Step-loading technique for tungsten carbide opposed anvil high-pressure setup

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A method is described to achieve a step loading of a tungsten carbide opposed anvil setup. The rise time of the pressure pulse is nearly 200 ms and the maximum height of the pressure pulse is 2 GPa. The use of this method in the study of the kinetics of pressure induced transformations under isobaric conditions is illustrated with the help of the data on fcc→bcc (face-centered-cubic and body-centered-cubic) transformation in ytterbium.

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INTRODUCTION

The kinetics of pressure-induced polymorphic transformations under isobaric-isothermal conditions have been studied recently by using a resistometric method.¹⁻³ To achieve the isobaric conditions it is essential to raise the specimen pressure to a desired value before any appreciable transformation can take place. The maximum pressurization rates in conventional static high-pressure equipments range from 0.02–0.2 GPa s⁻¹. Such rates of pressurization can be used to produce nearly isobaric conditions if the transformation times are a few minutes or more. A much larger rate of loading is obviously required to study the kinetics of faster transformations. This paper describes an experimental arrangement for step-loading a tungsten carbide opposed anvil setup. With this setup the specimen pressure can be increased in step in time intervals of ≈200 ms.

EXPERIMENTAL DETAILS

A. Opposed anvil setup

A tungsten carbide opposed anvil setup (12.7-mm anvil face) capable of going up to 10 GPa with pyrophyllite gasket and talc pressure transmitting medium was used to pressurize the specimen. The details of the high-pressure cell assembly are shown in Fig. 1. The specimen measuring 4×0.5×0.05 mm is placed in the cell and the resistance is measured by a four-lead arrangement. To keep track of the run-to-run variation in the applied load-versus-specimen pressure relation, a bismuth sample is placed at right angles to the specimen in each run; a thin sheet of mica is used to insulate specimen and bismuth sample. A two-lead measurement was found adequate to detect the Bi-I→II (2.54 GPa) and the Bi-III→V (7.6 GPa) transitions. A few experiments were done with Bi and Ti in the configuration shown in Figs. 1(a) and 1(b) for load-pressure calibration alone. These runs indicated that the load-pressure relation was very nearly linear. The pressure gradient in the cell was estimated to be less than 0.1 GPa mm⁻¹. Typically the distance between the voltage leads L₁–L₂ was 1 mm, and assuming that the stress distribution in the cell was cylindrically symmetric, the pressure at the middle of the specimen differed from the pressure at the leads L₁, L₂ by about 0.05 GPa.

B. Step-loading system

The step loading is achieved by using an arrangement shown schematically in Fig. 2. The solid lines indicate internally connected hydraulic lines. The bomb, the hydraulic ram, and the reservoir can be isolated by closing valves V₁, V₂, and V₃, respectively. The pump has a built-in nonreturn

FIG. 1. High-pressure cell assembly (a) as viewed normal to the anvil face and (b) as viewed parallel to the anvil face. P—pyrophyllite gasket; T—talc pressure transmitting disk; S—specimen; M—mica insulation; C, C—current leads for four-probe resistance measurement; L₁, L₂—voltage leads for four-probe resistance measurement; and L, L—leads for two-lead resistance measurement.

FIG. 2. The step-loading arrangement (schematic). Solid lines represent internally connected hydraulic lines. V_1 , V_2 , and V_3 —hydraulic valves.

valve. The hydraulic ram is first pressurized to a desired initial pressure p_i by closing V_1 and opening V_2 and V_3 ; the bomb also gets pressurized simultaneously. The ram is then isolated by closing V_2 and the bomb is pressurized to p_b ($> p_i$). When V_2 is suddenly opened, the ram pressure experiences a jump and finally reaches p_f such that $p_i < p_f < p_b$. The opposed anvil setup can be operated in the conventional manner by closing V_1 which isolates the bomb. The magnitude of the ram pressure p_f depends on both p_b and p_i in a manner shown in Fig. 3.

C. Pressure calibration

The applied load-versus-specimen pressure relation for continuous loading was established using Bi-I→II (2.54 GPa), Ti-III→IV (3.67 GPa), and Bi-III→V (7.6 GPa) transitions. The load-pressure relation was found to be linear. In over one hundred experiments made during the studies of kinetics of phase transformations¹⁻³ and the thermoelectric power⁴ it was observed that though the load at which the Bi-I→II transition occurred varied within 10%, the ratio X of the loads at which the Bi-I→II and the Bi-III→V occurred was remarkably constant, the average value of X being

FIG. 3. Experimentally determined final ram pressure p_f as a function of bomb pressure p_b for different initial ram pressure p_i .

3.00 ± 0.05 .

