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EFFECTS OF CYCLIC BENDING ON MICROSTRUCTURE OF HYBRID CARBON-METAL CONDUCTIVE INKS

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ABSTRACT

Conductive inks are widely employed in flexible electronics and circuits that undergo repeated bending or flexing, which can significantly affect the electrical and mechanical performance. This research presents the effects of cyclic bending on the microstructure of hybrid carbon-metal conductive ink. Using an ultrasonic bath, a sonication technique was employed to fabricate the hybrid carbon-metal powder using Graphene Nanoplatelets and silver flakes. The hybrid carbon-metal conductive ink was developed using a different ratio of organic solvent, cured at 260 °C, and underwent a cyclic bending test for 16000 cycles. Mechanical properties were examined using a Scanning Electron Microscope to evaluate the microstructure of the ink. The findings indicate that the ratio of 50:50 of 1-butanol to terpineol shows dense and compact filler networks with minimal voids and well-dispersed Graphene Nanoplatelets and silver flakes with the lowest value of resistance and resistivity after 16000 cycles. In conclusion, the carbon-metal conductive inks cured at 260 °C for five hours show improved electrical performance during the cyclic bending test. For further investigation, the curing temperature should remain at 260 °C for different curing durations.

1. Introduction

The growth of flexible and printed electronics has catalyzed the demand for advanced conductive inks, which enable the fabrication of low-cost, lightweight, and large-area electronic devices through additive manufacturing techniques. These inks are increasingly applied in a varied range of applications, including thin-film transistors, electromagnetic shielding, radio-frequency antennas, wearable biosensors, and graphene-based absorbing media. The functionality and reliability of such systems are basically linked to the physicochemical and mechanical properties of the conductive inks employed.

Among the available conductive materials, silver nanoparticles are widely used because of the high electrical conductivity, good surface morphology, and strong resistance to oxidation (Al-Gburi et al., 2024). Conventional silver-based conductive inks are composed of a dispersion of silver nanostructures in polymeric binders and organic solvents, typically accompanied by surfactants and rheological modifiers. High-temperature sintering (≥150 °C) is generally required post-deposition to decompose organic stabilizers and establish a percolative conductive network. However, this requirement imposes constraints on the choice of substrates, as many polymeric and flexible materials exhibit thermal deformation or degradation at elevated temperatures (Zulfiqar et al., 2021)

With the intention of lessening these limitations, hybrid ink systems combining carbon-based nanomaterials have been developed. Materials such as graphene nanoplatelets (GNPs), carbon black (CB), and carbon nanotubes (CNTs) offer a unique combination of mechanical flexibility, thermal stability, and environmental flexibility. The addition of these nanomaterials can enhance ink flexibility, reduce sintering temperature requirements, and expose stretchability to printed structures. However, carbonaceous materials typically exhibit lower electrical conductivity than metallic fillers and tend to agglomerate in dispersion because of the hydrophobic and π–π stacking interactions properties, cause difficulties ink formulation and stability (Qin et al., 2023). Hybrid conductive inks that combine metal nanoparticles with carbon-based additives aim to manipulate the high electrical conductivity of metals with mechanical compliance and interfacial adhesion of carbon materials. Combination of silver nanoparticles into a carbon-rich matrix can establish more efficient charging transport pathways, reduce inter-particle contact resistance, and improve film properties under strain. Furthermore, such hybrid systems may enable a reduction in metal content, decrease cost without affecting performance (Camargo et al., 2021).

