Mechanical properties and plasma erosion resistance of \( \text{BN}_p/\text{Al}_2\text{O}_3-\text{SiO}_2 \) composite ceramics

DUAN Xiao-ming(段小明)\(^1\,2\), JIA De-chang(贾德昌)\(^1\), ZHOU Yu(周玉)\(^1\), YANG Zhi-hua(杨治华)\(^1\),
WANG Yu-jin(王玉金)\(^1\), REN Feng-qin(任凤琴)\(^1\), YU Da-ren(于达仁)\(^3\), DING Yong-jie(丁永杰)\(^3\)

1. Institute for Advanced Ceramics, Harbin Institute of Technology, Harbin 150001, China;
2. Key Laboratory of Metal Precision Processing, Harbin Institute of Technology, Harbin 150001, China;
3. School of Energy Science and Technology, Harbin Institute of Technology, Harbin 150001, China

Abstract: \( \text{Al}_2\text{O}_3-\text{SiO}_2 \) system ceramic matrix composites with different volume fractions (10\%–60\%) of hexagonal BN particulates (\( \text{BN}_p \)) were prepared by hot-press sintering technique. Phase components, microstructure, mechanical properties and plasma erosion resistance were also investigated. With the increase of \( h-\text{BN}_p \) content, relative density and Vickers' hardness of the composite ceramics decrease, while the flexural strength, elastic modulus and fracture toughness increase and then decrease. The plasma erosion resistance linearly deteriorated with the increase of \( \text{BN}_p \) content which is mainly determined by the density, crystal structure and atomic number of the elements.

Key words: \( \text{BN}-\text{Al}_2\text{O}_3-\text{SiO}_2 \) composite ceramics; microstructures; mechanical properties; plasma erosion resistance

1 Introduction

The exploration and utilization of space require effective and reliable propulsion devices. Hall thrusters are a category of electric propulsion devices that originated in the 1950’s and 1960’s in both the United States and the former Soviet Union. Hall thrusters have mostly been used for satellite station-keeping duties, and their near-optimal specific impulse and thrust-to-power ratio have also led them to be used for orbit insertion [1–4]. There are several kinds of failure modes that limit the life time of Hall thrusters, for example, erosion of the cathode by ion bombardment, evaporation of the thermo-emitter and heater materials due to high temperature, degradation of insulating and structural materials in space conditions, deformation and cracking from thermal shocks and etc [5–8].

Boron nitride (BN) and BN containing composites are considered as the best candidate materials for manufacturing Hall thrusters channel because of their good chemically inert, thermal properties, and good electric insulation properties. Furthermore, BN has suitable secondary electronic emission factors and easy machinability [9]. However, the plasma erosion resistance of BN ceramic is worse, and thus BN based composites were developed in order to solve this problem. Now, there are many kinds of BN composites systems, such as \( \text{BN}/\text{SiO}_2 \), \( \text{BN}/\text{Al}_2\text{O}_3 \), \( \text{BN}/\text{ZrO}_2 \), \( \text{BN}/\text{Si}_3\text{N}_4 \) and \( \text{BN}/\text{AlN} \) [10–18], that can be used as Hall thruster channel wall materials. However, most of the studies involve only the preparation process, mechanical properties and microstructure characteristics of these composite materials. But the sputtering resistance performance and the plasma erosion damage mechanisms were rarely reported, and cannot fundamentally enhance erosion resistance properties of materials.

In the present work, \( \text{BN}_p/\text{Al}_2\text{O}_3-\text{SiO}_2 \) composites with different \( h-\text{BN}_p \) contents were prepared by hot-press sintering, the phase components, microstructures, mechanical properties and plasma erosion resistance were studied.

2 Materials and methods

The starting materials used in this work were hexagonal BN powders (particle sizes are 0.1–1 \( \mu \text{m} \) and purity >98\%), amorphous SiO\(_2\) powders (particle sizes are 2–10 \( \mu \text{m} \) and purity >98\%) and \( \alpha-\text{Al}_2\text{O}_3 \) powders...
The volume fraction of amorphous SiO$_2$ remains a fixed value of 30% in original composite powders, while the Al$_2$O$_3$ content decreases with the increase of BN content. The detailed compositions of raw materials are provided in Table 1. The starting powders were mixed by ball-milling with $\alpha$-Al$_2$O$_3$ balls for 12 h using ethanol as milling medium, and then dried. Finally, the powder mixture was hot-press sintering at 1650 °C for 60 min under a pressure of 30 MPa in 1×10$^5$ Pa Ar atmosphere.

