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Recent Joining Materials Options for Ceramic Matrix Composites

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ABSTRACT

The urgent need to mitigate climate change has driven the exploration of innovative materials and processes to achieve net-zero carbon emissions. This work investigates the potential of ceramic matrix composites (CMCs) as alternatives to traditional metals in high-temperature industrial applications, within the European Union project CEM-WAVE. Various joining materials, including glass-ceramics, metallic brazes, and pre-ceramic polymers, are evaluated for their effectiveness in creating robust CMC joints. The study demonstrates the viability of CMCs in steelmaking and other energy-intensive sectors, highlighting their resistance to extreme conditions and potential to replace metallic alloys. The findings underscore the need for continued research to optimize manufacturing processes and reduce costs, paving the way for broader adoption of CMCs in achieving sustainable industrial practices.

1 | Introduction

The escalating challenges posed by climate change, particularly the environmental impacts of human activities, are receiving increasingly urgent attention. This urgency is reflected in the European Commission's recent policy direction, the "*European Green Deal*", which aims to create a modern, resource-efficient, and competitive economy with net-zero greenhouse gas emissions by 2050, and to decouple economic growth from resource consumption [1].

In this context, research and innovation are being directed toward various approaches, including the transformation of processes to utilize clean energy sources and achieve lower CO_2 emissions. For energy-intensive sectors, where extremely high temperatures are required (e.g., steelmaking), the pathway involves using energy derived from hydrogen-based processes.

Hydrogen is experiencing unprecedented political and business momentum, with numerous policies and projects emerging globally. However, fuels containing hydrogen (mixtures of hydrogen and bio-methane) can cause detrimental phenomena such as hydrogen embrittlement in metals and hydrothermal attack in ceramics. These conditions cannot be sustained by the metallic materials currently in use. Therefore, there is a need for novel materials capable of enduring such extreme conditions over long periods while maintaining high performance.

This work summarizes some results obtained on this topic within the European Union project CEM-WAVE (Novel Ceramic Matrix Composites produced with Microwave-assisted Chemical Vapor Infiltration Process for Energy-Intensive Industries) [2]: details about the experimental activity have been reported in the cited references.

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CEM-WAVE focused on the use of ceramic matrix composites (CMCs) to replace metals: CMCs exhibit fracture toughness comparable to metals like cast iron, yet they are much lighter and designed for applications in severe environments, often combining high temperatures, high stress levels, and corrosive atmospheres.

Thanks to their exceptional properties, including relatively high toughness, low density, high temperature resistance, wear resistance, and corrosion resistance, CMCs are increasingly being considered for use in applications requiring reliable operation under extreme conditions [3–12].

Currently, CMCs are being introduced into many new fields, and their range of applications will expand if manufacturing and integration costs are significantly reduced.

Joining techniques remain a critical tool for reducing CMC assembly time, cost, and excessive material use. The development of suitable joining processes is essential for creating high-performance and high-efficiency components for the European industry. A systematic description of the various joining methods for ceramic matrix composites can be found [13].

If the challenges related to joining CMCs to themselves and to dissimilar materials are resolved, CMCs will be more extensively used to produce both new components and 'hybrid' structures. These hybrid structures, constructed from two or more different types of materials, combine the unique properties of each material to complement one another. Such components can be used in a range of applications across industries, including aerospace, gas turbines, furnace technologies, and nuclear plants [14, 15].

2 | Results and Discussion

2.1 | Context

The innovative approach of the CEM-WAVE project focused on drastically reducing the production costs of CMCs to enable their sustainable use in sectors where their benefits would be significant, both from an environmental and production viewpoint.

In the steelmaking sector, most blast furnaces (BF) and basic oxygen furnaces (BOF) currently reduce iron ore using high-temperature synthetic gas derived from coal and coke. Specifically, in steel processing lines, indirect heating of products in a protective atmosphere is carried out with radiant tubes equipped with gas-fired burners. These components are made from metallic materials, such as various grades of Inconel and stainless steel, depending on their location (first leg or end leg).

CEM-WAVE aims to validate the use of non-oxide (SiC/SiC) (silicon carbide fiber-reinforced silicon carbide matrix composites) and oxide CMCs (Al_2O_3/(Al_2O_3-ZrO_2)) (alumina fiber-reinforced alumina-zirconia matrix composites) in radiant tube furnaces instead of the currently used metallic alloys. Establishing the joining capability of these composites is essential to validate their use in radiant tube furnaces.

