

Novel oxide fiber composites by freeze casting

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Abstract

A freeze casting process with gelatine as gelling agent is presented to manufacture novel oxide fiber composites. The growth of ice crystals causes an in situ densification of the green matrix when the composite is frozen after the lamination of 2D-fabrics. This freeze casting process leads to a porous matrix with a cellular morphology, showing no shrinkage during sintering. The porous structure is suitable for a subsequent infiltration with a precursor to densify the matrix. As the matrix is crack-free, the sintered composites have high mechanical properties. The processing is demonstrated by manufacturing laminated NextelTM610/YAG-ZrO₂ oxide fiber composites.

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1. Introduction

Oxide fiber composites (OFC) are a promising material class for structural and functional applications in corrosive atmospheres at high temperatures. The high strength combined with damage tolerant fracture behavior, good thermal shock resistance and low density promise their use in future gas turbines.^{1,2}

The matrix design is a very important issue regarding the manufacturing of porous oxide fiber composites. The matrix decouples the fiber filaments and stress concentration after local fiber failure is reduced by crack deflection into the porous matrix. The porosity of this weak matrix is normally about 40 vol%. The matrix, which does not shrink during sintering, is realized with a network of coarse non-sintering particles. Mixed oxides like mullite or YAG with a sluggish sintering activity are advantageously.^{3,4} The particle size of the non-shrinking particle network is about 1 μm to guarantee a good packing density with respect to the fiber diameter of about 12 μm. Usually, this

network is consolidated with sinteractive nanoparticles or an additional infiltration with a precursor.¹

Aqueous freeze casting or aqueous gelation freezing processes are near-net shape techniques to produce bulk ceramics with different microstructures.⁵ To avoid drying cracks and uncontrolled drying shrinkage, special gelling agents or drying processes are necessary. Dependent on the parameters of the process like particle volume content of the suspension or the organic additives and freezing rate, the microstructure of the sintered parts can be totally dense or highly porous and structured. The porous morphology occurs due to the growth of ice crystals during the freezing process. The shape and dimension of the pores formed by ice-crystals can be adjusted in a wide range. Optionally, gelling agents like an inorganic sol⁶ or an organic additive like gelatine⁷ can be added to the slurry to modify the microstructure. Advantages of gelatine are that it is nontoxic and burns out without any residues in a subsequent heat treatment. If no gelling agent is added, the parts have to be freeze-dried.^{8,9} With the growth of the ice crystals during freezing an in situ densification of the green body and a reduced sintering shrinkage occur.^{10,11} This effect is used in the following process to design a non-shrinking matrix with a particle composition described above. A freeze casting process with gelatine is suitable, because the viscosity of the slurry has to be rather low for the infiltration and lamination of the fiber cloths and critical drying cracks

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are inhibited by the gelling agent gelatine as well as by freeze-drying. Gelling and evolution of the microstructure can be easily influenced by the selection of the Bloom-value of the gelatine. The goal is to design pores in the matrix, which are spherical and smaller in size than the fiber diameter. If the pores are spherical, critical stresses within the matrix can be reduced. A small pore size is adjusted to guarantee the mechanical decoupling of the fiber filaments.

In contrast to the known colloidal processing routes, the new matrix design derived from freeze casting with in situ densification due to the growth of ice-crystals during freezing enables a crack-free matrix. Low viscosity makes the slurry infiltration of large complex three-dimensional samples or a sandwich-concept with areas without fiber possible. In addition, requirements on the mold are low and the composite can be shaped by vacuum-bagging.

A YAG ($\text{Y}_3\text{Al}_5\text{O}_{12}$, yttrium aluminum garnet) matrix, stabilized with ZrO_2 is applied, because YAG and ZrO_2 are corrosion resistant materials in hot gas atmosphere.¹² Furthermore, a network of YAG particles can be stabilized with sintering necks of ZrO_2 at low sintering temperatures, the coefficient of thermal expansions of YAG ($8.0 \times 10^{-6} \text{ K}^{-1}$ for polycrystalline YAG with 5% porosity) and Al_2O_3 are similar¹³ and a system of Al_2O_3 , YAG and ZrO_2 is thermodynamically stable.¹⁴

