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Research Article

Keywords: Nano-silver sintering, Composite materials, Shear strength, Mechanical interlocking, Thermal aging

Posted Date: October 23rd, 2026

DOI: <https://doi.org/10.21203/rs.3.rs-11259596/v1>

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Low-temperature sintering and thermal-aging reliability of nano-silver joints on cyanate ester composite substrates

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Abstract: Direct nano-silver sintering on cyanate ester composite substrates provides a promising route for integrating power chips with lightweight, thermally stable, and low-dielectric polymer composites. In this study, power chips were directly bonded onto fiber-reinforced cyanate ester composite substrates by low-temperature nano-silver sintering. Ag particles coalesced on the composite surface and formed a mechanically bonded heterogeneous interface. The joint achieved a maximum shear strength of 11.5 MPa, while the sintered Ag layer exhibited a porosity of 10.1% after sintering at 15 MPa for 15 min, while the porosity decreased to 6.5% at 20 MPa. After high-temperature aging for 600 h, the shear strength remained at 9.3 MPa, representing a decrease of only approximately 19%. The failure mode progressively shifted from separation at the Ag/composite interface to cracking and delamination within the composite substrate. These results demonstrate the feasibility of low-temperature nano-silver bonding on non-metallized cyanate ester composites and identify substrate thermal degradation as the principal limitation on long-term joint reliability.

Keywords: Nano-silver sintering; Composite materials; Shear strength; Mechanical interlocking; Thermal aging

1. Introduction

Next-generation aerospace platforms increasingly require active electronic modules, such as conformal antennas, microwave circuits, and power-conditioning components, to be integrated directly into load-bearing composite structures. Such

structural-functional integration can reduce the mass, volume, and assembly complexity associated with conventional electronic enclosures and mounting hardware [1-3]. Successful implementation, however, requires the composite platform to retain its structural and environmental performance while satisfying the electrical, thermal, and manufacturing requirements of the integrated electronics.

Cyanate ester (CE) resin composites have long been established as a preferred matrix material for aerospace structural skins, radomes, and antenna structures [4, 5]. The highly cross-linked triazine-ring network formed upon CE curing is inherently insensitive to polarization relaxation, resulting in an extremely low dissipation factor ($\tan\delta \approx 0.002-0.005$) that remains stable across a wide frequency and temperature range [6], making CE-matrix composites a natural choice for RF-transparent and microwave-integrated structural applications [4]. CE resins also exhibit glass transition temperatures (T_g) typically exceeding 250 °C, significantly higher than those of conventional epoxy and bismaleimide systems. This high T_g is essential for withstanding the elevated process temperatures involved in nano-silver sintering [7, 8]. Combined with extremely low moisture absorption, good mechanical properties, excellent radiation resistance, long shelf life, and favorable processability [9-13], CE-based composites have been widely adopted in aerospace engine fairings, thrust reversers, and structural skins [14, 15]. These outstanding properties collectively position CE composite substrates as a structurally and electrically viable platform for directly integrating power devices into lightweight airframe structures.

Integrating wide-bandgap (WBG) power devices, such as SiC and GaN chips, onto CE composite substrates imposes two key requirements on die-attach materials: compatibility with the processing-temperature limitations of the resin matrix and reliable performance under the high-temperature operating conditions of WBG devices. Conventional Pb-based solders are increasingly restricted because of environmental concerns, whereas Sn-based solders generally have relatively low melting points and insufficient high-temperature reliability [16, 17]. Owing to the high surface activity of Ag nanoparticles, nano-silver pastes can be sintered at relatively low temperatures to

form joints with high melting points, high electrical and thermal conductivities, and good oxidation resistance [18-21]. These properties make sintered Ag joints particularly suitable for WBG devices operating under high-temperature, high-current-density, and high-power conditions [22-26]. Moreover, the typical sintering temperature range of 180-250 °C is compatible with the high glass transition temperature of CE resins, indicating good process compatibility between nano-silver sintering and CE composite substrates.

