Comparison of Vacuum and Pressure-Assisted Sintering of TiB_2-Ni

G. Suskin and G.I. Chepovetsky

A relatively new generation of cermet materials, based on TiB_2-Ni, has been investigated. These borides currently are being examined for industrial applications with the aim of exploiting their excellent wear resistance. Optimization of the liquid-phase sintering process for a TiB_2-Ni composition was studied. The mechanical properties of cermet materials prepared using vacuum and pressure sintering techniques were determined. Criteria for optimal sintering conditions were established according to hardness, strength, and fracture toughness properties.

Keywords
mechanical properties, pressure-assisted sintering, TiB_2, vacuum

1. Introduction

THE MECHANICAL properties of titanium diboride (TiB_2)-base cermets point to the potential excellent wear resistance of this material. Over the last few years, investigation into TiB_2-base material has broadened. Scientists and engineers in this field are primarily interested in achieving a composition with a suitable binder for the TiB_2 hard grains. Therefore, both an appropriate metallic binder and optimal sintering conditions are needed to obtain the desired properties. Recent investigations have been performed using iron, nickel, cobalt, chromium, and their combinations as metallic binders for TiB_2 (Ref 1-4).

Sintering of Ti_2B is a complicated process due to slow diffusion of this composition, and its undesirable reaction with the metallic binder, formatting brittle intermetallic phases (Ref 5). In this work on TiB_2-Ni, liquid-phase sintering was performed. In order to optimize the sintering conditions, experiments using vacuum and pressure-assisted techniques were compared.

2. Experimental Procedure and Results

The material tested in these experiments consisted of 90TiB_2-10Ni (wt%) and additional carbon. The powder composition passed through conventional stages of preparation before the sintering process: mixing, ball milling, and cold pressing. The objective was to compare results obtained from vacuum and pressure-assisted liquid-phase sintering. Vacuum sintering was conducted under a pressure of 1.3×10^{-3} MPa, while a 1.5 MPa argon gas atmosphere was applied for pressure-assisted sintering. The experimental range of sintering temperature was between 1450 and 1700 °C. The sintering time was set at 90 min.

Based on previous research (Ref 5), it is possible to assume the formation of additional phases in a TiB_2/metallic binder system during sintering. The effect of these phases can vary the actual density of the material compared to its approximate initial density. Therefore, optimal sintering conditions were chosen according to the highest mechanical properties, not necessarily near the theoretical density of the TiB_2/metallic binder. The mechanical properties chosen were transverse rupture strength (TRS), hardness, and fracture toughness.

For all experiments, rectangular specimens were pressed. Final sample dimensions of approximately 5.0 by 6.5 by 20 mm were achieved after the sintering process. Transverse rupture strength experiments were performed by means of a Zwick-1474 (Zwick Engineering Corp., Elkin, NC) testing machine under three-point bending with a crosshead speed of 2 mm/min, in order to define and evaluate the influence of sintering conditions on material strength. Vickers macrohardness of the bulk and fracture toughness, using a load of 98 N, were determined using Matsuzawa (Matsuzawa Seiki Co., Ltd., Tokyo, Japan) hardness testing equipment. The fracture toughness of TiB_2 cermet was assessed based on the detection of crack propagation and the calculation of the K_{Ic} crack resistance parameter (Ref 6).

A series of trials were run in increments of 50 °C starting from an initial sintering temperature of 1450 °C. In the first stage of the experiment, the results from vacuum and pressure sintering until 1600 °C were compared. Analysis of mechanical properties indicated an improvement after pressure-assisted sintering of TiB_2-Ni alloy. Further tests to explore and optimize pressure-assisted sintering were continued up to 1700 °C. The mechanical properties obtained in this research are shown in Fig. 1 to 3.

