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Joining of oxide/oxide composites by a preceramic polymer

Monica Ferraris [a,b,*], Aurora Pizzinat [a], Alessandro Benelli [a,b], Stefan Schafföner [c], Georg Puchas [c], Kevin Nordengren [d]

[a] Department of Applied Science and Technology, Politecnico di Torino, Torino I-10129, Italy
[b] J-TECH @PoliTO, Torino, Italy
[c] Chair of Ceramic Materials Engineering, University of Bayreuth, Prof.-Rüdiger-Bormann-Str. 1, Bayreuth 95447, Germany
[d] Almatis, C4701 Alcoa Road, Benton, AR 72015, USA

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ABSTRACT

Ceramic matrix composites (CMC), particularly oxide fiber-reinforced oxide matrix (ox/ox) composites, offer a viable alternative to traditional materials, due to their high temperature thermo-mechanical stability, intrinsic oxidation resistance and low density compared to metals.

However, a joining material having *the same* thermo-mechanical behavior and oxidation stability as the ox/ox composites is still an open issue.

In this respect, joining by a preceramic polymer offers a unique option, in principle enabling the fabrication of robust ceramic joints; since the process can be done at lower temperatures in respect to traditional methods such as brazing, it can also be attractive from an energy saving point of view.

This study investigated the use of a polysilazane-based preceramic polymer (Durazane 1800) filled with an increasing amount of alumina particles and 1 wt % chopped alumina fibers to join and coat ox/ox composites. The process was the same for joining and coatings: i.e. curing at 180°C and pyrolysis up to 1200°C in air, followed by microstructural and mechanical characterization on each sample.

Lap-shear tests were done on joined samples at room temperature, at 300°C and at 600°C, in air. Fracture surfaces exhibited cohesive failure, indicating sound adhesion between the joining material and the ox/ox composites. However, residual porosity and incomplete covering of the joined area were identified as a limiting factor affecting the joint strength.

X-ray computer tomography (CT) was used to measure the volume of residual porosity, cracks and lack of coating material after curing and pyrolysis on coated samples.

1. Introduction

The industrial sector remains one of the most challenging to decarbonize, accounting for approximately 30 % of global greenhouse gas emissions and a proportional share of global energy consumption [1]. Energy-intensive industries, including steel, cement, and aluminum production, are major contributors to these emissions [1,2]. Increasing energy efficiency is a key mitigation strategy, but technological advancements in energy, material, and end-use efficiencies are reaching their limits, underscoring the need for further innovation [1,3].

Ceramic matrix composites (CMC), particularly oxide fiber-reinforced oxide matrix (ox/ox) composites, offer a promising solution by enhancing energy efficiency and enabling high-temperature operations [4]. Ox/ox composites exhibit excellent thermal stability, maintaining long-term performance at up to 1150°C [5]. Their ability to replace traditional materials as well as steel and superalloys in various applications reduces production demands [6–8].

This paper explores an approach to joining ox/ox composites, recently re-considered for its potential, i.e. the use of a preceramic polymer filled with alumina particles and alumina fibers as joining material for ox/ox composites.

Preceramic polymer-based joining methods present significant advantages over conventional energy-intensive techniques, such as brazing and glass joining, primarily because the joining material obtained from preceramic polymers, when loaded with fillers such as particles and fibers, forms a ceramic matrix composite itself. Additionally, joining by preceramic polymers requires lower processing temperatures, thereby reducing energy consumption [9]. Through appropriate thermal

* Corresponding author at: Department of Applied Science and Technology, Politecnico di Torino, Torino I-10129, Italy.
E-mail address: monica.ferraris@polito.it (M. Ferraris).

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treatments (i.e., curing and pyrolysis), preceramic polymers transform into ceramics, enabling the fabrication of joints and coatings with tailored properties for high-temperature applications [10,11].

However, the polymer-to-ceramic conversion process entails gas release, isotropic volume shrinkage, and the formation of micro- and macro-porosity. These factors often lead to defects, such as cracks or pores, hindering the direct formation of dense ceramics unless component dimensions are very small [12]. To address this challenge, fillers including metallic and ceramic materials in various forms such as powders, platelets, nanotubes, and fibers can be incorporated into preceramic polymers to control shrinkage and enhance mechanical properties [12].

Preceramic polymer-based joining was successfully applied to monolithic SiC, SiC composites, and reaction-bonded SiC [13]. The use of silicone resins like GE SR350 has enabled the fabrication of ceramic joints with bending strengths up to 220 MPa and shear strengths of 39 MPa. The resulting joining material is an amorphous silicon oxycarbide.

Notably, tailoring filler properties can lead to minimal or even net-zero shrinkage during ceramization [14,15].

A very limited amount of research has been published on joining ox/ox composites by preceramic polymers: a recent study [16] presented a new method for joining ox/ox based on ionotropic gelation technique, i.e. the gelation of slurries with a polysaccharide polymer alginate as joining material. This is not a preceramic polymer, but it was used to obtain a ceramic-based joining material with a process at 1200°C. In this case, the presence of cracks in the joints gave a low double-notch shear strength of just 0.8 ± 0.2 MPa at room temperature.

In polymer-derived ceramic systems, residual micro and macro porosity is a key limiting factor for mechanical performance. Filler content and particle size distribution strongly affect shrinkage compensation, particle packing and gas transport during pyrolysis, and therefore the final porosity of the joint. In this context, X-ray computer tomography (CT) provides a powerful, non-destructive tool to quantify the three-dimensional distribution of voids and to correlate porosity to mechanical strength.

In the present work, CT was used to measure the volume of residual porosity, cracks and lack of coating material of Durazane 1800 based coatings with different alumina content, both after curing and pyrolysis.

