

THE STRENGTH OF SINGLE CRYSTALS OF INORGANIC SALTS UNDER HIGH PRESSURE, II

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Introduction

For the measurements of the optical properties of materials under high pressure, the various alkali halide single crystals are used as the optical windows of high pressure vessels. Previously, we reported on the bursting process of the window under high pressure¹⁾. Generally, the optical window of high pressure vessel is exposed to high pressure during the time of the optical measurements, so the time affects the strength of the crystal window. In this report, therefore, the plastic deformations of the alkali halide single crystals under hydrostatic pressures were observed as a function of the time, in order to investigate the fundamental properties of the optical windows.

Apparatus and Procedures

The triaxial pressure testing apparatus were reported by Griggs²⁾, Handin³⁾, and others. In this experiment, the apparatus of the same principle as Griggs' was used, and shown in Fig. 1. It consists of three main parts; high pressure cylinder part C, hydrostatic pressure intensifier part H, and part of differential pressure loading axially, D. The cubic specimen (a) is set in the double cylinder combined with taper (b) of 6 mm inner diameter. Then, petroleum or silicone oil is filled around the specimen as a pressure transmitting liquid, and the oil is passable to the lower space through the small side grooves of the plate (c).

The part H of hydrostatic pressure intensifier consists of two pistons and cylinders, one of which has 4 mm in diameter (d), and the other 20 mm (e), and gives the hydrostatic pressure intensified according to the piston ratio to the specimen. Bridgman type sealings were used in their piston heads. The 4 mm diameter piston (f) contacted with the upper surface of the specimen is fixed to the frame (g), so that when the piston (e) is elevated, the specimen is stressed by the hydrostatic pressure.

Then, the differential pressure is applied by the axial loading part, the cylinder (b) moves upwards against the piston (f) by means of the piston (h) and the movable frame (i), consequently, axial pressure is applied to the specimen in addition to the above hydrostatic pressure.

The change of the height of the specimen is known from the piston displacement to the cylinder, and is measured by means of the optical lever (j)⁴⁾, an accuracy of which, 5 mm shift on the scale corresponds to 0.001 mm piston displacement.

1) R. Kiyama and K. Inoue, *This Journal*, **21**, 78 (1951)

2) D. T. Griggs and W. B. Millers, *Bull. Geol. Soc. Am.*, **62**, 853 (1951)

3) J. Handin, *Trans. ASME*, **75**, 315 (1953)

4) R. Kiyama, J. Osugi and H. Teranishi, *This Journal*, **24**, 81 (1954)

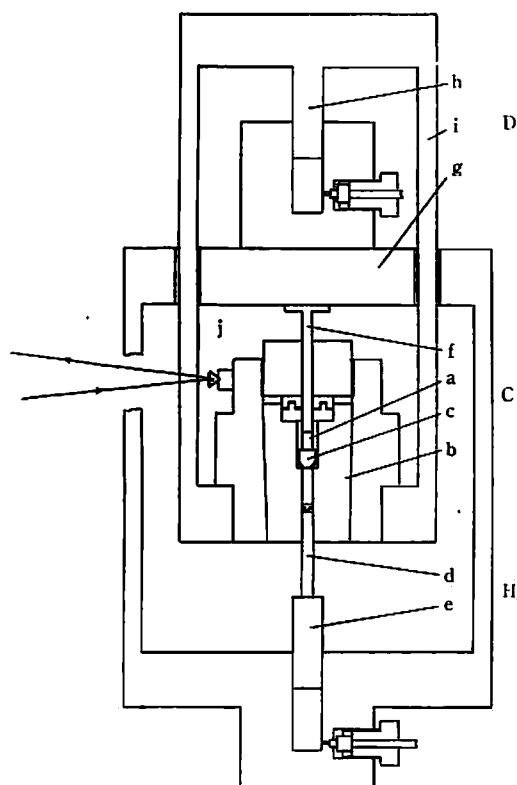


Fig. 1 Apparatus

D: differential pressure loading part
 C: high pressure cylinder part
 H: hydrostatic pressure intensifier part

The specimen of the crystals is of cubic form; its edge length is about 2.7 mm and each surface of the cube is parallel to the planes of cleavage. The accuracy of strain measurement corresponds to 0.04 per cent of that initial height.

The Bourdon type pressure gauges calibrated with a free piston gauge are used for the pressure measurements and their accuracy is 0.25 per cent of each measured pressure.

Results and Considerations

The strain-time relations of five kinds of alkali halide single crystals: lithium fluoride, sodium chloride, potassium chloride, potassium bromide, and potassium iodide were measured at definite constant differential pressures and under constant hydrostatic pressures of 200 and 1,000 kg/cm², and compared with the results under atmospheric pressure as shown in Fig. 2. In this figure, the higher the hydrostatic pressure, the lesser is the strain for the same kind of crystal at constant differential pressure. The decreases of the strain with the elevation of hydrostatic pressure were most remarkable in potassium iodide, and were hardly observed in lithium fluoride. The increase of the strength of material with hydrostatic pressure is larger in the ductile material.