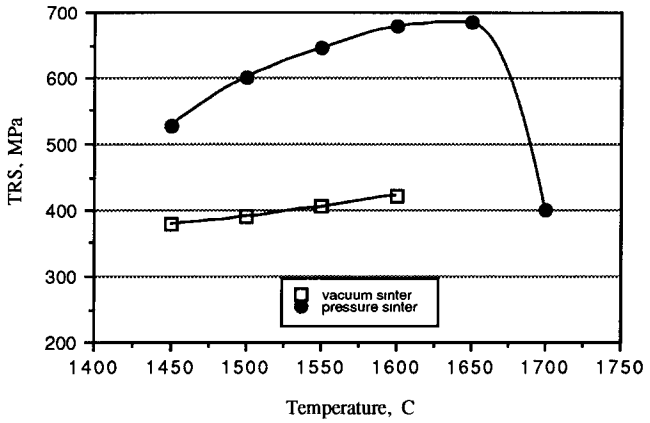

Fig. 1 Influence of sintering temperature on TRS

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A scanning electron microscope (SEM) (JSM-6400) (Jeol Ltd., Tokyo, Japan) equipped with a chemical microanalyzer (Link Analytical Ltd., Bucks, England) was used to define the microstructure of TiB_2 cermet after the sintering processes. A third phase, in addition to TiB_2 and nickel basic phases, was found present in a significant amount and was evident after each sintering test. Figure 4 shows the typical microstructure of the sintered material. Based on observation of the microstructure, TiB_2 grain growth up to 20% was achieved within the sintering temperature range (1450 to 1700 °C).

An effort was made to characterize the structure and composition of the material created by pressure-assisted sintering. Both chemical element analysis and x-ray diffraction (XRD) were used to define the phases and also to identify the additional third phase. An XRD plot obtained using Philips-XRD (PW-1729) (Philips Electronic Instruments, Inc., Alpharetta, GA) equipment is shown in Fig. 5. Based on the results of the microstructural investigation, it can be concluded that an additional phase, TiC, is present in the sintered material; this phase is visible as gray shaded areas in Fig. 4, within the major TiB_2-TiC-Ni three-phase system.

It should be noted that different surface structures are apparent after vacuum sintering compared to pressure-assisted sintering. Furthermore, a normal phenomenon is recognized between the as-sintered surface and the bulk structure, whereby the internal and boundary environmental conditions of the sintered material are different. Figure 6 shows that the effects from vacuum and pressure-assisted sinterings on the surface of the sample were obviously different. According to results gathered from element microanalysis, an enriched binder surface was obtained after vacuum sintering and a lack of binder was evident on the pressure-sintered surface (surface zone thickness in the range of a few microns). These microanalysis results were confirmed by microhardness surface testing using an applied load of 2.94 N ($HV_{0.3}$). For example, for samples sintered at 1600 °C, the vacuum-sintered sample at the surface was 90% Ni and the hardness, $HV_{0.3}$, was 13.0 GPa. In contrast, the surface of the pressure-assisted sintered sample was 5% Ni and its hardness was 19.5 GPa.