Existing studies on nano-silver sintering have primarily focused on metallic substrates and metallized ceramic substrates, such as direct-bonded copper (DBC) and direct-bonded aluminum (DBA), with particular emphasis on interfacial diffusion, nanoparticle coalescence, and reliability under thermal shock and high-temperature aging conditions [27-34]. These studies have shown that metallic contact, interfacial diffusion, and the formation of Ag sintering necks are essential to achieving reliable bonding [35-37]. More recently, a limited number of studies have explored nano-silver sintering on conventional organic printed circuit board substrates and demonstrated its preliminary feasibility, although reliability issues associated with the polymer matrix have also been reported [38-41]. In contrast, non-metallized CE composite substrates lack the conditions required for metal–metal diffusion, and their interfacial bonding is therefore expected to rely primarily on mechanical interlocking and interfacial adhesion. Nano-silver sintering on such substrates has not yet been systematically investigated, and its bonding feasibility and high-temperature reliability remain to be established.

To address this gap, Ag-plated Cu dummy dies were directly bonded to non-metallized quartz-fiber-reinforced cyanate ester (QF/CE) composite substrates by pressure-assisted nano-silver sintering at 200 °C. The effects of sintering pressure and dwell time on the microstructure, porosity, electrical resistivity, and die-shear strength of the joints were systematically evaluated. High-temperature aging was then conducted at 220 °C for up to 1000 h to investigate the evolution of joint strength and failure behavior. The bonding mechanism at the Ag/QF/CE interface and the role of composite-substrate degradation in long-term reliability were further clarified. This

study demonstrates the feasibility of direct nano-silver bonding on non-metallized QF/CE composites and provides an experimental basis for structurally integrated electronic packaging in aerospace applications.

2. Experimental procedures

Fig. 1 shows the micro-scale Ag flakes and micro-/nanoscale Ag particles used in the preparation of the nano-silver paste. The thickness of silver flakes was about 50 nm, and the average diameter of particles was 400 nm. The mass ratio of these two components was 1:3 in the nano-silver paste, while an appropriate amount of organic solvent composed of ethyl acetate, terpineol, and polyvinylpyrrolidone (PVP) was added and mixed evenly.

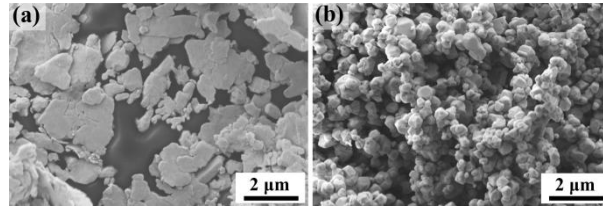


Fig. 1. Particles for preparing nano silver paste: (a) micron-scale silver flakes; (b) micro and nano scale silver particles.

Copper blocks used as dummy chips had dimensions of $5 \times 5 \text{ mm}^2$ and were coated with a $1 \text{ }\mu\text{m}$ -thick Ag layer. Quartz-fiber-reinforced cyanate ester composite boards with dimensions of $10 \times 10 \text{ mm}^2$ served as the substrates. As shown in Fig. 2a, the silver paste was directly printed on the quartz fiber-reinforced cyanate ester composite substrate, with a thickness of $100 \text{ }\mu\text{m}$. Fig. 2b shows a schematic diagram of the sintering structure of nano-silver paste. Sintering was performed using a custom-built hot-pressing sintering machine. The sample was first preheated at $140 \text{ }^\circ\text{C}$ for 10 min, followed by pressure-assisted heating and sintering, yielding the specimen shown in Fig. 2c. Considering the poor thermal resistance of the composite substrate and the characteristics of the silver paste, the sintering temperature was set to $200 \text{ }^\circ\text{C}$, with a sintering time of 10-15 min and an auxiliary sintering pressure of 5-20 MPa.