Sustainable steel production requires extremely high temperatures, often higher than 900°C, and involves exposure to green fuels such as hydrogen and biogas mixtures. Under these conditions, the joints used in production must withstand severe wear, corrosion, hydrothermal degradation, and long-term exposure to extreme environments. Therefore, the joining materials must meet several critical requirements:

- *Thermal and Mechanical Compatibility*: They should have a coefficient of thermal expansion (CTE) like that of CMCs.
- *Chemical Stability*: The materials must be thermodynamically stable and maintain compatibility with CMCs under thermal and chemical stress, and specifically to corrosion.
- *Wettability*: Good wettability on the composite surfaces is essential for reliable joints.

At Politecnico di Torino (Italy), various joining strategies for advanced ceramics and CMCs have been studied and designed as sustainable manufacturing processes for radiant tube components used in steelmaking.

The joining materials were selected based on their comparable CTE, stability, and wettability. The joints were characterized using single-lap offset mechanical shear tests and direct flame exposure tests to simulate in-service conditions.

2.2 | Joining of CMCs by Glass-Ceramics

One promising approach for joining CMCs is the use of glass-ceramics. Glass-ceramic materials combine the favorable properties of both glass and ceramics, offering a unique balance of high-temperature stability, mechanical strength, corrosion resistance, and resistance to thermal shock. Their ability to form strong, durable bonds with ceramic materials makes them an attractive choice for joining CMCs, especially in applications where conventional metal-based joining methods may not be suitable [16]. Glass-ceramics can be engineered to match the thermal expansion coefficients of CMCs, minimizing stresses at the joint interface [17–19].

Moreover, glass ceramics offer several advantages as joining materials for ceramic-based composites, particularly in harsh environments. These benefits stem from their unique properties and the ability to tailor their composition to meet specific requirements. They exhibit excellent high-temperature stability (i.e., showing a high softening point), making them suitable for applications in extreme thermal conditions. This high-temperature resistance is crucial for applications such as fusion reactors and thermal protection systems. They also provide excellent oxidation resistance, which is essential for maintaining the integrity of joints in oxidative environments. This property is particularly beneficial for high-performance structures exposed to oxidative conditions, as in the CEM-WAVE project.

In the present paper, two glass-ceramic systems, named T1 and T2M, were successfully used to join Al_2O_3/(Al_2O_3-ZrO_2) ceramic matrix composites, enabling them to withstand direct flame exposure at 900°C in combustion environments [20]. The oxide/oxide ceramic matrix composite was supplied by the

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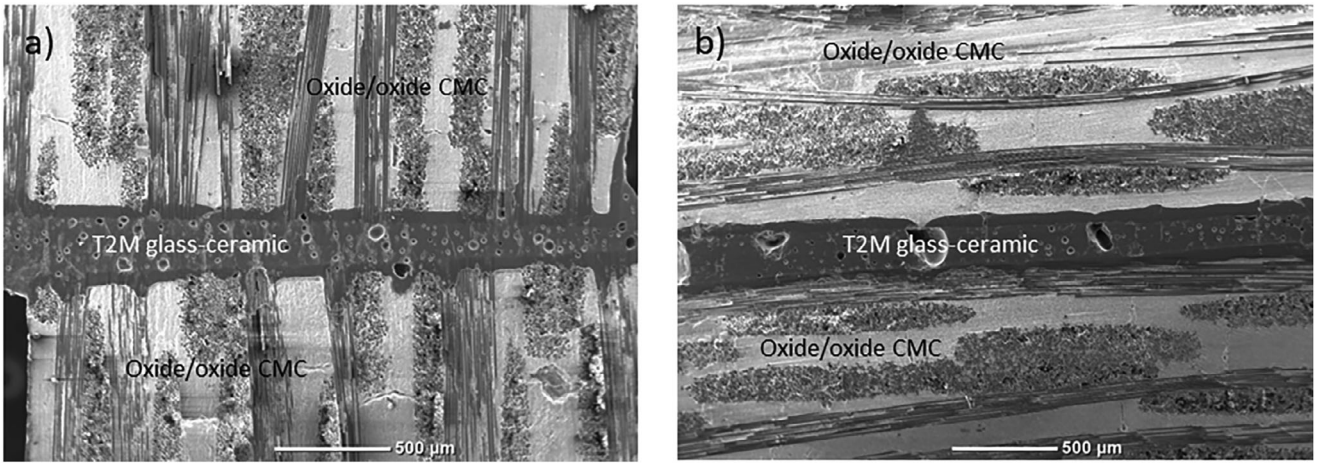

FIGURE 1 | Scanning electron microscopy (SEM) cross-sections of the butt-joint (a) and flat-joint (b) configurations for the T2M glass-ceramic oxide/oxide ceramic matrix composite (CMC) joints heat-treated at 1250°C for 10 min, followed by 960°C for 30 min under flowing Ar.

