

Deformation of bcc Metals to 15-GPa Pressure

W.B. Durham,¹ S. Mei,¹ Y. Wang,² D. Weidner,³ I. Getting,⁴ P. Burnley⁵

¹Lawrence Livermore National Laboratory (LLNL), Livermore, CA, U.S.A.

²GSECARS, The University of Chicago, Chicago, IL, U.S.A.

³State University of New York at Stony Brook, Stony Brook, NY, U.S.A.

⁴University of Colorado, Boulder, CO, U.S.A.

⁵Georgia State University, Atlanta, GA, U.S.A.

Introduction

This report describes the results of work at the APS during the year 2002 with a new type of high-pressure deformation apparatus called the deformation-DIA, or D-DIA.

The deformation of materials at substantial levels of confining pressure has long suffered from a lack of quality experimental data [1-3], especially at pressures above 5 GPa. For earth sciences research, this means that the rheology of much of the upper mantle has been beyond experimental reach. More recently, problems in metallurgy — in particular, those related to science-based stockpile stewardship (SBSS) [4] — have also demanded characterization of the rheology of metals at pressures far beyond the 3-GPa levels reached to date [5]. The D-DIA is a multianvil apparatus intended specifically for deformation and was designed by three of the authors (Durham, Getting, and Wang). The ability of this apparatus to control pressure and differential stress independently sets it apart from other deformation machines that function at these very high pressures. It also allows testing of specimens that are large enough ($\sim 1 \text{ mm}^3$) to allow strain to be measured with satisfactory precision and all relevant scale-dependent factors, such as grain size and preferred crystallographic orientation, to be determined precisely. The D-DIA can impose strain that is high enough (to >1) to allow strain-dependent phenomena, such as work hardening, to be characterized.

Methods and Materials

The D-DIA (Fig. 1) duplicates the configuration of a hydrostatic DIA, a cubic multianvil device. Under an axial force applied in a hydraulic press to the large platen faces at the top and bottom, the six anvils in the hydrostatic DIA close uniformly on a cubic volume roughly 5 mm on a side to generate hydrostatic pressures approaching 15 GPa. Nonhydrostatic stress is generated in the D-DIA by allowing independent movement of the top and bottom anvils. Contained within the pressurized volume are the solid pressure medium (a boron-epoxy composite), insulator, furnace, and deformation column. The deformation column is cylindrical and 1.2 mm in diameter; it stands between the top and bottom anvils and

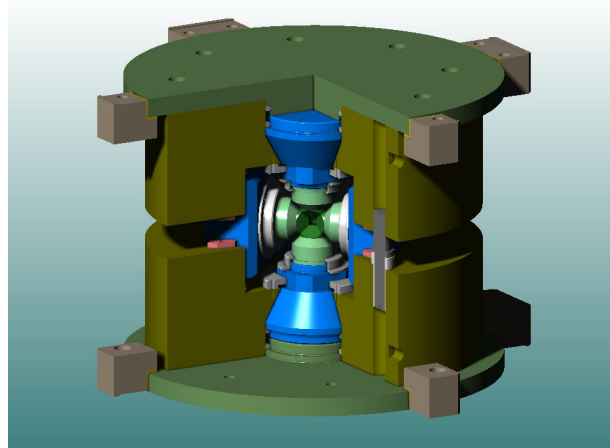


FIG. 1. The D-DIA in cut-away view. Six anvils, two of which (top and bottom) have independent control, pressurize and plastically deform the quasi-cubic assembly at the center. The height of the D-DIA as shown is about 250 mm.

consists of hard alumina pistons surrounding the sample assembly. For these experiments, the sample is a strongly x-ray absorbing polycrystalline bcc metal, so a stress gage of polycrystalline MgO (grain size of approximately 5 μm) is inserted in series with the sample. The sample and MgO stress gage are each approximately 1 mm in length. The DIA provides a line of sight for x-ray diffraction, and it is the press of choice for structural studies that use synchrotron x-rays.

The experiment was carried out by first hydrostatically pressurizing to run pressure and heating to run temperature, then initiating the differential displacement of the top and bottom anvils in order to plastically deform the sample (Fig. 2). As the top and bottom anvils compress the sample, the four horizontal anvils are retracted such that the mean stress remains constant. Plastic strains up to 0.5 (more is possible) are imposed on the sample at rates of 0.0001/s and slower, so runs typically last 24 hours or longer. During the experiment, measurements of the length of the sample are made

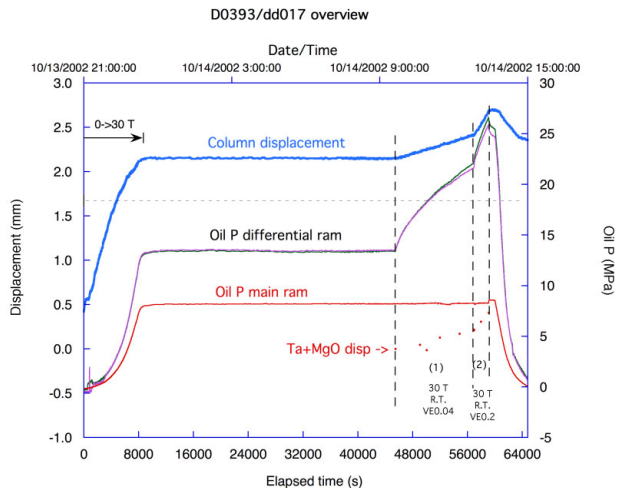


FIG. 2. Parameters related to the performance of the D-DIA. The solid curves represent measurements with sensors on the D-DIA. The points labeled Ta+MgO displacement were made by using radiography.

radiographically with a resolution better than $1\ \mu\text{m}$, alternating with measurements of the elastic strain in the MgO by using powder diffraction techniques.

