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Synthesis and characterization of moldable electric conductive ceramic matrix composites

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Abstract – A polysiloxane network prepared *in situ* from poly(methylsiloxane), PMS, and 1,3,5,7-tetravinyl,1,3,5,7-tetramethylcyclotetrasiloxane, D_4V, by hydrosilylation reaction catalyzed by Pt and filled with graphite or tungsten silicide powders, gave rise to electrical conductive ceramic bodies, by controlled pyrolysis at 950, 1300 and 1500°C. A high ceramic yielding at 1000ºC was observed for both materials. For graphite-containing composites, the high ceramic yielding, of 96%, suggests the trapping of organic volatile released during the pyrolysis process, in the graphite layers. The electrical conductivity values for the composites are in the range of electrical conductors, although these values decrease with the increase in the pyrolysis temperature.

Ceramic matrix composites (CMC) have been successfully synthesized by controlled pyrolysis of polymeric green bodies, which can be prepared in different shapes. The possibility of filling the polymeric bodies with inert or reactive fillers can promote different properties for the resulting CMC, such as electric conductivity. In this study, silicone network was obtained by Pt-catalyzed hydrosilylation reaction between 1,3,5,7-tetravinyl-1,3,5,7-tetramethylcyclotetrasiloxane, D_4Vi, and poly(methylhydrogensiloxane), PMHS, using graphite or tungsten silicide powders, 50 and 80 wt%, respectively, as fillers. The green bodies were pyrolysed under argon atmosphere in a tubular furnace, at 950, 1300 and 1500ºC, with heating rate of 2ºC/min.

The ceramic yielding at 1000ºC, determined by thermogravimetry, is 96 and 98%, for GCM (filled with graphite) and WCM (filled with WSi_2) composites, respectively. X-ray diffraction, XRD, shows a shift in the graphite [002] plane from 26,6 to 25,5º (2θ) for GCM composites. Raman spectra obtained present a strong fluorescence for the sample pyrolysed at 950ºC, probably related to trapped organic radicals into ceramic matrix layers. On the other hand, the samples pyrolysed at 1300 and 1500ºC exhibit well-defined D and G bands at 1317 and 1595 cm^{-1} respectively, typical of carbon-based materials. ^{13}C HPDEC nuclear magnetic resonance, NMR, for GCM sample obtained at 950ºC, shows a broad signal centered at ~115 ppm attributed to Csp^2 and a peak at ~69 ppm attributed to Csp^3 from silicon oxycarbide matrix.

The WCM composite prepared at 950ºC presents diffractions peaks typical of the WSi_2 filler. However, samples pyrolysed at 1300 and 1500ºC show only diffractions peaks typical of to WSi_2, nevertheless, the ^{13}C NMR of WCM composite obtained at 950ºC shows a strong signal at ~300 ppm, suggesting an early formation of high electrical conductor tungsten carbide, which was not detected by XRD, probably in amorphous phase. The electrical conductivity of the WCM composites show values from 219 to 175 $S.cm^{-1}$, with the increase of the pyrolysis temperature. For the GCM composites, the electrical conductivity varied from the 9 to 7 $S.cm^{-1}$, with the increase of the pyrolysis temperature.