University of Bayreuth, Germany. The compositions of these glass-based joining materials were designed using the SciGlass 6.6 software database, with key parameters such as thermal expansion and glass transition temperature (T_g) guiding the design process.

The glass designated as T1 (SiO_2, Al_2O_3, TiO_2, Y_2O_3, CaO, Na_2O, and K_2O) was previously developed as an oxidation protective coating for titanium suboxide thermoelectric materials [21].

The glass referred to as T2M contained the same oxides as T1 but in different weight percentages. However, the TiO_2/Y_2O_3 ratio was maintained to ensure that $Y_2Ti_2O_7$ remained the primary crystalline phase after the devitrification process. Building on the established composition and properties of T1, the T2M glass was specifically designed to exhibit lower characteristic temperatures, facilitating the joining process at reduced temperatures. A comprehensive investigation of the thermo-mechanical properties, including the glass transition temperature, coefficient of thermal expansion, and crystalline phases of the glass-ceramics, was conducted and documented [20]. The surfaces of the CMCs were initially cleaned in an ultrasonic bath, then a slurry containing glass powders (70% vol) and ethanol (30% vol) was deposited on the sample's surface using a spatula, and then another CMC was placed on top to create a sandwich-like structure. The average thickness of the glass-ceramic layer was around 300 µm. As an example, the cross-sections of T2M joined CMCs in butt- and flat-configurations are shown in Figure 1.

Upon devitrification, the softening temperature of both parent glasses increased by approximately 300°C. Mechanical tests were performed on samples with an average joined area of 10 × 7.5 mm, with a loading rate of 0.5 mm/min at room temperature, via single-lap offset shear tests. A detailed description of this test and the test reference standard can be found [22, 23]. The results showed that thermal treatment did not affect the material properties of the oxide/oxide CMC joints, and the mean ± SD apparent shear strength values of 18 ± 5 MPa for T1 glass-ceramic joints after delamination and 12 ± 5 MPa for T2M glass-ceramic joints after mixed cohesive failure with delamination. Flame and thermal cycling tests were conducted on the glass-ceramic joined CMC, revealing good resistance at the composite interface when exposed to a direct oxy-acetylenic C_2H_2/O_2 flame positioned to reach 900°C on the samples' surface for 30 min. The T1 glass-ceramic composites exhibited excellent thermal cycling resistance, after being exposed to 10 cycles between 400 and 900°C, compared to the formation of vertical cracks in T2M joints (Figure 2).

Additional research [24] focused on joining SiC/SiC ceramic matrix composites using an yttrium aluminosilicate glass-ceramic (YAS). YAS systems are characterized by high electrical resistivity, chemical durability and stability, low thermal conductivity, and a low coefficient of thermal expansion, comparable to that of the SiC/SiC CMC substrates, minimizing thermal stress in the joint. These unique properties make YAS systems promising for coatings and joining materials for SiC/SiC composites in high-temperature applications. They also exhibit high glass transition temperatures, hardness, and elastic moduli. The yttrium aluminosilicate glass powders have been supplied by Fraunhofer ISC, Bayreuth, Germany. Two different SiC/SiC composites were used in this work to interrogate the properties of the joining material. Archer Technicoat Ltd (ATL), United Kingdom, supplied the first SiC/SiC composite. The second one, Keraman SiC/SiC, was supplied by BJS Composites GmbH, Germany. Two joining processes were conducted, with one being selected as the most effective, which was carried out up to 1450°C, for 20 min, followed by a treatment at 1105°C for 60 min under flowing Ar. The glass-ceramic exhibited a high softening point (undetectable up to 1400°C) and a coefficient of thermal expansion similar to the SiC/SiC CMCs. The joints showed enhanced adhesion with the non-oxide substrate and low porosity within the glass-ceramic layer (Figure 3). Mechanical testing revealed an apparent shear strength of around 61 ± 12 MPa, leading to delamination of the composite.

