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Bridging the Thermal Gap: Ag-MWCNT Reinforced Silver Paste as an Interfacial Interconnect for High-Power Electronic Packaging

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ABSTRACT

With the rapid development of high-power-density electronic devices, the demand for thermal management in electronic packaging has become increasingly urgent. Graphene films (GFs) exhibit excellent thermal conductivity, but their practical heat transfer efficiency is severely restricted by the large interfacial thermal resistance between the film and the substrate. To mitigate this thermal resistance, this study developed an interfacial interconnection material comprising a nano-silver paste reinforced with silver nanoparticle-decorated multi-walled carbon nanotubes (Ag-MWCNTs). The results indicate that the introduction of Ag-MWCNTs effectively enhanced the thermal performance of the sintered silver paste. Specifically, at an Ag-MWCNTs loading of 5 wt%, the thermal conductivity reached a maximum value of $10.16 \pm 0.23 \text{ Wm}^{-1}\text{K}^{-1}$, representing a 175% enhancement compared to the pure silver paste. When the composite silver paste was applied as the interfacial interconnect between GFs and the copper substrate, the total thermal resistance of the system was minimized at $2.11 \pm 0.12 \text{ cm}^2\text{KW}^{-1}$ with 1 wt% Ag-MWCNTs addition, achieving a 59% reduction compared to the pure silver paste interconnect. This study provides an effective interconnection strategy for the interfacial thermal management of two-dimensional high-thermal-conductivity materials in advanced microelectronic packaging.

Keywords: Vertically aligned graphene; Thermal interface materials; Liquid metal; Contact thermal resistance

1 Introduction

With the rapid advancement of microelectronic packaging technologies toward higher integration and power density, thermal accumulation during chip operation has emerged as a critical challenge limiting device performance and reliability. This necessitates enhanced thermal transport efficiency in advanced thermal interface materials [1, 2]. Graphene, characterized by its exceptionally high theoretical in-plane thermal conductivity ($\sim 5300 \text{ Wm}^{-1}\text{K}^{-1}$) and superior mechanical stability, is considered an ideal candidate for next-generation high-performance thermal dissipation films [3, 4]. However, in practical packaging applications, the microscopic physical contact defects between graphene films (GFs) and traditional metal heat dissipation substrates e.g., copper), which arise from lattice dynamics mismatch and surface roughness, easily trigger intense interfacial phonon scattering. This significant interfacial thermal resistance substantially reduces the system's actual heat transfer efficiency, representing the primary barrier to realizing the inherent advantages of two-dimensional carbon-based materials in thermal management systems[5, 6].

Leveraging the low activation energy sintering mechanism enabled by nanoscale effects, nanosilver pastes achieve

low-temperature interconnection while forming continuous metallic networks with high thermal conductivity. This capability positions them as promising candidates for high-power electronic packaging applications [7]. However, under extreme thermo-mechanical cyclic loading conditions, pore coalescence in fully sintered nanosilver layers remains a critical issue impeding long-term reliability [8]. Introducing multi-walled carbon nanotubes (MWCNTs), which possess a one-dimensional tubular structure and an ultra-long phonon mean free path, as a reinforcing phase into the metal matrix has proven to be an effective approach to synergistically enhance the thermal properties of composite materials. However, due to their massive specific surface area and strong inter-tube van der Waals forces, MWCNTs are highly susceptible to agglomeration within the paste system [9]. Moreover, the chemically inert basal planes of carbon structures exhibit poor interfacial wettability with silver matrices, which not only disrupts the formation of continuous thermal pathways but also generates additional microstructural defects at interfaces [10]. Research has demonstrated that surface metallization of MWCNTs effectively addresses these limitations. Metal nanoparticle-decorated MWCNTs enhance carbon nanotube dispersion through steric hindrance mechanisms while serving as thermodynamically active sites during sintering. This enables the transition of interfacial interactions between MWCNTs and silver matrices from weak van der Waals forces to robust metallurgical bonds. Consequently, multiscale hierarchical heat transfer is achieved, significantly lowering interfacial phonon and electron transport barriers [11-13].

This study proposes a novel interfacial interconnection material based on silver nanoparticle-decorated MWCNTs (Ag-MWCNTs) reinforced nano-silver paste, aiming to optimize thermal interfacial coupling between graphene films (GFs) and copper substrates. The methodology involves four key stages: (1) optimization of sonication parameters through systematic evaluation of mixed-acid functionalization duration effects on MWCNT surface chemistry; (2) in situ synthesis of Ag nanoparticles on functionalized MWCNTs via controlled chemical reduction; (3) comprehensive characterization of composite nano-silver paste properties including microstructure evolution, electrical resistivity, and thermal conductivity as functions of Ag-MWCNT content; and (4) application demonstration through fabrication of GF/copper interconnects to quantify thermal interfacial resistance reduction. This research establishes a viable pathway for interfacial engineering and provides experimental validation for deploying two-dimensional high-thermal-conductivity films in next-generation microelectronic packaging systems.

2 Experimental Section

2.1 Materials

MWCNTs (length: 50 μm , diameter: 8-15 nm) were purchased from Jiangsu XFNANO Materials Tech Co., Ltd. Silver nanoparticles (60-120 nm), silver nitrate, ascorbic acid, polyvinyl butyral (PVB), triethylene glycol and terpineol were acquired from Aladdin Biochemical Technology Co., Ltd. Concentrated sulfuric acid, concentrated nitric acid, absolute ethanol and acetone were supplied by Sinopharm Chemical Reagent Co., Ltd. GFs with a thickness of 0.05 mm were sourced from Jiaxing Zhixian Thermal Information Technology Co., Ltd. Copper substrates with dimensions of $5\times5\times0.5\text{ mm}^3$, $30\times30\times0.3\text{ mm}^3$ and $10\times10\times0.5\text{ mm}^3$ were obtained from Xinghua Wanling Hardware Products Co., Ltd.

