Microstructure and Mechanical Properties of SiCf/SiC Brazing Joints

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Abstract

Continuous silicon carbide fiber-reinforced silicon carbide composites (SiCf/SiC) can be applied to nuclear reactor cladding tubes, control rod sleeves, intermediate heat exchangers, piping, etc., and their highly reliable joining is crucial for nuclear safety. This study employs active brazing to join SiCf/SiC, aiming to achieve high-strength joining performance through analysis of microstructure, interfacial phases, and mechanical properties of brazed joints at various temperatures. The results demonstrate that AgCuTi filler metal can achieve stable joining of SiCf/SiC, and that surface grinding treatment of SiCf/SiC is conducive to improving joint shear strength. As brazing temperature increases, the reaction between Ti, Si, and C becomes more vigorous; when the brazing temperature reaches 890 °C, the Ti_5Si_3 brittle phase gradually diffuses and is dispersedly distributed within the brazed seam, while the strengthening phase TiC becomes the primary constituent of the reaction layer, effectively improving the microstructure and enhancing brazed joint strength.

Full Text

Microstructure and Mechanical Properties of SiCf/SiC Brazed Joints

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Abstract

Continuous silicon carbide fiber-reinforced silicon carbide matrix composites (SiCf/SiC) are promising materials for nuclear reactor components including fuel cladding, control rod guide tubes, intermediate heat exchangers, and piping systems, where highly reliable joining is critical to nuclear safety. This study investigates the active brazing of SiCf/SiC composites, analyzing the microstructure, interfacial phases, and mechanical properties of joints fabricated at different temperatures to achieve high-strength bonding. The results demonstrate that AgCuTi filler metal can achieve stable joining of SiCf/SiC, and surface grinding of the SiCf/SiC substrates significantly improves joint shear strength. With increasing brazing temperature, the reaction between Ti and both Si and C becomes more vigorous. At 890 °C, the brittle Ti5Si3 phase gradually diffuses and disperses throughout the weld seam, while the reinforcing TiC phase becomes the dominant component of the reaction layer, effectively improving the microstructure and enhancing joint strength.

Keywords: SiCf/SiC; Brazing; Interface layer; Shear strength

1. Experimental Procedures

1.1 SiCf/SiC Composite Fabrication

To facilitate brazing experiments and mechanical testing, plate-shaped SiCf/SiC composites were designed and fabricated. Domestic third-generation SiC fibers (diameter: 10 m, density: 3.21 g/cm^3) from Liya New Materials Company were woven into 2.5-dimensional plate preforms (dimensions: 200 × 300 × 3.5 mm^3, fiber volume fraction: 40%). The interphase and matrix were subsequently deposited via chemical vapor deposition (CVD) and chemical vapor infiltration (CVI) processes, with detailed parameters summarized in . The deposition procedure consisted of two steps: First, a pyrolytic carbon (PyC) interphase layer (thickness: 200 nm) was deposited on the SiC fiber surfaces via CVD using methane as the carbon source. Second, the SiC matrix was infiltrated via CVI using methyltrichlorosilane as the precursor, yielding final CVI-SiCf/SiC plates (density: >2.1 g/cm^3) suitable for brazing.

The surface and cross-sectional morphology of the SiCf/SiC composites were characterized using scanning electron microscopy (SEM), and their composition was determined by energy-dispersive X-ray spectroscopy (EDS). [Figure 1: see original paper]a-b show the surface SEM micrographs of SiCf/SiC, revealing a dense surface composed of granular SiC deposits approximately 50 m in size. EDS area analysis of these deposits indicated a Si:C atomic ratio of 1.09 (Si: 49.24%, C: 45.29%, O: 5.47%), demonstrating high-quality SiC deposition. [Figure 1: see original paper]c-d present cross-sectional SEM images, showing that the SiCf/SiC interior consists of SiC fiber bundles oriented in different directions. While the fibers within each bundle are relatively dense, some pores remain between fiber bundles (with maximum sizes exceeding 100 m).