2.3 | Joining of CMCs by Metallic Brazes

Metallic brazes were also investigated as joining materials for CMCs in harsh environments.

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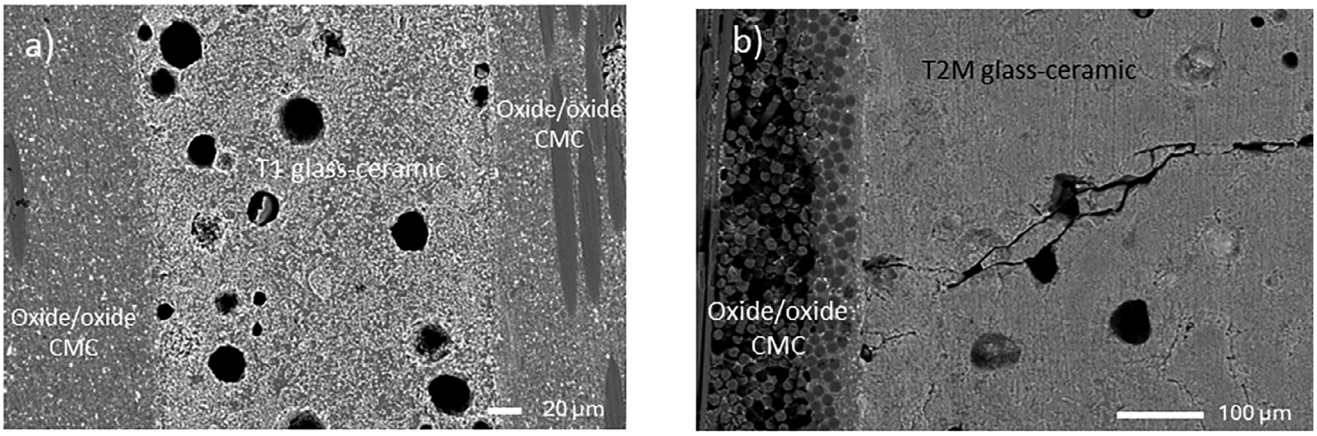

FIGURE 2 | Scanning electron microscopy (SEM) cross-sections of the T1 oxide/oxide ceramic matrix composite (CMC) joint (a) and the T2M oxide/oxide CMC joint (b) after the flame exposure treatment.

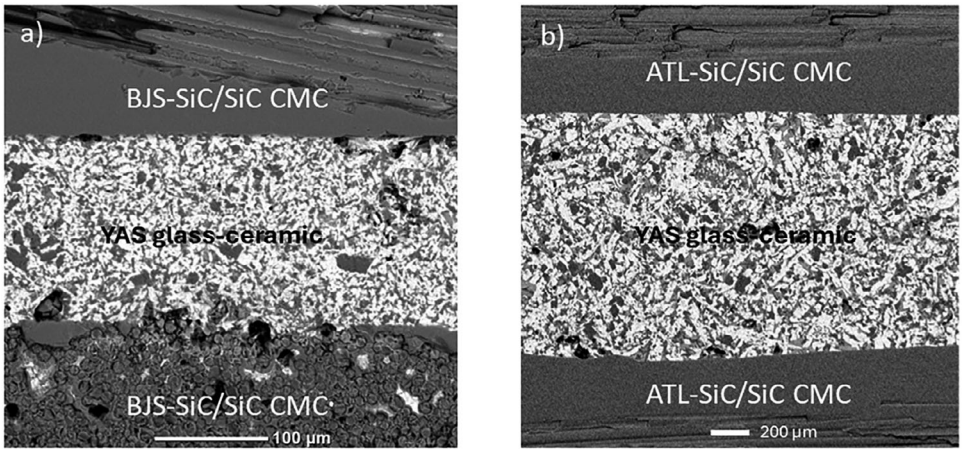

FIGURE 3 | Field-emission scanning electron microscopy (FE-SEM) cross-sections of BJS-SiC/SiC (a) and ATL-SiC/SiC (b) joined with yttrium aluminosilicate glass-ceramic (YAS) in flat-joints. Images a and b were obtained in BSE (back-scattered electrons) mode. Archer Technicoat Ltd (ATL) and BJS indicate the suppliers of the ceramic matrix composites (CMCs).

In the case of oxide-based CMCs, there are relatively few solutions available in the scientific literature. In contrast, various joining and brazing materials, including high-entropy alloys (HEAs), have been proposed, particularly for non-oxide-based CMCs.

