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Wear mechanisms of TiN–TiB_2 ceramic in sliding against alumina from room temperature to 700 °C

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Abstract

TiN–TiB_2 ceramic was prepared by the reactive hot-pressing method using titanium and BN powders as raw materials. The friction and wear properties of TiN–TiB_2 ceramic were evaluated in sliding against alumina ball from room temperature to 700 °C in air. The TiN–TiB_2 ceramic has a relative density of 98.6%, a flexural strength of 731.9 MPa and a fracture toughness of 8.5 MPa $m^{1/2}$ at room temperature. The TiN–TiB_2 ceramic exhibits a distinct decrease in friction coefficient at 700 °C as contrasted with the friction data obtained at room temperature and 400 °C. Wear mechanisms of TiN–TiB_2 ceramic depend mainly upon testing temperature at identical applied loads. Lubricious oxidized products caused by thermal oxidation provide excellent lubrication effects and greatly reduce the friction coefficient of TiN–TiB_2 ceramic at 700 °C. However, abrasive wear and tribo-oxidation are the dominant wear mechanisms of TiN–TiB_2 ceramic at 400 °C. Mechanical polishing effect and removal of micro-fractured grains play important roles during room-temperature wear tests.

Keywords: A. Hot pressing; C. Friction; C. Wear resistance; D. Borides; D. Nitrides

1. Introduction

In recent years, a major challenge in advanced structural ceramics is to develop reproducible, long-lifetime ceramic sliding components for use in mechanical systems that involve high temperatures, heavy loads and high velocities. Refractory materials such as borides, nitrides and carbides are natural candidates for these tribological applications due to their exceptional hardness, wear resistance and thermal stability at high temperatures. Titanium diboride (TiB_2) is one kind of refractory compounds with high melting point, high elastic modulus, and high hardness. Titanium nitride (TiN) has some attractive properties, such as high hardness, good electrical conductivity and excellent wear resistance. However, both TiB_2 and TiN exhibit poor sinterability due to their high Peierls barriers to the dislocation movement, which restricts their industrial applications [1].

In combination the extreme resistance to plastic deformation of TiB_2 with the high–temperature plasticity of TiN, TiN–TiB_2 ceramics have attracted great attention on industrial applications as jet engine parts, armor plates, cutting tools, dies, and other high-temperature ceramic components [2,3]. TiN–TiB_2 ceramics were fabricated with self-propagating high-temperature synthesis (SHS) method using Ti (or TiH_2), B and BN powders as raw materials [4–6]. However, previous studies focused mainly on processing and microstructure; there is almost no report on mechanical and tribological properties of TiN–TB_2 ceramics in open literatures till now.

For brittle ceramics, the fracture toughness and hardness are two important factors for us to evaluate and understand their dry sliding wear behavior [7]. In the present paper, dense TiN–33.3 mol%TiB_2 ceramic was fabricated via the reactive hot-pressing process. Friction and wear properties of TiN–TiB_2 ceramic were then investigated in sliding against Al_2O_3 ball from room temperature to 700 °C in air to obtain a better understanding of wear mechanisms.

2. Experimental

In the present paper, commercial titanium (~30 μm, 99.9%) and hexagonal boron nitride powders (~150 nm, 99%) were used as the starting reactants in the reactive hot-pressing

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process. The composition of reactive hot-pressed TiN–TiB_2 ceramic was designed according to a TiN/TiB_2 molar ratio of 2:1. The weighed powders were mixed by wet ball milling for 24 h in a polyethylene bottle with ZrO_2 balls and acetone as media. After mixing, the slurries were dried in a rotary vacuum evaporator, and then screened through a 120-mesh screen. The powder mixtures were heated to 1300 °C and held for 30 min, and then followed by hot pressing at 1800 °C for 1 h in vacuum with an applied pressure of 30 MPa. The bulk density of the specimens was measured by the Archimedes method, while the theoretical density was estimated by applying the rule of mixture. Specimens with dimensions of 3 mm × 4 mm × 36 mm were cut out from the sintered bodies using high-speed diamond cutter. These bars were ground with 1500-grit emery paper, and were finally polished with diamond slurries down to 1 μm finish. Three-point bending mode was used to measure flexural strength of samples over a span of 30 mm at a crosshead speed of 0.5 mm/min. Test pieces with the dimensions of 2 mm × 4 mm × 20 mm were prepared for measurements of single-edge notched beam (SENB) fracture toughness. Vickers hardness tests were carried out at a normal load of 98 N and room temperature. Each specimen was measured six times at selected load.

