High Temperature Stability of Bulk Ti_3SiC_2 Materials in Air

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The ternary carbides, Ti_3SiC_2, have attracted an enormous attention because of their unique combination of both metallic and ceramic properties [1,2]. Like metals, they have excellent electrical and thermal conductivities, high toughness, high fatigue-crack growth threshold, low hardness, good machinability, and high-thermal shock resistance. Like ceramics, they display excellent chemical resistance, high Young's modulus, high temperature strength, and high melting point.

Ti_3SiC_2 has been synthesized from various starting powders such as Ti/SiC/graphite [1-3], Ti/Si/graphite [4-6], Ti/Si/TiC [7], TiC/Si [8], etc. The employed processing methods were hot pressing, hot isostatic pressing [1-3,7,8], *in situ* hot pressing/solid-liquid process [4-6], etc. However, it has been known that the synthesis of bulk Ti_3SiC_2 with high purity is not easy because relatively a long reaction time at high temperatures is required to synthesize via powder metallurgical routes. Recently, we have successfully synthesized dense Ti_3SiC_2 with a relatively high purity by hot pressing newly developed TiC_x/Si powder mixtures.

The starting powders of TiC_x (x=0.6) and Si (<45 μmϕ, 99.7% purity) were weighed in a molar ratio of 3 : 1.1 using a microbalance, mixed in a SPEX™ shaker mill apparatus for 10 min, and hot pressed at 1360°C to 19 mmϕ x 10 mm bulk specimens under 25 MPa Ar pressure for 90 min. During hot pressing, Ti_3SiC_2 was synthesized by a direct reaction between TiC_x and Si without forming any intermediate phases. Bulk Ti_3SiC_2 was cut into small coupons with a size of 10x8x0.5 mm^3, ground to a 1000 grit finish, and then ultrasonically cleaned in acetone and methanol. Oxidation tests were performed in the temperature range between 25°C~1200°C in atmospheric air for up to 100 hr. The Ti_3SiC_2 samples were etched using a HF-HNO_3-H_2O solution for microstructural evaluation. Furthermore, they were investigated by a thermogravimetric-differential thermal analyzer (TG-DTA), a scanning electron microscope (SEM) equipped with an energy-dispersive spectroscope (EDS), an Auger electron spectroscope (AES), and an X-ray diffractometer (XRD) with Cu-K_α radiation.

The ternary layered carbide, Ti_3SiC_2, began to oxidize above 850°C appreciably. The oxidation resulted in the formation of an outer, coarse TiO_2 grains, and an inner, fine (TiO_2+ SiO_2)-mixed grains. During the initial stage of oxidation, oxygen diffused inward seriously, and carbon escaped from Ti_3SiC_2. Si tended to diffuse outward more than Ti. The oxide layer was softer and less stiff than the Ti_3SiC_2 matrix.

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