

improved value of the pressure at equilibrium between the two phases.

METHOD

The single crystal samples in the high pressure ambient were observed optically by means of single crystal sapphire windows in the pressure vessel shown in Fig. 1, similar to that described by

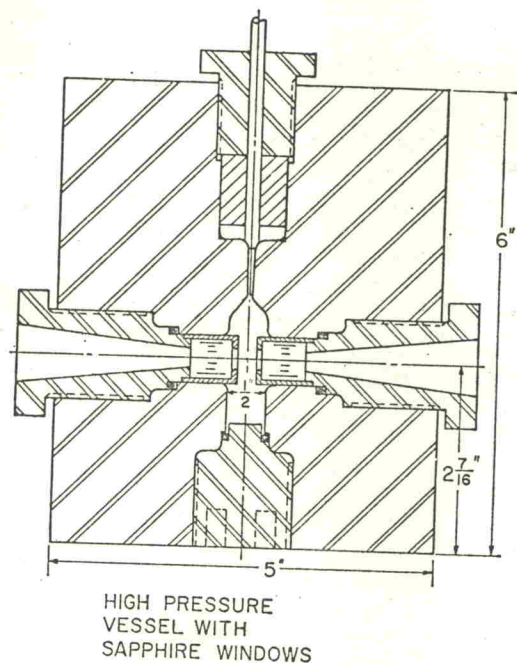


FIG. 1. High pressure optical cell with oriented single crystal synthetic sapphire windows. (Crystal "C" axis along cylinder axis.)

FISHMAN and DRICKAMER⁽¹²⁾ but using armored O-ring seals⁽¹³⁾ on the plugs and connecting with the pressure generator by 0.125 in. o.d. \times 0.26 in. i.d. type 316 ss tubing. The vessel has been used over a pressure range to 10 kb. Pressure was measured using a Manganin cell calibrated against the freezing pressure of mercury at 0°C, taken to be 7640 kg/cm² following Bridgman. The vessel was mounted on the stage of a Vickers metallographic microscope. The focal length of the Vickers' objective lens was lengthened using a double concave lens resting on top of the normal objective lens. The final magnification of the sample was 10–40 dia. depending on the eyepiece used. The single crystal sample rested unconstrained on a plexiglass frame in the pressure

vessel. Arrangements were made to take still, time lapse or moving pictures of the transforming crystal. The time lapse photography which permitted observations to be carried out continuously over periods as long as 48 hr was particularly useful at pressures only slightly above the equilibrium pressure, where the transformation proceeded quite slowly.

RESULTS

In crystals with contaminated surfaces (caused for example by long storage under oil), the observations qualitatively resembled those of JACOBS⁽¹⁰⁾ that is, at pressures well above the equilibrium pressure (i.e. at approximately 5000 b, the equilibrium pressure being about 3500 b) the crystals were observed suddenly to become opaque to white light. In some cases an audible click accompanied this phenomenon. As the pressure was then released, the crystal became approximately transparent again at pressures below 1500–2000 b. Crystals examined microscopically at atmospheric pressure, after having been transformed, revealed a definitely polycrystalline character.

Quite different behavior was observed in freshly cleaved or etched single crystals. In these the transformation was observed to start from relatively few centers at the surface of the crystals which grew to cover the entire surface of the crystal with a rough appearing layer before opaque regions in the interior began to spread through the volume of the crystal. These effects are shown on Figs. 2 through 6. The photographs were taken with transmitted white light using a microscope magnification of approximately 30 dia. The samples are cleavage blocks. The pressure was maintained at a constant value for this series of photographs. These photographs illustrate first the growth of the surface transformation in Figs. 2–5; then that of the bulk in 5 and 6. The crystal becomes completely opaque during final stages of the transformation, but clears slightly on long exposure to pressures above the equilibrium. Use is made of this latter phenomenon in establishing a value for the equilibrium pressure to be discussed later. By comparison with dimensions of samples and by differential focusing, the thickness of the transformed surface layer is estimated to be 150 μ .