Friction and wear tests were conducted on a reciprocating ball-on-block high-temperature tribometer in sliding against sintered Al_2O_3 ball. The specimens with the dimensions of 4 mm × 8 mm × 16 mm were cut, ground and polished with emery papers and diamond slurries down to 1 μm finish. Before wear tests, polished specimens were ultrasonically cleaned first in acetone and then in ethanol. The test specimen was placed on a SUS310 stainless steel holder heated by a high-frequency induction heating coil. The paired Al_2O_3 ball, which is fixed on the upper holder in the tribo-tests, has a diameter of 9.5 mm and a Vickers hardness of 16 GPa. The displacement stroke, normal load, frequency, and testing time are the external variables associated with the tribometer. The wear tests were performed at loads of 5, 10 and 20 N, test temperatures of room temperature, 400 and 700 °C, a reciprocating frequency of 1 Hz, a linear stroke of 10 mm, and a test duration of 1200 s. The humidity during room-temperature wear tests is 40% in laboratory air. In addition, each specimen and its paired ball were heated from room temperature to testing temperature by high-frequency induction heating coil at a heating rate of 50 °C/min, and held at testing temperatures for 5 min before starting a new wear test. During the wear runs, the test temperature of the specimens was monitored using a thermocouple inserted at the near-surface position through a small hole in the SUS310 stainless steel holder. The friction coefficients were calculated by measuring the friction force at 1-ms intervals, obtaining the average friction force (absolute value) of one cycle of the reciprocation (1 s) and dividing the average friction force by the load.

Before and after wear tests, X-ray diffraction (XRD) analyses were conducted to determine the structure of reactive hot-pressed TiN–TiB_2 ceramic and to identify the possible structural changes of worn surfaces after wear tests at elevated temperatures. The XRD patterns were recorded using a Rigaku D/max 2550 diffractometer with a Cu Kα radiation. In order to estimate wear loss of the specimens after wear tests, the profilometry of worn surfaces were carried out by confocal laser scanning microscope (Olympus OLS3100, Japan). Morphologies of worn surfaces on each specimen were observed using a scanning electron microscope (FEI Quanta 200F, USA). The compositional analyses of worn surfaces were carried out using the energy dispersive spectroscopy (EDS) attachment. For SEM observations, the samples were coated with a thin Pt coating in order to obtain sufficient conductivity on the surface and avoid charging in the SEM.

3. Results and discussion

Fig. 1 shows the XRD pattern of reactive hot-pressed TiN–TiB_2 ceramic. The as-sintered sample consists only of TiN and TiB_2. Clearly, no residual BN and Ti from raw materials are detected. The reactive hot-pressed TiN–TiB_2 ceramic has a relative density of 98.6%, and exhibits excellent mechanical properties with a flexural strength of 731.9 MPa and a fracture toughness of 8.5 MPa $m^{1/2}$ at room temperature. As contrasted with the TiN–TiB_2 ceramics reported in previous studies [8,9], the reactive hot-pressed TiN–TiB_2 ceramic in present study shows remarkably high fracture toughness. The fracture toughness of TiN–TiB_2 ceramic obtained by Rangaraj et al. [8] via hot pressing the mixture of Ti and BN powders at 1850 °C is 6 MPa $m^{1/2}$, while the value obtained by Shobu et al. [9] via sintering the mixture of TiB_2 and TiN powders at 1900 °C is only 4 MPa $m^{1/2}$.

The backscattered electron image of reactive hot-pressed TiN–TiB_2 ceramic is shown in Fig. 2. The distribution of TiN (grey grains) and TiB_2 (dark grains) is very uniform. The average grain size of the TiN–TiB_2 ceramic is about 3 μm, which is smaller than that of monolithic TiB_2 hot pressed at 1800 °C for 1 h reported by Park et al. [10]. The fine microstructure and high flexure strength are closely related to the in situ synthesis of TiB_2 and TiN and mutual growth restriction of these two phases. Clearly, the fine microstructure and uniform distribution of TiB_2 and TiN phases are responsible for its high fracture toughness.

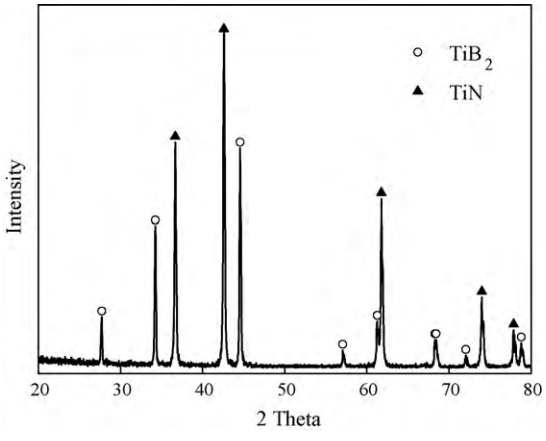

Fig. 1. XRD pattern of the reactive hot-pressed TiN–TiB_2 ceramic.