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Synthesis and Characterization of HfC/SiC Ceramic Nanoparticles

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Hafnium carbide (HfC) has been investigated in the last ten years, due to its great potential in applications of ultra-high temperature, because has unique properties such as high melting point (3900°C), high absorbance values, surficial area, good thermomechanical and thermochemical properties. Hence, it is a promising material for tools, rocket nozzles, aircraft, spaceships and transmitters [1, 2]. Feng et al. [3] synthesized HfC/SiC nanocomposites via spark plasma sintering (R-SPS) of HfC and $HfSi_2$ nanomaterials, using high energy ball mill during 2h in dry conditions, which were densified at 1750°C-1850°C by SPS during 10 minutes under vacuum axial pression of 40 MPa, obtaining a homogeneous microstructure.

The reagents used to synthesize the HfC precursor, were: hafnium tetrachloride (HfCl4, 98%), pectin ($C_6H_{10}O_7$, 74%) and distilled water as solvent. The reagents used to synthesize the SiC precursor, were: calcined SBA-15, sucrose ($C_{12}H_{22}O_{11}$, 99.5%), distilled water as solvent and sulfuric acid (H_2SO_4) as catalyst. The synthesis of HfC/SiC composite was realized in 3 steps: i) $HfCl_4$ and pectin were mixed through constant agitation at 40°C for 3h using sol-gel process. Next a drying is realized at 110°C for 24h obtaining the HfC precursor powder; ii) SBA-15, sucrose and H_2SO_4 were mixed and dried at 100°C for 6h, next the temperature is raised at 160°C under Argon atmosphere for 3h, obtaining the SiC precursor powder; iii) The HfC and SiC precursor powders, obtained to low temperature, were milled and mixed in a agate mortar (70% wt HfC and 30% wt SiC), next a heat treatment was carried out at 1600°C under Argon atmosphere for 3h, obtaining the HfC/SiC composite.

The phases composition of the samples was investigated by XRD, with the results shown in Figure 1. Characteristic peaks of crystalline HfC, at 2θ=33.5° (111), 38.8° (200), 56.1° (220), 66.8° (311) and 70.2° (222) and the peaks corresponding to β-SiC, at 2θ=35.5° (111), 59.9° (220), and 71.7° (311) were observed for the sample synthesized. It should be noted that characteristic peaks of m-HfO_2 (2θ= 28.3° (-111), and 30.4° (111)), which are dominant phases, were detected at very low intensity for this ceramic sample. This can be attributed to the non-oxygen containing system.

The morphology of HfC–SiC ceramics is shown in Figure 2. It can be seen that the particle size was to nanoscale and microscale. The EDS analysis (Figure 3) reveals that the main elements composing the powders are Hf, Si, C and O, indicating the presence of HfC and some oxygen forming oxycarbide compounds with Hf(C,O) formulae according to the XRD results shown in Figure 1.

Novel sources for the obtaining of HfC–SiC ceramic particles were synthesized using: hafnium tetrachloride, pectin and distilled water as solvent, as well as, calcined SBA-15, sucrose, distilled water as solvent and sulfuric acid as catalyst. The reaction of the precursors at 1600°C resulted in the formation of crystalline phases of HfC, SiC and HfO_2. The products obtained were mainly nanoparticles, with few microparticles.

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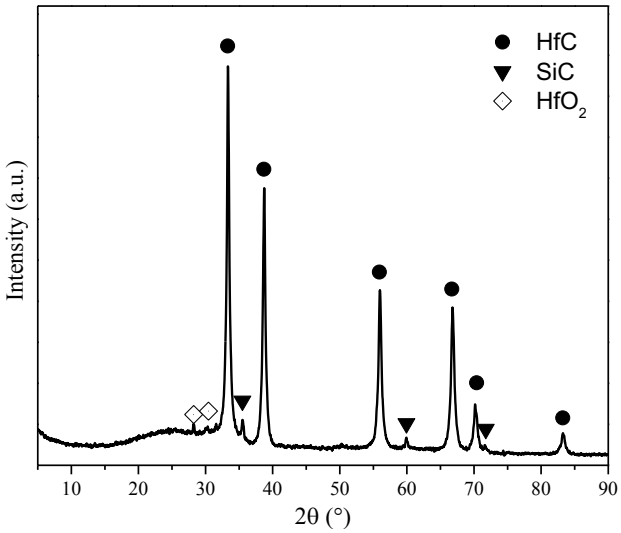

Figure 1. XRD patterns of HfC-SiC ceramic powder.

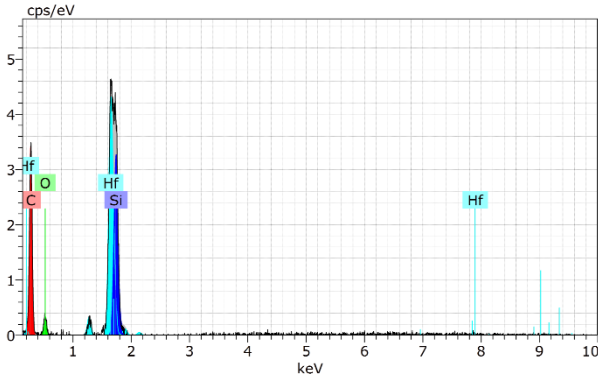

Figure 3. EDS analysis of HfC-SiC ceramic powder.

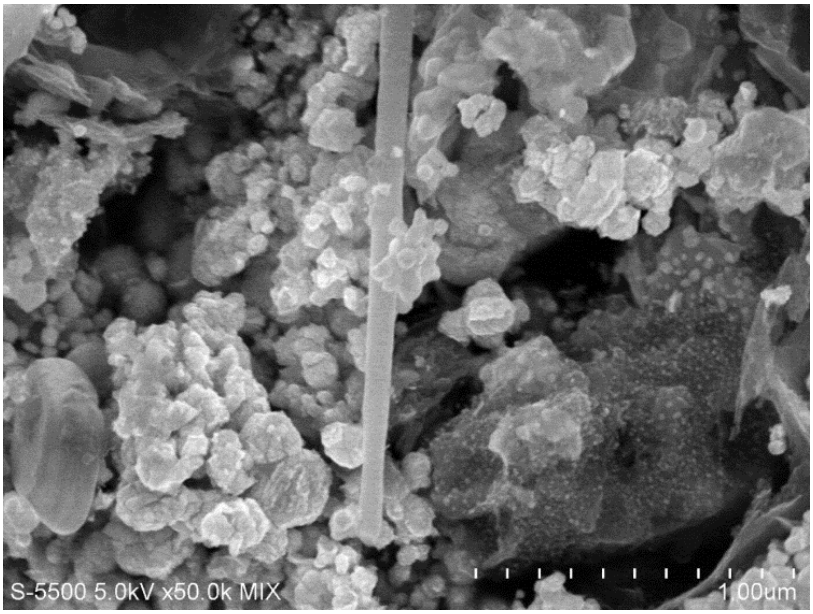

Figure 2. SEM micrography of HfC-SiC ceramic powder.

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