1.2 Brazing Process

For brazing experiments, the SiCf/SiC plates were cut into two sizes (10 × 10 × 3.5 mm^3 and 5 × 5 × 3.5 mm^3) using water-jet machining. To achieve better interfacial bonding and strength, the SiCf/SiC surfaces were ground prior to brazing to promote filler metal spreading. The filler metal was Ag-26.77Cu-4.4Ti (wt.%) foil with a thickness of 0.1 mm, cut into 5 × 5 mm^2 pieces. The SiCf/SiC substrates and filler were stacked in a SiCf/SiC-AgCuTi-SiCf/SiC configuration and fixed in a vacuum furnace for brazing ([Figure 2: see original paper]a). Brazing was performed in a VBF-113 vacuum furnace (Shenyang Vacuum Technology Research Institute) at temperatures of 850 °C, 870 °C, and 890 °C for 10 min; the brazing thermal cycle is shown in [Figure 2: see original paper]b.

1.3 Microstructural and Mechanical Characterization

After brazing, the joints were sectioned perpendicular to the filler layer for cross-sectional observation. The specimens were cold-mounted, ground with abrasive paper, and polished. SEM was used to examine the joint microstructure, and EDS was employed to identify phases in the weld seam. The mechanical properties of the brazed joints were evaluated through shear strength testing at room temperature using a GLEEBLE-1500 thermal simulation testing machine. Specimens were mounted in a custom fixture and tested at a shear rate of 0.5 mm/min. The reported shear strength values represent the average of five tests for each brazing condition.

2. Results and Discussion

2.1 Microstructure of SiCf/SiC Brazed Joints

[Figure 3: see original paper] shows optical micrographs of SiCf/SiC joints brazed at different temperatures for 10 min. The gray regions correspond to SiCf/SiC, the light-gray intermediate layer is the brazed seam, and the black areas represent inherent pores in the SiCf/SiC composite. At 850 °C, the filler metal exhibited good wetting and spread across the entire joint interface ([Figure 3: see original paper]a). As the brazing temperature increased (870 °C and 890 °C), the molten filler, benefiting from improved wettability and fluidity, infiltrated into the internal pores of the SiCf/SiC ([Figure 3: see original paper]b-c), creating a mechanical anchoring effect that enhances joint strength. Notably, at 890 °C, the overall weld width showed a distinct narrowing trend ([Figure 3: see original paper]c), attributed to enhanced filler spreading at higher temperatures within the optimal process window.

[Figure 4: see original paper] presents cross-sectional SEM images and corresponding elemental maps of SiCf/SiC joints brazed at different temperatures. Due to micro-pores present within the fiber bundles (some being closed pores), minor unbonded defects were observed at the SiCf/SiC-weld interface. At 850 °C, the molten filler reacted with SiCf/SiC to form a sound bonded interface

([Figure 4: see original paper]a). Elemental analysis ([Figure 4: see original paper]a1) revealed that blocky compounds in the weld consisted of Ag-Cu solid solutions, with Ti segregation at the SiCf/SiC-weld interface and infiltration of Ti, Cu, and Ag into the SiCf/SiC. At 870 °C ([Figure 4: see original paper]b-b1), Ti segregation persisted at the interface, but elongated rod-like structures decreased significantly, replaced by numerous blocky phases. The most notable change was the refinement of AgCu solid solutions in the weld, with localized Cu-rich regions. At 890 °C ([Figure 4: see original paper]c-c1), a well-bonded interface formed between SiCf/SiC and the filler, with further refined solid solutions in the weld center and disappearance of Cu segregation, resulting in uniform elemental distribution.

The reaction at the SiCf/SiC side enabled dense bonding between the base material and weld. For detailed microstructural analysis, higher-magnification observations were performed ([Figure 5: see original paper]a-c). EDS analysis results () indicate that the continuous compound layer at the interface primarily consists of C, Ti, and Si, forming a banded TiC + Ti5Si3 phase structure, while the weld seam is filled with Ag and Cu solid solutions.

Elevated brazing temperatures promote atomic diffusion and interfacial reactions. Thermodynamic calculations show that the Gibbs free energy changes (ΔG^0) for these reactions are all negative, confirming that Ti can react with Si, C, and SiC [38, 43]:

$$SiC + Ti \rightarrow TiC + Si \quad \Delta G^0(k \cdot mol) = -136.9 + 0.0095T$$
$$Ti(s) + C(s) \rightarrow TiC(s) \quad \Delta G^0(k \cdot mol) = -186.6 + 0.0132T$$
$$5Ti(s) + 3Si(s) \rightarrow Ti5Si3(s) \quad \Delta G^0(k \cdot mol) = -194.14 + 0.0167T$$

Higher brazing temperatures effectively increase the driving force for Ti-SiC reactions. At 890 °C, the interface layer on the SiCf/SiC side became continuous and uniformly distributed, with more dispersed Ti5Si3 dark spots forming. [Figure 6: see original paper] shows Ti elemental distribution in joints brazed at different temperatures, revealing that higher temperatures promote more dispersed Ti5Si3 distribution toward the weld center. This dispersion of Ti5Si3 phase contributes to interfacial structural stability and joint strength enhancement. Based on the microstructural and compositional analysis, the interfacial structure of AgCuTi-brazed SiCf/SiC joints consists of TiC + Ti5Si3 + Ag(s,s) + Cu(s,s).

