

# Ceramic composites derived from poly(phenylsilsesquioxane)/Al<sub>2</sub>O<sub>3</sub>/Nb

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## Abstract

Active filler controlled reaction pyrolysis of polymers (AFCOP) is a recent method for obtaining near-net shaped ceramic bodies from a mixture of polymers, reactive filler and inert filler. Samples containing 60 wt.% of poly(phenylsilsesquioxane) (PPS) and 40 wt.% of metallic niobium and alumina powders were homogenized, uniaxially warm pressed and subsequently pyrolyzed in flowing argon at 1200, 1400 and 1600 °C. Ceramic bodies were characterized by X-ray diffraction (XRD), thermogravimetry (TGA), differential thermal analysis (DTA), fourier transform infrared (FTIR), porosity measurements and scanning electron microscopy (SEM).

The results indicated that the AFCOP process is potentially very suitable for obtaining multiphase composite materials of the Al<sub>2</sub>O<sub>3</sub>–Nb system at low temperatures.

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## 1. Introduction

Investigations have been made into alumina-based composite materials with the purpose of finding technological alternatives to cemented carbides [1–3]. Today, WC–Co material is still the most commonly used material for cutting tool applications owing to its great hardness, flexural strength and fracture toughness. The addition of hard refractory particles, such as TiC, WC or NbC reduces alumina grain growth (pinning effect), thereby improving the mechanical performance of these composite materials [4–6]. The presence of these particles offers also the additional advantage to retard the sintering process [1,2,7]. The densification of such composites requires either pressure-assisted sintering or high sintering temperatures [8,9].

Manufacturing of composite materials by active filler controlled polymer pyrolysis reaction is a recent process developed because it allows for attractive shaping properties and low sintering temperatures [10–12]. Recently, poly(siloxanes) have received special attention as precursors for ceramics and special silicon carbide [13–14], and com-

posites derived from Nb/Al<sub>2</sub>O<sub>3</sub> filled poly(methylsiloxane) have already been investigated [15]. The formation of NbC gives rise to a material with good hardness values, but the low carbon content of polymer does not prevent niobium oxidation [15].

The objective of this work was to study the formation of reaction bonded niobium carbide ceramic composite from Nb/Al<sub>2</sub>O<sub>3</sub> powder mixtures filled in poly(phenylsilsesquioxane) resin with high carbon content.

The resulting ceramic composites were characterized by thermogravimetry (TGA) and differential thermal analysis (DTA), fourier transform infrared (FTIR), X-ray diffraction (XRD), scanning electron microscopy (SEM) and porosity measurements.

## 2. Experimental procedure

The starting powders consisted of Nb (Chemical Engineering University of Lorena-SP, Brazil), D50 = 16 μm and α-Alumina powder (CT 2000, SG, Alcoa), D50 = 0.1 μm as fillers and a commercial poly(phenylsilsesquioxane) (PSS) (Waecker Silicone AG, Germany) with a carbon content of 56 wt.%. The polymer/filler ratio was set at 60 wt.%

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Table 1  
Composition of the composite materials investigated (wt.%)

Ceramic composites	PSS (%)	Nb (%)	Al <sub>2</sub> O <sub>3</sub> (%)
CMC 1	60	40	0
CMC 2	60	15	25

polymer and 40% filler (40 wt.% Nb-CMC 1 and 15 wt.% Nb + 25 wt.% Al<sub>2</sub>O<sub>3</sub>-CMC 2). Table 1 lists the compositions used in this work. The polymer and fillers were mixed with a solvent (acetone) and a curing catalyst (0.1 wt.% aluminum acetylacetonate). The solvent was removed in a rotary evaporator (Rotavapor) and dried at 120 °C. Rectangular specimens of 50 mm × 50 mm × 3 mm were warm pressed at 200 °C at a pressure of 1 MPa. Pyrolysis was carried out under argon flow (100 mL/min) at temperatures between 1200 and 1600 °C, at 5 °C/min during 1 h.

The green compacts as well the pyrolysed samples were characterized by means of some analytical techniques as infrared spectrum (IR), thermogravimetry, differential thermal analysis, X-ray diffraction, scanning electron microscopy, flexural strength and porosity measurements. The fourier transform infrared (FTIR) spectra transmission technique with KBr discs was applied, using a Bomem B100 Spectrometer operating from 4000 and 400 cm<sup>-1</sup> with a resolution of 4 cm<sup>-1</sup>. TGA (Dupont 2100 equipment) and DTA (TA Instruments) analyses were performed at a heating rate of 10 °C/min in argon flux, in a temperature range of 30–1500 °C. X-ray diffraction analysis was performed in an X-ray spectrometer (Diffrac 500, Siemens) using monochromated Cu K $\alpha$  radiation to identify the formation of new crystalline phases. Microstructure of the pyrolyzed material was analyzed by SEM (Stereoscan MK II, Cambridge Instr., Cambridge, GB). Porosity was measured by Hg porosimetry (Porosimeter 2000, Carlo Erba Instruments). The fracture strength was determined by four-point bending, using an Instron Universal testing machine with a crosshead speed of 0.1 mm/min.