2. Experimental procedure

The ox/ox composites were provided by the Chair of Ceramic Materials Engineering at the University of Bayreuth, Germany. These composites consist of Nextel^{TM} 610 [17] high-purity α-alumina fibers embedded in a matrix of alumina (75 wt%) and zirconia (25 wt%) and are manufactured with a patented prepreg process, which is explained in detail elsewhere [18]. Desized Nextel^{TM} DF-19 fabrics were infiltrated with a slurry containing 67 wt. % solid content, consisting of 70 wt% coarse alumina (CT 3000 SG, Almatis, Germany), 5 wt% fine alumina (Taimicron TM-DAR, Taimei Chemicals, Japan) and 25 wt% fine 3YSZ (TZ-3Y-E, Tosoh, Japan). The liquid phase of the slurry comprised 26 wt % glycerol in regard to the solid content, 1.5 % Sokalan PA 15 (BASF, Germany) as dispersing agent as well as deionized water. After infiltration, the prepregs were conditioned at 53 %RH and 25°C for 20 hours to obtain a defined water content and tack. Eight conditioned prepreg layers were stacked and laminated manually with a cold roll laminator. The laminates were dried at 100°C for 20 h and subsequently sintered at 1225°C for 2 h in air. The resulting ox/ox composites had a fiber volume content of 42.5 % a porosity of around 29 % and an interlaminar shear strength of 11.8 ± 2 MPa as reported in [19].

As shown in Fig. 1, the ox/ox composites are cut into 12.5 mm × 25 mm samples using a BRILLANT 2000 (ATM Qness GmbH, Germany) mechanical cutting machine. They were then polished to remove the top layer and expose the fibers, to enhance the infiltration of the preceramic polymer-based slurry. The ox/ox composites were ultrasonically cleaned twice (PROCLEAN 4.5S, ULSONIX, Germany) in acetone at 40°C for up to 10 minutes prior to joining or coating operations.

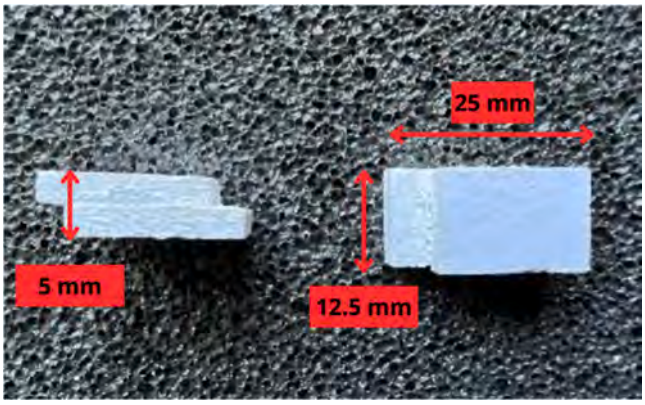


Fig. 1. Size and visual appearance of joined samples after pyrolysis at 1200°C: joining material is a 70 wt% alumina particle and 1 wt% alumina fibers loaded Durazane.

The joining material is prepared using Durazane 1800, a commercially available silicon-based preceramic polymer. This organo-polysilazane, produced by Merck KGaA (Germany), is a low-viscosity, solvent-free polysilazane resin [18]. The structure of Durazane 1800, which at ambient temperature is a low viscosity liquid (0.01- 0.04 Pa*s at 20°C, density 0.950-1.050 g/cm^3 at 25°C), consists of a silicon and nitrogen backbone [19]. When used as a silica precursor, the pyrolyzed material shows excellent high-temperature stability, withstanding peak temperatures of up to 1000°C, with a high ceramic yield of 80-90% depending on the atmosphere used [18]. In air, the crosslinking reactions are mostly hydrolysis and polycondensation reactions, i.e. the ≡Si–NH–Si≡ group reacts with water or oxygen molecules to form silanol groups, which subsequently polymerize to polysiloxane via polycondensation [20]. The presence of a certain degree of humidity on the surfaces to be joined play a crucial role in the formation of oxygen bridges [21].

The alumina powder used as passive filler, i.e. it does not participate in reactions during the pyrolysis of the preceramic polymer, is provided by Almatis (USA) and functionalized through a proprietary process [22]. Its denomination is “CT3000 SG” and it is characterized by high purity (99.7% Al_2O_3), a mono-modal particle size distribution with a mean particle size (D50) of 0.55 μm, and a typical specific surface area of 7.5 m^2/g.

The fibers used are 3M Nextel^{TM} 610 produced by a sol-gel spinning process. They are structural-grade ceramic fibers based on a fine-grained single-phase composition. The as-received fibers are coated by sizing, a processing aimed at providing both protection and easier handling for subsequent textile processing steps. The Nextel^{TM} 610 has a sizing composed of polyvinyl alcohol (PVA) and other additives (plasticizers, lubricants, etc). The sizing is removed by heating the fibers at 700°C for two hours in air, then fibers are chopped to a final length of about 15 mm.

Both alumina powder and fibers are incorporated into the preceramic polymer and thoroughly mixed using a mechanical mixer (Ultraturrax IKA T10 Basic, Germany) equipped with an immersion tip to ensure uniform dispersion and homogeneous slurry formation. The alumina fiber content is kept constant at 1 wt %, while the alumina powder content ranged from 50 to 90 wt %. The resulting slurry is degassed in a vacuum chamber for 10 minutes to eliminate air bubbles introduced during mixing.

As reported in the technical data sheet for the polymer, Durazane 1800 is cured in air using a CARBOLITE HTF16/27 (Verder Scientific GmbH and Co. KG, Germany) furnace preheated to 180°C for four hours, followed by slow cooling at a rate of less than 5 K/min to minimize crack formation. Thermogravimetric analysis (Mettler-Toledo TGA/DSC 3+STARe System) of the cured Durazane 1800 is performed in air, from

30°C to 1200°C, with a heating rate of 20 K/min.

Pyrolysis is then conducted in the same furnace under a controlled heating sequence in an air atmosphere, following a specific thermal cycle: heating at 5 K/min up to 400°C, followed by a 15-minute dwell; heating at 1 K /min up to 800°C, followed by another 15-minute dwell; further heating at 5 K/min up to 1200°C, or eventually to 850°C for lower temperature pyrolysis, followed by a 1-hour dwell; and finally, cooling at a rate of at least 5 K/min.

X-ray diffraction (Panalytical X'Pert3 Powder) analysis is performed after pyrolysis to determine the final composition.

