

# Deformation of BCC Metals to 15 GPa Pressure: Reports of Work Conducted at the APS, January 2003-December 2003

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## Introduction

During two sessions in March and July 2003 (see Table 1) on beamline 13-BM-D we began serious characterization of the rheology of BCC metals Ta and Mo at high pressures in support of DOE's stockpile stewardship program. We also performed further diagnostics, begun in 2002, of the new instrument used in these studies, the Deformation-DIA (D-DIA). In a remarkably short amount of time, we have designed, built, tested, refined, and proven the D-DIA, which resembles the hydrostatic DIA, and shares its pressure capabilities (~15 GPa for sample volume of ~1 cubic mm), but has the additional ability to shorten the sample in one direction and lengthen it in another. This allows us to directly measure rheology, i.e., characterize the relationship between strain rate and level of non-hydrostatic stress, under conditions of constant pressure, and through large strains. Before the D-DIA, the practical limit for doing such precise work deformation was about 3 GPa.

## Methods and Materials

We use synchrotron x rays in imaging mode to measure sample length as a function of time, which thus gives strain rate; and in diffraction mode to measure both pressure, as a process variable for controlling anvil movement in the D-DIA, and deviatoric stress, for direct use in the rheological law. Pressure and deviatoric stress can be measured in the axisymmetry of the D-DIA by diffracting at a range of chi angles. We accomplish the latter by using x-ray transparent cBN anvils with energy dispersive or wavelength dispersive measurements of the Bragg law. The first D-DIA operation was in late January, 2002, and the initial behavior was most encouraging. Subsequent beamline exercises at NSLS and APS have allowed us to refine operational techniques and the design of an effective cell assembly. Recently, we used cBN anvils for the first time and thus could make the first rudimentary rheological characterizations of the samples.

Table 1. D-DIA Experiments at APS during 2003.

D0438	Ta/BN assembly
D0439	Ta/BN assembly
D0448	NaCl
D0449	test of low P limit (blowout)
D0450	test of high P limit (blowout)
D0451	Olivine [calibration difficulties]
D0453	Ta/NaCl assembly

A typical sample assembly is shown in Figure 1. The sample, in this case polycrystalline tantalum, is 1 mm thick and not sufficiently x-ray transparent to allow diffraction. Therefore, as the sample is shortened by the action of the D-DIA, the state of stress within it is determined by using a proxy material, in this case MgO, whose equation of state parameters are known. The polycrystalline MgO is part of the compression column and thus

in mechanical series with the sample, and the presumption is that the state of stress in the Ta will be approximately the same as that in the MgO. One of the results of this work was that this assumption was quite poor. The design of later experiments in 2003 was therefore changed somewhat. These changes and subsequent results are described below.

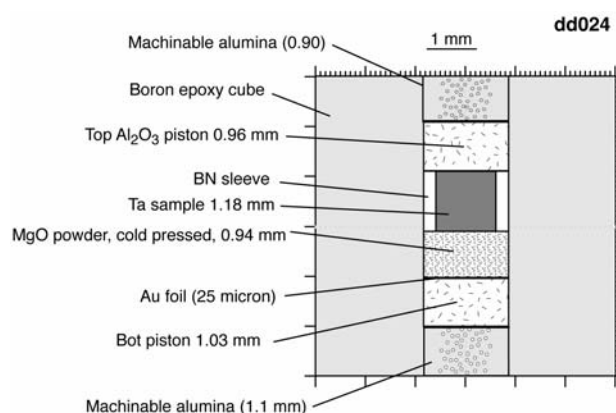


Fig. 1. Sample assembly.

We look to measure the strength,  $\sigma$ , of these materials as a function of strain rate ( $\dot{\epsilon}$ ), temperature ( $T$ ), and mean stress ( $P$ )

$$\sigma = f(\dot{\epsilon}, T, P)$$

where  $\sigma$  is the differential or shape-changing stress on the sample. We can also expect that BCC metals work harden significantly, that is, that  $\sigma$  is also a function of the total amount of plastic strain ( $\epsilon$ ). The experimental procedure is thus to operate the D-DIA in hydrostatic mode to bring the assembly to run pressure  $P$  (and  $T$  if applicable), and then use the differential motion of the D-DIA anvils to shorten the assembly of Figure 1, while allowing it to broaden. A permanent and large plastic strain is thus imposed on the sample at fixed  $P$ , and the synchrotron x-rays allow us to monitor the values of  $\sigma$  and  $\epsilon$  as a function of time during the several-hour duration of the experiment.