2.2 Mechanical Properties of SiCf/SiC Brazed Joints

[Figure 7: see original paper] illustrates the effect of brazing temperature and surface condition on the shear strength of SiCf/SiC joints. Surface grinding prior to brazing significantly improves shear strength by increasing the effective bonding area under constant filler metal conditions. With increasing brazing temperature, the joint strength initially increases and then stabilizes. The maximum average shear strength of 40 MPa was achieved at 890 °C for 10 min.

At lower brazing temperatures (850 °C), the reaction layer on the SiCf/SiC side was relatively thick and primarily composed of TiC reinforcement phase and Ti5Si3 brittle phase, which could not withstand large external loads, causing joint failure under modest shear forces. As the temperature increased (870 °C), the Ti5Si3 brittle phase gradually diffused into the weld region, the interfacial layer thinned ([Figure 6: see original paper]b), and joint strength improved accordingly. At 890 °C, the Si content decreased, brittle phases diminished, reinforcement phases increased, and Ti5Si3 brittle phase dispersion became more pronounced ([Figure 6: see original paper]c), though joint strength did not increase further (failure occurred in the base material). Therefore, controlling joint microstructure through brazing parameters and surface preparation can optimize joint performance.

[Figure 8: see original paper] shows cross-sectional fracture morphologies of SiCf/SiC joints brazed at different temperatures. [Figure 8: see original paper]a depicts the joint cross-section before shear testing. At 850 °C ([Figure 8: see original paper]b), fracture occurred near the interfacial layer. At 870 °C ([Figure 8: see original paper]c), fracture initiated near the interfacial layer and partially propagated along the ceramic interface, exhibiting a step-like fracture path. At 890 °C ([Figure 8: see original paper]d), fracture occurred entirely within the SiCf/SiC composite. [Figure 9: see original paper] schematically illustrates the fracture behavior at 890 °C, showing that failure within the SiCf/SiC base material indicates that the brazed joint strength equals or exceeds that of the parent material.

The high joint strength results from the combined contribution of both base material and weld seam. Weld strength depends on filler metal selection, elemental diffusion, and phase formation, while base material strength relies on composite fabrication quality. Observations revealed extensive filler infiltration paths, which, while beneficial for wetting, also indicate relatively low base material density and strength. Therefore, future work should focus on improving SiCf/SiC density through optimized CVI processes to enhance base material strength and achieve higher overall joint performance.

3. Conclusions

Based on in-house designed and fabricated CVI-SiCf/SiC composites, brazing studies were conducted using AgCuTi filler at 850 °C, 870 °C, and 890 °C. Microstructural analysis and mechanical testing yielded the following conclusions:

1. AgCuTi filler enables stable joining of SiCf/SiC composites, with surface grinding treatment beneficial for improving joint shear strength. The joint microstructure consists of TiC + Ti5Si3 + Ag(s,s) + Cu(s,s).

2. Increasing brazing temperature intensifies reactions between Ti and both Si and C, promoting diffusion and dispersion of the brittle Ti5Si3 phase throughout the weld region and improving microstructural uniformity. Simultaneously, the reinforcing TiC phase becomes the dominant component

in the reaction layer, significantly enhancing joint strength.

3. At a brazing temperature of 890 °C, fracture occurs within the SiCf/SiC base material, demonstrating that the brazed joint strength equals or exceeds that of the SiCf/SiC composite itself.

Author Contributions

Baoliang Zhang: Conceptualization, writing–original draft and editing, SiCf/SiC design and fabrication, participation in brazing test analysis. Hongqiang Zhang and Yu Zhang: Brazing experiments and corresponding content writing. Menghe Tu: Participation in SiCf/SiC fabrication. Wei Guo: Mechanical property testing, overall brazing experimental design and supervision.

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Note: Figure translations are in progress. See original paper for figures.