The slope of the tangent of each curve shows the rate of deformation. The maximum differential pressures, where the rate may be taken as zero within one hour experiment, are not changed with hydrostatic pressures, and the maximum pressure values are about 50 kg/cm² for

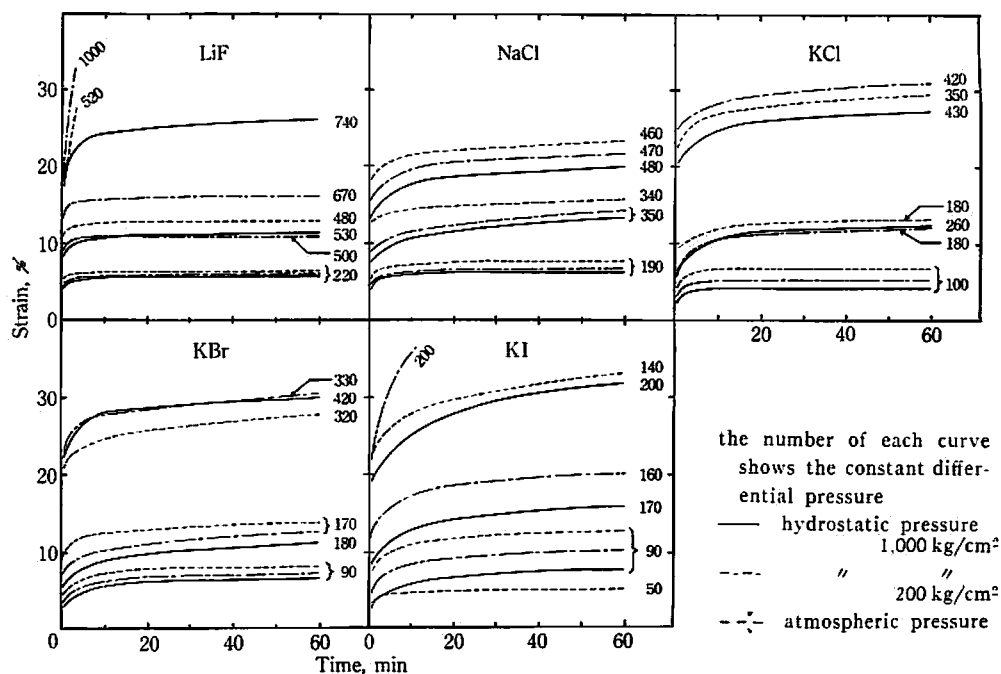


Fig. 2 Strain-time relations at constant differential pressure

potassium iodide, 100 kg/cm² for potassium bromide and potassium chloride, 200 kg/cm² for sodium chloride, and 500 kg/cm² for lithium fluoride respectively.

Fig. 3 shows the relations between differential pressure and strain after one hour loading cited from the curves in Fig. 2, and elucidates that the strain at the same differential pressure is the least for lithium fluoride and increases in order of sodium chloride, potassium chloride, potassium bromide, and potassium iodide.

For the constant strain value, the number of the cracks observed decreases in the above order. On lithium fluoride, many cracks are observed in the specimen and it becomes opaque when the strain exceeds 10 per cent, and crushes at about 20 per cent strain, but the other four kinds of crystals do not crush at less than 30 per cent strain, and on potassium iodide crystal, cracks are not observed even above 30 per cent strain. As for sodium chloride and potassium chloride, when above 10 per cent strain, it is found that the minute stripes run on the (100) and (010) surfaces of the specimen in the direction parallel to the loading axis [001]. In the middle part of the specimen, the cross section cut normal to axis exhibits a cross stripe pattern. The stripes observed on the surface are considered to be the lines intersected between the surface plane and gliding planes which are parallel to (110) and ($\bar{1}\bar{1}0$) planes. The uneven planes which do not exhibit clear stripes are observed in potassium bromide and potassium iodide under lower pressures.

As for sodium chloride, its plastic deformation was measured as a function of the time at finite axial pressures, and was shown in Fig. 4 where hydrostatic pressure did not apply. It seemed that the slope of strain against pressure is smaller at the fourth step than that at the first step.

