Room Temperature Slip in Titanium Diboride Produced by High Pressure

Abstract. *Prismatic slip occurs in polycrystalline titanium diboride after applying 5 to 15 kilobars of hydrostatic pressure. Specimens were subjected to these pressures inside silver chloride matrices by means of a simple cylindrical piston device. The slip was of the type {10$\bar{1}$0} <11$\bar{2}$0>. Microhardness indentations produced similar results.*

Slip has been reported to occur at room temperature in ceramic materials such as MgO (*1*) and WSi_2 (*2*). Slip is the macroscopic appearance of plastic deformation on a crystal concentrated on certain crystallographic planes or "slip planes," slicing the crystal into lamellae. The step produced on the surface by the relative slip of two lamellae is called a "slip line." The planes and directions of slip can be determined by comparing the slip lines on several faces of a crystal with the x-ray diffraction patterns obtained with single crystals in which exact plane orientations can be established. Several investigators (*3*) have studied slip in hexagonal titanium metal. The slip planes found were of the type {10$\bar{1}$0} and {10$\bar{1}$1} with the diagonal axis of indices <11$\bar{2}$0> as the effective slip direction.

Cylindrical samples of hexagonal titanium diboride pressed at high temperature (approximately 0.5 cm in diameter and 0.5 cm in length) with Ti plus B content of 99.3 percent and having a density of 99.6 percent of the theoretical density were polished mechanically with diamond paste and Linde A powder. Before being subjected to high pressure, polished specimens were studied by optical and electron microscopy to determine whether slip had occurred prior to the high pressure treatment (*4*). No evidence of slip was found. Specimens were placed inside cylindrical silver chloride sleeves which on the outside were enclosed in lava cylinders. Top and bottom of the lava cylinders were closed by suitable lava disks. In this manner, quasi-hydrostatic conditions were obtained. These cells in turn were placed in a cylindrical pressure device and subjected to pressures ranging from 1 to 15 kbar. The high pressure device was calibrated against the well-known Bi I–II and Bi II–III transitions at 25.4 and 27.0 kbar, respectively, at room temperature. Specimens then were repolished and the same areas as before were compared by microscopy. Samples pressed below 5 kbar showed little or no traces of slip. Specimens pressed at 5 to 15 kbar showed evidence of slip formation (see Fig. 1). No cracks of any kind were produced by the applied pressure, indicating that the pressure conditions during the pressing procedure were of the hydrostatic type.

Single crystals of TiB_2, containing 99.7 percent Ti plus B, of theoretical density, and grown by the Verneuil process (*5*), were previously studied for hardness relative to crystal orientations (*6*). Knoop and Vickers microhardness indenters were used at loads ranging from 100 to 1000 g. Microhardness indentations at room temperature on these single crystals also caused slip (see Fig. 2). Laue and cylindrical-rotation patterns on cleaved sections of TiB_2 single crystals were compared with microstructural features. From these data the exact crystal planes were determined. The microstructural features present in TiB_2 crystals as grown are in the form of a Widmanstätten precipitate substructure (*6*), which forms along the crystallographic axes. After being annealed above 2300°C, this precipitate started dispersing into the matrix and was brought out, by etching with nitric acid, as etch pits and polygon walls (see Fig. 3). These

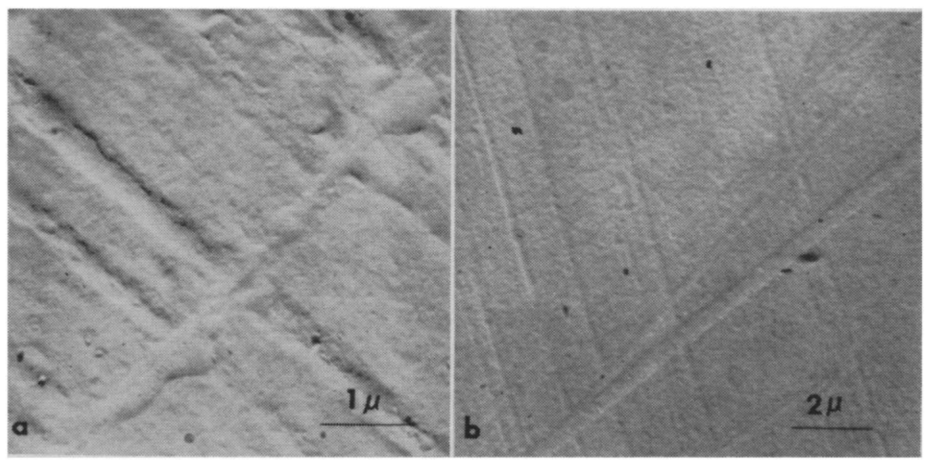

Fig. 1. Electron micrographs of polycrystalline TiB_2, subjected to a pressure of 10 kbar; (*a*) showing slip lines separated by a grain boundary, and (*b*) showing cross-slip.

