B. STATIC MEASUREMENTS -- X-ray STUDIES

1. Introduction

High pressure X-ray diffraction studies have been performed with a camera similar to that described by Mariano. In this camera, high pressure is achieved between diamond anvils. A collimated beam is brought through one diamond. The diffracted rays fan out through the other diamond and infringe on a flat photographic plate of film. The apparatus is shown in Figure 27 and in Figure 28.

With powder specimens, Debye rings are formed and the interplanar spacing d may be calculated from the formula

$$d = \frac{\lambda}{\sqrt{2} \left[1 - \left(\frac{1}{1+x^2}\right)^{1/2}\right]^{1/2}} \tag{14}$$

where λ is the wavelength of the radiation employed and x is the ratio of the measured radius of the Debye ring to the sample-to-film distance.

The compressibility β_T is defined as $-(1/V)(\partial V/\partial P)_T$ where V is the volumen, P, is the pressure and T is the temperature, but this is just three times the linear dilatation $(1/d)(\partial d/\partial p)$ where d is an interplanar spacing. Differentiating the expression given earlier we can obtain

$$\beta = \frac{3x\,(\partial x/\partial p)}{2(1+x^2)[(1+x^2)^{1/2}-1]} \tag{a}$$

or for finite changes (15)

$$\beta_{av} = \frac{3x\,(\Delta x/\Delta p)}{2(1+x^2)[(1+x^2)^{1/2}-1]} \tag{b}$$

2. Experimental Techniques

To measure the compressibility we measure the line shift observed between exposure at different pressures. By superinforcing exposures at different pressures on the same film, errors due to changes in the sample-to-film distance or film shrinkage, etc. are avoided. The line displacement which is proportional to the compressibility is easily estimated to one-twentieth of a millimeter. Attempts have been made to resolve the shifted lines with a densitometer in order to achieve even greater precision. However, at convenient exposure levels this could not be done.

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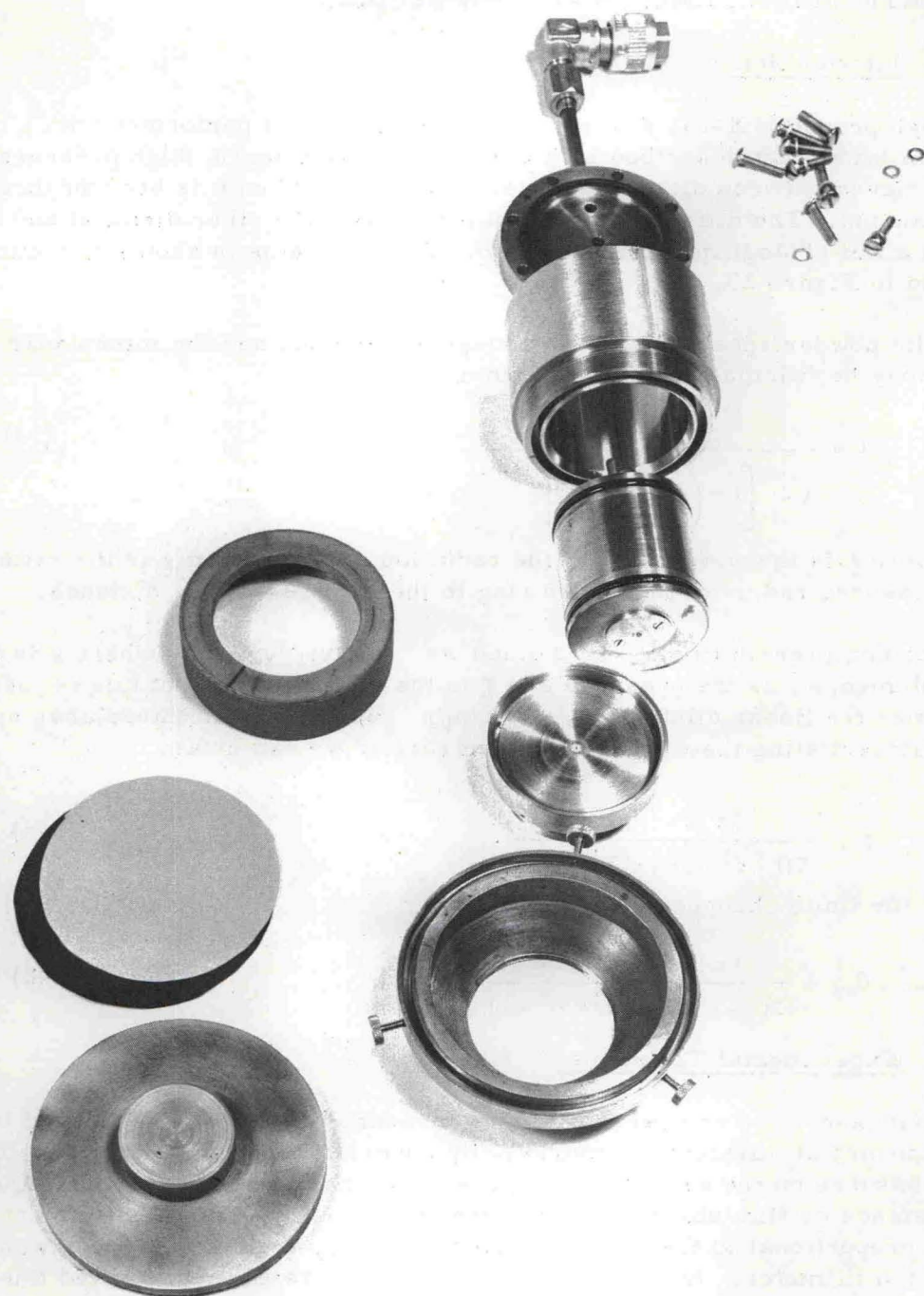
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Figure 27 EXPLODED VIEW OF HIGH PRESSURE X-RAY CAMERA