2.2 Preparation of Ag-MWCNTs

First, raw MWCNTs were dispersed in a mixture of concentrated sulfuric acid: nitric acid (3:1 v/v) and subjected to ultrasonication for 3, 5, 10, and 15 min, respectively. The suspension was then centrifuged at 4000 rpm for 15 minutes.

The supernatant was decanted, and the resulting precipitate underwent repeated washing with deionized (DI) water before being dried in an oven at 150°C for 5 hours. Next, the acid-treated MWCNTs were uniformly dispersed in a 0.1 mol/L aqueous silver nitrate solution under continuous magnetic stirring. A 0.1 mol/L aqueous ascorbic acid solution was then introduced dropwise into the mixture at a controlled rate of ~1 drop per minute. The reaction mixture was maintained under continuous stirring for 30 minutes. Post-reaction, the product was centrifuged at 3000 rpm for 10 minutes to isolate the precipitate, which was thoroughly rinsed with DI water and dried in an oven at 150°C for 4 hours to yield the Ag-MWCNT composite.

2.3 Sample preparation of Nano-silver paste

Polyvinyl butyral (PVB) powder was incrementally added to absolute ethanol under continuous magnetic stirring at 500 rpm for 15 minutes using a mechanical stirrer. introduced dropwise into the solution at a predetermined ratio, followed by an additional 15-minute stirring period. Subsequently, the pre-prepared Ag-MWCNTs composites and silver nanoparticles were added sequentially to the mixture, and the solution was stirred for 15 minutes. The total mass fraction of conductive fillers was maintained at 82 wt%, with Ag-MWCNTs mass fractions set to 0, 0.5, 1, 3, 5, and 10 wt%. The formulated nano-silver paste was then sonicated in an ice-water bath for 30 minutes. The comprehensive synthesis procedure for Ag-MWCNTs is illustrated in Fig. 1.

2.4 Characterization

The synthesized Ag-MWCNT composites were characterized using transmission electron microscopy (TEM; JEM-2100 Plus, JEOL), X-ray diffraction (XRD; D8 Advance, Bruker), Raman spectroscopy (LabRAM HR Evolution, Horiba), Fourier-transform infrared spectroscopy (FTIR; Nicolet iS 10, Thermo Fisher Scientific), and scanning electron microscopy (SEM; GeminiSEM 300, ZEISS). Electrical and thermal properties of the nano-silver paste were evaluated via a linear four-point probe system and a steady-state thermal conductivity analyzer (DRL-V, Xiangtan Xiangyi Instrument Co., Ltd.), respectively.

3 Results and Discussion

3.1 Materials Characterization

Figures 2 and 3 present the low- and high-magnification TEM morphologies of Ag-MWCNTs composites prepared with varying carboxylation durations. Figure 2(a) displays pristine MWCNTs, characterized by entangled fibrous tubes with smooth surfaces and hollow interiors [14]. Figures 2(b) and (c) depict untreated MWCNTs and those treated for 3 minutes, respectively. While scattered AgNPs are visible at low magnification, high-resolution TEM (Fig. 3(b) and (c)) reveals that AgNPs remain largely unattached to the MWCNT surfaces due to insufficient active sites. Extending the ultrasonic carboxylation time to 5 minutes (Fig. 2(d) and 3(d)) introduces discrete black dots on the MWCNT walls, indicating initial electrostatic adsorption of Ag⁺ ions facilitated by carboxyl groups. The optimal modification is achieved at 10 minutes: Figures 2(e) and 3(e) show densely distributed AgNPs with uniform coverage. Despite localized agglomeration from high loading density, this arrangement ensures sufficient metallic contacts for efficient thermal/electrical bridging in applications [15]. In contrast, prolonging treatment to 15 minutes (Fig. 2(f) and 3(f)) reduces AgNP loading density despite eliminating agglomeration. This results from severe MWCNT oxidation, which damages

structural integrity and degrades functional groups, impairing Ag⁺ adsorption capacity [16].

As shown in Fig. 4, the XRD pattern of pristine MWCNTs reveals a diffraction peak at $2\theta=26.02^\circ$, corresponding to the (002) crystal plane [17]. In contrast, the Ag-MWCNTs composites synthesized with varying ultrasonic carboxylation durations exhibit distinct diffraction peaks of metallic Ag, attributed to the (111), (200), (220), and (311) crystallographic planes, confirming the presence of silver nanoparticles with a face-centered cubic (FCC) structure [18]. Furthermore, by calculating the integrated area of the primary Ag (111) diffraction peak for each sample, it is found that the peak area of the 10-min treatment group reaches a relative minimum. According to the Scherrer equation [19]:

$$D = \frac{K\lambda}{\beta \cos \theta}$$

where D is the average crystallite size, K is the Scherrer constant, λ is the incident X-ray wavelength, β is the full width at half maximum (FWHM), and θ is the Bragg diffraction angle. Due to the inverse relationship between β and D , the diffraction peaks exhibit significant physical broadening when the nanoparticle size is extremely small, leading to a statistical attenuation of the coherent scattering intensity and the integrated area [20, 21]. The 10-minute sample's minimal peak area suggests the formation of ultra-small primary nanocrystals due to burst nucleation triggered by the highest density of carboxyl groups on the MWCNT surface.

Raman spectroscopy was employed to analyze structural modifications and defect densities in MWCNTs and Ag-MWCNTs (Fig. 5). The Raman spectra of pristine MWCNTs exhibit characteristic D-band (1344 cm^{-1}) and G-band (1592 cm^{-1}) peaks. The D-band intensity arises from structural defects and disorder, while the G-band corresponds to stretching vibrations of sp^2 -hybridized C=C bonds. The I_D/I_G ratio quantifies defect density, with lower values indicating fewer structural defects[22]. Pristine MWCNTs show an I_D/I_G ratio of 1.19, which increases to 1.27 for Ag-MWCNTs synthesized via 10-minute ultrasonic carboxylation. This rise, alongside minor peak shifts, stems from the introduction of defect sites and oxygen-containing functional groups during carboxylation. Extending the treatment time to 15 minutes reduces the I_D/I_G ratio to 1.18, likely due to severe structural degradation of the carbon nanotubes from prolonged strong-acid ultrasonication. This degradation may cause exfoliation or dissolution of highly defective outer carbon lattices.

