A Kinetic Study of the Densification of TiB$_2$ at High Pressure and High Temperature

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High-pressure technology has been used to prepare dense, fine-grain specimens of TiB$_2$ at temperatures as low as 1600°C, as compared to temperatures of the order of 2300°C which are required to prepare dense, coarse-grain specimens by vacuum-sintering. Experiments were performed to describe the temperature and the pressure coefficients of the rate of densification and the effect of temperature and pressure on grain growth. Fully dense specimens of TiB$_2$ can be fabricated without significant grain growth. A mechanism for densification under these extreme experimental conditions is suggested.

INTRODUCTION

Several fabricating procedures were studied in the initial phase of a fundamental investigation [$^1$] of the physical, chemical, and mechanical properties of the diborides of titanium, zirconium, hafnium, niobium, and tantalum. During the early stages of this program, it became apparent that neither vacuum- nor inert-atmosphere sintering [$^2$] nor conventional hot-pressing procedures could provide suitable specimens for the above program. The floating-zone-melting technique [$^3$] was used to prepare pure samples of all the above diborides except TiB$_2$, the lack of mechanical integrity of zone-refined NbB$_2$ and TaB$_2$ precluded the use of these materials for physical and mechanical property measurements. Concurrently, a limited study was initiated to ascertain the feasibility of preparing material suitable for various property measurements by using high-pressure technology to extend the pressure range of conventional hot-pressing procedures to the range 100,000–300,000 psi. The successful application of this technique was demonstrated by the fabrication of specimens for oxidation evaluation and for physical and mechanical property measurements [$^4$].

The present program was undertaken to study the mechanism of densification of TiB$_2$ at high pressures and high temperatures; TiB$_2$ was chosen as representative of the hard-metal compounds, which are difficult to fabricate without the use of low-melting binder materials.

EXPERIMENTAL

Characterization of Starting Material

The TiB$_2$ powder was a high-purity product supplied by Millmaster Chemical Company. The chemical and spectroscopic analysis data given in Table I indicate