### 3. Results and discussion

The curves obtained from the TGA analysis of the pure poly(phenylsilsesquioxane) and CMC 1 and CMC 2 samples are illustrated in Fig. 1. The poly(phenylsilsesquioxane) showed a multi-step weight loss process during pyrolysis. The first stage showed a  $T_{\max}$  at 250 °C, which is related to the condensation products [10,11]. The second step ( $T_{\max}$  = 450 °C) is less accentuated than the first one and not completely distinct from the third degradation process ( $T_{\max}$  at 600 °C). These processes involve the organic to inorganic conversion, leading to the formation of silicon oxycarbide glasses, with general formula of SiC<sub>x</sub>O<sub>y</sub> [10,16]. From 800 °C up to 1500 °C the weight of the samples remained unchanged. After 1350 °C, the CMC 1 and CMC 2 samples, displayed a new weight loss associated to the carbothermic re-

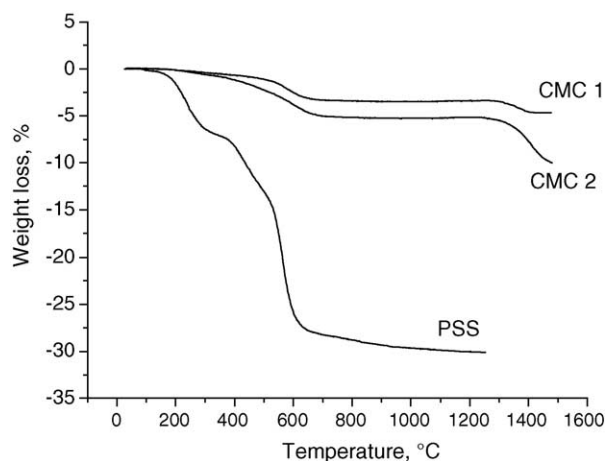


Fig. 1. TGA curves of pure poly(phenylsilsesquioxane) (PSS), CMC 1 and CMC 2 in an argon atmosphere.

duction of SiO<sub>4</sub> sites [11]. The thermal behavior observed led to a high ceramic yield of the ceramic composite at 1000 °C (93–97 wt.%), rendering this polymer an appropriate precursor for use in CMC technology.

The DTA curves obtained for the green compacts are shown in Fig. 2. The composite materials displayed an exothermic peak at around 500–600 °C, resulting from the reactions among the polymer degradation products and the niobium powder, which led to the formation of NbC, Nb<sub>3</sub>Si, Nb<sub>5</sub>Si<sub>3</sub> [15,16]. Mineralization of the polymer also occurs in this range of temperatures, forming an amorphous silicon oxycarbide matrix [10]. A study published in the literature demonstrates that, in this step, the polymer material presents a random distribution of SiO<sub>4</sub>, SiO<sub>3</sub>C, SiO<sub>2</sub>C<sub>2</sub>, SiOC<sub>3</sub> and SiC<sub>4</sub> [10,11]. The CMC 2 material (Fig. 2) also reveals an exothermic peak at around 1400 °C, which was attributed to the reaction of alumina with the silicon oxycarbide matrix and crystallization of the mullite phase.

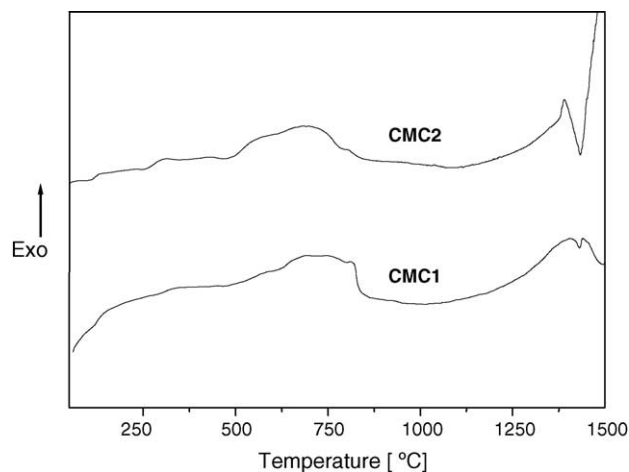


Fig. 2. DTA curves of pure poly(phenylsilsesquioxane) (PSS), CMC 1 and CMC 2 in an argon atmosphere.

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