<table>
<thead>
<tr>
<th>Sample</th>
<th>BN (%)</th>
<th>Al$_2$O$_3$ (%)</th>
<th>SiO$_2$ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>10BN</td>
<td>10</td>
<td>60</td>
<td>30</td>
</tr>
<tr>
<td>20BN</td>
<td>20</td>
<td>50</td>
<td>30</td>
</tr>
<tr>
<td>30BN</td>
<td>30</td>
<td>40</td>
<td>30</td>
</tr>
<tr>
<td>40BN</td>
<td>40</td>
<td>20</td>
<td>30</td>
</tr>
<tr>
<td>50BN</td>
<td>50</td>
<td>20</td>
<td>30</td>
</tr>
<tr>
<td>60BN</td>
<td>60</td>
<td>10</td>
<td>30</td>
</tr>
</tbody>
</table>

X-ray diffraction (XRD) analysis was carried out using Cu K$_\alpha$ radiation. Density of the specimen was determined by the Archimedes method. On polished surfaces, Vicker’s hardness ($H_v$) was measured with a load of 5 kg using an HBV-30A tester. Flexural strength and elastic modulus were measured on chamfered bars with size of 3 mm×4 mm×36 mm, in three-point bending, with a span length of 30 mm and at a cross-head speed of 0.5 mm/min. Single-edge-notched-beam (SENB) samples were fabricated by notching the segments of tested flexure specimens with a 0.15 mm thick diamond wafer saw. The SENB samples with dimensions of 2 mm×4 mm×20 mm were tested in three-point loading with a 16 mm span at a cross-head speed of 0.05 mm/min. Plasma erosion resistance of the composites were measured in the plasma sputtering test equipment at Steady Plasma Advanced Technology Laboratory of Harbin Institute of Technology, China. The samples with size of 10 mm×10 mm×2 mm were placed 18 mm away from Kr plasma emission source. In the process of sputtering test, the plasma is perpendicular incident to the sample surface, and sputtering time is 4 h. Microstructure of fracture surfaces and plasma sputtered surfaces of the composite samples, sprayed with gold film was observed by scanning electron microscopy (SEM, FEI Sirion). Fine microstructures of composite ceramics were characterized in transmission electron microscope (TEM, Phillips CMH2) with accelerating voltage of 120 kV.

3 Results and discussion

3.1 Phase component and microstructures

Figure 1 shows XRD patterns of the hot-pressed BN$_p$/Al$_2$O$_3$-SiO$_2$ ceramic composites. The composite with low $h$-BN$_p$ volume fraction of 10% consists of $h$-BN, Al$_2$O$_3$ as well as mullite, which originates from the reaction between Al$_2$O$_3$ and SiO$_2$ during hot-pressing. While there are no Al$_2$O$_3$ diffraction peaks in the ceramics with 20%−60% BN$_p$ volume fractions. The molar ratio of Al$_2$O$_3$ to SiO$_2$ is 3 to 2 according to the chemical formula of mullite (3Al$_2$O$_3$·2SiO$_2$). For the composites with different $h$-BN volume fractions, as indicated in Table 1, the specific value of Al$_2$O$_3$ to SiO$_2$ are 2/1, 5/3, 4/3, 1/1, 2/3, 1/3, respectively. So, for the composites with 10%−20% BN$_p$, there are too much Al$_2$O$_3$ to totally react with SiO$_2$ to form mullite. While for the composite with 30%−60% BN$_p$, the Al$_2$O$_3$ can completely react with SiO$_2$ to produce mullite, and a certain amount of SiO$_2$ retains which tends to be amorphous.

![Fig. 1 XRD pattern of hot-press sintered BN$_p$/Al$_2$O$_3$-SiO$_2$ ceramic with different $h$-BN$_p$ volume fractions: (a) 10% BN; (b) 20% BN; (c) 30% BN; (d) 40% BN; (e) 50% BN; (f) 60% BN](image_url)