Sintering behaviour of alumina–niobium carbide composites

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Abstract

Ceramic cutting tools have been developed as a technological alternative to cemented carbides in order to improve cutting speeds and productivity. Al\textsubscript{2}O\textsubscript{3} reinforced with refractory carbides improve fracture toughness and hardness to values appropriate for cutting applications. Al\textsubscript{2}O\textsubscript{3}–NbC composites were either pressureless sintered or hot-pressed without sintering additives. NbC contents ranged from 5 to 30 wt\%. Particle dispersion limited the grain growth of Al\textsubscript{2}O\textsubscript{3} as a result of the pinning effect. Pressureless sintering resulted in hardness values of approximately 13 GPa and fracture toughness around 3.6 MPa m\textsuperscript{1/2}. Hot-pressing improved both hardness and fracture toughness of the material to 19.7 GPa and 4.5 MPa m\textsuperscript{1/2}, respectively. © 2000 Elsevier Science Ltd. All rights reserved.

Keywords: Al\textsubscript{2}O\textsubscript{3}; Composites; Cutting tools; Hardness; NbC; Sintering

1. Introduction

Structural ceramics are characterised by high hot-hardness and superior chemical stability. Nevertheless, their brittle nature restrains their use as cutting tools to a present 5\% of a consolidated market dominated by high-speed steels (AISI class T and M), and cemented carbides (Co-WC).\textsuperscript{1} The growing interest in ceramics for cutting tools is justified from both an economical and technological perspective. Wear consists basically in a systemic phenomenon, which implies that each class of wear-resistant materials may find use in different applications, according to the counterpart of the tribological couple. Ceramics resist higher temperatures than metals without deforming. This allows tools to cut at faster speeds and deeper depths, resulting in increased removal rates and, consequently, cost efficient machining.\textsuperscript{2} Recent developments in cutting tool ceramic materials include escalating improvements on the strength, fracture toughness, and wear-resistance of Al\textsubscript{2}O\textsubscript{3} and Si\textsubscript{3}N\textsubscript{4}. Albeit its high-strength and thermal-shock resistance, Si\textsubscript{3}N\textsubscript{4} has proven effective only for machining a limited number of materials, including cast irons and nickel-based alloys.\textsuperscript{3} Furthermore, machining with Si\textsubscript{3}N\textsubscript{4} usually requires the use of coolants, which represent three times the current cost of cutting. On the other hand, Al\textsubscript{2}O\textsubscript{3}-based ceramic composites do not require cutting fluids yielding both economic and environmental benefits.\textsuperscript{1}

The development of new Al\textsubscript{2}O\textsubscript{3}-based composites has been accompanied by a significant improvement in properties. This increases the range of applications for such materials as cutting tools, from widely used steels and cast irons to very hard steels and superalloys for the aerospace industry.\textsuperscript{2} Aspects such as processing and microstructure have been extensively investigated. Fracture toughness, strength, hardness, and wear resistance have been particularly improved by the dispersion of hard carbide particles, such as TiC,\textsuperscript{4-7} WC,\textsuperscript{8} and NbC.\textsuperscript{9,10} Moreover, the presence of dispersed particles can produce a pinning effect\textsuperscript{11} and inhibit the grain growth of the matrix, which further contributes to the final performance of the composite.
2. Experimental procedure

The starting powders consisted of Alumina APC-2011 SG (Alcoa, Brazil), D50 = 2.3 μm, surface area 1.5 m²/g; and NbC (Hermann Starck, Berlin, Germany), D50 = 2.3 μm. Al₂O₃ and NbC were dry-mixed during 4 h in a planetary ball milling containing Al₂O₃ grinding media. Compositions containing 5, 10, 20 and 30 wt% NbC were prepared. Subsequently, powders compacts were uniaxially pressed into pellets of 8 mm in diameter under 100 MPa. Next, specimens were pressureless sintered under flowing argon either at 1650°C or 1800°C/min in a graphite-resistance-heated furnace. The heating and cooling rates were set to 20°C/min. Second series of samples was hot-pressed at 1650°C/30 min in flowing argon. The density of sintered specimens were measured by the Archimedes method.