The load-pressure relation during step loading was established by using manganin and lead as the pressure gauges. A manganin wire specimen (0.12-mm diam.) was mounted together with the Bi specimen as shown in Fig. 1(a) and 1(b). The load was increased at a rate 0.5 GPa min^{-1} until the Bi-I→II transition was observed. The resistance of the manganin specimen increased during this loading as shown in Fig. 4. The cell was then step loaded in the manner described in Sec. I A. The resistance-load point marked by the open circle in Fig. 4 was obtained. The load was then increased at a rate 0.5 GPa min^{-1} until the Bi-III→V transition occurred. The datum point (open circle in Fig. 4) after step loading fell close to the resistance-load curve defined by the data obtained during continuous loading before and after step loading. The experiments conducted with lead as the pressure gauge also gave similar results; a typical set of data is shown in Fig. 4.

A closer examination of the data indicated that the resistance of manganin and lead after step-loading were slightly time dependent. The point (open circle in Fig. 4) after step loading fell nearly 0.003 below the curve defined by the points obtained by continuous loading. The resistance increased with time and after nearly 100 s it fell in line with the rest of the data. In the case of lead, the point after step loading stayed 0.005 above the curve defined by the rest of the data and the resistance decreased with time. The time dependence of resistance may arise because of a slight redistribution of pressure after step loading. If so, the changes in resistances of both manganin and lead indicate a small pressure increase ($< 0.1 \text{ GPa}$).

The ratio X for the runs with an intermediate step loading was 3.00 ± 0.05 , which is very close to the value obtained in continuous-loading experiments. This together with the data shown in Fig. 4 suggests that the load-pressure relation is not affected by step loading. The gauge pressure p_f obtained after the step loading can be used to compute the specimen pressure, knowing the ram pressures at which the two Bi transitions occur.

FIG. 4. The resistance R of manganin and lead as a function of ram pressure for continuous slow loading and step loading. R' denotes the specimen resistance at 3.45 MPa. The occurrence of Bi-I→II and Bi-III→V transitions are marked, respectively, by A and B.

FIG. 5 The resistance-time response for manganin (a), and the ram pressure (b) during a step loading

D. Rise time of the pressure pulse

The rise time of the pressure pulse was estimated from the resistance-versus-time response of the manganin specimen during a pulse loading. The voltage across the leads L_1 - L_2 (Fig. 1) was recorded during step loading on a strip-chart recorder at a chart speed of 1200 mm min^{-1} . The traverse time of the recorder pen during the recording of the pressure pulse was estimated to be less than 10 ms. A typical record is shown in Fig. 5(a). The rise times in a number of runs made with different bomb pressures and the initial ram pressures were $200 \pm 25 \text{ ms}$. The changes in the hydraulic pressure of the ram during step loadings were also recorded using a pressure transducer [Fig. 5(b)]. The shapes and the rise times of the hydraulic pressure pulse in the ram and the pressure pulse experienced by the manganin specimen placed between the anvils were the same within the experimental error.

E. Temperature rise during step loading

The temperature of the specimen is expected to rise because of the rapid compression of the gasket and the pressure transmitting medium. The temperature rise during a step loading was estimated by placing a chromel-alumel thermocouple on the specimen. A typical trace of the thermocouple output during a step loading is shown in Fig. 6. After the step loading the temperature returned rapidly to the ambient. The temperature rise Δt ($^{\circ}\text{C}$) was found to depend mainly on the height of the pressure pulse AP (GPa) and satisfied the relation, $\Delta t = 1.45 AP$. Thus, the temperature rise during the step loading is small, and can be neglected for most kinetics studies.

Fig. 6. A record of the thermocouple output during step loading: $AP = 2 \text{ GPa}$.

FIG. 7. The variation of voltage across L_1 - L_2 (Fig. 1) in the case of ytterbium during a pulse loading and the fcc $\rightarrow$ bcc transformation. The details are given in the text.

II. SOME RESULTS

The sequence of loading used in the study of the kinetics of phase transformation is illustrated with the help of the data on the fcc $\rightarrow$ bcc transformation in ytterbium. First, the pressure is continuously increased up to a predetermined value (2.54 GPa in this case). This region is marked A in Fig. 7. The voltage across the leads L_1 - L_2 (Fig. 1) (and hence the resistance of the specimen) increases during this loading. The ram is then isolated by closing the valve V_2 (Fig. 2). The bomb pressure which would give the required final pressure is read from Fig. 3 and the bomb is then pressurized to the required pressure. The region B indicates the pause during the pressurization of the bomb. The hydraulic pressure in the ram remains constant during this period. The resistance of the fcc phase shows an increase during the step loading. The region C indicates the incubation period. The start of the fcc $\rightarrow$ bcc transformation is marked by a decrease in the specimen resistance.

III. DISCUSSION

The present method can be used to raise the specimen pressure in a step-wise manner in nearly 200 ms , the maximum height of the pressure step being 2.0 GPa . It may be noted that the rise time of the pressure pulse is the characteristic of the hydraulic lines and the valves, but the maximum height of the pressure pulse can be increased by reducing the anvil face. This technique has found application in the study of the kinetics under isobaric conditions of pressure-induced transformations. Since the change in the electrical resistance occurring at the transformation is used to calculate the fraction transformed, this method is useful for electrically conducting or semiconducting materials.

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