For each slurry composition, joints were prepared on ox/ox composites. Before applying the slurry to the ox/ox composites in a sandwich-like structure, their surface is moistened with a drop of distilled water. After a few seconds to allow for water absorption, the excess water is removed by drying with laboratory paper. The slurry is then manually applied with a spatula and sandwiched between two ox/ox composites, which are kept in place by applying very limited pressure during curing by a weight placed on top. Due to the low viscosity of the slurry, the final joint thickness is in the range of a few hundred micrometers.

After joining, the samples undergo mechanical testing (in triplicate) using a universal testing machine (Zwick Roell Z050, Germany) with a custom-designed "reversal cage" fixture (Fig. 2).

The reversal cage fixture is designed so that, by applying tensile force on the two ends of the fixtures (gray parts in Fig. 2), the sample placed in the middle of the fixture (in red in Fig. 2) is subjected to compressive stress. The fixture is designed to keep external surfaces of the substrates parallel while loading, in order to minimize bending moments during test. This type of test is a modification of ASTM D905, commonly used for wood substrates. The test speed is set to 1 mm/min. The test is a single-lap offset test, and the nominal area of 12.5 mm × 25 mm is used to calculate the joints' apparent (lap) shear stress. The lap shear strength is measured at room temperature, at 300°C and 600°C. The temperature gradually increased with a ramp of 15 K/min to the set point and then held for 5 minutes to allow the sample's temperature to stabilize before testing.

Scanning electron microscopy (SEM) (JEOL JCM-6000 PLUS, Japan) is used to analyze the top views, the joint cross-sections, and the fracture surfaces after mechanical tests.

To quantitatively measure the residual porosity, some coatings were deposited on ox/ox composites by manually applying slurries with alumina powders content of 70 wt%, following the same procedure used to prepare the joined samples; coatings were then analyzed by X-ray computer tomography (CT) after both curing and pyrolysis.

The volumetric porosity was quantified by segmenting pores, cracks and regions lacking coating material within a defined *ideal* coating volume of interest and calculating the ratio between total void volume and ideal coating volume. CT also identified particles' agglomeration and heterogeneities in the entire coating volume.

3. Results and discussion

Unlike other preceramic polymers, which are usually in solution with solvents, Durazane 1800 can be used directly, thereby foregoing the need for additional processing steps to eliminate solvents before application. TGA measures the cured polymer mass loss during heat treatment, allowing the shrinkage compensations achieved by adding fillers to be estimated; furthermore, it helps define the optimal pyrolysis sequence: as can be observed in Fig. 3a, most of the mass loss occurs between 350°C and 850°C; at higher temperatures, further mass loss is negligible. The mass change of Durazane 1800 is caused by the release of volatile oligomers, ammonia, hydrogen and methane, while the incorporation of oxygen occurs to form Si-O-Si bridges after pyrolysis at 1000°C in air [23,24].

In this work, pyrolysis was performed at 1200°C in air and the XRD of Durazane 1800 after curing and pyrolysis (Fig. 3b) has the typical amorphous silica broad peak at 20°.

The incorporation of fillers is well known in the literature to be the most effective solution to reduce shrinkage, crack formation and porosity after curing and pyrolysis of preceramic polymers: thus, to obtain the joining material, Durazane 1800 was processed by incorporating alumina fibers (1 wt%) and an increasing amount of functionalized alumina powder (50 – 90 wt%).

Ideally, the volume of fillers added should match the volume loss during pyrolysis. However, accurately correlating mass loss from thermogravimetric analysis to the volume loss is challenging, due to materials' transformations and density changes during the thermal process. Therefore, the approach used in this work was based on adding an increased amount of alumina powders: the following alumina percentages (50, 60, 70, 80, 90 wt%) were investigated, together with 1 wt% of alumina fibers, which was kept constant for this study.

As an example, the thermogravimetric analysis (TGA) in air of Durazane 1800 with 70 wt% alumina particles and 1 wt% alumina fibers after curing is shown in Fig. 4a. It can be observed that the mass loss with 70 wt% alumina fillers is much lower compared to the one measured for the pure Durazane 1800 (Fig. 3a) without fillers.

XRD performed on Durazane 1800 with 70 wt% alumina particles and 1 wt% alumina fibers after curing and pyrolysis, Fig. 4b, shows the presence of alumina peaks as well as the amorphous silica broad peak. The final composition of the joining material after pyrolysis, as identified by XRD analysis, is thus composed of amorphous silica and crystalline alumina particles and fibers.

Fig. 5 shows the SEM cross-sections and higher magnification of relevant parts of the joined region after curing and pyrolysis at 1200°C, for 50, 60, 70, 80, and 90 wt% alumina loaded Durazane 1800 (alumina fibers are kept constant at 1 wt%).

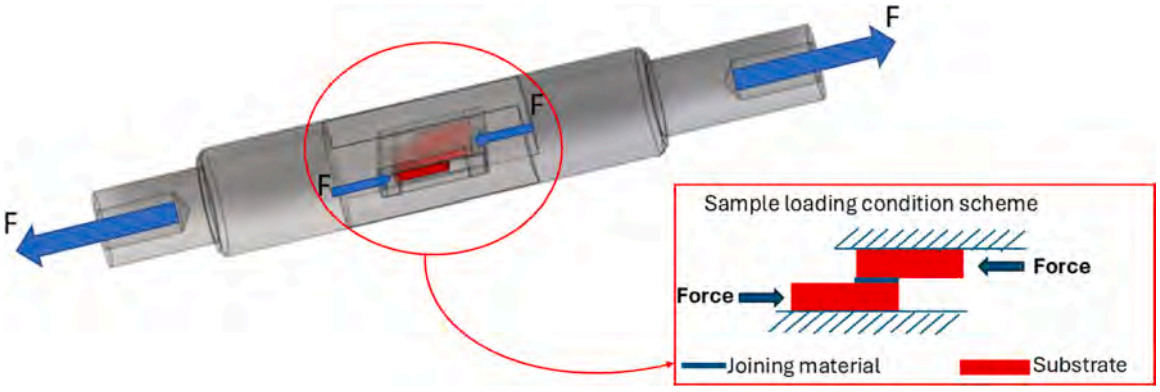


Fig. 2. Scheme of the mechanical test setup using a reversal cage system. In gray the two parts of the fixtures where the tensile movement is applied, while in red the sample that is instead subjected to compressive loading.

