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Short communication

Synthesis and microstructure evolution during vacuum sintering of Mo_2FeB_2 based cermets

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ABSTRACT

Mo_2FeB_2 based cermets were prepared by vacuum sintering at different temperatures and with different holding times. The phase transformation and microstructure evolution were studied by using scanning electron microscopy (SEM), energy dispersive X-ray analysis (EDX) and X-ray diffraction analysis (XRD). It was found that the hard phase Mo_2FeB_2 was produced in the compact at the stage of solid phase sintering by the reaction Mo and Fe_2B, which exhibited an equiaxed morphology. An extremely rapid densification occurred in the temperature ranging from 1050 °C to 1080 °C, and almost full densification was obtained at the temperature of 1080 °C without holding time. With an increase of liquid phase sintering temperature, the cermets exhibited a relatively homogenous microstructure, accompanied by an in situ growth of elongated Mo_2FeB_2 grains. Abnormally large and faceted Mo_2FeB_2 grains occurred during the isothermal hold at 1320 °C. These abnormal grains usually coalesced with surrounding grains and grain boundaries were formed between them.

Introduction

Borides, especially transition metal borides, have a high hardness, high melting point and relatively high electrical conductivity among hard materials. Hence, borides are promising candidates for wear resistant applications and have been intensively studied [1,2]. However, borides show a poor sinterability, extreme brittleness and strong reactivity with metals, which make it difficult to prepare boride based cermets from borides and metals. Reaction boronizing sintering is a novel strategy to form a ternary boride coexisting with a metal matrix in the cermets during liquid phase sintering. This new sintering technique has successfully developed ternary boride based cermets with excellent mechanical properties, such as Mo_2FeB_2 [3], Mo_2NiB_2 [4] and WCoB [5] based cermets, and has been applied in wear resistant applications such as injection molding machine parts, can forming tools, and dies for the extrusion of copper. In particular, Mo_2FeB_2 based cermets have attracted much attention because of cheap raw material, simple preparation method and superior properties [6,7].

Mo_2FeB_2 based cermets consisted of the Mo_2FeB_2 hard phase and Fe based binder phase. Previous work revealed that the mechanical properties of Mo_2FeB_2 based cermets could be improved by introducing Cr and Ni additions [8,9]. Further studies showed that Mn and V additions decreased the grain size and remarkably increased TRS [10,11]. Although many studies had been conducted on the improvement of mechanical properties, little attention had been paid on the phase transformation and microstructure evolution during the vacuum sintering. It was obvious that the studies on phase transformation and microstructure evolution were important to the determination of the sintering process, which also had a notable effect on the mechanical properties.

In the present work, the Mo_2FeB_2 based cermets were prepared by vacuum sintering at different temperatures and with different holding times. The phase transformation and microstructure evolution were studied by using scanning electron microscopy (SEM), energy dispersive X-ray analysis (EDX) and X-ray diffraction analysis (XRD).

Experimental procedure

Commercially available Mo, Cr, FeB, carbonyl Fe, Ni and pure graphite powders were used as raw materials. Characteristics of these powders are listed in Table 1. The nominal compositions of Fe–48Mo–6.0B–2.9Ni–2.5Cr (wt.%) were used for the present investigation. A small amount of graphite was also added up to 0.5 wt.% to the powder mixture to facilitate reduction of oxides during the sintering.

These powders were mixed in a planetary ball-mill in ethanol together with cemented carbide balls for 24 h at a speed of 150 rpm (rotations per minute). After milling, the slurries were dried at 80 °C in an infrared stove, and then sieved through 200 mesh and pelletized with 7 wt.% polyvinyl alcohol as a binder. The powders were pressed into green compacts with dimensions 39 mm × 8 mm × 8 mm at 100 MPa for 30 s. The green compacts were dewaxed and sintered in a vacuum. The sintering experiments of powders were carried out at temperatures from 600 to 1320 °C in order to obtain information on the

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Table 1
Characteristics of the raw powders.

Power	Mean particle size (μm)	Chemical composition (wt.%)
Mo	4	Fe < 0.002, O < 0.1, Si < 0.001
Fe	3.5	C_{free} < 0.1, N < 0.1, O < 0.2
FeB	45	C_{free} < 0.27, Si < 0.71, B = 20.21
Cr	45	O < 0.2, Fe < 0.18, N < 0.045
Ni	3.5	Fe < 0.006, Si < 0.0036
C	3.5	N < 0.015, O < 0.3

crystalline phase and microstructures in the different states of sintering. Fig. 1 shows a typical heating curve for the specimen sintered at 1320 °C for 40 min, and an isothermal hold at 400 °C, 600 °C, 800 °C and 1000 °C was set up in order to obtain a relative high vacuum degree during the sintering. A similar heating curve was carried out for the other specimens when the required sintering temperature and holding time were reached.

The density of the specimens sintered at low temperatures (≤1050 °C) was measured using the geometry method. At higher temperatures, the specimens were polished and then weighed in air, before and after being coated with a thin layer of lacquer, and in distilled water using an electronic balance to an accuracy of ±0.0001 g. The coat of lacquer was applied to prevent the distilled water from entering any surface connected porosity. The densities of the specimens were deduced according to the formula proposed by Tham LM [12]. The X-ray diffraction analysis was performed using Cu Kα radiation (D8ADVANCEX X-ray diffractometer). The microstructure was observed by a scanning electron microscope (SEM, QUANTAN200/FEI), and the distribution of the elements was determined by energy dispersive X-ray analysis (EDAX, GENESIS2000) in combination with the SEM.