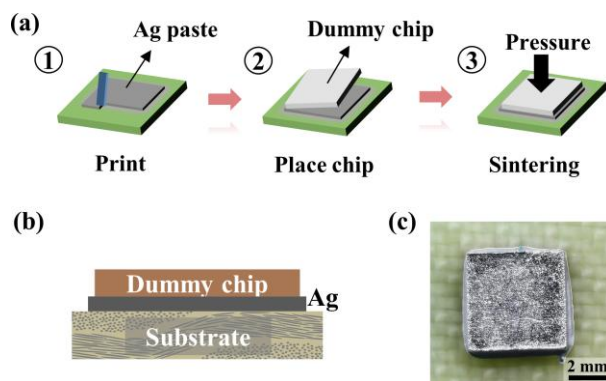


Fig. 2. (a) Sintering process; (b) the schematic diagram of the sintering structure; (c) the physical diagram of sintering.

The joints were subjected to high-temperature aging in a muffle furnace. The shear strength of the sintered joints were tested using a tensile shear testing machine (Dage 4000). For each set of processing parameters, the reported shear strength was the average value obtained from five joints. The microstructures of the sintered joints were examined using an optical microscope (Axio-LAB, Zeiss) and a field-emission scanning electron microscope equipped with energy-dispersive X-ray spectroscopy (FE-SEM/EDS; Sigma 500, Zeiss, Germany), with EDS used to obtain elemental distribution maps of the joint interfaces. The microstructure of the sintered Ag layer was further characterized by transmission electron microscopy (TEM; JEM-2100). The porosity was statistically analyzed using Image J software. The cross-section of the sintered joints was finely polished using ion beam polishing technology (Leica, EM TIC 3X).

3. Results and discussion

3.1 Design and analysis of nano-silver paste

The thermal behavior of the organic vehicle in the Ag paste was investigated by TG-DSC to determine an appropriate sintering temperature. As shown in Fig. 3a, the mass-loss behavior of the paste can be divided into two stages. Below approximately 150 °C, the paste lost 7.74% of its initial mass, mainly because of the evaporation of low-boiling-point organic components. Between 150 and 270 °C, distinct endothermic and exothermic features were observed, accompanied by only limited additional mass loss. These thermal events were associated with the volatilization and decomposition

of the residual organic constituents. The progressive removal of the organic vehicle facilitated closer contact between adjacent Ag particles and provided favorable conditions for subsequent neck formation. Considering both the thermal behavior of the Ag paste and the thermal tolerance of the composite substrate, 200 °C was selected as a compromise sintering temperature. Fig. 3b shows the microstructure of the silver paste after sintering at 200 °C in air. Diffusion at the contact interfaces between Ag flakes and particles promoted the formation and growth of sintering necks, resulting in a uniform and interconnected Ag network [43].

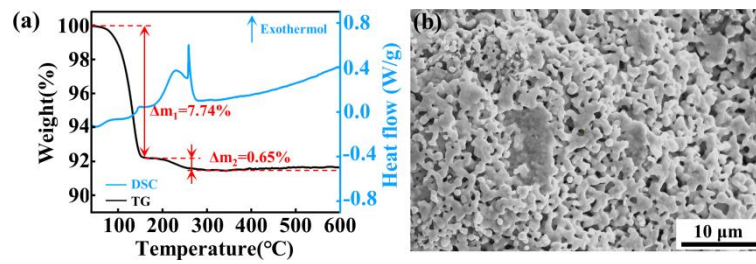


Fig. 3. (a) TG-DSC curves of nano-silver paste; (b) morphology of the nano-silver paste after sintering

3.2 Microstructure of the sintered joints

Fig. 4a shows the macroscopic morphology of the cross section of the sintered joint. Figs. 4b and 4c illustrate the SEM micrographs and corresponding elemental distribution maps of the joint. The sintered Ag layer formed effective interconnections with both the dummy chip and the quartz-fiber-reinforced cyanate ester composite substrate. Fig. 4d shows the interface between the dummy chip and the sintered silver layer, where silver particles diffused and bonded to the dummy-chip surface. Fig. 4e reveals that the sintered silver layer formed a heterogeneous interface with the composite substrate, attributable to the inherent surface roughness of the composite. Figs. 4f and g display the distributions of Ag and C at the interface between the sintered silver layer and the composite substrate; the resulting interface exhibited pronounced mechanical interlocking, thereby enhancing the interfacial bonding strength.