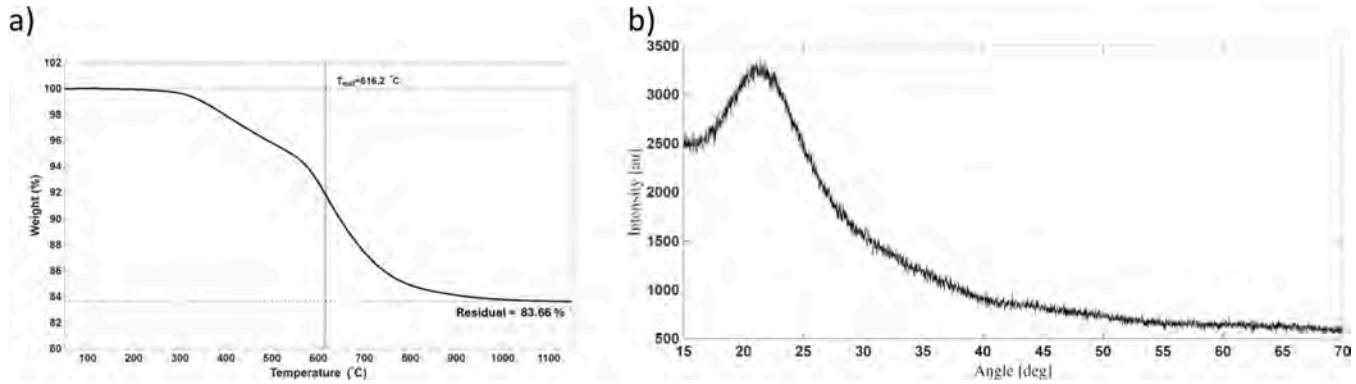


Fig. 3. TGA in air of the cured Durazane 1800 mass loss versus temperature (a) and XRD after pyrolysis at 1200°C (b).

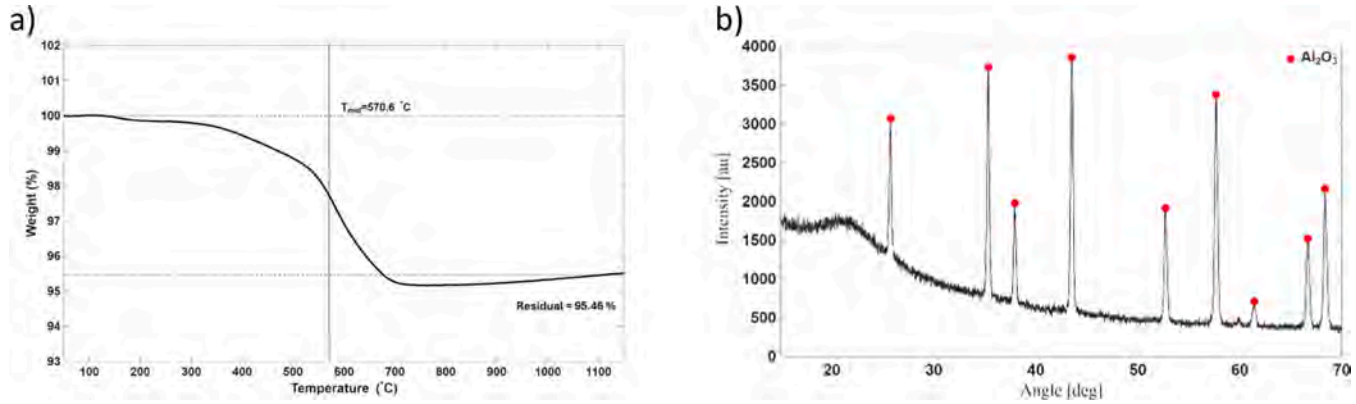


Fig. 4. TGA in air of the cured Durazane 1800 (with 70 wt% alumina particles and 1 wt% alumina fibers) mass loss versus temperature (a) and XRD after pyrolysis at 1200°C (b).

Upon examination of the cross-section images of the representative samples (Fig. 5), a certain reduction of the joint porosity seems to occur by increasing the mass content of alumina particles from 50 to 80 wt%; interestingly, the higher magnification images show that it is sometimes impossible to distinguish the interface between the ox/ox composites and the joining material, demonstrating the potential of this joining material for ox/ox composites.

While 50 and 60 wt% samples seem to have a very high residual porosity (Fig. 5 a–d), in the 90 wt% samples, the increased slurry viscosity resulted in a reduced infiltration and adhesion to the ox/ox substrate. The joint is almost completely separated from the ox/ox composites (Fig. 5 i-j).

Joints containing 70 wt% alumina particles (Fig. 5 e,f) exhibited in general lower porosity than the others. Additionally, preliminary lap shear tests at room temperature showed a better mechanical performance of the 70 wt% samples compared to the others, thus the mechanical test at 300°C and 600°C were performed on samples joined with 70 wt% alumina particle loadings (with 1 wt% fibers).

The geometry and visual appearance of the joined specimens after pyrolysis at 1200°C are shown in Fig. 1.

The mechanical behavior of the joints pyrolyzed at 1200°C is summarized in Fig. 6a. The average maximum lap-shear stress is approximately 3.5 ± 1.4 MPa at room temperature, 4.0 ± 0.8 MPa at 300°C and 3.0 ± 0.9 MPa at 600°C. The slightly higher strength at 300°C and the limited scatter at this temperature indicate reasonably robust behavior under these thermal loads.

Notably, the joint strength measured at 600°C is comparable to that at room temperature, demonstrating that the ceramic joining material retains its integrity and load-bearing capability over the explored temperature range. As a reference value, it must be considered that the ILSS of these ox/ox composites is about 11.8 ± 2MPa [19].

Although these strength values are relatively low compared to those reported in [4] for brazing alloys or glass-ceramic joining materials for ox/ox composites, they remain competitive with alternative polymer-derived joining approaches and are achieved with a joining material that is compositionally similar to the ox/ox substrate.