HEAs are composed of five or more elements in equimolar or near-equimolar ratios. These alloys are known for their low brazing temperatures combined with high operating temperature stability, making them ideal for brazing superalloys, refractory metals, and carbon fiber composites across a range of industrial applications [25–31].

The careful design of HEA compositions, with tailored properties and a homogeneous microstructure, is essential for optimizing the mechanical strength and corrosion resistance of the joints for specific applications [32].

Achieving the best balance in the brazing method requires considering process parameters and selecting the most appropriate materials, particularly with respect to the operating environment and the maximum temperature of the brazed joints.

In this study [33], the $Al_2O_3/(Al_2O_3\text{-}ZrO_2)$ supplied by the University of Bayreuth, Germany, ceramic matrix composites were brazed using a commercial brazing alloy named TiBraze200Nb from Titanium Brazing Inc., USA. This alloy was supplied in the form of powders (140 mesh) with a water-based S-Gel binder. The TiBraze200Nb powder was manufactured as a mixture of the TiBraze200 brazing alloy from the same company, with the addition of Nb powder, a refractory element with a BCC crystal structure that increases the heat resistance of the brazed joints. The composition of the braze is as follows (wt%): Ti-17Zr-17Cu-17Ni-17Nb; the brazing temperature range is between 1030 and 1100°C, and the maximum working temperature for brazed joints is 1100°C.

The brazing process was carried out under vacuum conditions (10^{-5} mbar) at temperatures up to 1050°C, held for 10 min. The TiBraze200Nb alloy, containing 16 wt% binder, demonstrated excellent brazeability. Field-emission scanning electron microscopy imaging revealed a well-formed interface with an interlocked structure at the brazing alloy/CMC boundary in the butt-joint configuration (Figure 4).

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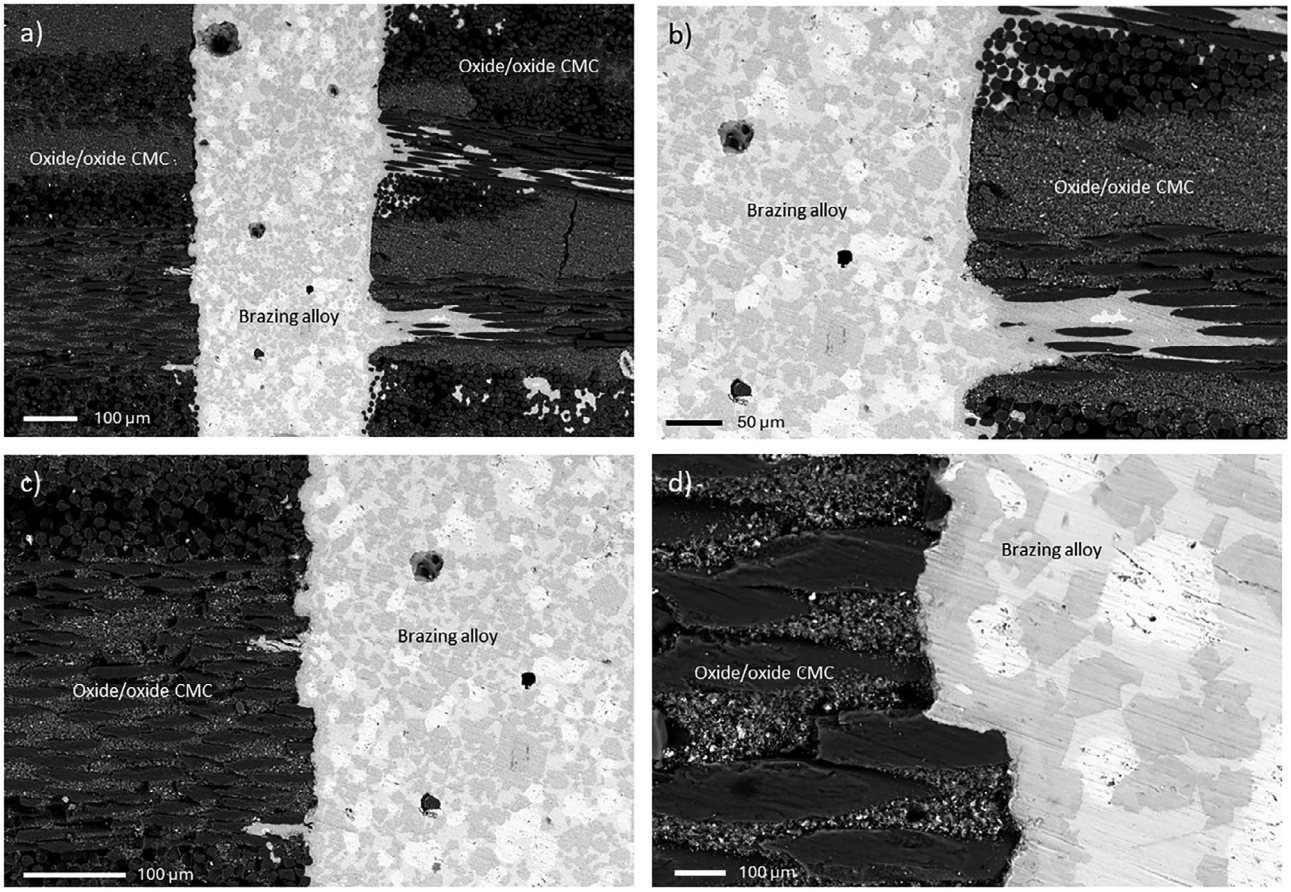

