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Effect of microstructural features and properties of constituents on the thermo-elastic properties of ceramic matrix composites

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ABSTRACT

It is proposed that the effect of microstructural features and constituents on thermo-elastic properties in ceramic matrix composites (CMCs) can be identified by using descriptive modeling tools. In this work, three CMC materials – N720/AS, SiC/SiNC and SiC/SiC-B_4C, were modeled and tested. A hybrid FEA code was used to calculate thermo-elastic properties as a function of porosity, constituent volume fraction, and fiber coating thickness. Model inputs were obtained from properties of as-manufactured composite constituents (when possible), literature values of constituent materials, and quantitative microscopy. The results illustrate the sensitivity of the composites' physical properties to the microstructural features of interest and demonstrate a way of using modeling tools when critical input values are not available.

1. Introduction

Ceramic matrix composites (CMCs) are light-weight structural materials that perform well at and above 1000 °C, which make them candidate materials for hot-section components and thermal protection systems in next generation turbine engines and space-access vehicles. CMCs are manufactured with iterative, and operator intensive, processes that lead to a complex microstructure with variability in spatial distribution of constituent materials, processing defects, and behavior. A major impediment to application of CMCs is the limited knowledge base regarding relationships between processing variables, microstructure, and properties as well as a lack of physics based descriptive models to accurately predict behavior and life.

Many models have been developed to calculate the mechanical and thermal properties of composites utilizing analytical, numerical, or statistical approaches (see for example, Refs. [51,56,58,14,25,61,31,22,24]). The most desirable modeling tools are numerically efficient, and include the physical and chemical attributes of the materials being studied. A significant challenge to accurately model CMC materials is that the processes and constituent formulations are not only difficult to characterize in the form that develops within the composite during manufacture, but their properties are also considered proprietary to the manufacturer and typically not published. The approach used in this work is to utilize property input values from literature when possible and to use ranges of possible values for key input properties in order to observe the changes in composite properties using a combination of physics-based modeling tools and a hybrid Finite Element Analysis (FEA) technique.

This work demonstrates the effects of microstructural features and constituent properties on thermo-elastic behavior for three different CMC material systems. The materials studied are manufactured with widely different constituents, processing techniques, and microstructures and include: 1) an oxide-oxide material (N720/AS) densified using sol-gel techniques [13,28,32]. 2) a silicon-carbide reinforced system (SiC/SiNC) densified using autoclave and polymer infiltration and pyrolysis (PIP) process [60,4,12]; and 3) a silicon-carbide reinforced system (SiC/SiC-B_4C) densified using constituent layering via chemical vapor infiltration (CVI) process [52,53,54]. The FEA code pcGINA© [22] was used to calculate elastic and thermal properties using ranges of values for key microstructural parameters including porosity, constituent volume fraction, and fiber coating thickness. Experiments were performed to measure the elastic and thermal properties for each composite and to compare collected data to calculated values. A sensitivity analysis was carried out to assess the impact of total porosity, percent of alumina in the N720/AS composite, effect of the fiber-matrix interface/interphase and fiber coating thickness on the SiC based materials. Results illustrate the sensitivity of the composite thermo-elastic behavior to the microstructural features of interest and

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demonstrate a way to utilize available data and modeling tools when critical input values are not available.

2. Material description

Three CMC composite systems, comprised of different constituent materials and made by different densification processes, were investigated. Those studied include an oxide/oxide, a SiC/SiNC, and a SiC/SiC-B_4C composite. Materials and manufacturing processes are described in the ensuing sections.

2.1. Oxide-oxide (N720/AS)

The oxide-oxide material is N720/AS, which is comprised of woven Nextel™ 720 (85% Al_2O_3 – 15% SiO_2) fiber preforms in a sol-gel matrix of alumino-silicate. Oxide based composites are a lower-cost family of CMCs that do not require expensive fiber interphase coating systems for crack deflection but rely on nano-porous matrices and weak fiber-matrix bonding to impart toughness. The N720/AS material studied was manufactured by COI Ceramics, Inc. (COIC)[1] and made from fabrics of fiber tows woven in a balanced, 8-harness satin weave pattern, with 27 ends per inch (400 filaments per tow). The composite was constructed using 12 plies with a $[0/90]_{3s}$ layup for a total composite thickness of approximately 2.54 mm and a fiber volume fraction of 45%. Optical images that provide examples of this N720/AS material microstructure are shown in Fig. 1.

The alumino-silicate matrix is introduced via a sol-gel technique in which a solution containing a mixture of fine Al_2O_3 powder and a SiO_2-forming polymer is infiltrated into the fabric. The wet fabric layers are stacked and placed in a vacuum bag and autoclave cured under low pressure and a temperature of around 150 °C. Once cured, the part is then placed in a high temperature furnace and the matrix is converted to ceramic. This alumino-silicate matrix is sintered at ~1200 °C [28]. During heating, joints between matrix particles are formed providing stiffness and strength [65] while at the same time extensive microporosity and matrix shrinkage cracks are formed [40] as seen in Fig. 1.