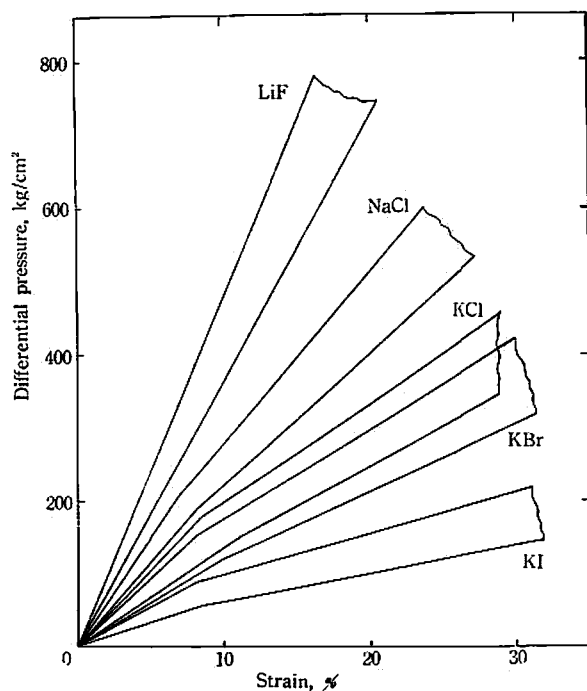


Fig. 3 Stress-strain relations after one hour loading

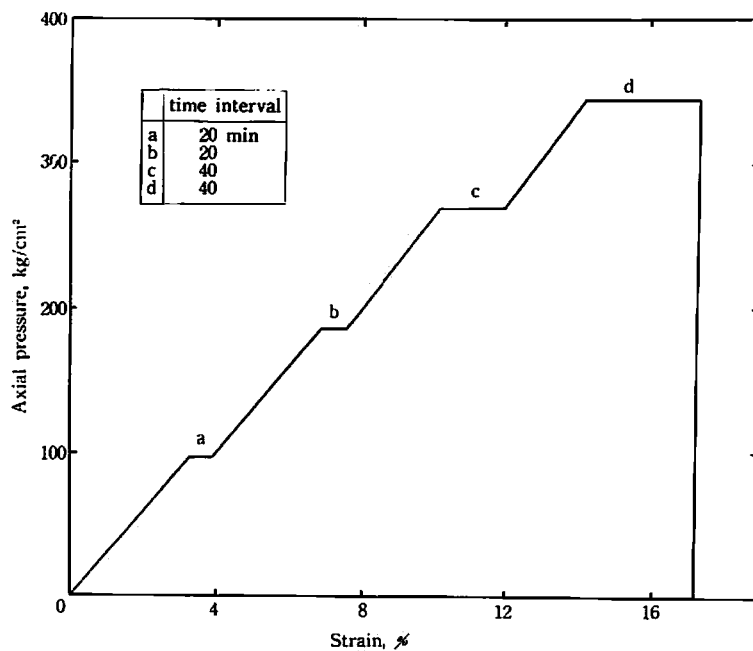


Fig. 4 Stress-strain-time relation of sodium chloride under atmospheric pressure

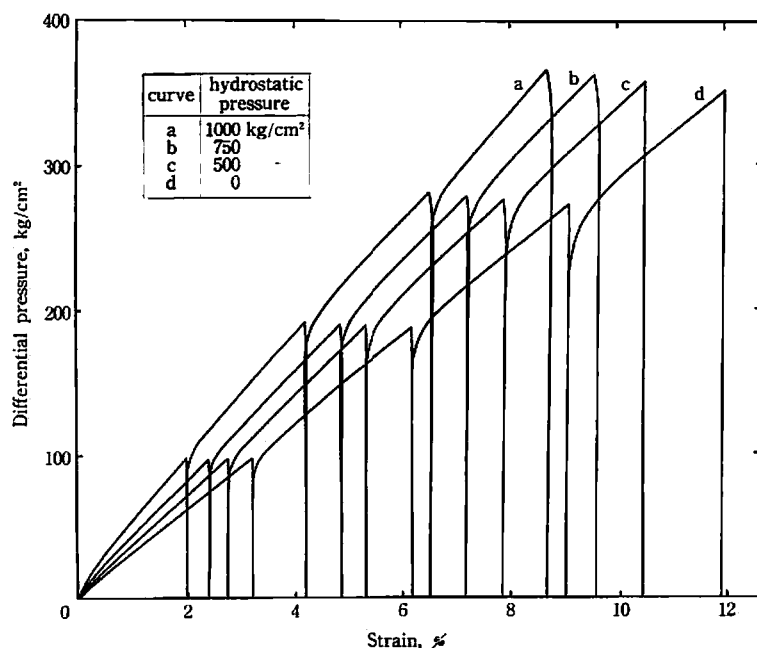


Fig. 5 Stress-strain relations of sodium chloride under several hydrostatic pressures

It may be considered that the specimen is influenced by "workhardening effect".

The results of the continuous compression on sodium chloride under three different hydrostatic pressures are shown in Fig. 5, and in each experiment, differential pressure is released at each elevation of 100 kg/cm², and reloaded continuously at each step. It is found that the amount of strain at fixed differential pressure is smaller with the increase of hydrostatic pressure as the results on the hydrostatic pressure effect mentioned above.

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