In fact, the joint itself is a particle- and fiber-reinforced ceramic matrix composite, which provides chemical and microstructural continuity with the ox/ox composite. Furthermore, the joining process is performed at 1200°C, which corresponds to the maximum service temperature of the ox/ox composite, making the process attractive from a compatibility standpoint.

To reduce the joining process temperature, additional joints were prepared and pyrolyzed at 850°C, a temperature above which TGA in Fig. 3a indicates negligible mass loss. The same 70 wt% alumina / 1 wt% fiber composition was used, and the joints were tested at room temperature, 300°C and 600°C; the corresponding results are shown in Fig. 6b. The joints pyrolyzed at 850°C exhibit significantly lower mechanical strength at all the tested temperatures. Average maximum stresses of 1.5 ± 0.8 MPa at room temperature, 1.0 ± 0.1 MPa at 300°C and 2.0 ± 0.2 MPa at 600°C were measured. Although a modest increase in strength is observed at 600°C compared to room temperature, the values remain clearly lower than those of the joints pyrolyzed at 1200°C. This indicates that pyrolysis at 850°C does not provide a sufficiently developed ceramic network and is therefore not suitable for producing mechanically robust and thermally stable joints. Overall, the comparison between Figs. 5 and 6 confirms that pyrolysis at 1200°C is more effective in achieving a mechanically reliable joining material.

Fractographic analysis after mechanical testing was performed on all joints. In all cases, cohesive failure was observed within the joining

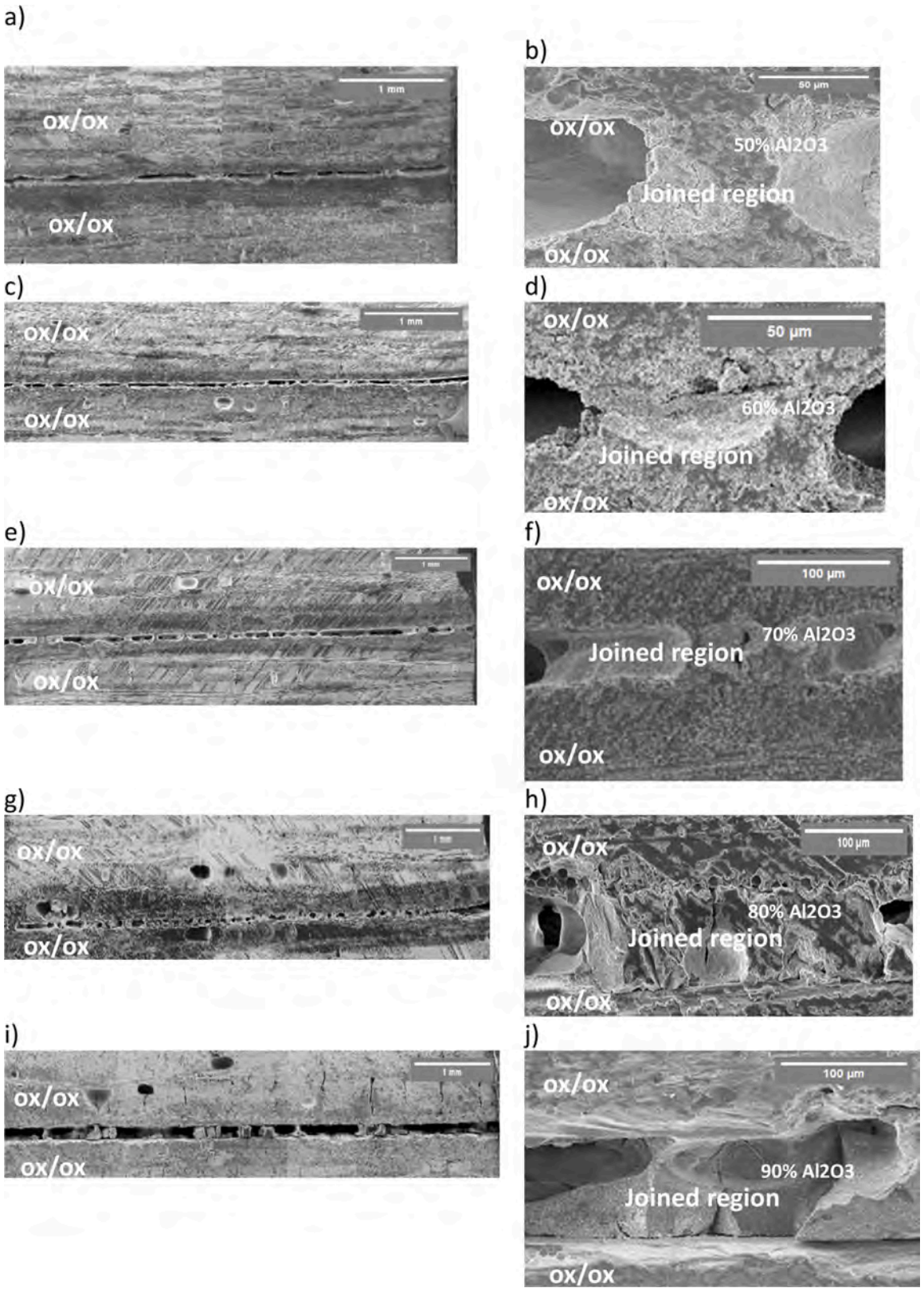


Fig. 5. Joined ox/ox samples: SEM cross-sections and higher magnification of relevant parts of the joined region (b,d,f,h,j) after curing and pyrolysis at 1200°C: joining material is a 50 (a,b), 60 (c,d), 70 (e,f), 80 (g,h), and 90 (i,j) wt% alumina particle loaded Durazane 1800 (alumina fibers are kept constant at 1 wt%).

layer, with joining material present on both fracture surfaces, indicating good adhesion to the ox/ox substrates even after testing at 600°C.

A representative macroscopic view of the fracture surface of a joined sample with 70 wt% alumina particles, 1 wt% alumina fibers, after curing and pyrolysis at 850°C, as well as a higher-magnification SEM image are shown in Fig. 7 (a,b).

