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Original Article

Crack healing of ferrosilicochromium-filled polymer-derived ceramic composites

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ABSTRACT

In this study, SiOC/FeSiCr/SiC-ceramics were derived by using polymethylsilsesquioxane as a polymeric precursor and FeSiCr (iron-chromium-silicide) and SiC powders as ceramic fillers in a polymer-extrusion process followed by pyrolytic conversion in a nitrogen atmosphere. The crack healing properties of the ceramic in an oxidising atmosphere were subsequently investigated. The SiOC/FeSiCr/SiC-ceramics showed crack closure and strength recovery behaviour after oxidation treatments in air from 600 to 1300 °C with various holding times from 2 to 1000 min. The crack healing mechanisms at different oxidation temperatures and with various dwell times are discussed.

1. Introduction

Advanced ceramics with superior mechanical strength and excellent temperature resistance are being used in a wide range of applications [1–3]. However, the brittle nature of ceramics, which makes ceramics sensitive to surface cracks, can limit their applications [4]. To overcome this limitation, ceramic materials with crack healing properties have been intensively studied in recent years [5–11]. Gupta [10] and Evans et al. [11] demonstrated the crack closure effect in oxide ceramics due to grain growth during "resintering" at temperatures above 1400 °C. Nakatani et al. [12] reported the crack healing effect in Si_3N_4 caused by surface oxidation in a temperature range between 700 and 1000 °C. Schlier et al. [5,13] have demonstrated crack filling and surface strengthening of polymer-derived ceramics (PDCs) in a nitrogen atmosphere at temperatures above 1000 °C. PDCs are typically silicon-based oxide or non-oxide ceramics, which are synthesized from polymeric precursors (preceramic polymers such as organo-silicon) [14–16]. Preceramic polymers can yield ceramics like SiC, Si_3N_4 or SiOC via cross-linking and pyrolysis in an inert atmosphere [5,17]. The synthesis and processing of PDCs has gained increasing interest due to various advantages compared with conventional ceramic processing routes [14,18]. The use of a ceramic filler combined with preceramic polymers offers the possibility of using plastic shaping technologies (e.g. extrusion) and near net shape manufacturing techniques [13,16]. Moreover, the low ceramic conversion temperatures of PDCs compared to traditional technical ceramics offers both cost and energy saving potentials. In this work, ferrosilicochromium-filled polymer-derived ceramics were fabricated via extrusion, cross-linking and pyrolysis in a nitrogen atmosphere at 1300 °C. The crack healing behaviour of these FeSiCr-based ceramics in air was investigated by mechanical testing and by microstructural analysis. The mechanisms of crack healing were discussed by using crystalline phase analysis and thermochemical calculations.

2. Experimental

The feedstocks for extrusion were composed of 50 vol.% preceramic polymer of polymethylsilsesquioxane ($[(CH_3)SiO_{1.5}]_n$ with n = 130–150; density: 1.1 g/cm^3; melting temperature: ~55 °C; cross-linking temperature: ~200 °C; Silres MK, Wacker Chemie AG, Burghausen, Germany), 34 vol.% FeSiCr powder (average particle size: 1.7 µm; density: 5.0 g/cm^3; Mineralmühle Leun Rau GmbH & Co. KG, Leun, Germany) and 16 vol.% SiC powder (SiC UF-05 green; average particle size: 1.4 µm; density: 3.2 g/cm^3; H.C. Starck GmbH, Selb, Germany). The powder blend was homogenized at room temperature in an intensive batch mixer (Laborator EL1, Gustav Eirich GmbH, Hardheim, Germany) for 2 min. by using a rotational speed of 2000 rpm. Continuous filaments with rectangular cross-section (3.5 × 3.4 mm^2) were extruded on a laboratory-scale twin-screw extruder (Rheomex, ThermoHaake, Karlsruhe, Germany) operating at an extrusion temperature of 140 °C with an extrusion velocity of 1 mm/s. After extrusion the filaments were cut to the desired length and then cross-linked in an autoclave (Dunze GmbH, Hamburg, Germany) at an air pressure of 1 MPa at 250 °C. Subsequently, the cross-linked filaments