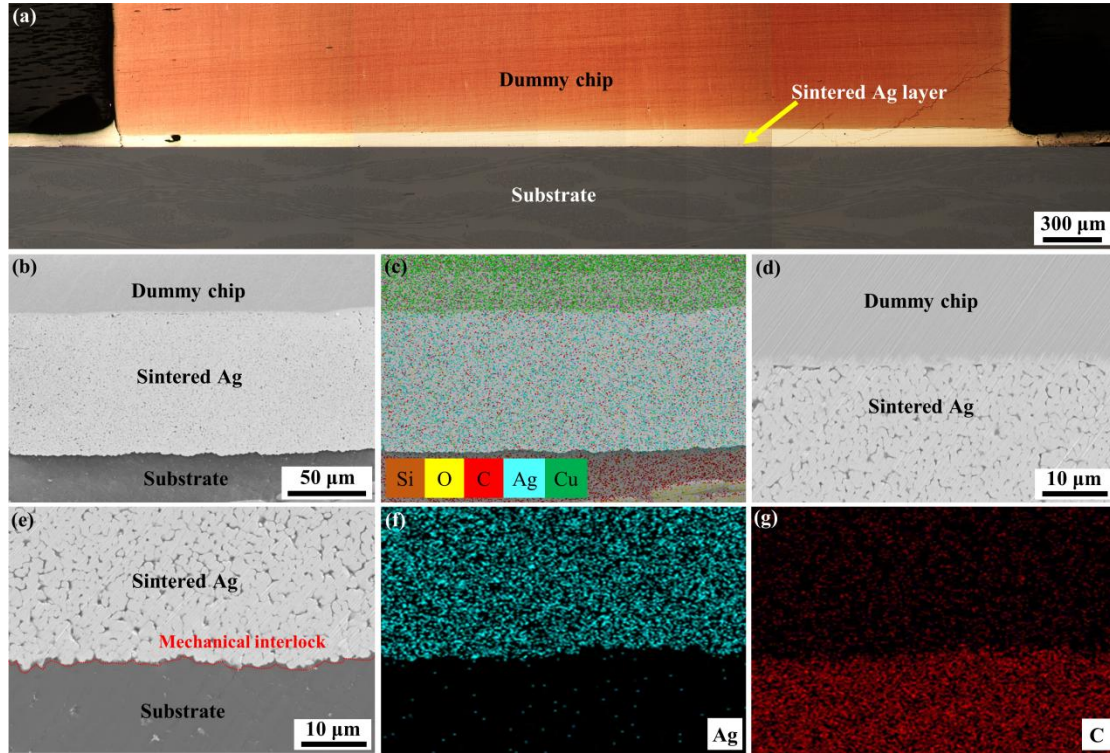


Fig. 4. Cross-sectional characterization of the sintered joint: (a) optical microscopy image; (b) SEM image; (c) element distribution across the joint; (d) bonding interface on the dummy-chip side; (e) bonding interface on the substrate side; (f) Ag distribution; (g) C distribution.

Fig. 5 schematically illustrates the joining mechanism responsible for the interfacial bonding between the sintered silver layer and the cyanate-based composite substrate. As shown in Fig. 5a, the nano-silver paste was uniformly deposited onto the substrate surface and partially penetrated into the surface asperities and gaps of the composite substrate, while the silver nanoparticles were homogeneously dispersed in the organic vehicle. During preheating at 140 °C (Fig. 5b), the organic constituents gradually volatilized, leading to a reduction in the interparticle spacing. This process promoted close contact between adjacent silver particles and provided favorable conditions for subsequent sintering. When the temperature increased to 200 °C (Fig. 5c), further removal of the organic components facilitated atomic diffusion among the silver particles. Driven by the reduction in surface energy, adjacent nanoparticles underwent diffusion, coalescence, and neck growth, resulting in the formation of a continuous conductive network and strong bonding within the sintered silver layer. Meanwhile, the penetration of silver into the surface irregularities of the cyanate-based

composite substrate produced effective mechanical interlocking, thereby enhancing the interfacial adhesion between the sintered silver layer and the substrate (Fig. 5d).