Nevertheless, optimal dispersion, solvent compatibility, binder interaction, and curing behavior must be carefully tuned to maintain uniformity, prevent phase separation, and achieve desirable electrical and mechanical properties. In practical applications, particularly in wearable electronics, flexible displays, and soft robotics, conductive inks are routinely subjected to cyclic mechanical deformation, including bending, twisting, and stretching. These dynamic loads introduce mechanical fatigue, which may manifest as interfacial delamination, microcrack spread, or disruption of the percolative network. While silver flakes have demonstrated enhanced mechanical compliance because of the planar morphology and broad interparticle contact areas (Zulfiqar et al., 2021), the fatigue behavior of hybrid carbon-metal inks remains insufficiently characterized, especially at the microstructural level. In order to achieve the best performance, the flexible and stretchable printed circuit with conductive ink must be defect free and have good adhesion between substrate and the ink as well as the mounted components to make sure the circuity can withstand all the loads such as bending, vibration, thermal shock and stretching during application. The development of cyclic bending test and cyclic stretchability test rig is required to investigate the functionality stretchable conductive ink under repeated loadings (Manaf et al., 2020).

In this study, the effects of cyclic bending on the microstructural integrity and electrical conductivity of hybrid carbon-metal conductive inks are investigated. A series of hybrid inks composed of GNPs and silver nanoparticles were formulated using controlled solvent ratio and the printed films were subjected to repeated bending cycles. This work provides insights into the mechanical reliability and design considerations for hybrid conductive inks, with implications for the development of high-performance materials for flexible and stretchable electronic systems.

2. Materials and Methods

The materials used in this study were shown in Table 1. The hybrid carbon-metal powder and carbon-metal conductive ink were prepared according to previous study conducted by (Syamsul Helmi, et al. 2024). The GNPs with 25 µm particles size and silver flakes (Ag) 10 µm particles size were used as conductive fillers whereas silver acetate (SA) as a precursor and 1-butanol and terpineol were used as organic solvent.

Table 1. Materials used in formulation of carbon-metal hybrid powder and carbon-metal conductive ink

Type	Material
Filler material	GNPs 25 µm particles size with the surface area of 120 to 150 m^2/g
	Ag 10 µm particles size, 99.9 % trace metals basis
Precursor	SA 99.99 % trace metal basis acid silver salt
Chemical solvent	Ethanol denatured 99%
Organic solvent as binder	1-Butanol 99.9% butyl alcohol
	Terpineol pine oil contains 65 % α, 10 % β, 20 % γ

Approximately, 0.005 g of GNPs powder was dispersed in 5 ml of ethanol in a beaker. The beaker was then covered with aluminum foil to avoid ethanol from evaporating and underwent sonication for 10 minutes using an ultrasonic machine. After that, the mixture was added with 0.4292 g of Ag and sonication continues for 60 minutes. Then, the mixture was added with 0.042 g of SA and the mixture was sonicate for another 60 minutes. Subsequently, the mixed solution was then heated on a hotplate at 70 °C under a constant stirring 200 rpm until most of the ethanol had evaporated and paste like mixture was gathered. The mixture was cured for 1 hour in a reflow oven at 250 °C. The cold dried mixture was then grounded into a fine powder using a mortar and pestle.

Roughly for every 0.1733 g carbon-metal hybrid powder, 1 drop of butanol, 0.02 g and 1 drop of terpineol, 0.03 g is used to fabricate the conductive ink. For sample 5-Terpineol, 1.733 g of carbon-metal hybrid powder was added with 10 drops of 1-butanol and 5 drops of terpineol, for sample 10-Terpineol, 1.733 g of carbon-metal hybrid powder was added with 10 drops of 1-butanol and 10 drops of terpineol and for sample 15-Terpineol, 1.733 g of carbon-metal hybrid powder was added with 10 drops of 1-butanol and 15 drops of terpineol. All samples were mixed using the Thinky mixer for three minutes at 2000 rpm.

Using manual stencil printing technique with the mesh stencil thickness of 60µm ± 2µm (Gholamalizadeh et al., 2022), conductive patterns of carbon-metal conductive ink were printed on copper substrates as illustrated in Figure 1. The ink underwent thermal curing in reflow oven at 260 °C for 5 hours.