Microstructural characterisation was carried out on specimens polished with diamond paste to a 1 μm finish, and thermally etched under vacuum at 1500°C/30 min. Grain size distributions were estimated by scanning electron microscopy and image analysis using the IMAGE-C computer program (INTRONIC, Germany). The identification of crystalline phases was carried out by X-ray diffraction. The parameters related to the intended application for the composites were gathered by the indentation method. Vicker’s microhardness (Hᵥ) and fracture toughness (Kᵥc) were evaluated by measuring the lengths of the cracks and the diagonal impressed by a Vickers indenter applying loads that varied from 30 to 100 N during 15 s. Loads varied according to the carbide content of the test specimen, aiming at the minimum necessary to produce radial cracks without ramification. Further details on the method used for fracture toughness evaluation can be found elsewhere.

3. Results and discussion

Table 1 summarises the physical and mechanical properties of Al₂O₃–NbC composites. Densification was a function of the temperature and the sintering process. Pressureless sintering at 1650°C without additives did not result in significant densification. Increasing the sintering temperature to 1800°C improved the density of the material from ~90% TD to ~96% TD. Hot-pressing resulted in density values in the range of ~98 and 99.5% TD. Similar densities have been reported for cold-isostatically pressed Al₂O₃–TiC composites pressureless sintered at 1870°C. It has also been shown that the relative density of Al₂O₃–TiC composites varies within a wide range (75%TD to >99) according to the sintering method and temperature.

The hardness values obtained were consistent with the composite processing method. Pressureless sintering resulted in relatively low hardnesses, ranging from ~9.6 to a maximum of 13.9 GPa obtained for Al₂O₃–30 wt% NbC sintered at 1800°C/15 min. Hardness did not increase at high NbC contents, probably because of the low density of the material. This is further confirmed by examining the density and hardness of hot-pressed specimens. As compared to pressureless sintering, hot-pressing resulted in denser materials also characterised by hardness in excess of 19 GPa, such as that obtained for

<table>
<thead>
<tr>
<th>Composition</th>
<th>Density (wt%)</th>
<th>Al₂O₃–D50 (μm)</th>
<th>Hᵥ (GPa)</th>
<th>Kᵥc (MPa m¹/₂)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Uniaxial pressing — sintered at 1650°C/30 min</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>93.0</td>
<td>3.0</td>
<td>9.5±0.8</td>
<td>3.0±0.5</td>
</tr>
<tr>
<td>Al₂O₃ + 5% NbC</td>
<td>91.8</td>
<td>1.9</td>
<td>12.9±0.7</td>
<td>3.6±0.6</td>
</tr>
<tr>
<td>Al₂O₃ + 10% NbC</td>
<td>91.6</td>
<td>1.6</td>
<td>9.6±0.9</td>
<td>3.2±0.5</td>
</tr>
<tr>
<td>Al₂O₃ + 20% NbC</td>
<td>89.6</td>
<td>1.8</td>
<td>9.8±0.8</td>
<td>3.1±0.6</td>
</tr>
<tr>
<td>Uniaxial pressing — sintered at 1800°C/15 min</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>96.1</td>
<td>–</td>
<td>9.8±0.6</td>
<td>2.5±0.8</td>
</tr>
<tr>
<td>Al₂O₃ + 10% NbC</td>
<td>96.5</td>
<td>–</td>
<td>12.6±0.5</td>
<td>3.1±0.7</td>
</tr>
<tr>
<td>Al₂O₃ + 20% NbC</td>
<td>96.2</td>
<td>–</td>
<td>9.9±0.4</td>
<td>3.7±0.4</td>
</tr>
<tr>
<td>Al₂O₃ + 30% NbC</td>
<td>95.3</td>
<td>–</td>
<td>13.9±0.6</td>
<td>3.0±0.8</td>
</tr>
<tr>
<td>Hot-pressed at 1650°C/30 min</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Al₂O₃ + 5% NbC</td>
<td>99.5</td>
<td>2.3</td>
<td>16.5±1.1</td>
<td>2.9±0.6</td>
</tr>
<tr>
<td>Al₂O₃ + 10% NbC</td>
<td>99.5</td>
<td>1.4</td>
<td>15.7±1.2</td>
<td>2.8±0.6</td>
</tr>
<tr>
<td>Al₂O₃ + 20% NbC</td>
<td>98.2</td>
<td>1.4</td>
<td>19.7±1.2</td>
<td>4.5±0.5</td>
</tr>
<tr>
<td>Al₂O₃ + 30% NbC</td>
<td>97.7</td>
<td>1.3</td>
<td>19.0±1.3</td>
<td>4.2±0.4</td>
</tr>
</tbody>
</table>
Al$_2$O$_3$ reinforced by 20–30 wt% NbC hot-pressed at 1650°C/30 min. These values are compatible to other cutting tools materials, such as cemented carbides (12–20 GPa)$^1$ Al$_2$O$_3$ (19 GPa)$^5$ Al$_2$O$_3$–TiC (19–23 GPa)$^5,15$ and Si$_3$N$_4$ (19 GPa)$^{16}$ Fracture toughness of pressureless sintered Al$_2$O$_3$–NbC composites remained roughly unchanged within the margin of error. Average values ranged from 2.5 to 3.7 MPa m$^{1/2}$, regardless of the sintering temperature and NbC contents. A slight increase to $\sim$4.5 MPa m$^{1/2}$ was obtained hot-pressing specimens with NbC contents in excess of 20 wt%. These values are compatible to those obtained from hot-pressed and hipped Al$_2$O$_3$–TiC (3.4–4.5 MPa m$^{1/2})^{15,17}$ and grant NbC particular interest in its application as reinforcement for Al$_2$O$_3$ cutting tools.