## Results

One of our first objectives, now that the basic operation of the new instrument has been established, was to measure the pressure dependence of the strength of BCC metals. During our March 2003 session, therefore, we designed two very simple experiments. In the first experiment, we shortened the Ta sample about 20% in two steps, the pressure in the second step being about twice that of the first step. In the second experiment, we did everything the same, but reversed the order of pressure, running at the higher pressure first, and following with the low pressure. If Ta work hardens at high pressure as it does at 1 atm, this order reversal would allow us to deconvolute

the effects of pressure and of strain on the value of differential stress. We ran these two tests at room temperature.

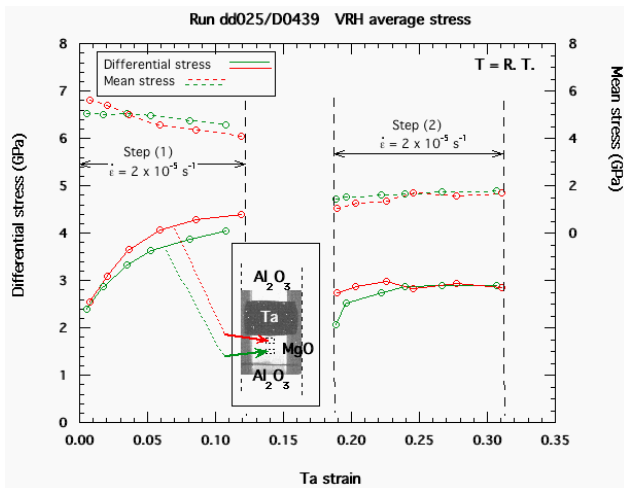
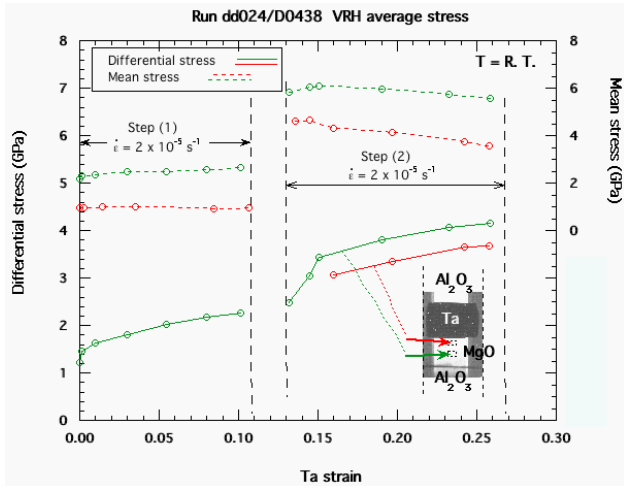


Fig. 2. Deformation results on polycrystalline Ta, using MgO as a stress proxy for the Ta for the first (top) and second (bottom) experiments. Only the order of pressurization was changed between the two experiments. Mean stress (dashed lines) are referenced to the right-hand vertical axis, and differential stress (solid lines) to the left-hand vertical axis. The two colors represent measurements at two different locations along the axis of the MgO.

The results in Fig. 2 appear to be fairly straightforward and easy to interpret. Depending on where in the MgO we placed our diffraction spot, we measured a mean stress (dashed lines in Fig. 2) of 1-2 GPa in one step, and 5-6 GPa in the other (the order being reversed in the two experiments). Differential stress in the MgO proxy was about 2 GPa at the lower mean stress and about 4 GPa at the higher mean stress, suggestive of a pronounced effect of pressure on strength of the Ta. The MgO also reflected some work hardening in the Ta (differential stress rising with strain), but the pressure effect is clear no matter what the order of pressure stepping.

