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Pressure Dependence of the Plastic Flow Stress of Alkali Halide Single Crystals*

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The change in the plastic yield stress of alkali halide single crystals due to applied hydrostatic pressure has been measured. The pressure dependence of the yield stress ($\delta\sigma/\sigma$) ranges from $\sim 9\times 10^{-2}$/kbar in soft RbI to ~ 0 in LiF. The experimental results suggest that in soft crystals point-defect generation and/or diffusion is effective in limiting dislocation mobility in the early stages of deformation, whereas in crystals hardened by irradiation or cold work, elastic interactions are rate limiting.

INTRODUCTION

In order to identify the various activated processes occurring during the plastic deformation of a crystal, measurements of the dependence of the plastic flow stress σ on the strain rate, temperature, and pressure are required. Observations of the strain rate and temperature dependence of σ are extensive for many materials, but the pressure dependence of σ has never been established experimentally. In fact, the early work of Schmid and Boas,[1] which introduced the concept of the critical resolved shear stress, suggests that the normal stress on the slip plane due to hydrostatic pressure should have no effect on σ; the experiments of Bridgman[2] and of Haasen and Lawson[3] on the plastic flow of metals under pressure reveal little, if any, pressure dependence of σ. There is such a pressure dependence, however. In the following we report the effect of hydrostatic pressure on σ in a number of alkali halide crystals as measured by interrupted compression tests.

EXPERIMENTS

Apparatus

Since σ is extremely structure sensitive, it is impossible to obtain accurate results from comparisons of compression tests performed at 1 atm with others performed at high pressure. Hence we use the method of interrupted stress–strain (σ–ϵ) tests on a single specimen. For experiments of this type a testing machine, the minitester, was designed[4] to operate while completely enclosed in a pressure vessel. The results are therefore free of problems of jerky loading and/or load weighing errors which arise if a loading device is brought through a system of high-pressure seals from outside a pressure vessel.

The original minitester has been modified and improved for this series of experiments. The tester, which has a diameter of about 3 cm and is about 38 cm long, consists of a synchronous motor and planetary gear box which turn a 0.3175-cm pitch screw. The rotation of the screw is converted to translation and advances the lower compression platen in the barrel of the tester. The moving platen bears on the sample being tested and the applied force is transferred by a steel rod to a load cell which is, essentially, a very stiff steel spring whose deflection is indicated by strain gauges. The load cell is made from fully hardened Vasco MA steel and contains two thin, parallel arms loaded in tension. Identical strain gauges are mounted on these arms and are connected in series to compensate for bending. The gauges are mechanically bonded to the load cell by the "Rockite" process by the Baldwin–Lima–Hamilton Corporation of Waltham, Massachusetts. For use under high pressure it is necessary that the load-cell material and the strain-gauge bonding material have similar compressibilities.[5] Gauges whose backing is highly compressible, such as epoxy gauges, strip off steel when subjected to a moderate pressure. Experience has shown that Rockite-bonded gauges are reliable through many applications of pressure. The load cells are calibrated at room pressure with an Instron testing machine equipped with an X–Y recorder, which allows simultaneous plotting of the applied force and the strain-gauge output of the load cell. The plot of strain vs load for the load cells is approximately linear with a slope of about 1.35×10^{-6}/N. Since a strain of 1×10^{-6} is easily detectable, load changes on the order of 0.75 N can be read. As specimens which average 0.6 cm^2 in area are generally used, it is possible to detect stress changes of about 0.14 bars.

The driving motor used in the minitester for the compression tests is a hand-wound, single-phase, hysteresis-type, synchronous ac device operating at 25 V and 1800 rpm. When connected through a 60 000:1 gear reducing system to the 0.3175 cm pitch screw, the rate of platen advance in the tester is approximately 0.009 cm/min. As the motor is almost perfectly synchronous, samples are subjected to a

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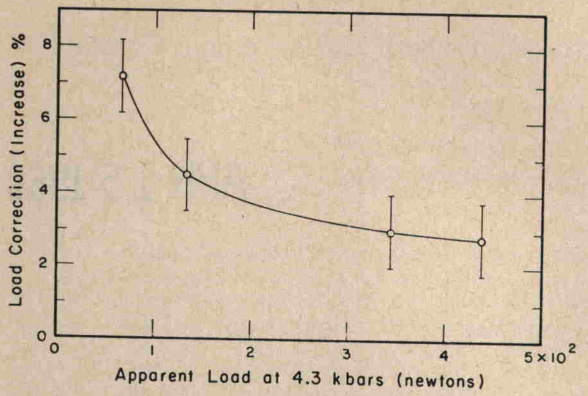