Despite the cohesive nature of the failure, the fracture surfaces reveal that only a limited fraction of the nominal overlap area is effectively

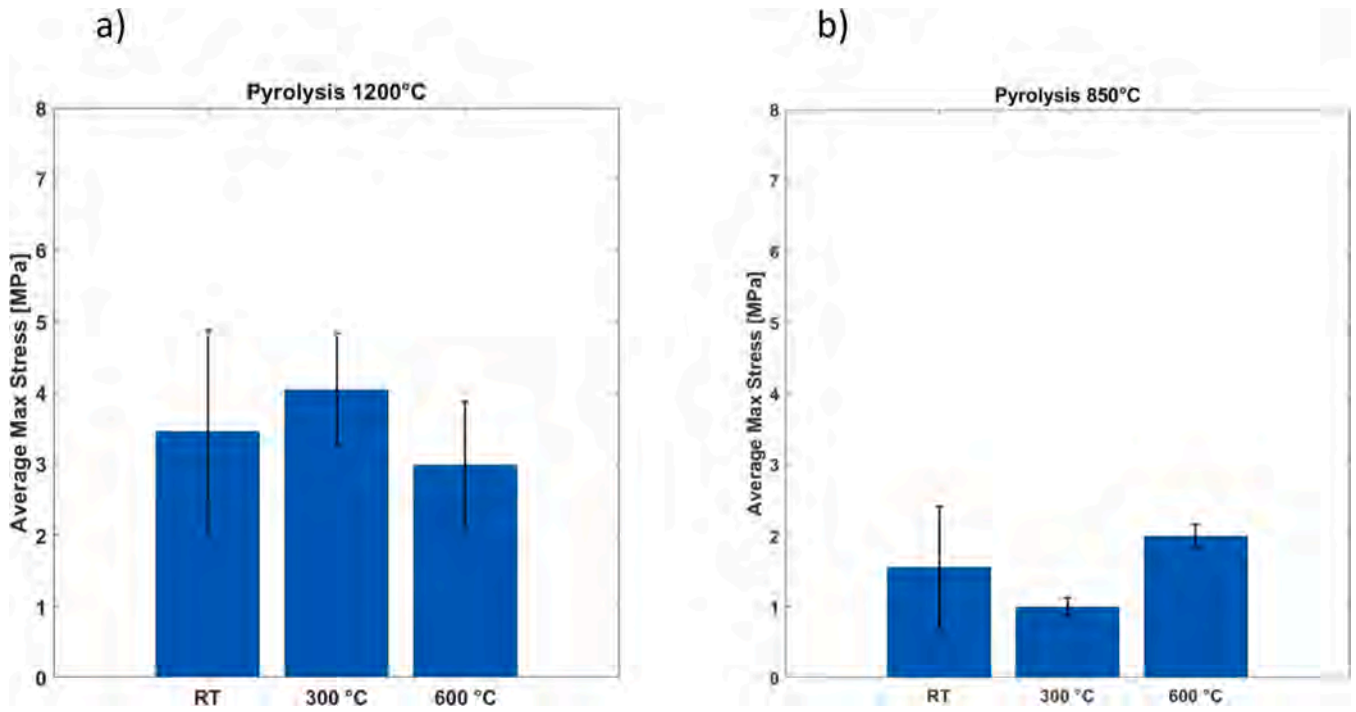


Fig. 6. Mechanical test results at different temperatures for joined samples with 70 wt% alumina particles, 1 wt% alumina fibers, after curing and pyrolysis at 1200°C (a) and after curing and pyrolysis at 850°C (b).

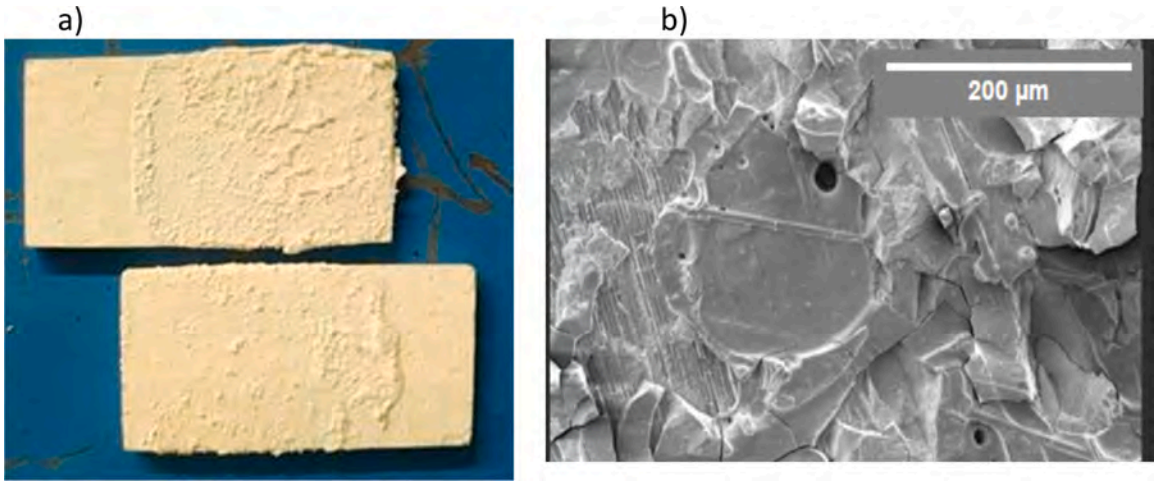


Fig. 7. Fracture surface after mechanical test at 600°C: visual appearance of the cohesive fracture (a) and SEM of the fracture surface (b): joined samples with 70 wt % alumina particles, 1 wt% alumina fibers, after curing and pyrolysis at 850°C.

covered by the joining material, while extended regions of the ox/ox composite remain exposed.

The combination of large voids and incomplete filling of the joined region reduces the real load-bearing area and promotes early crack initiation and propagation along the joining layer, thus explaining the relatively low lap-shear strength measured for the joints, despite the good chemical compatibility and adhesion between the joining material and the ox/ox composite.

To understand if the gaseous products released during curing and pyrolysis were somehow hindered in the sandwich configuration used for the joints, some coatings were prepared on ox/ox composites with Durazane 1800 loaded with 70 wt% alumina particles, 1 wt% alumina fibers, cured and pyrolyzed at 1200°C, with the same procedure used for the joined samples.

