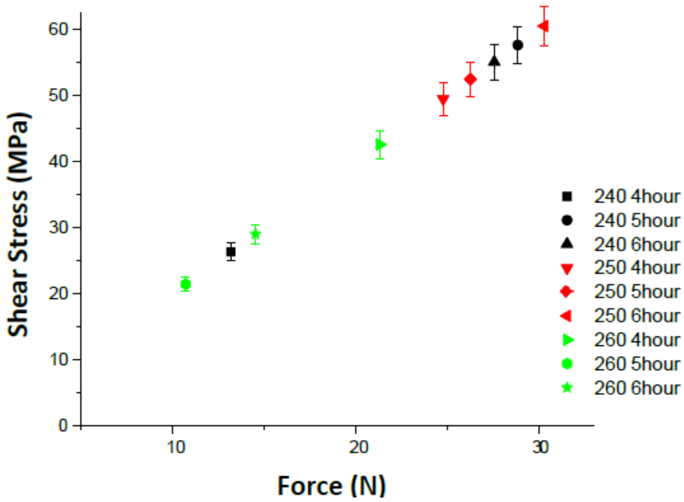


Fig. 8. Shear stress of different temperatures and curing durations

The examination of shear stress in relation to applied force under varied curing conditions reveals intriguing patterns. At a curing temperature of 240°C, there is a positive correlation between shear stress and the applied force. This is evident as the shear stress reaches a maximum of 26.32 MPa at 4 hours, 57.66 MPa at 5 hours, and 55.08 MPa at 6 hours. The existence of this positive relationship suggests that as the force exerted on the material increases, so does its resistance to shear deformation. The observed strain at peak force and the derived values of Young's modulus confirmed the material's ability to endure mechanical stress. From the graph, it can be observed that the value of shear stress increases with the increment of curing temperature at 250°C with values of 49.5 MPa at 4 hours, 52.48 MPa at 5 hours, and 60.54 MPa at 6 hours. This escalation in shear stress points to an improvement in the material's capacity to resist internal deformation, a conclusion supported by data on tensile displacement and Young's modulus.

Alternatively, at 260°C, the behaviour of shear stress becomes more complex. Initially, the shear stress increases for durations of 4 and 5 hours but then exhibits a decrease at 6 hours. Interestingly, the increase in applied force does not consistently translate to an increased shear stress at this temperature. This pattern indicates a multifaceted interaction between temperature and curing duration, significantly affecting the mechanical properties of the ink. In addition, the shear stress in this experience also influences the particle alignment. By increasing the shear stress enhances the dispersion, then promoting better conductivity. However, by excessing the shear, it can disrupt the ink’s structure and reducing the adhesion.

4. Conclusions

The results of the research demonstrate that increased curing temperatures have an adverse effect on the adhesive strength of the conductive ink, highlighting the correlation between curing conditions and adhesion qualities. The findings indicate that the shear strength of the conductive ink varies significantly with changes in curing temperature. The primary aim of varying the curing temperature and time experiment is to determine the optimal curing temperature and time and investigate the impact of various curing periods on the adhesion properties of the GNP hybrid conductive ink. Specifically, the shear strength increases as the curing temperature rises from 240°C to 250°C, suggesting that higher temperatures enhance the bonding strength of the ink. Alternatively, at 260°C, the shear strength decreases again, indicating that prolonged exposure to even higher temperatures may lead to a weakening of the mechanical properties. As a result, the optimal curing temperature for achieving the highest shear strength appears to be around 250°C.

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