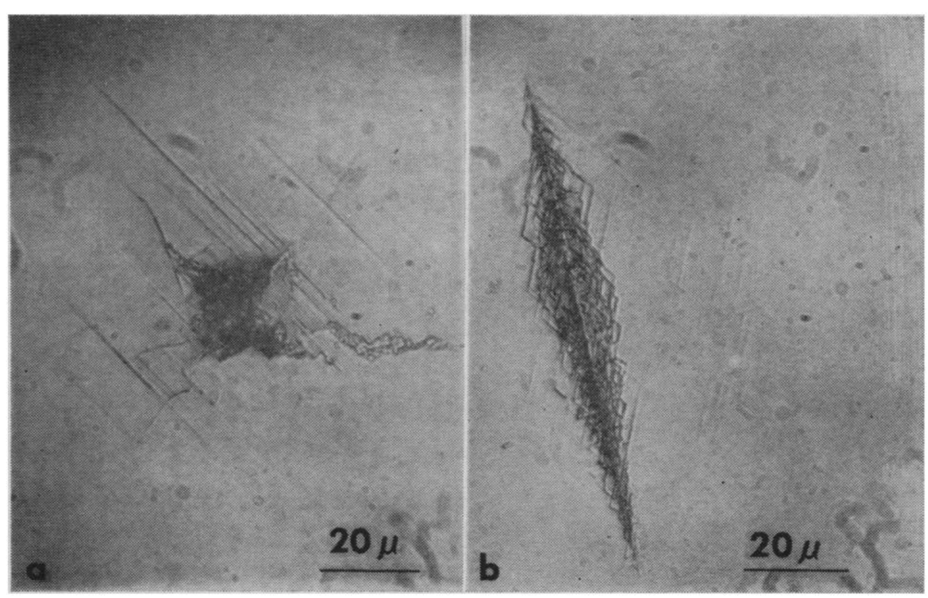

Fig. 2. Optical micrographs of single crystals of TiB_2 (*a*) showing slip produced by a Vickers microhardness indentation (at a 1000-g load) on the (11$\bar{2}$0) plane, and (*b*) showing slip produced by a Knoop microhardness indentation (at a 1000-g load) on the (0001) plane.

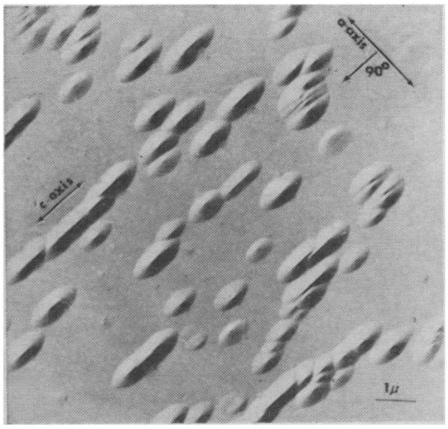

Fig. 3. Electron micrograph of a single crystal of TiB_2 annealed at 2300°C, showing etch pits on the $(10\bar{1}0)$ plane.

etch pits had the shape of triangles or hexagons on the basal or (0001) plane. Elliptical spiral etch pits (long axis parallel to the *c*-axis) were observed on the $(10\bar{1}0)$ plane. It was determined from the $(10\bar{1}0)$ plane that etch pits produced on $\{10\bar{1}0\}$ planes of annealed single crystals (2300°C) were at an angle of 30 degrees to the Burgers vector, which is parallel to the dislocation line (Fig. 3) (*7*). This means that the slip direction must be of the $\langle 11\bar{2}0\rangle$ type. The findings were then correlated with the results of the high-pressure experiments on polycrystalline TiB_2. Since we knew the plane orientations on the single crystals and could compare the slip traces of the single and polycrystals, we could also determine the planes of the corresponding polycrystalline grains. The slip occurring in both single and polycrystalline TiB_2 was found to be the same, namely, of the type $\{10\bar{1}0\}$ $\langle 11\bar{2}0\rangle$.