Figure 6 displays the FTIR spectra of MWCNTs and Ag-MWCNTs. Both the 5-minute and 10-minute treatment groups exhibit distinct C=O stretching vibration peaks near 1720 cm^{-1} , indicative of oxygen-containing functional groups (e.g., carboxyl groups, -COOH) introduced onto the MWCNT surfaces via ultrasonic carboxylation. Extending the treatment time to 15 minutes eliminates this characteristic C=O peak, signaling severe over-oxidation. This behavior arises from violent structural exfoliation and physical fragmentation of the carbon nanotubes due to prolonged strong-acid ultrasonication. Specifically, the highly functionalized outer carbon lattice formed at 10 minutes likely destabilized into oxidation fragments from excessive structural damage, which were subsequently removed during washing and filtration [23].

Figure 7 illustrates the sintered microstructures of nano-reinforced silver pastes with varying Ag-MWCNT loadings. The morphological evolution of sintered joints reveals a pronounced dependence on Ag-MWCNT content. At low loadings (0–1 wt%), the sintered surfaces exhibit dense, continuous, and planar morphologies with robust metallurgical bonding between silver particles. This is primarily attributed to the uniform dispersion of MWCNTs within the silver matrix, which minimally impedes atomic diffusion and neck growth [24]. Upon increasing the loading to 3 wt%, the surface begins to develop subtle undulations and roughness, signaling the onset of sintering inhibition caused by the volumetric influence of MWCNTs [25]. In the high-loading regime (>3 wt%), a marked degradation occurs: samples with 5 wt% display distinct

microcracks and isolated pores, while those with 10 wt% exhibit severe MWCNT agglomeration. The mismatch in thermal expansion coefficients between MWCNTs and the silver matrix exacerbates stress concentration, leading to an interconnected network of macrocracks and a highly porous, fragmented microstructure [26].

3.2 Electrical and Thermal Properties

Figure 8 depicts the correlation between Ag-MWCNT loading and the electrical resistivity of nano-silver paste. The pristine silver paste exhibits a resistivity of $1.22 \pm 0.23 \times 10^{-6} \Omega \cdot \text{m}$. As Ag-MWCNT content increases, resistivity rises progressively, driven by microstructural evolution and interfacial phenomena in the composite. The low resistivity of pure nano-silver paste originates from thermal-driven atomic diffusion at silver nanoparticle surfaces, enabling dense sinter neck formation and continuous metallic bonding. However, incorporating rigid, high-aspect-ratio Ag-MWCNTs introduces physical barriers within the silver matrix. These one-dimensional structures hinder long-range silver atom diffusion and coalescence during sintering, disrupting the conductive network continuity and increasing porosity, thereby elevating resistivity [27]. Additionally, MWCNTs' high specific surface area and strong intertube van der Waals forces promote agglomeration. While well-dispersed at low loadings (<3 wt%), they form clusters at 5 wt% and 10 wt%, creating heterogeneous high-impedance regions within the composite [28]. These agglomerates fail to establish effective conductive pathways due to insufficient filler contact, further exacerbating resistivity increase and conductivity decline.

Figure 9 presents the thermal conductivity trends of modified nano-silver pastes with varying Ag-MWCNT loadings. Thermal conductivity initially increases and subsequently decreases with rising Ag-MWCNT content. Sintered pristine nano-silver paste exhibits a thermal conductivity of $3.69 \pm 0.34 \text{ Wm}^{-1}\text{K}^{-1}$. At 5 wt% Ag-MWCNT loading, thermal conductivity peaks at $10.16 \pm 0.23 \text{ Wm}^{-1}\text{K}^{-1}$, marking a 175% enhancement over pure silver paste. This improvement arises from surface silver nanoparticles on MWCNTs forming stable phonon transmission nodes and efficient transport channels during curing. These structures enhance MWCNT-silver matrix interfacial compatibility, reducing carrier transport barriers and enabling Ag-MWCNTs to function as thermal bridges across silver nanoparticle gaps, thereby establishing multidimensional heat transfer pathways. The exceptionally long phonon mean free path of MWCNTs facilitates rapid thermal energy migration from silver nanoparticles to carbon tube axes, promoting wide-range diffusion and significantly boosting composite thermal diffusivity [29]. However, at 10 wt% loading, thermal conductivity declines sharply due to severe MWCNT agglomeration induced by strong van der Waals forces during sintering. Heat transfer is compelled to traverse MWCNT overlapping interfaces with inherently low radial thermal conductivity. Concurrently, agglomerates disrupt silver nanoparticle metallic bonding, fragmenting the continuous sintered silver network and undermining thermal transport efficiency.

Figure 10 illustrates the thermal resistance trends of GFs/copper substrate interconnects as a function of Ag-MWCNT loading, exhibiting a distinct U-shaped curve. The thermal resistance decreases from $4.26 \pm 0.17 \text{ cm}^2 \cdot \text{K} \cdot \text{W}^{-1}$ for the pristine sample to a minimum of $2.11 \pm 0.12 \text{ cm}^2 \cdot \text{K} \cdot \text{W}^{-1}$ at 1 wt% loading—a 59% reduction. This enhancement is primarily attributed to the thermal bridging effect, where the 1D Ag-MWCNTs span the gaps between silver nanoparticles to establish long-range phonon transport pathways, while the surface silver decoration optimizes interfacial compatibility and reduces contact resistance [13]. However, exceeding the optimal 1 wt% threshold triggers a reversal in performance. At 10 wt%, thermal resistance surges to $4.86 \pm 0.16 \text{ cm}^2 \cdot \text{K} \cdot \text{W}^{-1}$ at 10 wt%. This degradation arises from severe MWCNT agglomeration driven by strong van der Waals forces, which creates high-impedance interfaces and physically obstructs silver matrix sintering kinetics. Excessive fillers also increase porosity and fracture the metallic bonding network continuity, culminating

in the observed thermal performance decline.