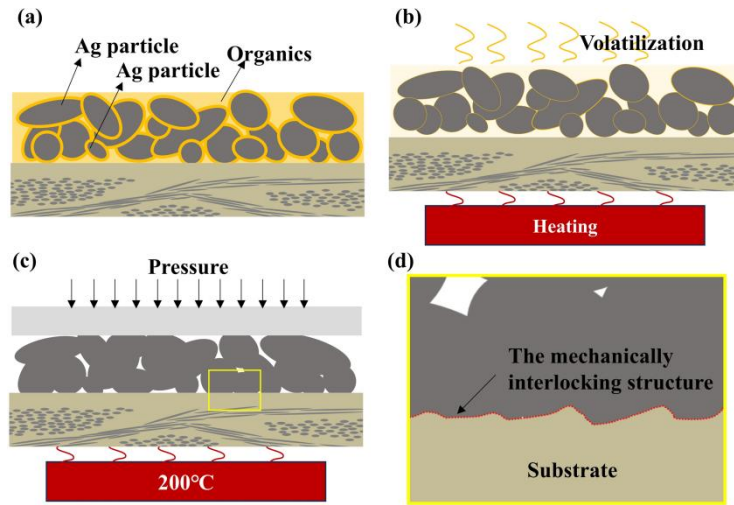


Fig. 5. Schematic illustration of the bonding mechanism between the sintered Ag layer and the composite substrate: (a) printing of the nano-silver paste; (b) volatilization of low-melting-point organic components; (c) diffusion and sintering of Ag particles under pressure; and (d) formation of mechanical interlocking at the Ag/composite interface.

To further investigate the microstructural evolution of the sintered silver layer, TEM sampling and analysis were conducted. As shown in Fig. 6, the continuous grain growth within the sintered Ag layer was driven by the combined effects of temperature and pressure, accompanied by the formation of a substantial number of twins and dislocations. These dislocations became entangled and accumulated, thereby impeding further dislocation motion and rendering particle deformation increasingly difficult. Concurrently, the twin boundaries also served as barriers to dislocation movement [44] and may contribute to the deformation resistance of the sintered silver layer.

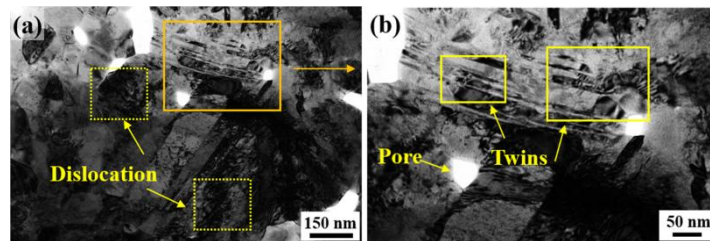


Fig. 6. TEM morphology of the sintered silver layer: (a) dislocations; (b) twins.

The porosity of the sintered silver layer is governed by the applied sintering pressure, which in turn exerts a direct influence on the morphological characteristics and the electrical conductivity of the layer. Fig. 7 illustrates the porosity of the sintered

silver layer as a function of sintering pressure. At a sintering pressure of 5 MPa, the porosity of the sintered silver layer was determined to be 15.35%. Upon increasing the pressure to 20 MPa, the porosity was significantly reduced to 6.53%.