We used both tungsten carbide (WC) and cubic boron nitride (cBN) anvils in the D-DIA. The former are strongly absorbing and provide a line of sight to the sample only through a narrow vertical gap between anvils. The cBN anvils, which became available only late in 2002, are x-ray transparent and allow diffraction measurements at all azimuths around the beam direction.

Results and Discussion

We carried out seven runs using the WC anvils. These allowed us to measure the length of the sample and so were very useful in helping us to learn and understand the behavior of this new instrument. In brief, we consider the results to be spectacular. The D-DIA design involves the coordinated motion of anvils — in particular, the simultaneous forward and reverse movement of different anvils — which had never been tested or even attempted before now. However, we encountered no significant difficulties operating the instrument. The seven runs were carried out on polycrystalline samples of Ta, Mo, NaCl, and olivine to mean stresses (pressures) of 9 GPa, temperatures to 1300K, and strains as high as 0.40 at sample strain rates from 5 to $1 \times 10^{-6}\ \text{s}^{-1}$. The anvil displacement was invariably smooth and reproducible from run to run, and the gaskets withstood the asymmetric displacement patterns. Operation was straightforward, allowing for very quick mastery by the operator.

With the availability of cBN anvils, we were able to move immediately from operational questions to actual measurements of the stress and strain rate, which is the

point of these rheological measurements. We did our final two runs in 2002 by using these anvils, and in the second run, we made our first measurement of differential stress. Figure 3 illustrates the plastic strain imposed on the Ta sample over the course of 4 hours of deformation in this final run.

Figure 4 shows the distortion of the Debye rings in the MgO stress gage (Fig. 3) when it is under deviatoric

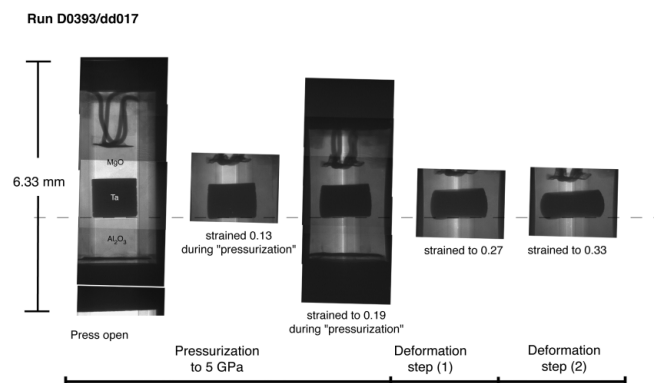


FIG. 3. Deformation of the sample and column at various stages of run D0393/dd017. The thermocouple is clearly visible in the top alumina piston.

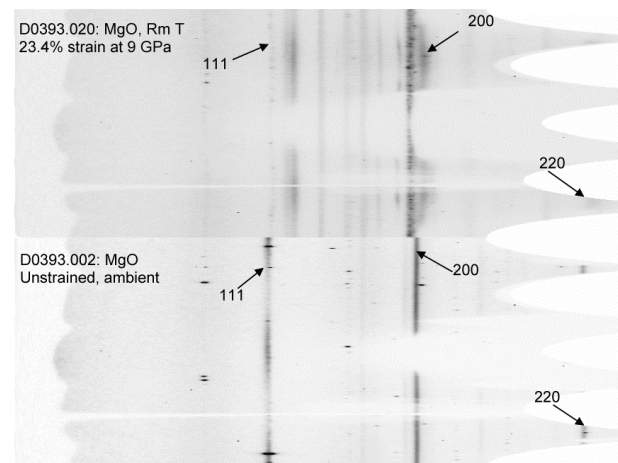


FIG. 4. Comparison of diffraction lines of MgO at 9 GPa and room temperature, after 24% shortening (upper half), and at ambient (stress-free) pressure and temperature (lower half). The Debye rings have been transformed into straight lines to allow easy recognition of the distortion. Horizontal axes are two-theta from 0° to 10° , and vertical axes are the azimuth angle measured from 0° (horizontal and perpendicular to the shortening axis) to 360° . Certain intensity variations are due to the shadowing of the cBN anvils. Modulated distortion in the (111) and (200) lines is evident.

stress. The distortion indicates a differential stress (maximum minus minimum principal stress) in the MgO of roughly 3.4 GPa. The strain rate in the Ta was $1.3 \times 10^{-5} \text{ s}^{-1}$.

Further details about the operation of and preliminary results from the D-DIA have been published [6, 7]. These early indications that the new D-DIA functions as designed and that the deviatoric stress in a polycrystalline material was successfully measured by x-ray diffraction, are promising signs that the instrument will shed new light on the rheology of materials at high pressures.

Acknowledgments

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