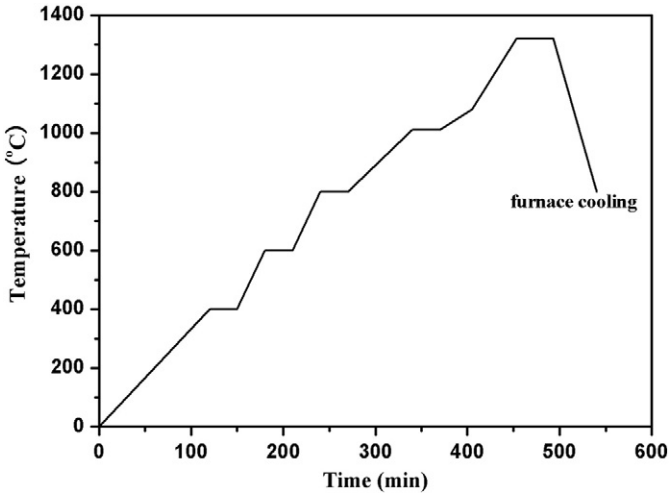

Fig. 1. Heating curve of the cermets.

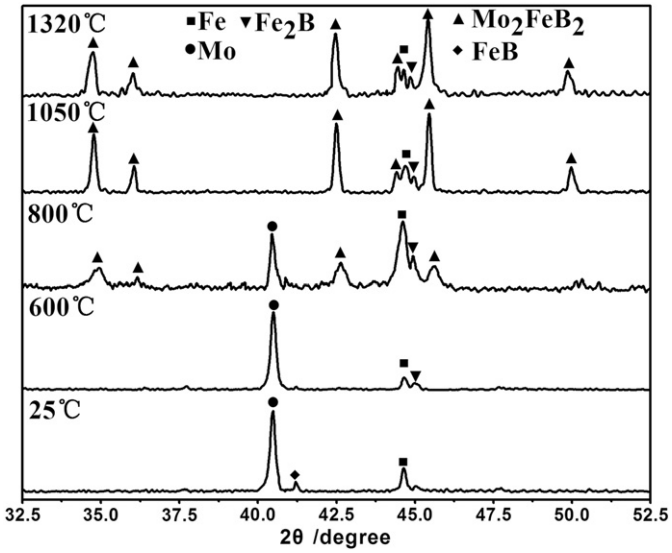

Fig. 2. XRD patterns of the cermets sintered at different temperatures.

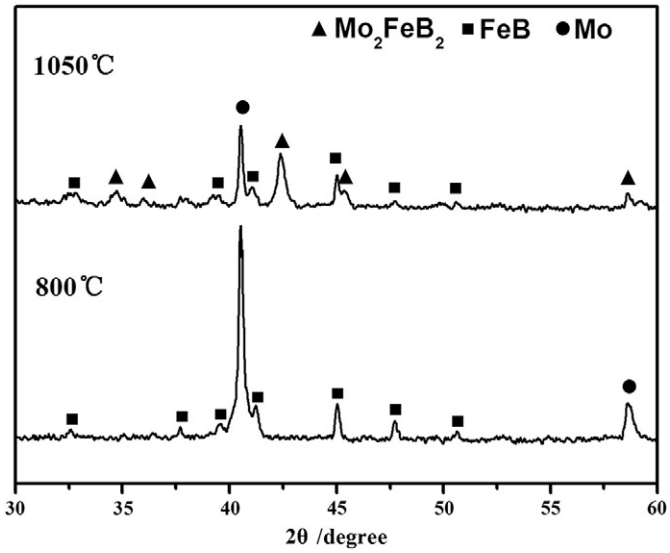

Fig. 3. XRD patterns of the new mixed powders.

Results and discussion

Crystalline phases

The XRD patterns of the samples with different sintering temperatures without holding time are shown in Fig. 2. It can be seen that the Fe_2B phase was formed with a decrease of the peaks of Fe when the sintering temperature was 600 °C. With an increase of the temperature from 600 °C to 800 °C, a new phase corresponding to Mo_2FeB_2 appeared, and the peaks of Mo weakened. The peak intensity of Mo_2FeB_2 was obviously strengthened and the peaks of Mo disappeared completely when the sintering temperature was 1050 °C. At a higher sintering temperature, no appreciable phase transformation could be observed.

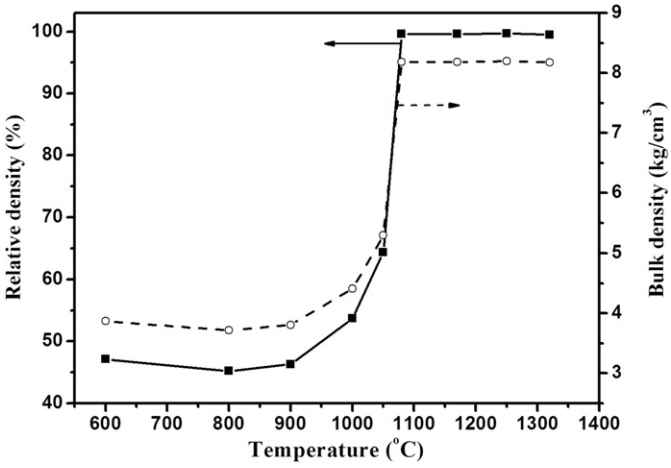

Fig. 4. Average relative density and bulk density as a function of sintering temperature.