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