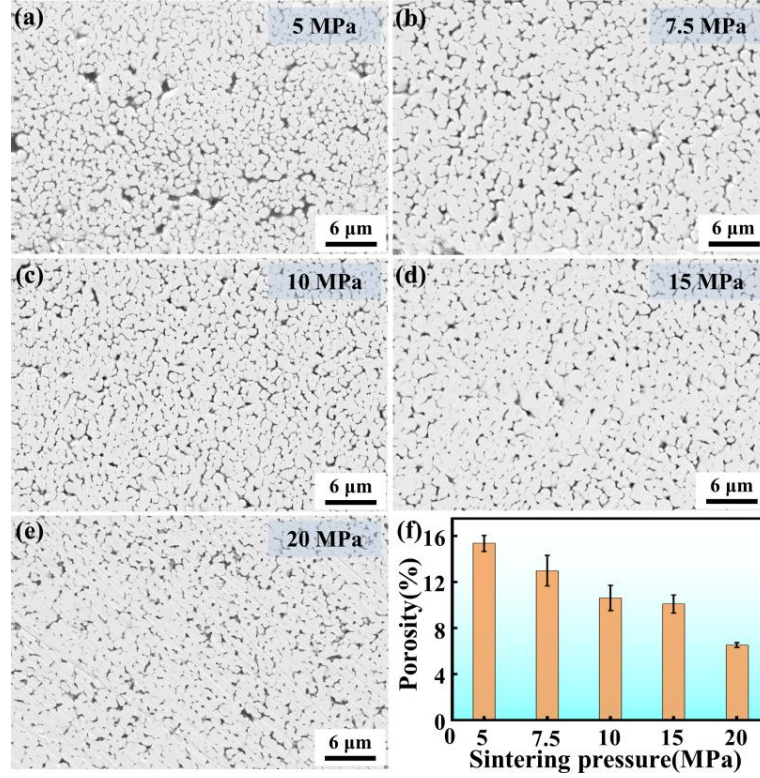


Fig. 7. Sintered Ag layer obtained under different auxiliary pressures: (a) 5 MPa; (b) 7.5 MPa; (c) 10 MPa; (d) 15 MPa; (e) 20 MPa; and (f) porosity of the sintered Ag layer as a function of sintering pressure. All samples were sintered at 200 °C for 15 min.

As shown in Fig. 8, the electrical resistivity decreased markedly after pressure-assisted sintering was introduced. Although the resistivity varied non-monotonically over the pressure range of 5-20 MPa, the minimum value was obtained at 15 MPa. At 20 MPa, the resistivity was approximately 50% lower than that of the pressureless sample. This overall reduction was mainly attributed to pressure-induced particle rearrangement and densification, which increased the interparticle contact area and facilitated the formation of electrically conductive pathways within the sintered Ag layer. The resistivity of the sintered silver layer was calculated using Eq. (1):

$$\rho = \frac{RS}{L} \quad (1)$$

where ρ is the electrical resistivity, R is the measured resistance, S is the cross-sectional area of the Ag line determined using ImageJ, and L is the length of the Ag line, fixed

at 0.01 m.

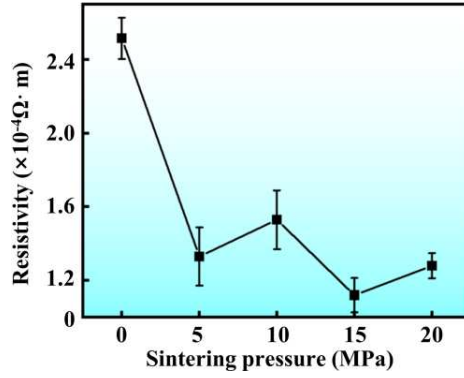


Fig. 8. Electrical resistivity of silver line as a function of sintering pressure at 200 °C for 15 min.