Fig. 8 shows representative top view (Fig. 8 a,b) and cross-section (Fig. 8 c,d) SEM pictures at different magnifications: a certain porosity is visible in Fig. 8 a,b, but alumina particles and fibers are very well embedded in the silica matrix. The cross-sections, Fig. 8 c,d show a fairly good adhesion of the coating to the ox/ox composites, with some cracks across the coating thickness, but apparently less porous than the joints (Fig. 5 e,f).

While qualitative information can be obtained from SEM cross-sections, two-dimensional observations on a single plane may underestimate the real void content and do not fully capture the three-dimensional morphology of the pore network. For this reason, the residual porosity of 70 wt% alumina loaded Durazane 1800 coatings was measured by X-ray computed tomography (CT) (no fibers were added in these coatings).

Representative CT slices of the three-dimensional reconstructions of the coating after curing and after pyrolysis are shown in Fig. 9 a,b: two

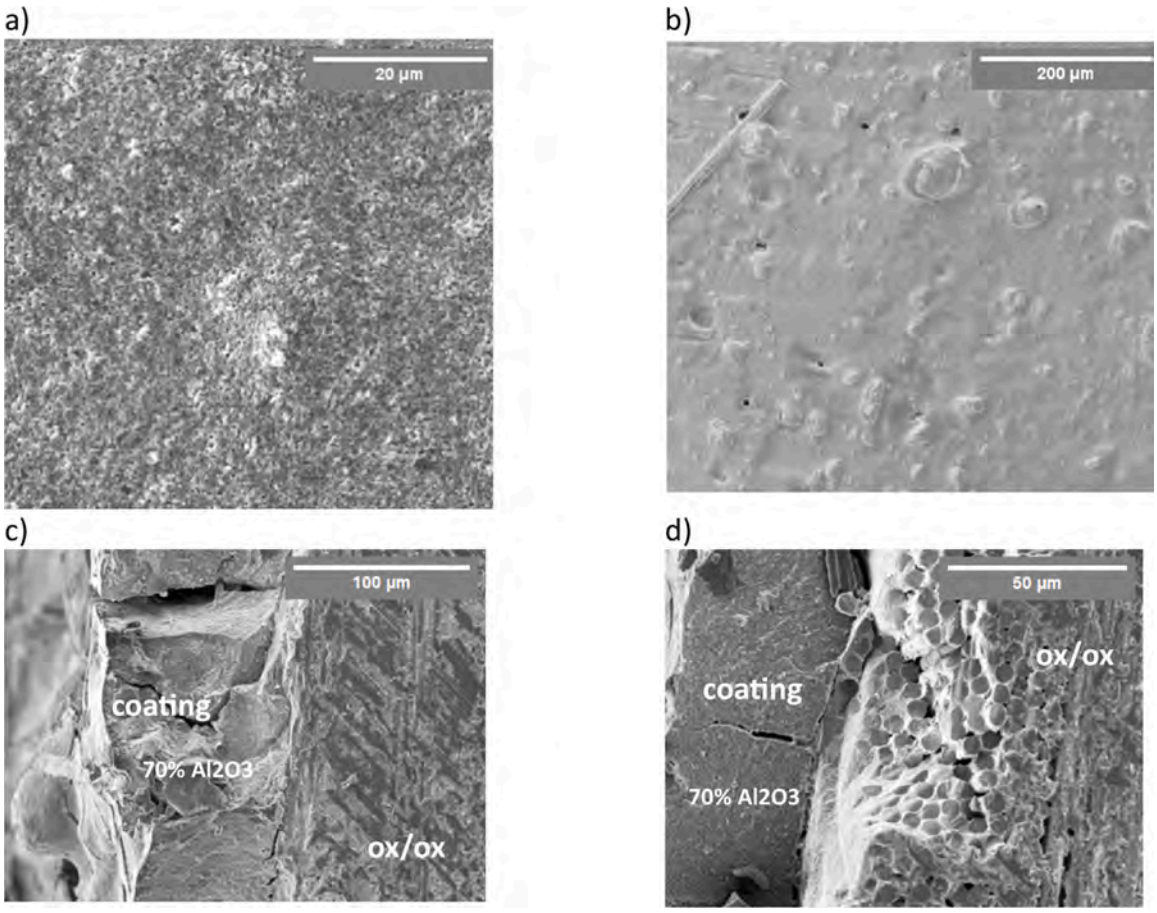


Fig. 8. Top view (a,b) and cross-sections (c,d) of coatings on ox/ox composites obtained by 70 wt% alumina loaded Durazane 1800 and 1 wt% alumina fibers, after curing followed by pyrolysis at 1200°C.

orthogonal sections taken at the mid-plane of the coating are displayed: a plan-view section parallel to the coating surface and a transverse section normal to it. Pores and cracks are already visible after curing and their size and density further increase after pyrolysis. The microstructure is clearly heterogenous and the distribution of alumina particles is far from optimal, with marked particle agglomeration likely due to a not optimized mixing process.

The corresponding maps obtained from CT segmentation are reported in Fig. 9 c, d: pores, cracks and lack of coating material (i.e. uncoated areas) detected in the specimens after curing are highlighted in blue, whereas after pyrolysis they are shown in green.

The total volume of pores, cracks and lack of coating material in the 70 wt% alumina loaded Durazane 1800 coatings was calculated as about 9 % after curing and 38 % after pyrolysis.

The combination of porosity, cracks, particle agglomeration and uncovered area in the joined region is responsible for severely reducing the effective load-bearing cross-section and introducing a dense network of stress concentrators in the joints.

Quantifying residual porosity, cracks, and uncoated regions after curing and pyrolysis by a non-destructive method such as CT-scan provides a basis for process optimization: ongoing work focuses on improving the mixing process and optimizing the filler size distribution.

4. Conclusion

This study investigated the feasibility of using a preceramic polymer-based joining material to join oxide fiber-reinforced oxide matrix (ox/ox) composites, presenting a unique way to obtain an ox/ox composite joining material to join ox/ox composites.

