Processing Ceramic-Matrix Composites Using Electrophoretic Deposition

A.R. Boccaccini and C.B. Ponton

A novel, cost-effective processing technique for manufacturing ceramic-matrix composites (CMCs) containing two-dimensional woven-fiber reinforcement has been developed. The technique relies on the electrophoretic deposition (EPD) of ceramic sols onto the fiber preforms to achieve the required impregnation. The laboratory-scale results achieved thus far indicate that the processing approach offers great potential for the manufacture of high-quality, composite products with dual-component oxide matrices for high-temperature structural applications.

INTRODUCTION

Based on a variety of performance advantages (e.g., high-temperature performance and light weight), considerable research effort is being applied to the development of ceramic-matrix composites (CMCs) containing two-dimensional woven-fiber reinforcement, with particular emphasis on the establishment of reliable and cost-effective fabrication procedures. Traditional ceramic processing inherently lacks a clear approach for controlling microstructural heterogeneities, and, thereby, achieving microstructural uniformity. Property variability and a lack of engineering reliability are the result of uncontrolled microstructures. Ceramic composites containing two- or three-dimensional (2-D and 3-D, respectively) fiber reinforcements are particularly prone to exhibiting uncontrolled microstructures, but their demonstrated improved intra- and interlaminar strengths offset this problem, which arises because it is extremely difficult to achieve complete infiltration of the matrix material into the fiber tows (where the openings are on the order of ≤100 nm). Hence, new processing approaches and novel manufacturing techniques using nanoscale ceramic particles in a nonagglomerated form are required.

A novel and simple method for achieving complete infiltration of tightly woven fiber preforms, which are candidate 2-D reinforcements for CMCs, is based on the electrophoretic deposition (EPD) of colloidal sols into the fiber preforms. The principles of the EPD process are described in the sidebar.

The complete flow diagram for a manufacturing route leading to SiC-Nicalon woven-fiber-mat-reinforced mullite-matrix composites is shown in Figure 1. In this article, only the processing steps prior to the firing stage are described fully. The densification of the prepregs via pressureless sintering or hot-pressing is the focus of ongoing investigations.
A silicon-carbide fiber preform type (Nicalon 607C) is available commercially from Nippon Carbon Company, Japan. It has a residual carbon surface layer (~100 nm thick) resulting from its manufacture, providing the fiber with sufficient electrical conductivity to be used as the deposition electrode during EPD. The fiber architecture considered is a 2-D plain-woven multifilament fiber. A plain weave is the tightest of the weaves available, making it the most difficult to infiltrate, and it is readily available in prewoven form from the manufacturer.

Different silica and alumina precursors, in the form of sols or fumed fine powders, have been tried in order to find the optimum starting materials for the complete impregnation of Nicalon fiber mats using EPD. NaOH-stabilized silica sols were not selected, since they are prone to crystallization and cristobalite formation at relatively low temperatures, which would prevent full densification of the mullite matrix as shown in previous studies. The easy availability, low cost, and environmental safety of the materials has also been taken into consideration during their selection. On the basis of these requirements, organometallic compounds were not considered.

The starting materials finally selected were:
- Fumed amorphous silica (Aerosil OX50 and Aerosil 200, Degussa Limited, United Kingdom). Aerosil OX50 has a broad particle-size distribution (10–100 nm) and an average particle size of 40 nm. Aerosil 200 has a narrow particle size distribution and an average particle size of 12 nm. The pH in water solution ranges from 3.8 to 4.8.
- Boehmite (γ-AlOOH) sol (Remal A20, Remet Corporation, United States), containing 20 wt.% solid. The boehmite particles have a mean particle size of 50 nm and a fibrillar morphology. The pH of the as-received sol is 4.

The precursors were mixed in proportion such that the resulting ceramic after firing would be stoichiometric mullite (i.e., 72 wt.% alumina, 28 wt.% silica). After the slow addition of the fumed silica to the boehmite sol (with continuous-magnetic stirring of the solution during 12 hours), the pH of the sol remained essentially unchanged at pH ~4.4. The relative spatial arrangement of the alumina and silica particles within the sol and the degree of mixing were investigated by transmission electron microscopy (TEM) since they are critical factors for obtaining dense mullite at relatively low temperatures (~1,300°C). As shown in Figure 2, the boehmite particles (fibrillar morphology) and the silica particles (spherical) are mixed intimately on a nanometer scale.