3.3 Mechanical Properties

Figure 11 presents the shear strength trends of sintered joints fabricated with nano-silver pastes modified by varying Ag-MWCNT loadings. The data demonstrate that the shear strength of the modified paste exhibits a distinct trend of initial increase followed by a subsequent decrease. Pure silver paste exhibits a baseline shear strength of 16.31 ± 0.94 MPa. Incorporating Ag-MWCNTs enhances mechanical performance, peaking at 23.36 ± 1.16 MPa at a loading of 1 wt%, which represents a 43% enhancement compared to the pure silver paste, demonstrating the excellent nano-reinforcement effect of Ag-MWCNTs. However, once the loading exceeds 1 wt%, the shear strength declines sharply. When the content is further increased to 10 wt%, the strength drops to 5.02 ± 0.51 MPa, which is significantly lower than that of the pure nano-silver paste, indicating that an excessive addition of MWCNTs has a pronounced negative impact on the mechanical properties of the silver paste.

To elucidate the relationship between Ag-MWCNT content and shear strength, the fracture microstructures of sintered joints were analyzed via SEM. Figure 12 displays cross-sectional morphologies of fractured joints with varying Ag-MWCNT loadings following shear testing. It can be observed that the evolution of shear strength with Ag-MWCNTs loading is highly consistent with the characteristics of the fracture microstructures. For the pure silver paste, the fracture surface exhibits a large number of interconnected nano-silver particles forming sintering necks; however, due to the volatilization and decomposition of organic binders, distinct micropores are visible on the sintering neck surfaces[30]. With the introduction of Ag-MWCNTs up to the optimal loading of 1 wt%, the sintered matrix demonstrates improved densification with reduced internal voids, and significant plastic deformation traces appear on the fracture surface. This indicates that an appropriate amount of MWCNTs effectively lowers the porosity and enhances interfacial bonding, thereby maximizing the load-bearing capacity of the joints [26]. However, when the Ag-MWCNTs content is further increased to 3 wt%, the volume effect of the MWCNTs begins to hinder the diffusion and sintering of silver atoms, leading to the initiation of micro-pores and localized loose regions on the fracture surface. At a loading of 10 wt%, the fracture morphology deteriorates further; excessive MWCNTs undergo severe agglomeration and induce network-like cracks. These structural defects reduce the effective load-bearing area, causing the failure mode of the sintered silver joints to shift completely to brittle delamination along the defect regions, which ultimately leads to the degradation of shear strength [31].

4 Conclusion

In summary, surface functionalization of MWCNTs via acid-mixture chemical treatment was optimized through systematic analysis of morphology and crystal structure variations under differing ultrasonication durations. An optimal ultrasonication time of 10 minutes was identified as the threshold for achieving maximal dispersion efficiency and functional group density. Key experimental findings demonstrate that the sintered nano-silver paste incorporating 5 wt% Ag-MWCNTs achieves a maximum thermal conductivity of $10.16 \pm 0.23 \text{ Wm}^{-1}\text{K}^{-1}$ —a 175% enhancement over pristine silver paste. This improvement stems from the synergistic effects of surface-decorated silver nanoparticles forming stable phonon transmission nodes and MWCNTs acting as axial heat conduits, enabling multidimensional thermal transport pathways. For interfacial thermal management applications, Ag-MWCNT-modified joints exhibit a minimum thermal resistance of $2.11 \pm 0.12 \text{ cm}^2\text{KW}^{-1}$, achieved at a 1 wt% loading, representing a 59% reduction compared to the unmodified counterpart. These results establish a robust interfacial interconnection strategy for next-generation microelectronic

packaging, particularly for two-dimensional high-thermal-conductivity systems. The methodology provides a scalable framework for optimizing hybrid thermal interface materials through tailored nanostructure design and processing parameter control.

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Author contribution

Study conception and design: YZ. Data collection: ZXF, WWL. Analysis and interpretation of results: WWL and YZ. Draft manuscript preparation: WWL and YZ. All authors reviewed the results and approved the final version of the manuscript.

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Date Availability

The data that support the findings of this study are available from the corresponding authors upon reasonable request.

Declarations

Conflict of interest The authors declare that there is no conflict of interests regarding the publication of this article.

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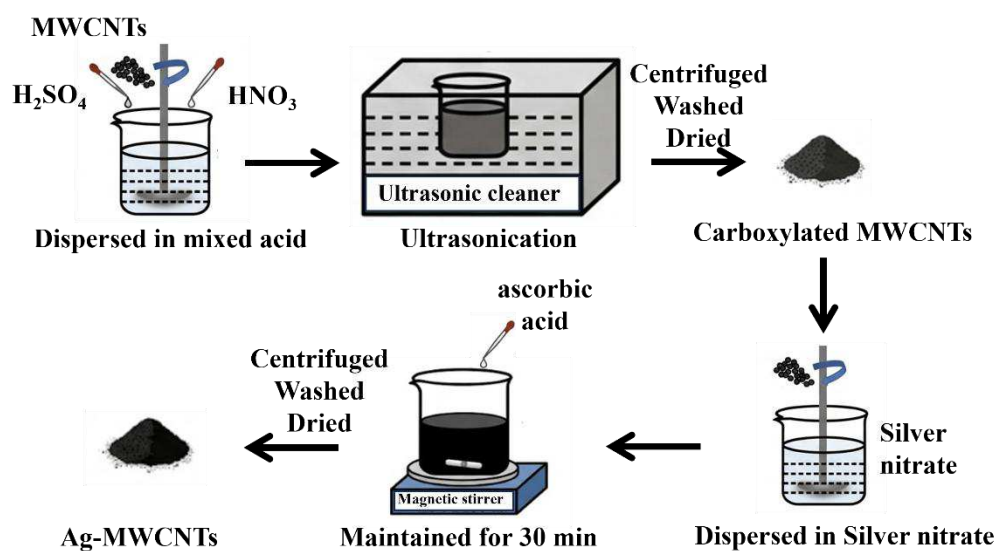