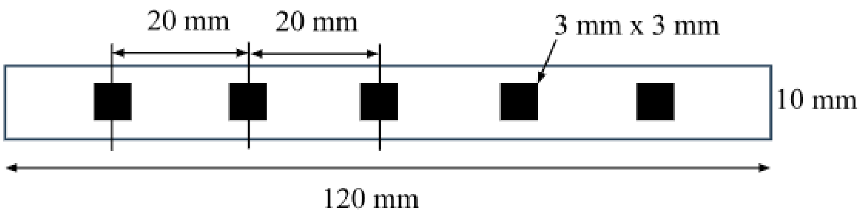

Figure 1. Conductive patterns of carbon-metal conductive ink printed on copper substrates

Printed conductive ink underwent a cyclic bending test (Manaf et al., 2020) for 16000 cycles using a cyclic bending test machine. The test sample pattern for 2-point probe method (Zulfiqar et al., 2021) was set at 3 mm x 3 mm at at four designated points of conductive ink (1,1) (2,2) (3,3) (4,4) as shown in Figure 2.

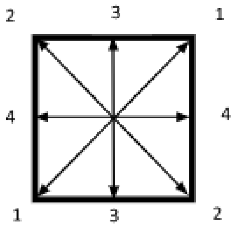

Figure 2. Four points measurement of resistance of carbon-metal hybrid conductive ink

The surface structure of the carbon-metal ink was observed using a light microscope. The printed ink was prepared for metallographic microstructure analysis after the cyclic bending test and cold mounting technique was selected for preparing the sample. The specifications of the epoxy resin and hardener used were shown in Table 2. The Scanning Electron Microscope at an accelerating voltage of 5kV was used to analyse the surface morphology of the ink.

Table 2. Epoxy resin and hardener specifications for cold mounting

Specifications	Epoxy Resin	Hardener
Colour	Clear	Clear
Physical	Liquid	Liquid
Curing time (10 °C)	40 hours	40 hours
Curing time (25 °C)	20 hours	20 hours
Ratio by weight	3	1
Hardness	1.14 g/cm^3	1.03 g/cm^3

3. Results

Table 3 shows the image of carbon-metal conductive ink cured at 260 °C for 5 hours before cyclic bending tests under a light microscope. For sample 5-Terpineol, the cured film shows grainy surface texture, visible porosity, and regions with non-uniform packing density. For sample 10-Terpineol the film appears smoother and more uniform, with closely compacted

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conductive areas and minimal visible porosity and for sample 15-Terpineol the film shows a slightly uneven and rougher texture than the 10-Terpineol sample, with evidence of lower compaction.

After exposing the carbon-metal conductive ink samples to 16,000 cycles of bending, clear differences in microstructural degradation were observed under the light microscope, as shown in Table 4. The sample 5-Terpineol exhibited severe crack spread, with visible fracture networks and potential delamination. In contrast, the 10-Terpineol sample demonstrated minimal microstructural damage, with only slight deformation lines and preserved conductive pathways. The 15-Terpineol sample showed moderate structural deterioration, with several visible crack lines and fragmentation of the conductive network.

Table 3. Light microscope images of carbon-metal conductive inks after curing at 260 °C for 5 before cyclic bending test

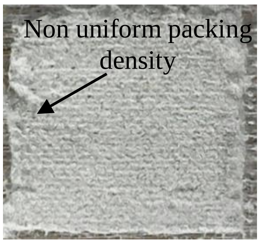
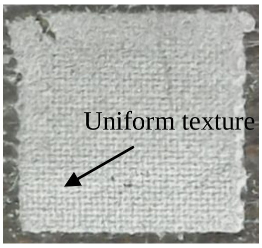
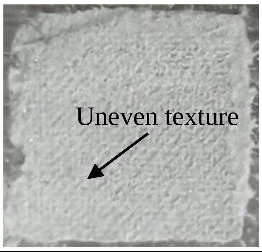
Sample	Carbon -metal (g)	1-Butanol (Drops)	Terpineol (Drops)	Ratio (1-Butanol: Terpineol)	Ratio (Filler: Solvent)	Observation
5-Terpineol	1.733	10	5	67:33	80:20	
10-Terpineol	1.733	10	10	50:50	72:28	
15-Terpineol	1.733	10	15	40:60	64:36	