The addition of NbC to Al$_2$O$_3$ restrained grain growth, resulting in pinned microstructures. This can be implied from an analysis of the D50 values included in Table 1. Although the increasing concentration of NbC did not result in a steady reduction in the average grain size of the Al$_2$O$_3$ grains for composites sintered at 1650°C, the difference in grain size distribution of plain and reinforced alumina is evident from Figs. 1 and 2. Grain growth inhibition has been observed in a variety of materials, as shown in Fig. 1 and 2.
of particulate composites, both metallic and ceramic, such as carbide-reinforced Fe–Ni–Cr alloys,\textsuperscript{18} Al\textsubscript{2}O\textsubscript{3}–ZrO\textsubscript{2}\textsuperscript{19} and Al\textsubscript{2}O\textsubscript{3}–SiC.\textsuperscript{11} Several models have been proposed to quantitatively describe the pinning effect in composite materials.\textsuperscript{20–22} However, they all apply to highly dense composites (<99.5%TD). In porous composites, not only dispersed particles but also remained pores restrain grain growth, which prevents a further analysis based on such models for the results obtained herein.

Figs. 3 and 4 show the microstructure of plain Al\textsubscript{2}O\textsubscript{3} and Al\textsubscript{2}O\textsubscript{3}–5 wt% NbC composite, respectively. Both were sintered at 1650°C/30 min. The reduction in Al\textsubscript{2}O\textsubscript{3} grain size caused by the addition of NbC particles is rather evident from the pinned microstructure of the composite. X-ray diffraction patterns from both sintered and hot-pressed specimens revealed that Al\textsubscript{2}O\textsubscript{3} and NbC were the only crystalline phases present, as it should be expected from such composites processed without additives.

4. Conclusions

The results obtained from the characterisation of pressureless sintered and hot-pressed Al\textsubscript{2}O\textsubscript{3}–NbC composites revealed that:

1. Pressureless sintering resulted in densities ranging from 90% TD to 96% TD. Hot-pressing resulted in denser specimens (~98 and 99.5% TD).
2. The hardness of the composites appeared to be a function of both NbC content and density. Hot-pressed materials resulted in the highest hardness values, exceeding 19 GPa, which is appropriate for cutting tool materials.
3. Hot-pressing increased the maximum fracture toughness from 3.7 to 4.5 MPa m\textsuperscript{1/2}. These values are compatible to similar hot-pressed and hipped Al\textsubscript{2}O\textsubscript{3}–TiC.
4. The addition of NbC restrained the grain growth of Al\textsubscript{2}O\textsubscript{3}.

Acknowledgements

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References