FIGURE 4 | Field-emission scanning electron microscopy (FE-SEM) cross-sections of oxide/oxide ceramic matrix composites (CMCs) joined with TiBraze200Nb brazing paste in the butt-configuration. Image (a) shows the overall view of the brazed joint, while (b–d) show the interface with the oxide/oxide CMC. Images obtained in back-scattered electrons (BSE) mode.

Mechanical testing showed that brazed butt-joints achieved higher apparent shear strength compared to flat-joint configurations. Furthermore, flame tests confirmed the superior performance of butt-joints over flat-joints when directly exposed to a C_2H_2/O_2 flame at 900°C for 30 min.

2.4 | Joining of CMCs by Pre-Ceramic Polymers

An alternative strategy for creating joints for CMCs involves the use of pre-ceramic polymers filled with ceramic particles and fibers, which are then thermally treated to eliminate the organic molecules through pyrolysis. The goal, still in its preliminary stages, is to develop reliable, high-temperature-resistant composite joints. Since the mechanical properties of polymer-derived joints primarily depend on the amount of residual porosity, it is crucial to introduce the optimum quantity of fillers into the polysilazane and to optimize the curing and pyrolysis processes. This approach aims to enhance the strength of the final joint, which benefits from being a CMC joint, similar to the materials being joined. From this point onward, newly obtained results concerning the use of pre-ceramic polymers for joining ceramic matrix composites are presented.

For the oxide-based CMC, $Al_2O_3/(Al_2O_3\text{-}ZrO_2)$, a commercial polysilazane, Durazane 1800, was used. Microstructural characterization of the joints, carried out by scanning electron microscopy, allowed for the evaluation of the adhesion between the components and the cohesion of the joint material (Figure 5). Two filler percentages of fine alumina powder (D50 = 0.55 µm) initially used (70% wt and 80% wt), degassed under vacuum for 10 min to reduce entrapped air and minimize residual porosity, showed the best results so far in terms of morphology and mechanical strength of the joints.

Mechanical tests in a single lap offset configuration were conducted to determine the strength of these joints. A lap-shear strength of about 4 MPa was achieved both at room temperature and at 600°C in the 70% wt filler-doped joint samples.

A commercial polycarbosilane, characterized by a Si-C backbone, was mixed with varying amounts of SiC particles and SiC fibers to join SiC/SiC composites: up to 80% wt SiC particles and 1% wt SiC short fibers were added. The curing and pyrolysis processes were conducted up to 1450°C in an argon atmosphere to prevent oxidation. Several joints were produced, and Figure 6 shows a typical cross-section.

The results indicated overall good adhesion to the SiC/SiC and fairly good cohesion of the SiC-based composite joint. However, the mechanical tests revealed that the joint strength is still far from acceptable levels.