FIG. 1. Pressure dependence of calibration of load cell No. 4 (at 4.3 kbar). The apparent load is computed using the 1-atm calibration.

constant rate of compression. The spring constant for the minitester is about 5×10^4 N/cm (comparable to an Instron-type machine). For the highest rate of work hardening observed in the samples tested here the ratio of the plastic compressive deformation rate of the sample to the total deformation rate is $\simeq 0.78$. Usually, however, the ratio is in excess of 0.9. Therefore, for the sample length used, 1.8 to 2.54 cm, the plastic compressive strain rate is about 0.5×10^{-4}/sec for the rate of platen motion specified above.

In order to compare the flow stresses of a crystal at high and low pressure, it is necessary to determine the effect of pressure on the calibration of the load cell. The procedure adopted for this purpose is to calibrate the load cell *in situ* in the minitester against a helical steel spring both at low and high pressures. Load is generated by compressing the spring against the load cell using the motor-driven platen and the pressure-induced change of calibration is determined by comparing plots of the strain output of the load cell vs time. The applied load, which need not be known explicitly, can be computed knowing the time of loading, the rate of platen advance, the minitester deflection and the spring constant. In comparing plots of load-cell strain vs time, allowance is made for the slight variation of motor speed from test to test (∼±2%) by noting the loading time and the total platen advance at maximum load. This means that the calibration point at the highest load is uniquely determined, but reliability of the points at lower load depends on the constancy of the motor speed with load. Correction for the elastic deflection of the minitester is also made but this is of lesser importance as the tester is rather "hard", i.e., its deflection is small. The load cell used (LC No. 4) is fairly sensitive to pressure, and, in addition, the pressure effect varies with load as shown in Fig. 1. Each point on the figure represents the average of numerous calibration runs. It is believed that the pressure-induced change in calibration is determined to ±1%, i.e., typically the change in calibration is 4±1% at 4.3 kbar for LC No. 4.

The minitester is operated inside a 63.5-cm long, 15.2-cm o.d., 3.17-cm-bore-diam pressure vessel. The vessel is of three-piece construction of a general type described previously.[6] The minitester is placed in the vessel with the motor end down; thus electrical leads brought through the bottom plug connect to the motor and those in the piston connect to the load cell. Pressure is generated by driving the piston with one of two simultaneous acting 500-ton hydraulic rams, while the vessel is advanced by the other ram. Because the effect of even large hydrostatic pressure on the flow stress of the materials studied is moderate, it is not essential to know the applied pressure with great accuracy. Accordingly, the oil pressure in the top 500-ton ram was calibrated against pressure within the vessel by using a 120 Ω manganin gauge whose calibration is based on readings taken with a Harwood DWT-1000 dead-weight tester. Observation of the ram oil pressure allows pressure determination to ±0.1 kbar.

The load-weighing system of an Instron testing machine is used for recording the stress–strain curves (actually, load-cell response vs time) of the samples tested in the minitester. This system allows detection of a force of 0.75 N.

Materials and Testing Technique

The effect of pressure of 4.3 kbar on the plastic deformation of single-crystal samples of the alkali halides LiF, NaCl, KCl, KBr, KI, RbI, and CsBr was studied. The samples, all of which were purchased from the Harshaw Chemical Co., are in the form of $\{100\}$ cleavage blocks except in the case of the CsBr which possesses no natural cleavages. CsBr is sawed to shape using a string saw. A great majority of the samples tested were received in the past year but two tests are performed on samples received in 1959 for use in a previous study. The new crystals measure $2.54\times0.64\times0.95$ cm. The older samples are about $1.78\times0.5\times0.5$ cm. The ends of each sample tested are polished flat and parallel by hand using a vee-block. Load is always applied parallel to the largest dimension. The "new" and "old" crystals show a significant difference in flow stress as, due to improved production techniques, the newer crystals are relatively purer and therefore softer. In order to do a controlled study of the effect of crystal strength on the pressure-induced change of flow stress, crystals of different strength were produced by subjecting "new" crystals to 0.1 MR/h ^{60}Co radiation. The specimens in this case were generally reduced in dimension to $2.54\times0.64\times0.47$ cm in order to lower the applied load necessary to cause yielding. All tests were conducted at room temperature.

The cleavage blocks of the fcc structure alkali halides have four equally stressed primary $\{110\}$ $\langle\bar{1}10\rangle$ slip systems when loaded in simple compression.

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