Fig. 1 . Preparation processes of Ag-MWCNTs

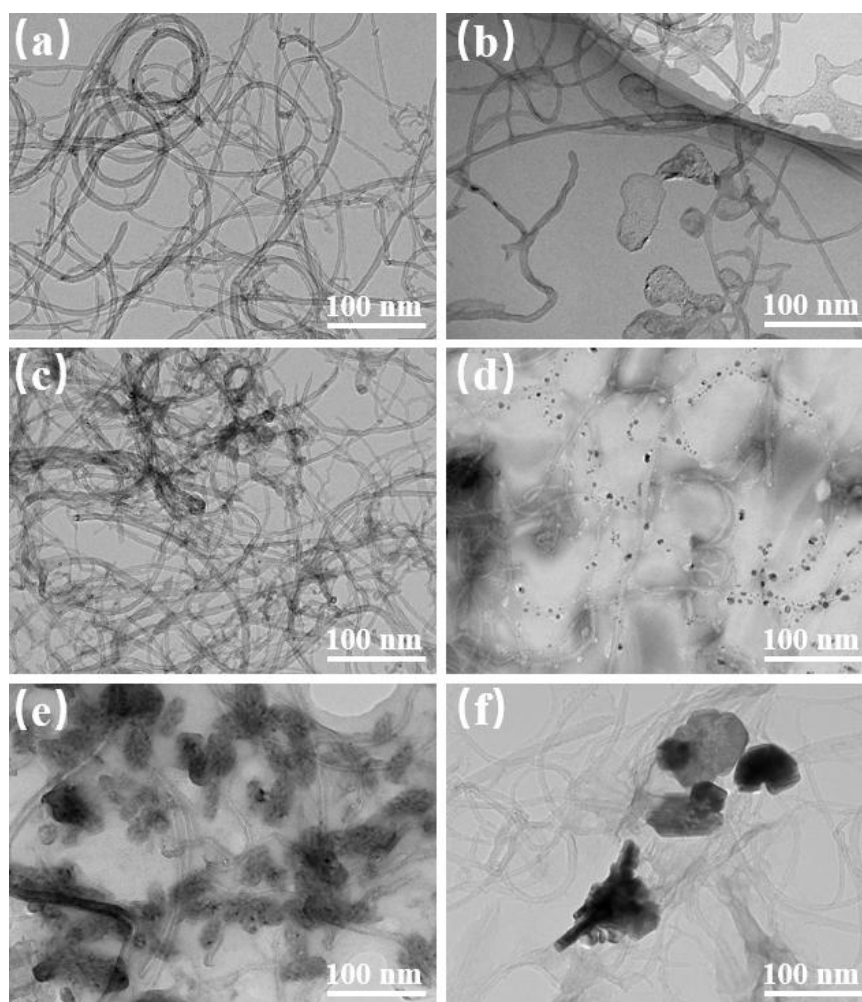


Fig. 2. TEM images of Ag-MWCNTs prepared under different carboxylation times at a 100 nm scale: (a) MWCNTs; (b) 0 min; (c) 3 min; (d) 5 min; (e) 10 min; (f) 15 min

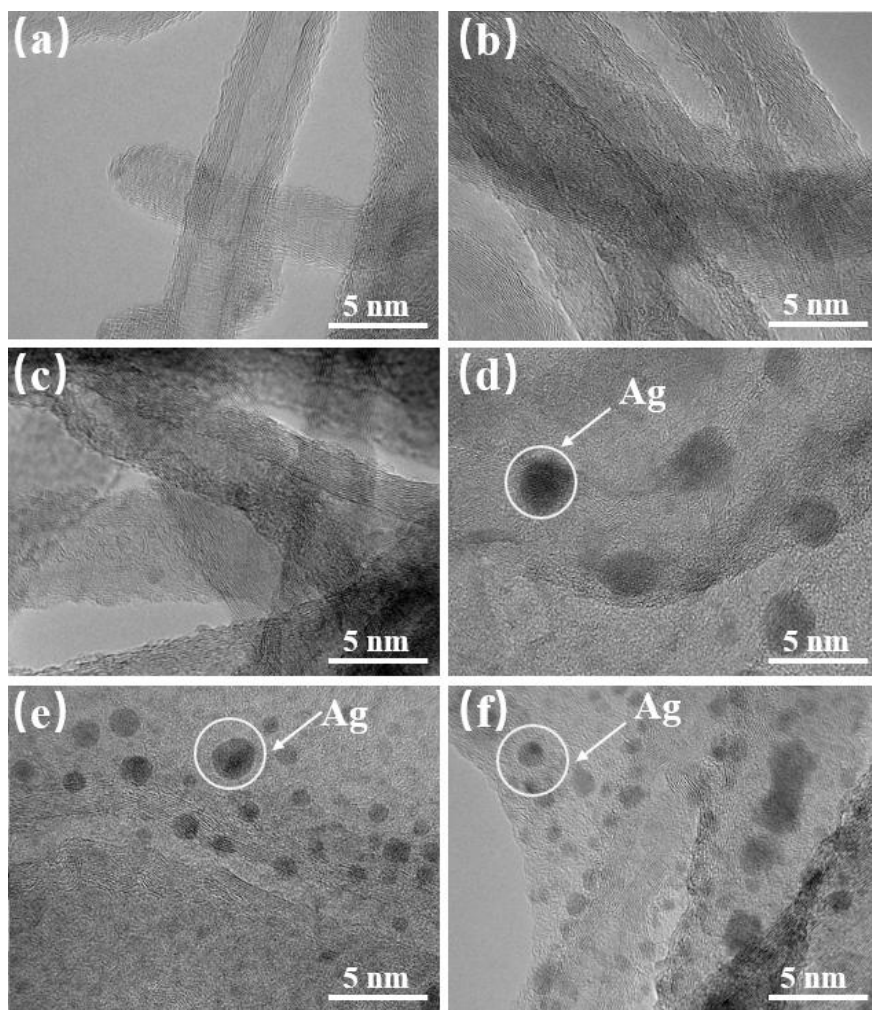


Fig. 3. TEM images of Ag-MWCNTs prepared under different carboxylation times at a 5 nm scale: (a) MWCNTs; (b) 0 min; (c) 3 min; (d) 5 min; (e) 10 min; (f) 15 min