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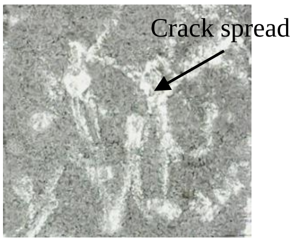
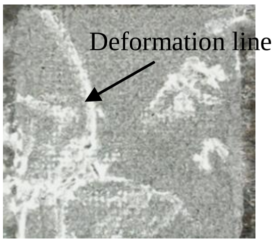
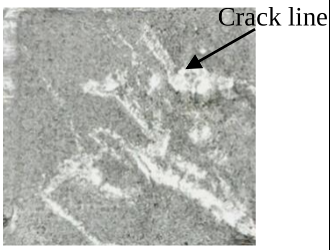
Table 4. Light microscope images of carbon-metal conductive inks after curing at 260 °C for 5 hours and subjected to 16,000 cycles of bending

Sample	Carbon-metal (g)	1-Butanol (Drops)	Terpineol (Drops)	Ratio (1-Butanol: Terpineol)	Ratio (Filler: Solvent)	Observation
5-Terpineol	1.733	10	5	67:33	80:20	
10-Terpineol	1.733	10	10	50:50	72:28	
15-Terpineol	1.733	10	15	40:60	64:36	

The inks were formulated with varying ratios of 1-butanol to terpineol 67:33, 50:50, and 40:60, to investigate the influence of solvent composition on the post-cyclic bending test microstructure. The cross-sectional SEM image of the carbon-metal conductive ink film cured at 260 °C and subjected to 16,000 bending cycles for sample 5-Terpineol are shown in Figure 3. The sample with ratio of 1-butanol to terpineol 67:33 exhibited a roughly packed and relatively non-uniform structure. Significant vertical alignment of filler particles and interfacial voids were observed, probably caused by fast solvent evaporation of the high instability of 1-butanol. Such a morphology suggests incomplete compaction and may lead to suboptimal electrical conductivity and poor mechanical stability under cyclic deformation.

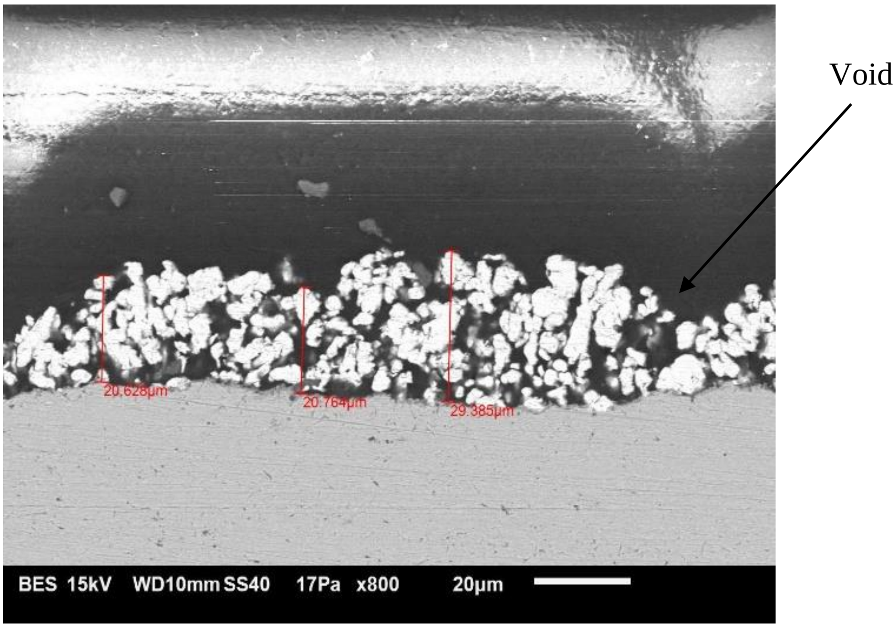


Figure 3. 5-Terpineol carbon-metal ink magnification x800 and particles distribution

In contrast, for sample 10-Terpineol with a ratio of 50:50 1-butanol to terpineol as in Figure 4 demonstrated a highly compact and uniform film structure. The GNPs and Ag particles were densely packed with minimal voids, indicate an optimal balance of solvent evaporation rate and ink viscosity. This improved microstructural integrity is expected to facilitate a continuous conductive network and enhance electrical conductivity and higher mechanical durability during repeated bending.

