Chapter 9
Self-Propagating High-Temperature Synthesis (SHS) and Spark Plasma Sintering (SPS) of Zr-, Hf-, and Ta-Based Ultra-High Temperature Ceramics

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ABSTRACT
The identification of efficient techniques for the fabrication of Ultra High Temperature Ceramics (UHTCs) is very crucial in view of their rapid and wider development. Along these lines, the use of the self-propagating high-temperature synthesis (SHS) technique in combination with the SPS technology is examined in this chapter for the obtainment of fully dense $\text{MB}_2\text{SiC}$ and $\text{MB}_2\text{MC-SiC}$ ($\text{M=Zr, Hf, Ta}$) ceramics. The starting reactants are first processed by SHS to successfully form the desired composites. The resulting powders are subsequently consolidated by spark-plasma sintering (SPS). Bulk products with relative densities $\geq 96\%$ can be obtained within 30 minutes, when the dwell temperature is $1800\, ^\circ\text{C}$ and $P=20\, \text{MPa}$. Hardness, fracture
The strong interest in ceramic composites based on transition metal (Zr, Hf, Ta, etc.) diborides and carbides, often referred in the literature to as Ultra High Temperature Ceramics (UHTCs), is due to the unique combination of suitable chemical-physical and mechanical properties, such as melting point above 2700°C, high hardness, good electrical and thermal conductivity, chemical inertness, good thermal shock resistance and resistance to ablation in oxidizing environments (Upadhya et al., 1997; Fahrenholtz et al., 2007). These characteristics make UHTCs interesting in several application fields where thermal, electrical, chemical, and wear resistance are required, like cutting tools, high temperature crucibles, microelectronics as well as in aerospace industry for the fabrication of thermal protection components (Fahrenholtz et al., 2007; Rapp, 2006). In this context, the beneficial effect provided by the introduction of SiC, as additive, in terms of oxidation resistance at high temperatures is also well established (Tripp, 1973; Hinze et al., 1975; Monteverde & Bellosi, 2003; Monteverde & Bellosi, 2005; Wu et al., 2006; Monteverde & Scatteia, 2007).

Bulk UHTCs are typically obtained in dense form by Hot Pressing (HP), through which commercially available powders are sintered (Fahrenholtz et al., 2004; Marschall et al., 2004; Monteverde & Bellosi, 2005). Alternatively, the synthesis and simultaneous densification can be also accomplished by reactive HP using appropriate reaction promoters (Opeka et al., 1999; Zhang et al., 2000; Monteverde, 2005). The critical aspect related to this processing route is represented by the high sintering temperatures, mechanical loads and, especially, prolonged processing times (on the order of hours), required to achieve acceptable relative density levels. Moreover, under these processing conditions, materials with residual porosity and rather coarse microstructure are typically obtained. One option successfully considered for making consolidation conditions milder is represented by the use of some sintering aids, such as Si$_3$N$_4$ (Monteverde & Bellosi, 2002), HfN (Monteverde & Bellosi, 2004), and MoSi$_2$ (Balbo & Sciti, 2008). Nevertheless, the total sintering time still remains high when using conventional HP.

In this regard, the relatively novel Spark Plasma Sintering (SPS) technology, where the starting powders to be only consolidated or also simultaneously reacted are crossed by an electric pulsed current (Orrù et al., 2009; Munir et al., 2011), provides a convenient alternative to overcome the drawbacks above. Indeed, the
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Investigation of the Effect of Cutting Conditions and Tool Edge Radius on Micromachining with the Use of the Finite Elements Method