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Laser-supported joining of SiC-fiber/SiCN ceramic matrix composites fabricated by precursor infiltration

Marion Herrmann[a,*], Katrin Schönfeld[b], Hagen Klemm[b], Wolfgang Lippmann[a], Antonio Hurtado[a], Alexander Michaelis[b]

[a] *Technische Universität Dresden, Institute of Power Engineering, Chair of Hydrogen and Nuclear Engineering, D-01062 Dresden, Germany*
[b] *Fraunhofer Institute for Ceramic Technologies and Systems (IKTS), D-01277 Dresden, Germany*

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Abstract

SiC-fiber/SiCN ceramic matrix composites were manufactured by means of polymer infiltration and pyrolysis. The fiber preform was made by slurry infiltration and winding using a computer-controlled winding module. Multiple infiltration steps using a Si–C–N precursor were included to increase the density. The influence of the sintering conditions on the microstructure of the CMC was demonstrated.

Pipe sections made of the CMC materials were joined using a laser-supported heating technology with an Y–Al–Si–O glass–ceramic filler. The thermal response of the CMC components was controlled by the anisotropic thermal conductivity. Fast heating by laser beam was achieved for elements rotating in the direction of the fiber winding. SEM micrographs of the joints showed the good wettability of the CMC by the glass–ceramic filler. Nearly defect-free joints were obtained using a nitrogen process atmosphere. The laser-supported technology was shown to be promising for the joining of CMC components.

Keywords: CMC; SiC fiber; PIP; Laser joining; Glass–ceramic filler

1. Introduction

Rising needs for high-temperature components in the energy industry cannot be met with conventional metallic material-based solutions alone. Ceramic matrix composites (CMCs) have a high potential for applications in ground-based and automotive gas turbine components such as combustors, liners, turbine vanes, and blades as well as aerospace engines and other industrial applications such as heat exchangers, hot gas filters, and radiant burners.[1]

CMCs can be divided into oxide and nonoxide composites. In oxygen-containing atmospheres at high temperatures, oxide composites exhibit high stability but inadequate mechanical properties (e.g., strength and creep resistance) for structural applications. In comparison, nonoxide composites are more susceptible to oxygen environments at elevated temperatures but exhibit considerably better mechanical properties at high temperatures.[2]

Nonoxide fiber composites based on SiC fibers were used in this study. CMCs can be manufactured using a variety of processes, including melt infiltration, chemical vapor infiltration, and polymer infiltration and pyrolysis (PIP).[3] Because of the low complexity of the required facilities and the low costs, the PIP process was used in this work.

However, the processes available only allow fiber-reinforced components of limited size and with simple geometries to be produced. Suitable joining technologies needed for fabrication of complexly shaped CMC components are currently being developed, with the main emphasis being placed on technologies using special materials for the joining of SiC_f/SiC as structural materials for fusion power reactors and for the Very High Temperature Reactors (VHTR) within the framework of

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* Corresponding author. Tel.: +49 351 463 32371; fax: +49 351 463 37161.
E-mail address: marion.herrmann@tu-dresden.de (M. Herrmann).

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the international Gen-IV program.[4,5] The joining materials for application at high temperatures can be divided into

- reactive materials,
- preceramic polymers, and
- glass–ceramic fillers.

Examples of reactive materials are technologies using solid-state displacement reactions for the formation of joints. For instance, mixtures of TiC and Si react to form an epitaxial layer of secondary SiC at the interface to the SiC matrix, thereby increasing the joint shear strength.[6,7]

Different authors described the application of preceramic polymers for the joining of SiC_f/SiC.[6,8,9] The intrinsic character of the joint was found to be advantageous; however, the pyrolysis process itself led to material shrinkage and gas formation. These effects could be partially reduced through the addition of inert filler materials.