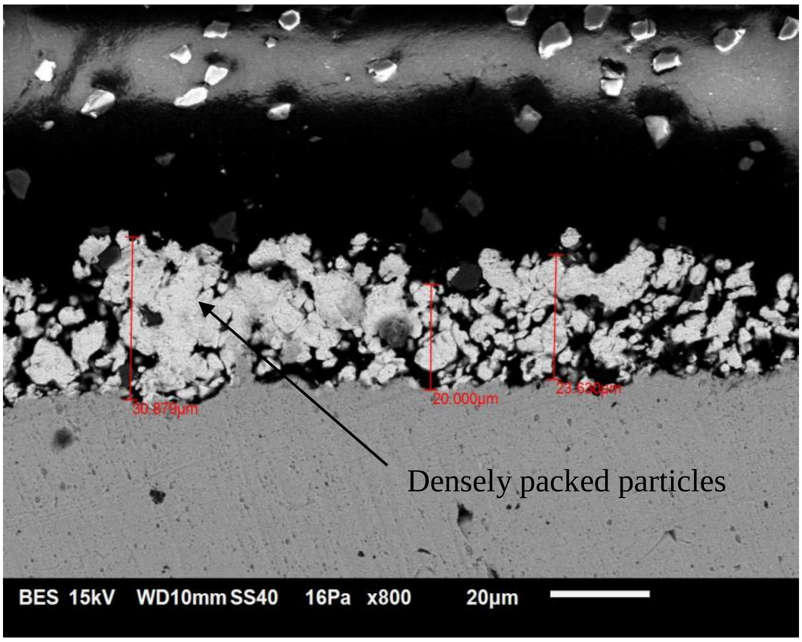


Figure 4. 10-Terpineol carbon-metal ink magnification x800 and particles distribution

For sample 15-Terpineol with a ratio of 1-butanol to terpineol 40:60 as in Figure 5, microstructure showed signs of agglomeration and reduced packing density compared to the 10-Terpineol sample. The higher terpineol content increases ink viscosity, potentially clogging uniform filler dispersion and helps the formation of larger particle clusters. These structural features may reduce the effectiveness of the conductive pathways.

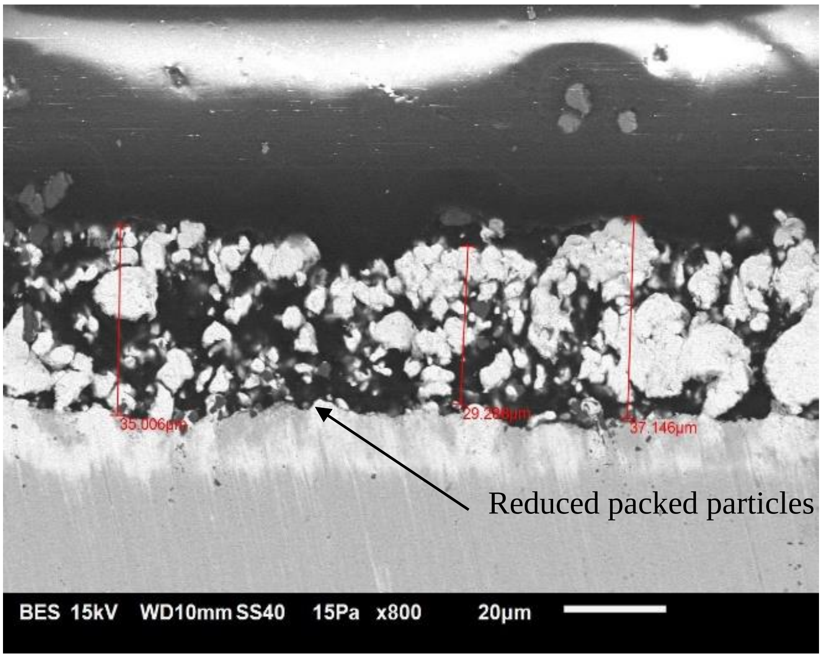


Figure 5. 15-Terpineol carbon-metal ink magnification x800 and particles distribution

The bulk resistance of the carbon-metal hybrid conductive inks was measured before and after 16,000 cycles of bending to assess the electrical durability under mechanical stress and the results were as shown in Table 5.

