



Optical characterization of hafnium boride and hafnium carbide-based ceramics for solar energy receivers

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ABSTRACT

The availability of spectrally-selective, thermally stable and more efficient sunlight absorbers represents a crucial aspect for the development of solar energy technologies able to operate at elevated temperature. In this regard, the optical properties of dense HfB_2 , HfC , $\text{HfB}_2\text{-SiC}$ and $\text{HfB}_2\text{-HfC-SiC}$ products are assessed, compared and discussed in the present work, in view of the possible utilization of such Ultra-High-Temperature Ceramics (UHTCs) for thermodynamic solar energy conversion. The materials above are first produced in bulk form by Spark Plasma Sintering (SPS), starting from ceramic powders preliminarily prepared by Self-propagating High-temperature Synthesis (SHS), or by reactive-SPS, using elemental reactants. All sintered specimens displayed relative density above 95%, with the composite systems approaching the theoretical density values, due to the beneficial role played by SiC as sintering aid, which also improves the oxidation resistance of the resulting UHTCs. The material composition is found to largely affect the related optical properties, as the SiC addition typically increases solar absorbance and decreases spectral selectivity with respect to pure UHTC boride and carbide phases. A final discussion is given about the best tradeoff material.

1. Introduction

The identification of innovative materials for improved sunlight absorbers, capable to withstand more severe temperature conditions with respect to those tolerable by the standard systems, represents a key issue for the future development of concentrated solar power (CSP) technologies Behar et al., 2013; Tian and Zhao, 2013. Indeed, while an increase of the operating temperatures is highly desirable to improve the efficiency of solar plants, currently available absorbers are not able to provide good performances and stability above 700–800 °C (Cao et al., 2017; Pitz-Paal et al., 2005).

In this context, taking advantage of the peculiar combination of high-temperature structural and optical properties shown by Ultra-High Temperature Ceramics (UHTCs), the potential use of this class of materials in solar energy exploitation has been proposed.

Starting from pioneering works in the Seventies-Eighties dealing with UHTC coatings (Seraphin and Meinel, 1976; Haygarth, 1980; Randich and Pettit, 1981), the interest in the use of such materials has quickly increased (Kennedy, 2002) and recently moved towards their

bulk form. Specifically, in addition to their extremely high melting point (above 3000 °C), chemical stability, high hardness, good electrical and thermal conductivities, etc. (Fahrenholtz and Hilmas, 2017), several members of this ceramic family also exhibit high absorbance and intrinsic spectral selectivity properties (Randich and Allred, 1981; Sani et al., 2016a–c, 2017; Musa et al., 2016). Therefore, improved performances can be, in principle, expected by the availability of novel solar receivers based on UHTCs.

Unfortunately, the covalent character of chemical bondings in UHTC compounds makes the related powders difficult-to-sinter, so that the fabrication of bulk components usually requires quite severe processing conditions, particularly if pressureless sintering or conventional hot-pressing are adopted (Opeka et al., 1999; Fahrenholtz et al., 2007). Benefits to improve the densification behavior of UHTC materials can be provided by the use of more efficient hot pressing methods, like the Spark Plasma Sintering (SPS) technique Orrù et al., 2009, especially when starting from highly sinterable powders like those ones for instance produced by Self-propagating High temperature Synthesis (SHS) Licheri et al., 2008; Musa et al., 2014.

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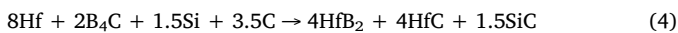
Alternatively, the SPS technology can be also exploited to perform synthesis and consolidation in a single stage, according to the Reactive SPS (R-SPS) route [Orrù and Cao, 2013](#). It is also well established that the presence of proper amounts of certain additives, such as SiC, MoSi₂, etc., further promotes powders densification, other than improving the oxidation resistance of the resulting material ([Licheri et al., 2009](#)).

Extensive studies have been carried out on monophasic and composite UHTCs for aerospace applications, as materials for thermal protection systems of hypersonic flights and atmospheric re-entry vehicles ([Fahrenholtz and Hilmas, 2017](#)). On the other hand, the evaluation of optical properties for the several constituents of such ceramics category is still confined only to few systems. In addition, the dependence of optical characteristics of the observed materials on their composition, microstructure and topological features, is still far from being well understood. Indeed, few detailed studies have been conducted only recently on this subject ([Sani et al., 2016a–d, 2017, 2013](#); [Musa et al., 2016](#); [Sciti et al., 2013, 2014](#); [Silvestroni et al., 2018](#)).

Along the same line, in this work we measured and compared the optical properties of highly dense hafnium-based monophasic and composite UHTCs, namely hafnium boride (HfB₂), hafnium carbide (HfC), and some of their silicon carbide (SiC) containing composites, i.e. the binary HfB₂-SiC and the ternary HfB₂-HfC-SiC, produced either by R-SPS or SHS-SPS routes. The most relevant topological and microstructural characteristics are investigated and discussed in view of the possible exploitation of these materials in the solar energy field.