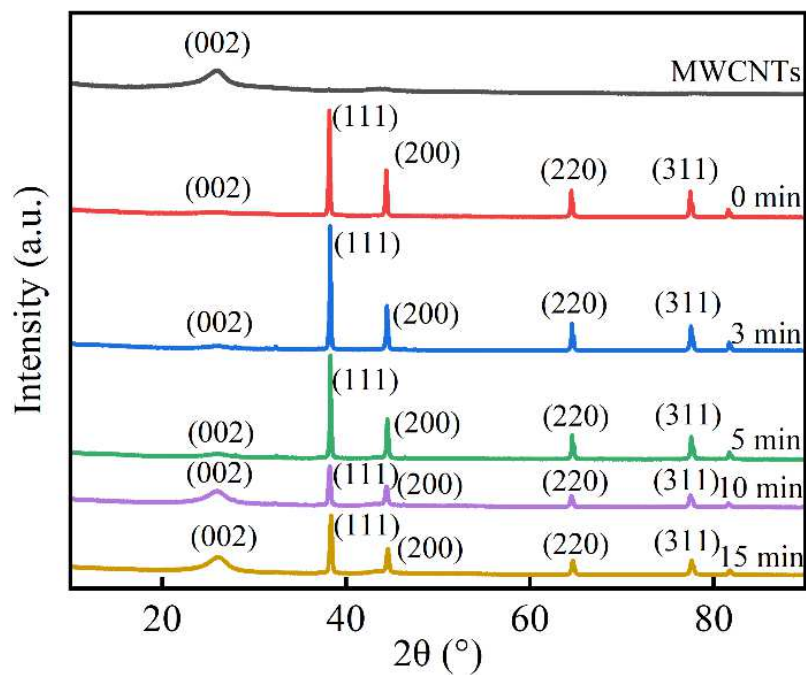


Fig. 4. X-ray diffraction patterns of MWCNTs and Ag-MWCNTs with different carboxylation times

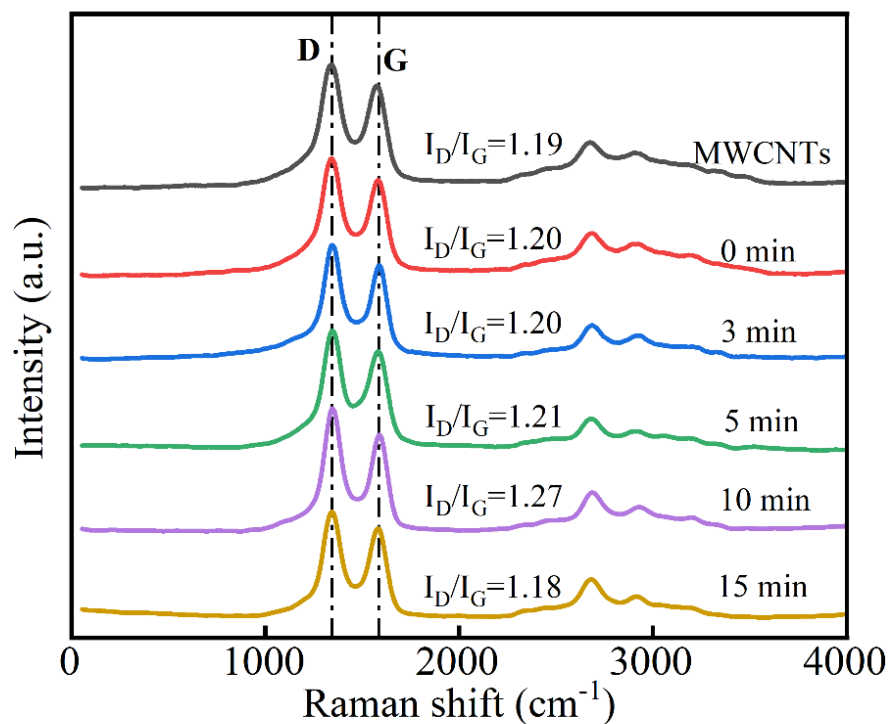


Fig. 5. Raman spectra of MWCNTs and Ag-MWCNTs with different ultrasonic carboxylation times