Katoh et al.[10] and Ferraris et al.[11] proposed the use of glass–ceramic fillers for joining. They identified the advantages as being the possibility of tailoring the properties and the chemical resistance of the glass–ceramics. The most frequently investigated glass–ceramic filler systems for SiC materials were Y_2O_3–Al_2O_3–SiO_2,[12] MgO–Al_2O_3–SiO_2,[11,13] and CaO–Al_2O_3.[10,14,15] The joints exhibited superior thermo-mechanical stability when thermally stable crystalline phases were formed. The coefficients of thermal expansion (CTEs) of the crystalline phases should be adjusted to the CTE of the matrix material for a high joint quality to be achieved. The residual glass phase shows self-sealing behavior if the glass–ceramic is applied at temperatures higher than the glass transition temperature.[10] As a consequence of their high glass transition temperatures, glasses in the Al_2O_3–Y_2O_3–SiO_2 system play an important role among glass–ceramic fillers for the joining of SiC.[16] These compositions were also extensively investigated in connection with liquid phase sintering of the nonoxide ceramic materials SiC and Si_3N_4.[17,18]

NITE (nano-infiltration and transient eutectic) sintering technology was shown to be promising for joining CMCs.[19] This technology requires temperatures of at least 1700 °C and pressures between 10 MPa and 20 MPa. The composition of the joining zone (mix of Y_2O_3, Al_2O_3, SiO_2, and β-SiC nano-powder) was adjusted to be close to that of the matrix material.[20] The joints exhibited very high thermal and mechanical stabilities, especially under conditions of irradiation (investigated at ~6 dpa at 800 °C).[5] For this reason, the technology was recommended for joining of SiC_f/SiC components under neutron irradiation.

With all of these joining methods, the components are heated and joined, in some cases under high pressure, in a furnace. In contrast, the laser-supported technology enables heat input to be localized. Thus, the dimensions of the components to be joined are not restricted through the dimensions of the heating furnace.[21] Superior results in joining of monolithic SiC components with laser-supported heating were obtained in previous studies.[21–23] A glass–ceramic filler composition in the Y_2O_3–Al_2O_3–SiO_2 system was used for these experiments. Short processing times and localized heating of components were the main advantages of the laser brazing method. Furthermore, the high thermal conductivity and the low thermal expansion coefficient of SiC were found to be beneficial for diminishing temperature gradients and transients introduced by the laser energy input. In comparison with other technologies, the laser-supported joining technology proved to be very energy-efficient.

The goal of the current study was to develop a laser-supported technology for joining CMC components. This involved two different tasks:

- investigation of the thermal response of the CMC components to laser heating and
- testing of glass–ceramic fillers for joining of the CMC material.

Focus was on the development of materials with properties suitable for application under corrosive conditions at temperatures of above 1000 °C and the implementation of these advanced materials in components and functional prototypes for energy technology (e.g., heat pipes).

2. Materials and methods

2.1. CMC fabrication process

Polycrystalline SiC fibers of type “Tyranno SA3” (UBE Industries Ltd.) were used to produce the fiber preforms. After being desized at 800 °C the fiber roving was wound onto differently sized mandrels at a winding angle of 85° according to a customized winding program. During this winding process the roving was infiltrated with a ceramic slurry comprising a SiC or Si_3N_4 powder (SNE-3, SNE-10, UBE Industries), a binder, and a sintering aid. The powder loading in the slurry was 20 wt%.

After the preform was dried PIP was performed using the commercially available polysilazane Si–C–N precursor “HTT1800” (Clariant Advanced Material GmbH). Pyrolysis was subsequently performed at 900 °C in argon. Conversion from the liquid precursor to the amorphous SiCN was accompanied by shrinkage of the matrix, which led to formation of pores and cracks in the matrix. A defined fiber spacing was obtained through the presence of powder particles between the individual fibers introduced during slurry infiltration in the winding process. Several reinfiltration and pyrolysis steps were performed in order to minimize the porosity of the matrix. At least a sintering process was performed in a gas pressure sintering furnace between 1500 °C and 1700 °C in a nitrogen atmosphere. The preparation route used is shown in Fig. 1.

The components for the laser joining experiments underwent three infiltration/pyrolysis cycles and were sintered at 1700 °C. Two sample configurations were prepared. Fig. 2a shows pipe section samples of outer diameter 30 mm, length 20 mm, and wall thickness 1 mm for the heating and simple joining experiments. The softening and wetting behavior of the filler was investigated on these samples; the joints were realized as butt