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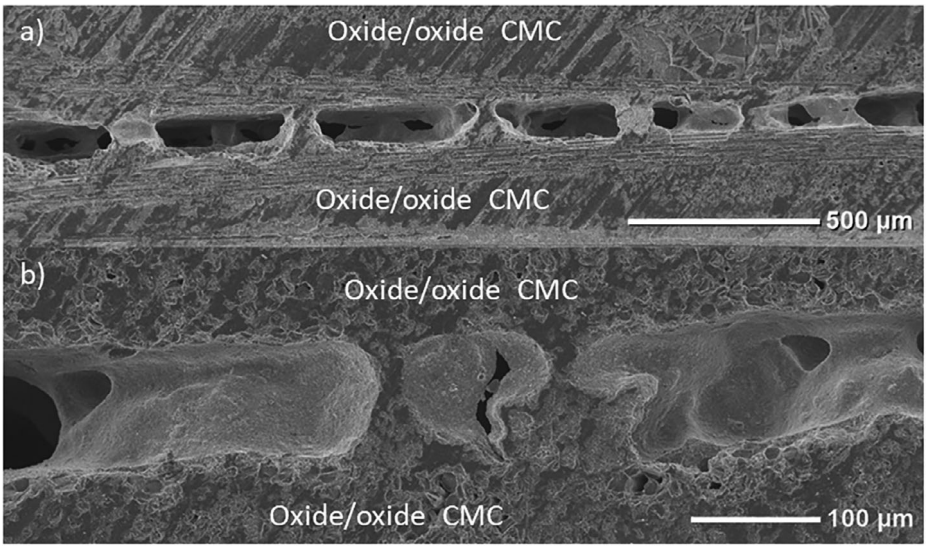

FIGURE 5 | Cross-sections of oxide/oxide ceramic matrix composites (CMCs) joined with preceramic polymer and 70% fillers. (a) shows the overall view of the joint, while (b) shows the higher magnification of the joint.

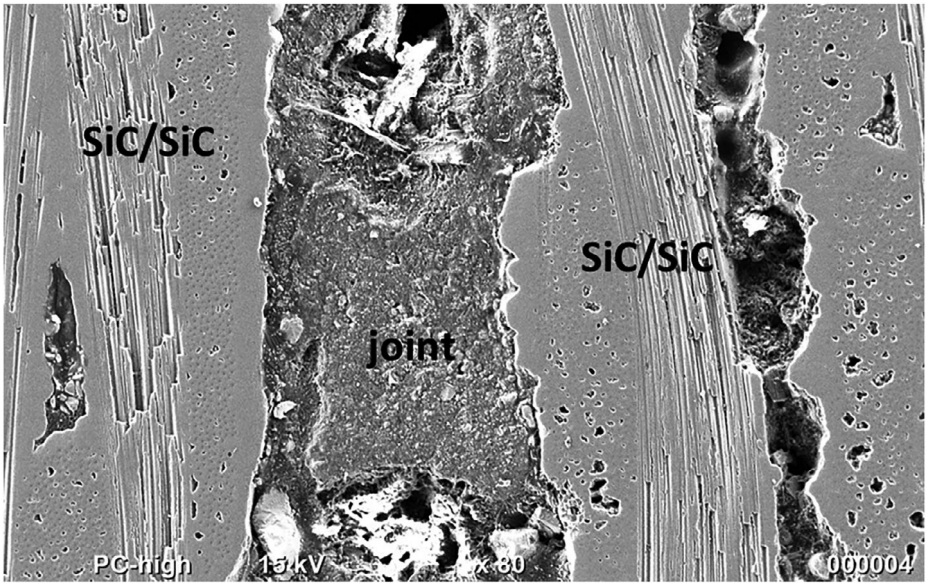

FIGURE 6 | Cross-sections of SiC/SiC joined with preceramic polymer and 80% of fillers.

To improve the mechanical properties of both these joints, further optimization of the filler content and the pyrolysis process is necessary. This includes fine-tuning the particle size distribution and the fiber amount and orientation within the pre-ceramic polymer matrix to reduce residual porosity. Additionally, exploring alternative additives that can promote better cross-linking and densification during pyrolysis may yield stronger joints.

3 | Conclusions

The work highlights the significant potential of ceramic matrix composites that demonstrated excellent performance in high-temperature and corrosive environments, making them promising for applications in steelmaking and other energy-intensive industries. This research identified glass-ceramics, metallic brazes, and pre-ceramic polymers as potential methods for joining CMCs, with ongoing measurements needed to assess their performance and reliability under industrial conditions.

Future studies could focus on comparing these approaches with current CMC connection methods to evaluate organizational performance, demonstrating the feasibility and potential superiority of the proposed techniques. Overall, the findings support the broader adoption of joining methods as a viable solution for CMCs in various industrial sectors.

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