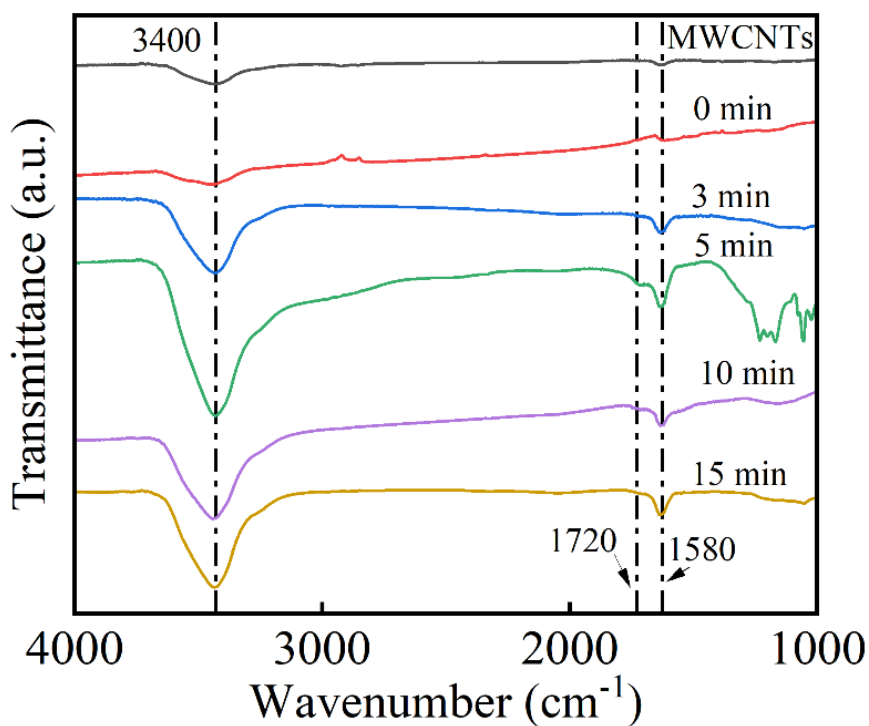


Fig. 6. FTIR spectra of MWCNTs and Ag-MWCNTs with different ultrasonic times

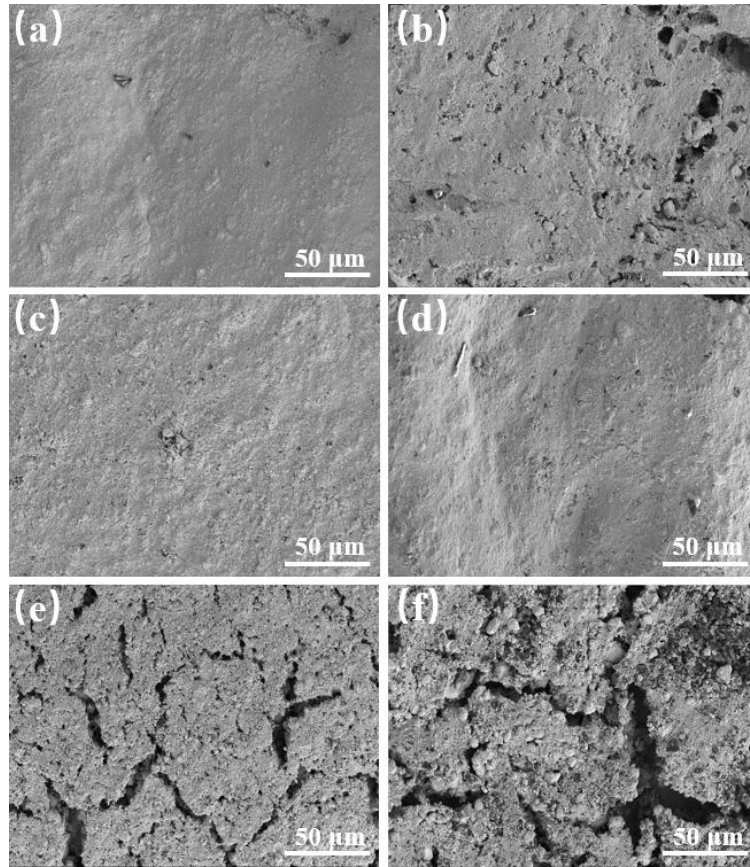


Fig. 7. SEM images showing the sintered morphologies of nano-silver pastes with various Ag-MWCNTs contents: (a) 0 wt%; (b) 0.5 wt%; (c) 1 wt%; (d) 3 wt%; (e) 5 wt%; (f) 10 wt%

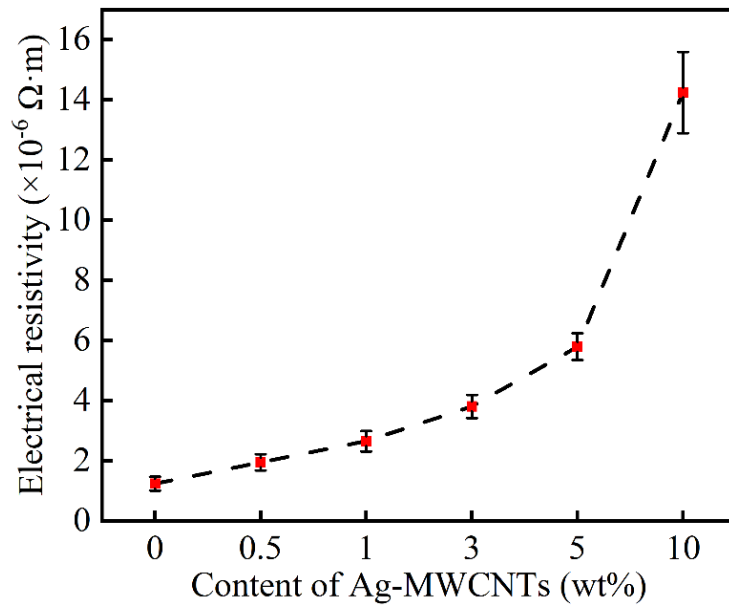


Fig. 8. Electrical resistivity of nano-silver paste enhanced with different Ag-MWCNTs loadings

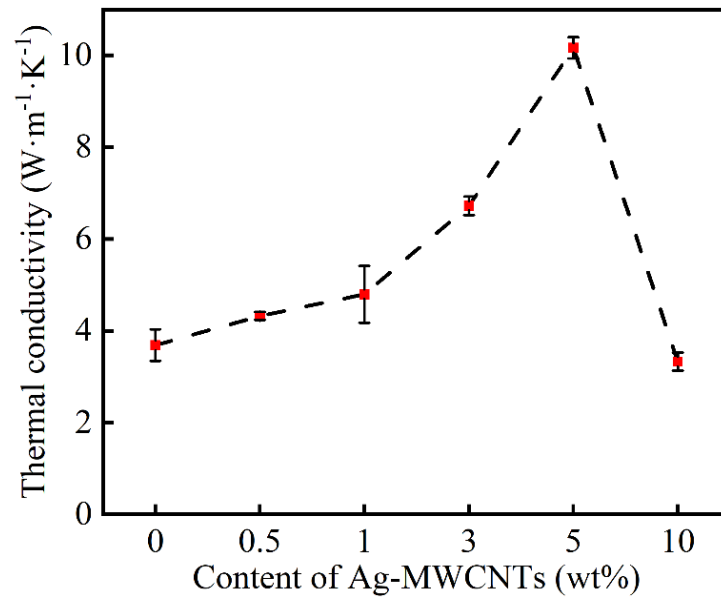


Fig. 9. Thermal conductivity of nano-silver paste enhanced with different Ag-MWCNTs loadings