We followed these measurements in March 2003 with our first serious attempt at modeling the dynamics of the D-DIA assembly shown in Fig. 1, with the eye-opening outcome that in conditions where materials surrounding the deformation column had moderate strength, that lateral support of certain parts of the column could be significant. For certain choices of material properties, the state of stress in the MgO in Fig. 1 can differ by

a large amount. Since the properties of some of the cell materials in Fig. 1 are largely unknown at the high pressures of the D-DIA, we have to be conservative. We therefore instituted a design change wherein a sleeve of fine grained rock salt, NaCl surrounded the deformation column. During the July 2003 session at 13-BM-D we tested polycrystalline Ta in one such configuration, and in addition tested a second assembly with polycrystalline NaCl substituted for the Ta. The purpose of the latter test was to provide input for the dynamical model of the Ta assembly with salt sleeve.

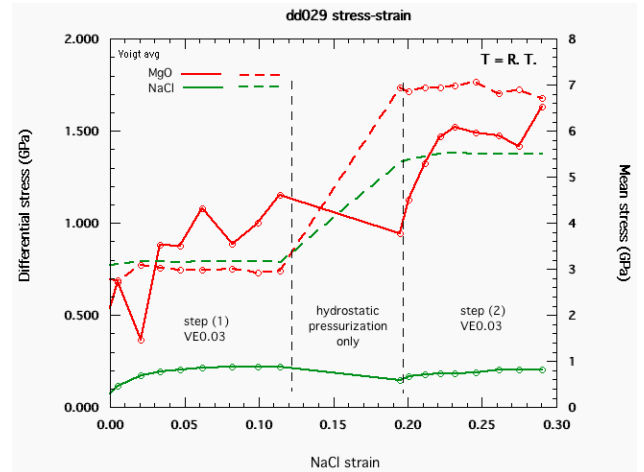


Fig. 3. Deformation results for polycrystalline NaCl. Mean stresses are indicated by dashed lines, differential stresses by solid lines. Green indicates measurements in the center of the NaCl sample, red the measurements in the MgO proxy in the deformation column.

The deformation results for the NaCl are shown in Fig. 3 and for Ta in the new salt assembly in Fig. 4, plotted in the same manner as Fig. 2. We tested the NaCl at two different values of mean stress, roughly 3 and 6 GPa, and found the strength to be approximately 0.2 GPa in both cases.

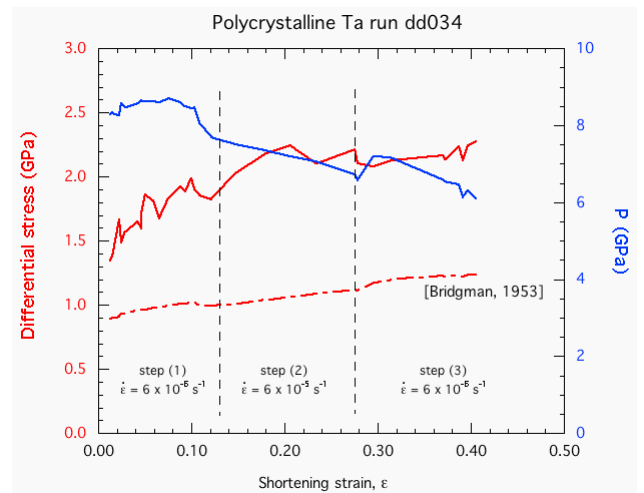


Fig. 4. Deformation results on polycrystalline Ta in the assembly with a NaCl sleeve surrounding the deformation column. Bridgman's (1953) *J. Appl. Phys.*, 24, 560 measurements from extension tests at 3 GPa are also shown.

In addition to examining the effect of the NaCl sleeve, the experiment in Fig. 4 was aimed at constraining the effect of strain rate on strength of polycrystalline Ta. While the effect of changing strain rate is weak—strength rises only slightly in step

(2) run at a 10 x higher rate than in step (1) and (3), an effect of the salt sleeve is apparent. The strength of polycrystalline Ta in Fig. 4, again reflected in the state of stress of the MgO, is somewhat lower than in Fig. 2.

As indicated by our dynamical simulation, the strength of the materials surrounding the deformation column does seem to matter. The suggestion of the trend of strength in actual experiments between Fig. 2 and 4 is that the boron-epoxy/BN environment of the first experiments has more inherent strength than the NaCl environment. It remains to be seen if these observations are borne out by the dynamical simulations. The dynamical simulation for the Ta in the NaCl assembly is still in progress

These experiments are the first of their kind on BCC metals, and have set the stage for definitive measurements of the effect of pressure on the strength of materials.

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