Some of the key parameters that describe this material and drive its thermo-elastic behavior include the fiber volume fraction as well as the amount of matrix porosity and shrinkage cracks. The amount of micro-level porosity in the matrix, which includes the pores and cracks within the fiber tows and within the matrix rich regions, is expected to affect both the thermal and elastic properties. The constituent ratio of alumina to silica in the matrix is also expected to influence the composite properties.

2.2. SiC/SiNC (S200H)

The SiC/SiNC composite studied was manufactured by COIC, under the trade name S200H. This composite is comprised of woven fabrics of Hi-Nicalon™ SiC fiber in an amorphous matrix of silicon, nitrogen, carbon. The matrix also contains a Si_3N_4 filler. Like all silicon fiber based CMCs, this material requires a fiber-matrix interface layer to promote crack deflection and protection from the environment. The S200H has a dual layer fiber coating system, with BN adjacent to the fiber for debonding and Si_3N_4 as the outer layer to protect the BN during processing. This composite was constructed using 8 plies of a balanced fabric, with an 8 harness satin weave pattern, that contained 24 ends per inch (500 filaments per tow) formed in a $[0/90]_{2s}$ layup. The total composite thickness was 3.17 mm, the fiber volume fraction was approximately 42%, and the volume fraction of open porosity was on average around 2%. Examples of the SiC/SiNC material microstructure are shown in Fig. 2.

S200H is manufactured using a polymer infiltration and pyrolysis (PIP) process. During this process a pre-ceramic polymer containing a filler is first prepregged into the fabric by hand. The fabric is then stacked and warm molded in an autoclave. The densified part is then put in an oven to cross-link and pyrolize the polymer. The pyrolysis causes an organic-inorganic transition forming a ceramic matrix around the fibers [4]. The polymer volume is significantly reduced in this process forming extensive cracks as seen in Fig. 2. Multiple infiltration and pyrolysis steps using a filler-free polymer are then performed to reach a target densification level for the composite that is less than 5% open porosity.

Some of the key microstructural features that describe this material and drive its thermo-elastic behavior include the fiber volume fraction, the amount of matrix porosity and shrinkage cracks, amount and type of filler used, and the thickness of the fiber-matrix interface coating system. The volume of matrix porosity can influence the thermal conductivity and stiffness of the material. The fiber coating system serves to protect the fibers during processing and allows cracks to deflect along the fiber direction instead of propagating through them (the source of the so-called graceful failure). Total thickness of the fiber-matrix interface/interphase coating systems will affect these functions.

2.3. SiC/SiC-B_4C (SiC/HYPR-SiC)

The SiC/SiC-B_4C composite was manufactured by Hyper-Therm High Temperature Composites Inc.,[2] under the trade name HYPR-SiC. This composite is reinforced with Hi-Nicalon™ SiC fibers that are coated via chemical vapor infiltration (CVI) with pyrocarbon (PyC). Nine plies of balanced plain-weave pattern with 16 ends per inch (500 filaments per tow) were used and resulted in a composite panel that was 2.8 mm thick and had a fiber volume fraction of 34.1%.

The matrix is comprised of alternating layers of SiC and B_4C also introduced via CVI. The basic idea behind the layered matrix system is to have a material with high temperature stability and stiffness such as SiC, and a sacrificial material that forms a viscous glass when oxidized such as boron and boron containing compounds. Boria glass formation allows for crack bifurcation and can flow to plug cracks limiting the accessibility of oxygen and other environmental gases to the fibers. In the material under study, alternating layers of SiC and B_4C were applied with a 5:1 thickness ratio along with a very thin layer of fugitive carbon to prevent chemical bonding between the layers [53,54]. The thickness of the layers is increased with each successive CVI run. Examples of the SiC/SiC-B_4C material microstructure are shown in Fig. 3.

Some of the key microstructural features that describe this material and drive its thermo-elastic behavior include the fiber volume fraction, the amount of matrix porosity, and the thickness of the fiber coating system. The amount of matrix porosity in the material can influence the thermal conductivity and stiffness. The CVI process creates larger type voids when the small inlets for the reactive gases get canned off. This can create large voids between fabric plies and can affect through thickness properties. Often these large voids are located near the center of the panels and have very sharp corners that serve as crack initiation sites. The fiber-matrix interface coating thickness is also considered a key parameter of the microstructure that can similarly impact thermo-elastic and high-temperature behavior like in the case of the SiC/SiNC material.

3. Experimental methods

The elastic and thermal properties were experimentally evaluated for the three composite systems by the University of Dayton Research

[1] COI Ceramics, Inc. is an ATK Space affiliate, 9617 Distribution Avenue, San Diego, CA 92121.

[2] Hyper-Therm High-Temperature Composites, Inc., now Rolls-Royce High-Temperature Composites, is a wholly-owned subsidiary of Rolls-Royce North America, 18411 Gothard St., Units B & C, Huntington Beach, CA 92648.