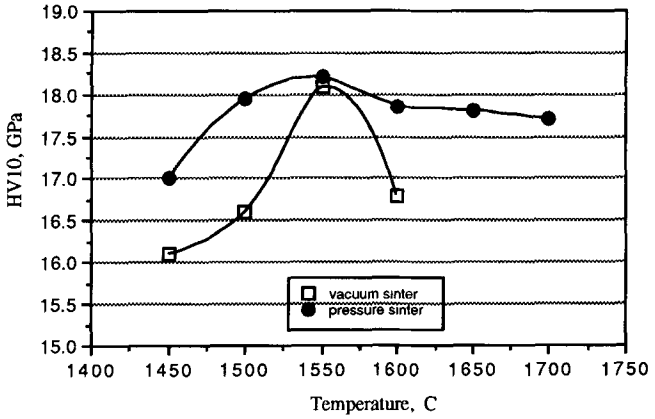


Fig. 2 Influence of sintering temperature on room-temperature hardness

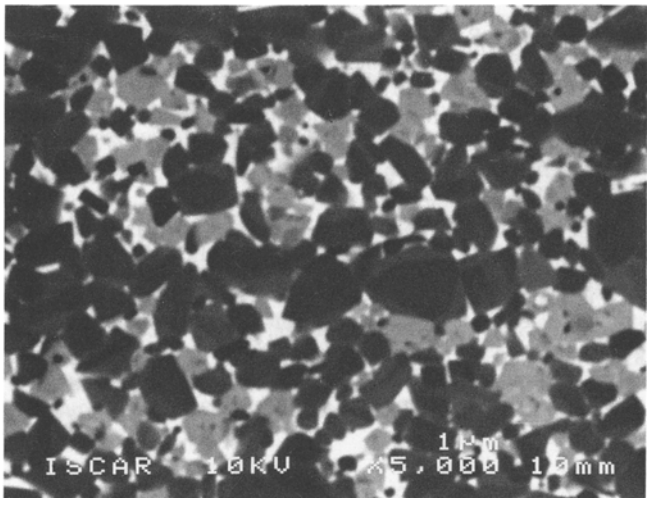


Fig. 4 SEM microstructure of TiB_2-base alloy after sintering for 90 min at 1600 °C

3. Discussion and Conclusions

The mechanical properties and microstructure of a TiB_2-Ni material were compared after sintering was performed in vac-