2. Experimental

2.1. Matrix concept and materials

As fiber reinforcement NextelTM610 fabrics (DF11, 1500 den, 8 HS Satin, 3 M Corp., USA) are utilized. The matrix is designed of a network of $1 \mu\text{m}$ coarse YAG powder (mixed-oxide synthesis with Al_2O_3 and Y_2O_3 via solid-state reaction), which is consolidated with fine ZrO_2 -nanopowder (TZ-3YE, Tosoh Corp., Japan; supplied from Krahn Chemie GmbH, Germany) with a mean particle size of $0.1 \mu\text{m}$. The composition of coarse YAG to fine ZrO_2 powder is 97 to 3 by weight. The particle size of the coarse powder with a mean particle size of $1 \mu\text{m}$ and a maximum particle size of $5 \mu\text{m}$ is chosen because a high fiber volume content and matrix strengthening via infiltration with a precursor can be realized. Up to three infiltration cycles with ZrO_2 precursor (zirconium(IV) butoxide solution (80 wt% in 1-butanol, Alfa Aesar GmbH & Co KG, Germany)) are used to further densify the matrix.

2.2. Processing

The process shown in Fig. 1 starts with the preparation of a suspension containing organic additives such as a netting agent and a dispersant. As dispersant and netting agent 1 wt% polyacrylic acid (Sokalan PA15, BASF, Germany) is used. The particle loading is adjusted to 75 wt% or 40 vol%, respectively. The slurry with a viscosity of about 5 Pa s at a shear-rate of 1 s^{-1} is homogenized for about 12 h on a rolling machine with 3 mm ZrO_2 beads. Because the process implies a freeze-drying step, glycerol is added as cryoprotectant. The water solidification modifier glycerol helps to prevent the expansion of the water

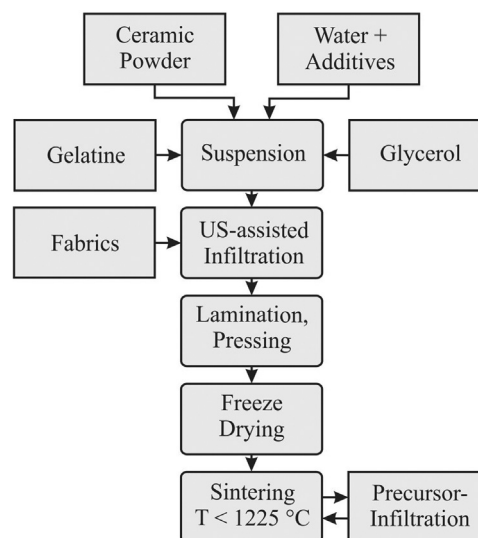


Fig. 1. Flowchart of the freeze casting process

during freezing and modifies the crystallization.¹⁴ An amount of 20 wt% of the water quantity is proofed to be suitable. Furthermore the crystallization of the water during freezing and the dimension of the ice-crystals are influenced by the addition of gelatine. An amount of 0.25 wt% gelatine (Gelita Novotec 100, 85 g Bloom, Gelita Deutschland GmbH, Germany) with respect to the slurry quantity is added to adjust the size of the ice-crystals or rather the pore size. With the addition of the gelatine, the green strength of the dried laminate is enhanced, too. The swollen gelatine can be dissolved in the suspension, which is heated up to 60°C . Then the slurry is cooled down to 40°C to avoid evaporation of water during the processing and put into a cuvette in an ultrasonic bath which is also tempered to 40°C and where the fabrics are infiltrated subsequently.

To adjust the fiber volume content to about 40 vol%, the laminated fabrics are pressed to a defined size between two plates with spacers. This moderate fiber volume content is chosen to avoid internal mechanical stresses in the composite which might cause delamination after drying. When 8 fabrics are used, the composite has a thickness of about 2 mm. The fabrics are arranged in a $0/90^\circ$ orientation. After cooling down to -70°C , the composite can be removed from the mold and freeze-dried (laboratory freeze drier ALPHA 1-4 LSC, Martin Christ, Germany). The removal of the mold after freezing is necessary to ensure the integrity of fiber and matrix and the demoulding leads to a high surface quality. The last step is the sintering process at 1225°C .

To densify the composite further, up to three vacuum infiltration cycles with zirconium-butoxide solution are performed. The composites are put into an exsiccator. After evacuation to 50 mbar the ZrO_2 precursor solution is added. For a complete precursor infiltration the composites stay about 1 h in the precursor-solution. Before sintering at 1225°C after each infiltration cycle, the infiltrated composites are stored for 12 h in air to hydrolyze the precursor. The yield of the precursor is about 25 wt%.

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