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Study of the sintering and oxidation behavior to the HfB$_2$/SiC system. Synthesis, reactivity and chemical-physical characterization.

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Introduction

The first aim of our work consists in synthesizing Ultra High Temperature HfB$_2$/SiC ceramics, used as structural materials in aeronautic and aerospace areas, by Spark Plasma Sintering (SPS). This process is used to obtain fully dense materials (>99% relative density) with fine grains, at lower sintering temperature and shorter dwell time than conventional techniques. Sintered materials are then oxidized in a solar furnace and characterized in order to better understand the oxidation mechanisms.

Study of sintering and oxidation behaviour

Spark Plasma Sintering (SPS)

<table>
<thead>
<tr>
<th>SIC content (% vol.)</th>
<th>Sintering temperature (°C)</th>
<th>Relative density (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>1850</td>
<td>100</td>
</tr>
<tr>
<td>5</td>
<td>1850</td>
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<tr>
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<td>1750</td>
<td>99</td>
</tr>
<tr>
<td>25</td>
<td>1700</td>
<td>99</td>
</tr>
</tbody>
</table>

Oxidation in solar furnace

- Mass gain rate for 80% vol. HfB$_2$ + 20% vol. SiC
- Slower mass gain rate for 80% vol. HfB$_2$ + 20% vol. SiC
- Slower mass gain rate for the composition 80% vol. HfB$_2$ + 20% vol. SiC
- Different microstructures observed depending on the oxidation temperature and ceramic composition.

Characterization of oxidized products

- Appearance of hafnium and silicon dioxides
- Increase of dioxides intensity peaks with temperature
- Silicon carbide grains growth with oxidation temperature
- External oxide layer growth with oxidation temperature

The milling was carried out in a planetary mill during 30 minutes containing 6 cycles of 6 minutes (1 minute of milling and 5 minutes rest).

Conclusion

- Sintering of fully dense HfB$_2$/SiC ceramics by Spark Plasma Sintering
- Decrease of sintering temperature with the addition of silicon carbide
- Oxidation tests carried out with a solar furnace up to 1800K under stagnant air

* H95S5: 95% vol. of milled HfB$_2$ + 5% vol. SiC