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References and Notes

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Metabolism of Iodine-131–Labeled Thyroxine-Binding Prealbumin in Man

Abstract. *The metabolism of ^{131}I-labeled thyroxine-binding prealbumin was studied in four normal subjects and in five hospitalized patients with nonthyroidal disease. The average half-time of total-body thyroxine-binding prealbumin, calculated on the basis of a two-compartmental model, was 1.90 days in normal subjects. Increased fractional degradation or diminished synthesis, or both, contributed to the depression of thyroxine-binding prealbumin in the serum of patients with nonthyroidal disorders.*

Thyroxine-binding prealbumin (TBPA) first described by Ingbar (*1*), is recognized as one of three serum proteins taking part in the peripheral transport of thyroxine (*2*). Experiments in our laboratory have indicated that this protein is important in regulating the concentration of free thyroxine in the serum (*3, 4*). In patients with systemic nonthyroidal illness a diminished concentration of TBPA frequently results in an increase of free thyroxine in the serum. (*3*). Furthermore, within 2 to 4 days after a major surgical procedure serum TBPA falls to approximately one-half of the preoperative value with a concomitant rise in the concentration of free thyroxine (*4*). The free thyroxine concentration is considered a better index of the overall thyroidal status of an individual than is the concentration of total circulating hormone (*5, 6*).

We have isolated TBPA in an immunologically pure form (*7*). The protein has a molecular weight of 73,000, a sedimentation coefficient of 4.58×10^{-13} sec, and a diffusion constant of 5.93×10^{-7} cm^2/sec. The protein is identical with prealbumin-1 (PA-1) separated by starch-gel electrophoresis. It has a single thyroxine-binding site per molecule, and like the prealbumin preparation of Schultze, Schonenberger, and Schwick (*8*), it is rich in tryptophan. We now describe results of metabolic studies in four normal male subjects and five hospitalized patients with varying concentrations of serum TBPA. There was rapid turnover of TBPA in comparison with that of other plasma proteins. Certain aspects of these studies have been reported (*9, 10*).

The TBPA was isolated from normal serum by means of cellulose-column electrophoresis and concentrated on diethylaminoethyl cellulose (DEAE) columns (*7*). In order to render the protein safe for administration to human subjects, the preparation was pasteurized (60°C, 10 hr) to inactivate any possible hepatitis virus. This procedure did not change the electrophoretic behavior of TBPA or alter the maximum thyroxine-binding capacity per milligram of protein. Similarly treated albumin has been generally used in turnover studies of radio-iodinated albumin in man. In a single study in which heat treatment of albumin was omitted (*11*) the overall metabolism of the iodinated protein in normal subjects was similar to that of human serum albumin which had been lightly iodinated and heated.

The initial studies were carried out on a single lot of TBPA iodinated (method I) at Abbott Laboratories, Oak Ridge, Tennessee (*12*). One milliliter of a solution of 10 mg TBPA in 0.1*M* phosphate buffer, *p*H 7.4, was mixed with K^{131}I and nonradioactive KI. The iodide was oxidized to iodine by dilute hypochlorite solution. Excess iodide was removed by passage through an ion-exchange column and subsequent dialysis against isotonic saline. The iodinated TBPA had an initial specific activity of 0.744 mc/mg and contained approximately 1.4 atoms of iodine per molecule. In order to minimize radiation damage, the final radio-iodinated product was immediately mixed with a 1-percent solution of human serum albumin (Albumisol; Merck, Sharp, and Dohme) in isotonic saline. When subjected to vertical starch-gel electrophoresis, 90 percent of radioactivity migrated as a sharp symmetrical peak anodal to serum albumin, but approximately 10 percent of the radioactivity was associated with a peak in the albumin area. When added to serum, ^{131}I-TBPA migrated with the leading edge of PA-1. Less than 2 percent of the radioactivity was dialyzable, suggesting neglible contamination with ^{131}I-iodide. The final material was sterilized by passage through a 0.22-μ Millipore filter. This preparation was used in studies on JO, MS, JS, HB, MK, and YF.

It became more convenient to iodinate TBPA in our laboratory as needed for individual studies (method II) by the technique of Greenwood, Hunter,