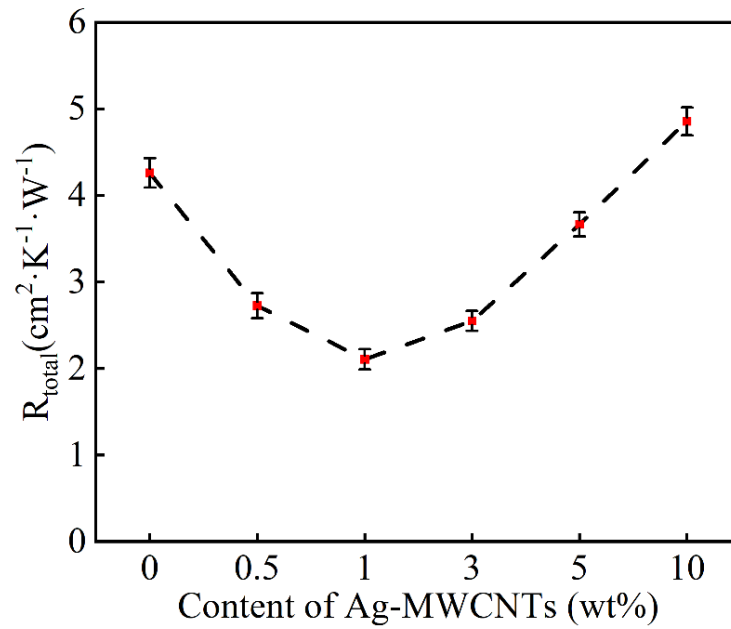


Fig. 10. Thermal resistance of GFs/copper substrate structures interconnected by nano-silver paste enhanced with different Ag-MWCNTs loadings

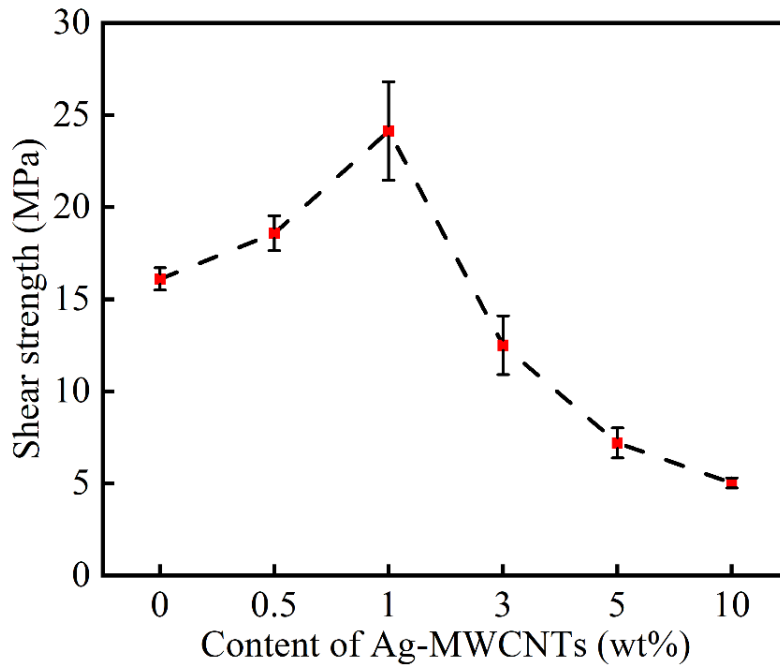


Fig. 11. Shear strength of sintered joints using nano-silver paste enhanced with different Ag MWCNTs loadings

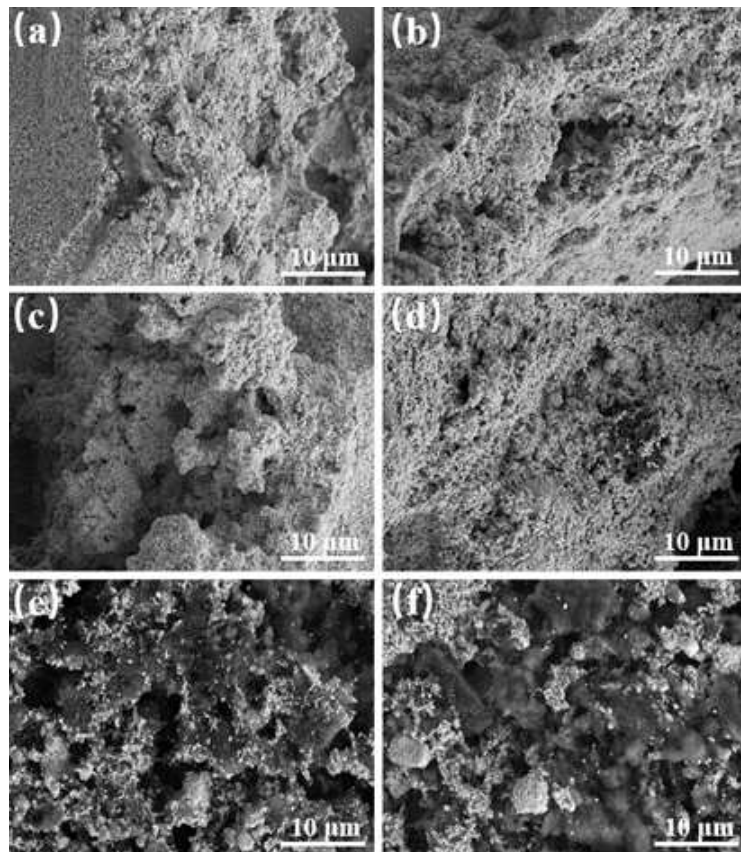


Fig. 12. Cross-sectional SEM images of sintered joints using silver paste enhanced with different Ag-MWCNTs loadings: (a) 0 wt%; (b) 0.5 wt%; (c) 1 wt%; (d) 3 wt%; (e) 5 wt%; (f) 10 wt%