3.3 Strength and fracture surface of the sintered joints

The mechanical integrity of the sintered joint is critical to the reliable operation of the power device. Fig. 9a presents the combined effects of sintering time and pressure on the joint shear strength. The maximum shear strength of 11.5 MPa was achieved at a sintering time of 15 min and a sintering pressure of 15 MPa. At a given sintering pressure, the joints sintered for 15 min generally exhibited higher shear strength than those sintered for 10 min. However, as the pressure increased, the shear strength initially increased and then decreased. This trend may be associated with two possible factors: (i) a relatively weak bonding interface near the composite substrate, and (ii) deformation or structural damage of the composite substrate under excessive pressure, which may have adversely affected the shear strength. Fig. 9b illustrates the shear force-displacement curves under various pressures. Notably, the joint did not fracture even after a displacement of 0.6 mm under shear loading, indicating that the sintered joint possessed considerable energy absorption capacity and deformation resistance.

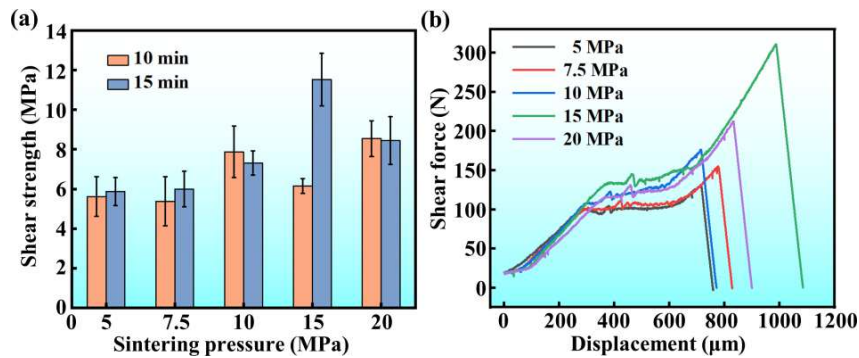


Fig. 9. (a) Shear strength of the sintered joints; (b) force-displacement curves of the sintered joints.

Fracture of the sintered joint was governed by its weakest link. Figs. 10a and c depict the fracture surfaces of the composite substrate and dummy chip after shear testing, demonstrating that failure propagated predominantly along the interface between the sintered silver layer and the composite substrate. The presence of residual sintered silver on the composite substrate side (Fig. 10b) suggests that the cohesive strength of the silver layer exceeded the interfacial bonding strength at this boundary. Figs. 10b and d further reveal shear-induced directional fracture features within the sintered silver layer. This oriented fracture morphology is indicative of substantial plastic deformation and is consistent with the pronounced deformation capacity observed in the force–displacement curves in Fig. 9b.

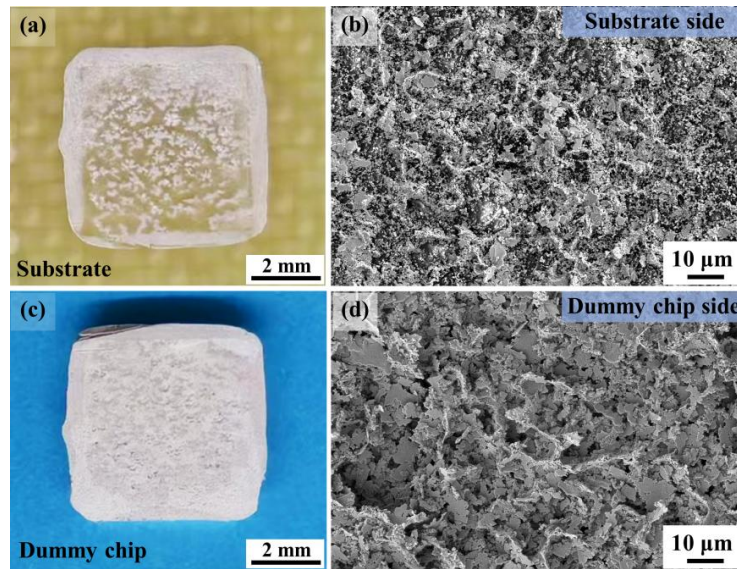


Fig. 10. (a, c) Morphology of the shear section; (b, d) microscopic morphology of the fracture surface.