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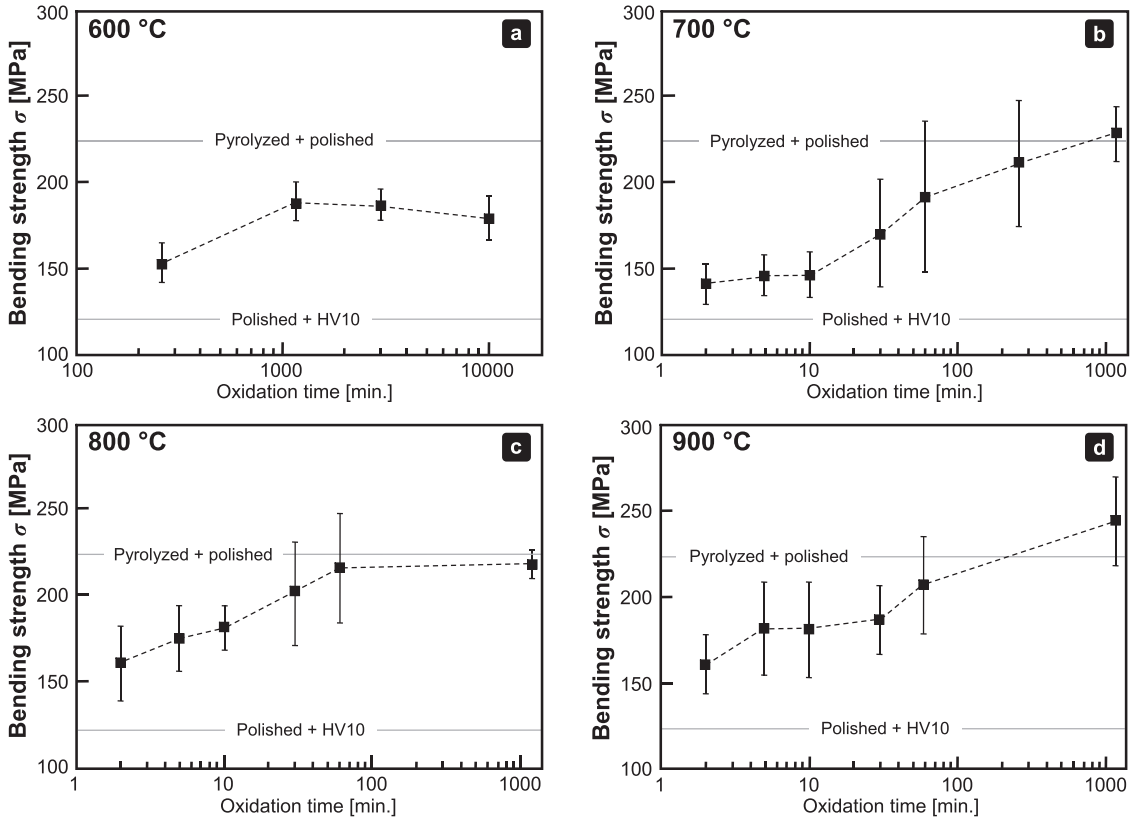

Fig. 1. Bending strength of pyrolyzed and polished SiOC/FeSiCr/SiC-samples after oxidation treatments at various temperatures (from 600 to 900 °C) and with different dwell times. Defects were introduced with a Vickers indenter prior to heat treatment.

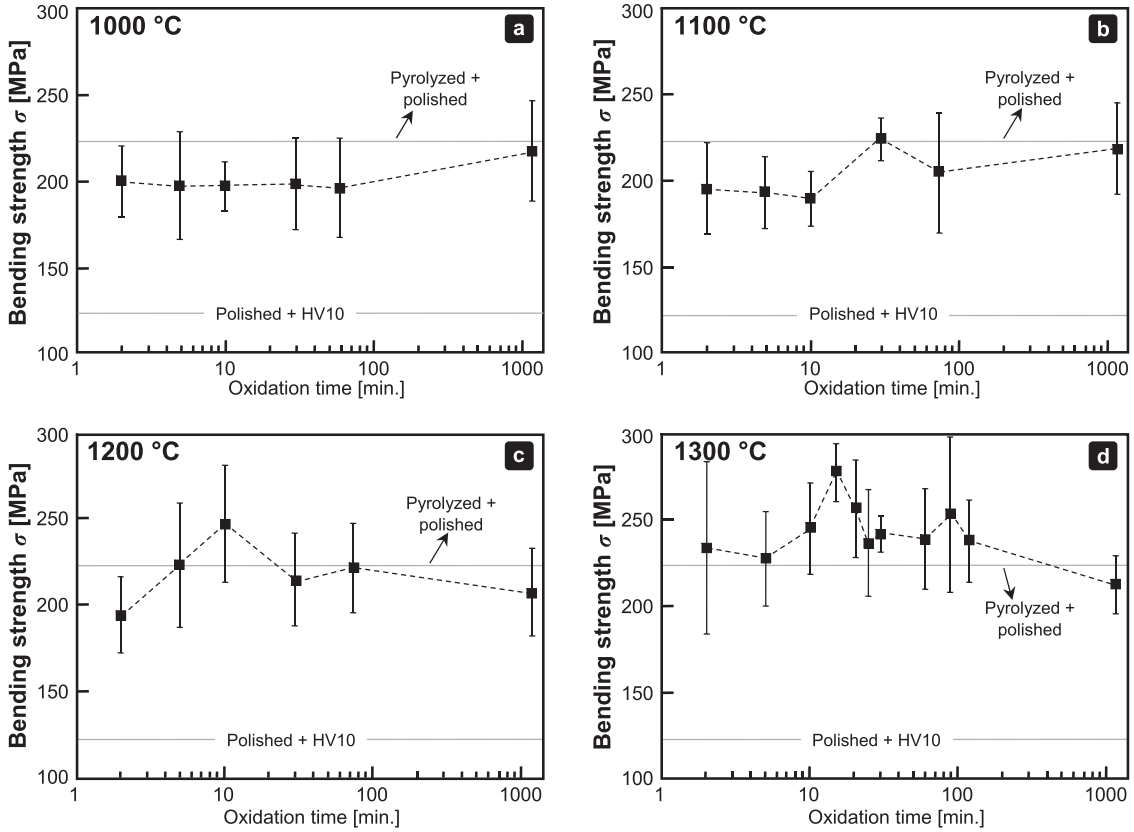

Fig. 2. Bending strength of pyrolyzed and polished SiOC/FeSiCr/SiC-samples after oxidation treatments at various temperatures (from 1000 to 1300 °C) and with different dwell times. Defects were introduced with a Vickers indenter prior to heat treatment.