2. Materials and methods

The characteristics of starting powders used for the synthesis of HfB₂, HfC, HfB₂-SiC and HfB₂-HfC-SiC products are reported in [Table 1](#). The initial mixtures to be processed by SHS or R-SPS were prepared by combining reactants in stoichiometric ratios according to the following reactions:



A slight excess of boron with respect to the stoichiometric value was used in reaction (1) to achieve the complete conversion of Hf powders into HfB₂. Indeed, as explained elsewhere ([Musa et al., 2013](#)), a certain amount of B was lost during reaction occurrence, due to the presence of adsorbed oxygen/moisture and other oxide impurities in the original powders, which determined the formation and the rapid expulsion of volatile B₂O₃. For sake of brevity, the four Hf-based UHTCs here investigated will be hereto after identified with the labels indicated in [Table 2](#), where the relative volume composition of each phase present in the synthesized products is also reported.

During SHS runs, cylindrical pellets of about 10 mm diameter and 20–30 mm high consisting of the various mixtures corresponding to reaction stoichiometries (1) to (4) were locally activated using an electrically heated tungsten filament. Detailed information on SHS experiments can be found elsewhere ([Licheri et al., 2009](#); [Musa et al.,](#)

Table 1

Characteristics of the starting reactants used in the present work for the preparation of Hf-containing ceramics by SHS or R-SPS.

Powders	Vendor	Particle size	Purity
Hf	Alfa-Aesar (10201)	< 44 µm	99.6%*
B ₄ C	Alfa-Aesar (40504)	1–7 µm	> 99.4%
Si	Aldrich (21,561–9)	< 44 µm	> 99%
Graphite	Aldrich (28,286–3)	1–2 µm	–

* Metals basis excluding Zr (2–3.5%).

Table 2

Designation and composition of the Hf-containing ceramic systems investigated in the present work.

System ID	HfB ₂ (vol.%)	HfC (vol.%)	SiC (vol.%)
HB	100	0	0
HC	0	100	0
HBS	73.5	0	26.5
HBCS	48	41	11

[2013](#)). About 4 g of the obtained porous products were then ground by ball milling (SPEX 8000 shaker mill, SPEX CertiPrep, USA) for 20 min, using a stainless steel vial with two steel balls (13 mm diameter, 8 g weight), to reduce them in powder form.

A laser light scattering analyzer (CILAS 1180, France) was used to evaluate particle size distribution of the resulting powders.

Phase analysis of the SHS products was performed by a Philips PW 1830 X-rays diffractometer equipped with a Ni filtered CuK_α radiation (λ = 1.5405 Å).

Both the consolidation of SHS powders and R-SPS experiments were carried out using a Spark Plasma Sintering equipment (515 model, Fuji Electronic Industrial Co. Ltd., Kanagawa, Japan) under vacuum conditions (20 Pa). This apparatus combines a uniaxial press (max 50 kN) with a DC pulsed current generator (10 V, 1500 A, 300 Hz), to concurrently provide an electric current through the processing powders and the graphite die containing them, together with a mechanical pressure through the die plungers. During each SPS run, the temperature was generally increased at a constant rate from the ambient value to a maximum level (T_{max}). Then, the T_{max} value was maintained for a certain duration t_D. Additional details related to SPS experiments can be found in references ([Licheri et al., 2009](#); [Musa et al., 2013](#)).

The relative densities of SPS products were determined by the Archimedes' method considering 11.18, 12.69, and 3.2 g/cm³, as the theoretical values for HfB₂, HfC, and SiC, respectively. In addition, a rule of mixture was used to evaluate the theoretical density of HBS and HBCS composites, i.e. 9.17 and 10.92 g/cm³, respectively ([Matthews and Rawlings, 1994](#)).

The microstructure of sintered samples was examined by SEM using a Hitachi S4000 FEG HRSEM. Image acquisition was obtained by the software Quartz PCI v. 5 (Quartz Imaging Corporation, Vancouver, BC, Canada).

To remove carbon contamination from the graphite foil, sintered samples were first ground using progressively finer abrasive paper down to 1000-grit. Subsequently, the surface of each specimen to be exposed to optical measurement was polished with diamond paste down to 0.25 µm.

Before performing the optical measurements, the surface texture of each polished sample was characterized to investigate the possible effects of the topological parameters on the optical properties of the synthesized materials.

The surface texture analysis was carried out with a non-contact optical profilometer (Taylor-Hobson CCI MP, Leicester, UK) equipped with a green light and a 20× magnification objective lens. For each samples, an area of 7.5 × 7.5 mm² was scanned and the collected surface data were processed with the Talymap 6.2 software (Taylor-Hobson, Leicester, UK).

In this work, the analysis of surface data was performed in terms of areal high parameters, as 3D parameters can provide a more comprehensive information about surface texture with respect to 2D ones. The evaluation of 3D texture parameters ([GPS, 2012](#)) was carried out on each of the 4 sections in which the scanned area was divided, so that standard deviations can be obtained. Before calculating the 3D parameters, the data collected were denoised (median filter 5 × 5 in X and Y), the form was removed and a robust gaussian areal S-filter was applied.

Hemispherical reflectance spectra were acquired using a double-

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