Table 5. The average bulk resistance and resistivity of carbon-metal hybrid conductive ink

Sample	Before cyclic bending test		After 16000 cyclic bending tests	
	Resistance (Ω)	Resistivity (Ωm)	Resistance (Ω)	Resistivity (Ωm)
5-Terpineol	2.6	1.5×10^{-4}	1.6	1.0×10^{-4}
10-Terpineol	1.1	0.66×10^{-4}	0.7	0.42×10^{-4}
15-Terpineol	0.8	4.8×10^{-5}	1.18	7.08×10^{-5}

Among the formulations, the 15-Terpineol sample exhibited the lowest initial resistance 0.8 Ω and resistivity 4.8×10^{-5} Ωm, suggests excellent initial electrical conductivity. However, after cyclic bending, this formulation showed a significant increase in resistance to 1.18 Ω, and resistivity increased to 7.08×10^{-5} Ωm, demonstrate poor mechanical stability of the conductive network. In contrast, the 10-Terpineol formulation shows balanced electrical and mechanical performance, with relatively low initial resistance 1.1 Ω and moderate change after bending 0.7 Ω, resistivity dropped slightly to 4.2×10^{-5} Ωm. This behavior proposes improved particle-particle connectivity and better structural integrity under repeated deformation, attributed to optimal viscosity and filler dispersion. The 5-Terpineol sample, while initially having the highest resistance of 2.6 Ω, showed a reduction in resistance to 1.6 Ω after bending. This decrease may be because of the compaction of the ink microstructure under compressive strain, enhanced contact between conductive fillers. However, the visible microstructural damage observed previously suggests this effect may not be consistent or reliable across samples.

4. Discussion

The effect of varying 1-butanol to terpineol ratios on the mechanical and electrical stability of carbon-metal hybrid conductive inks was investigated through SEM imaging and cyclic bending tests up to 16,000 cycles. The morphological evaluation of carbon-metal conductive inks shown that solvent composition plays a critical role in determining film integrity and performance. Among the tested formulations, the ink with a 50:50 ratio of 1-butanol to terpineol exhibited the most uniform and compact microstructure, characterized by dense packing of conductive fillers and minimal voids. This structure is conducive to stable electrical conductivity and mechanical durability during repeated flexing. In contrast, the inks with 67:33 and 40:60 ratios showed less satisfactory morphologies, including void formation and particle agglomeration, which may compromise both conductivity and flexibility. These findings highlight the importance of optimizing the solvent ratio to achieve balanced drying kinetics, filler dispersion, and structural cohesion in stretchable and printed electronic applications.

5. Conclusion

In summary, the 50:50 1-butanol to terpineol solvent ratio was found to be optimal for balancing solvent evaporation kinetics, chemical interactions, particle dispersion, and thermal-electrical performance. This formulation yielded a strong, conductive, and thermally stable ink film suitable for flexible electronic applications subjected to repeated mechanical deformation. The carbon-metal conductive inks cured at 260 °C for five hours show improved electrical performance during the cyclic bending test.

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Authors declare that there is no conflict of interests regarding the publication of the article.
During the preparation of this manuscript, the author(s) used OpenAI's ChatGPT to assist in improving the readability and language of the text. All content generated by ChatGPT was

subject to thorough review, editing, and revision by the author(s) to ensure its accuracy, completeness, and alignment with the research objectives. The authors take full responsibility for the integrity and content of published work. This declaration complies with ICGESD 2025 guidelines on the use of generative AI in scientific writing.

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