3.4 Thermal aging behavior of the sintered joints

Given that the operating temperature of power devices can exceed 250 °C and the glass transition temperature of the cyanate ester composite ranges from 240 °C to 260 °C, an aging temperature of 220 °C was selected for the sintered joints in this study to evaluate their high-temperature reliability. Fig. 11a reveals that the shear strength of the joints initially increased and subsequently decreased with prolonged aging time.

This behavior is attributed to the continued diffusion of nano-silver particles within the sintered layer, concurrent with pore enlargement and progressive degradation of the composite substrate. Notably, even after the initiation of cracks in the substrate at 600 h, the joint shear strength remained at 9.3 MPa, representing a modest degradation of approximately 19% relative to the as-sintered condition (Fig. 11b). However, upon extending the aging duration to 1000 h, severe structural damage was observed, including delamination of the quartz fiber reinforcement and fiber/matrix debonding, as well as severe degradation of the cyanate ester matrix (Fig. 11c).

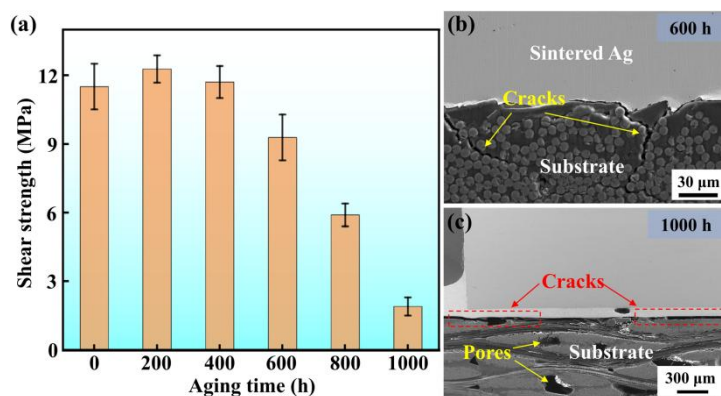


Fig. 11. Thermal aging behavior of the sintered joints at 220 °C: (a) shear strength as a function of aging time; (b) cross-sectional damage after 600 h; (c) cross-sectional damage after 1000 h.

4. Conclusions

(1) When sintered at 20 MPa for 15 min, the sintered silver layer exhibited a reduced porosity of 6.5%, accompanied by an approximately 50% decrease in electrical resistivity. The applied pressure facilitated particle rearrangement, plastic deformation, and interparticle diffusion, thereby promoting the densification of the silver layer. In addition, the infiltration of sintered silver into the surface asperities of the composite substrate contributed to the formation of mechanical interlocking at the interface.

(2) The shear strength of the joint increased initially and then decreased with increasing sintering pressure. The highest shear strength of 11.5 MPa was achieved at 15 MPa, owing to improved densification of the silver layer and enhanced interfacial contact. Further increasing the pressure may induce local stress concentrations or structural damage in the composite substrate, resulting in reduced bonding strength.

(3) After thermal aging at 220 °C for 600 h, the shear strength decreased to 9.3 MPa, corresponding to a reduction of approximately 19% compared with the unaged joint. The observed cracking in the substrate suggests that the strength degradation was primarily related to thermal-aging-induced damage in the composite substrate and the consequent weakening of interfacial adhesion.

These results demonstrate the feasibility of direct nano-silver bonding on non-metallized QF/CE composites and indicate that the long-term reliability of the joints is ultimately limited by the thermal stability of the composite substrate.

Authors' contributions

Xiaoli Li: Formal analysis, Writing- original draft, Writing- review & editing. Wei Guo: Funding acquisition. Shan Jiang: Funding acquisition. Zilong Peng: Methodology. Long Bai: Funding acquisition. Chen Shu: Formal analysis, Writing- original draft. Yu Zhang: Data curation, Resources, Writing-review & editing. Hongqiang Zhang: Data curation, Resources, Writing-review & editing.

Funding

This work was supported by the Joint Key Laboratory under Grant 637-2024-70-F-052.

Data availability

The datasets used and analysed during the current study are available from the corresponding author upon reasonable request.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Ethics Approval

Not applicable.

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