The approach involved a simple mixing and dispersion procedure using Durazane 1800, a commercial polysilazane, combined with alumina particles and fibers as fillers. The prepared slurry subsequently underwent ceramic conversion through curing and pyrolysis at 850 and 1200°C.

By varying the alumina particles' content, the quality of the joints was enhanced, with the primary objective being the determination of the optimal amount of particle filler material needed to achieve maximum possible strength.

Various joints with different mass fractions of particle fillers were produced, morphologically and compositionally analyzed, and mechanically tested.

The results showed that the maximum strength was achieved with a filler content of 70 wt% of alumina powders and 1 wt% of fibers and with pyrolysis at 1200°C; a lap shear strength of about 3-4 MPa was measured, remaining essentially constant from room temperature to 600°C.

X-ray computer tomography on coatings enabled quantification of residual porosity, cracks and uncovered areas after curing and pyrolysis, also revealing marked particle agglomeration and non-optimized packing, likely associated with the use of a monomodal particle size distribution. These microstructural features are identified as the main limitations to joint strength.

In conclusion, while the findings of this work provide valuable insights into the potential of preceramic polymer-based joining for oxide fiber-reinforced oxide matrix (ox/ox) composites, further research is needed to improve the joints' strength by reducing porosity within the joining layer.

Future work will focus on reducing residual porosity by optimizing

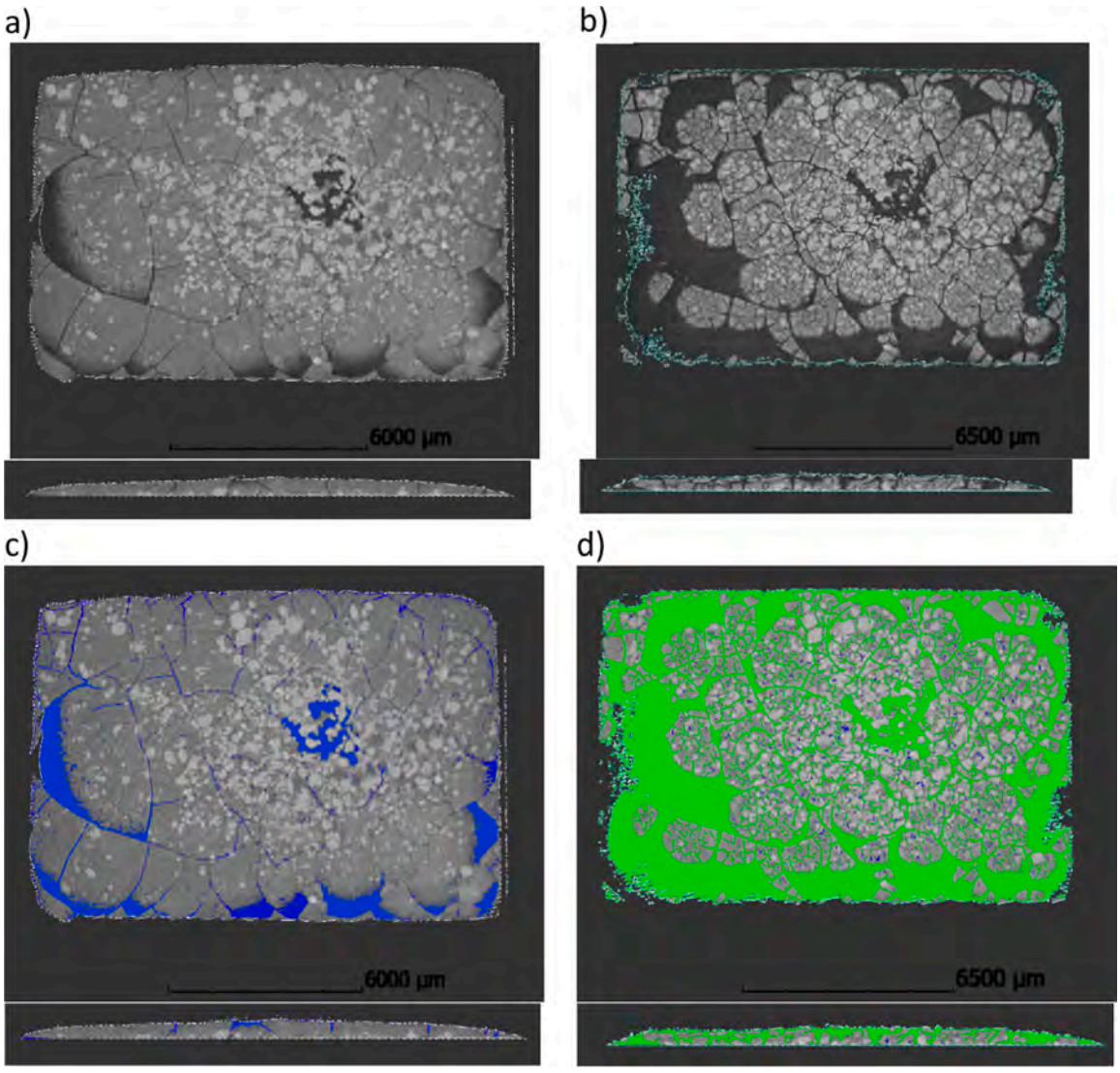


Fig. 9. Representative X-ray CT slices of Durazane 1800-based coatings with 70 wt% alumina after curing and after pyrolysis (a,b): plan-view section parallel to the coating surface and transverse section. Porosity maps obtained from CT segmentation (c,d): highlighted porosity, cracks and uncovered area after curing (blue), and after pyrolysis (green).

particle packing (e.g. by adopting bi- or multi-modal particle size distributions and tailored mixing procedures) and by controlling gas release during curing and pyrolysis, with the goal of significantly increasing the mechanical performance of the joints.

CRediT authorship contribution statement

Monica Ferraris: Writing – review & editing, Writing – original draft, Supervision, Funding acquisition, Conceptualization. **Aurora Pizzinat:** Data curation. **Alessandro Benelli:** Methodology, Investigation, Formal analysis, Data curation. **Stefan Schafföner:** Supervision. **Georg Puchas:** Writing – review & editing, Resources, Investigation, Data curation. **Kevin Nordengren:** Writing – review & editing, Resources, Conceptualization.

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.

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