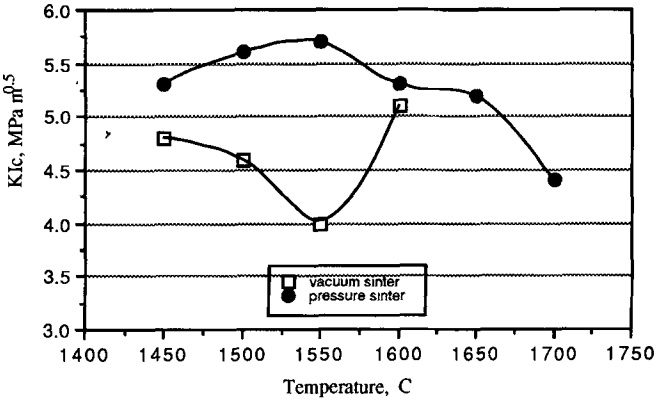


Fig. 3 Influence of sintering temperature on room-temperature fracture toughness

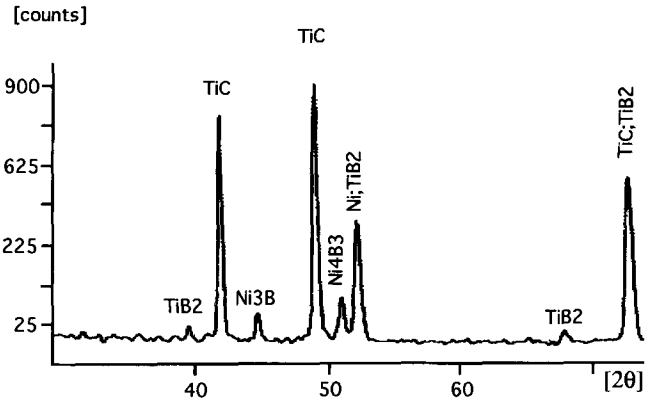
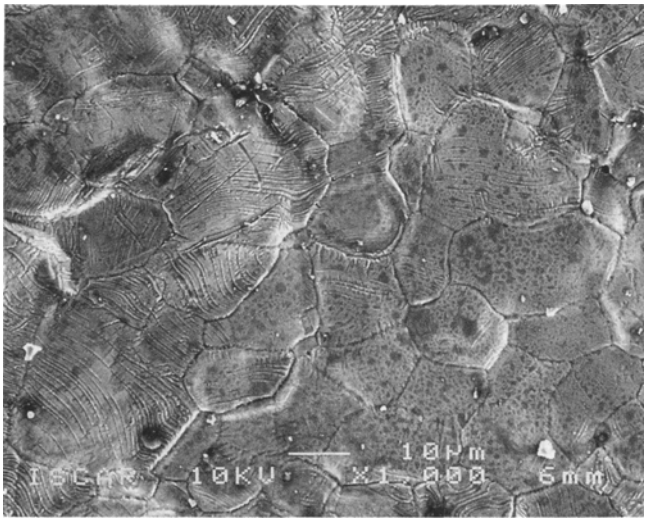
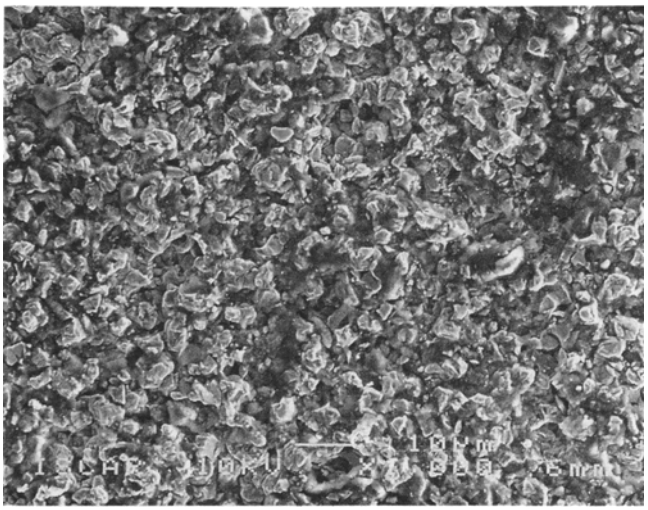


Fig. 5 X-ray diffraction plot of TiB_2-Ni cermet heated to 1600 °C



(a)



(b)

Fig. 6 Effects of sintering on surface morphology. (a) After vacuum sintering. (b) After pressure-assisted sintering

uum and in gas pressure sintering environments. Over the course of this experimental work, the advantages of pressure-assisted sintering compared to vacuum sintering became obvious. One significant benefit of pressure-assisted sintering is relatively high strength values. It should be noted that the present research established a good direct correlation between strength and sintered density. These results confirm previous investigations, showing the improvement of densification after pressure-assisted sintering, with a consequent increase in the strength of brittle materials (Ref 7).

Other tested mechanical properties (K_{Ic} and HV) generally remained at similar levels, and fluctuations according to the varying sintering conditions were indicated. However, superior and more stable results were obtained with pressure-assisted sintering within the tested temperature range.

Based on mechanical properties results, the optimal conditions for sintering of TiB_2-Ni alloy are pressure-assisted processes at a temperature range of 1600 to 1650 °C. The sintering temperature is limited mainly by the level of material strength received. Within the recommended sintering temperature region, TiB_2 cermet revealed a maximum density; temperatures above 1650 °C lead to a drop in properties of the studied material. The sharp decrease of strength at 1700 °C is probably linked with an oversintering phenomenon, such as damage to cohesive joining between hard grains and binder, grain growth, and evaporation of the metallic phase.

It should be noted that a three-phase system was evident in the cermet after sintering. In addition to the initial two phases (TiB_2 and Ni), a third phase (TiC) was present in a significant amount. However, Ni_xB_y phases, which have been found in previous TiB_2-Ni sintering investigations, also were recorded in insignificant amounts (Ref 5). The TiC phase formation most certainly originates from the active reaction between TiB_2 grains and carbon during the sintering process; hence, the contribution of the carbon balance must be considered in TiB_2-Ni sintering. Similar conclusions regarding the effect of carbon during sintering of TiB_2-Ni have been reached in previous work (Ref 8).

The studied material possesses excellent hardness, wear resistance, and chemical stability. It is thus a potential candidate for modern cutting tool applications in the field of high-speed machining.

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