NON-DISPERSIVE HIGH PRESSURE HIGH TEMPERATURE X-RAY DIFFRACTION ANALYSIS

P. J. Freud and P. N. LaMori

Battelle Memorial Institute, Columbus Laboratories
Columbus, Ohio

INTRODUCTION

All current high pressure X-ray diffraction apparatus uses dispersive techniques. These techniques are characterized by use of monochromatic radiation and measurements of Bragg angles, and, in general, they can measure lattice parameters to only 0.1% and require exposures of 1/4 to many days. Accurate intensity measurements are obtainable in only a few systems.

Recently several semiconductor device manufacturers have reported a remarkable improvement in solid state detector systems for measurements of X-ray energies. Giessen and Gordon[1] reported use of a Li drifted silicon detector to analyze X-ray energy spectra. This technique is somewhat analogous to time of flight neutron diffraction. For X-rays the crystal analyzer is at a constant angle and analyzes the continuous energy spectrum with a multichannel analyzer. This technique can be used to accurately measure intensities and gather data at a faster rate than dispersive techniques on the same apparatus. In addition, lattice parameters can be determined with improved accuracy by simple curve fitting techniques.

The purpose of this paper is to describe the use of the energy analysis technique, especially with a particular high pressure apparatus. This apparatus has only recently been described in detail.[2] Design of this apparatus and its use for dispersive analysis will therefore be briefly described.

APPARATUS

Dispersive Technique

The high pressure cell is based on the high compression belt developed by Bundy.[3] In the application used here the belt die is split in half on a plane perpendicular to the die axis. A series of splits and fan shaped grooves are ground into the mating surfaces of the die halves to permit entrance and collimation of the X-ray beam, exit of the undiffracted beam and exit of

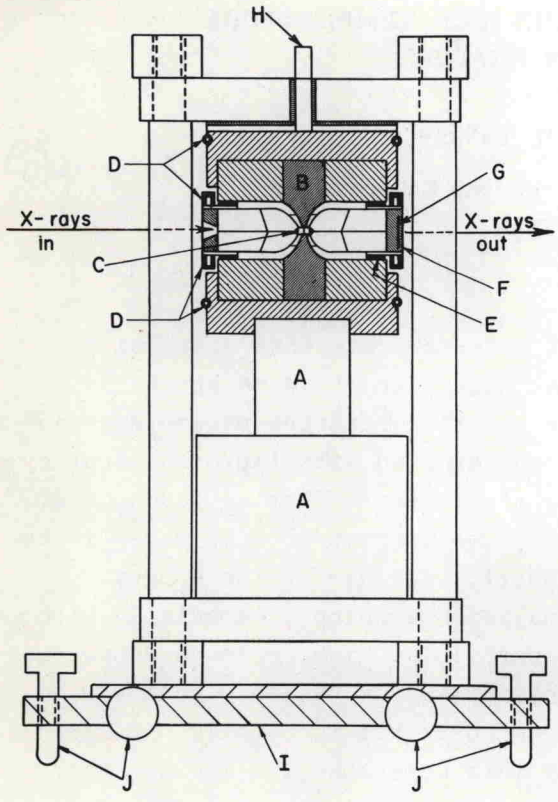

Fig. 1. Schematic drawing of the high-pressure apparatus. A--hydraulic 50-ton ram; B--WC pistons; C--die assembly; D--water cooling tubes; E--rubber shim to position die water-cooling tube; F--film cassette; G--X-ray film; H--insulated current lead; I--pressure positioning table; J--adjusting screws for vertical and horizontal positioning.

the diffracted beam. This apparatus is described fully in (2).

Figure 1 is a schematic of the high pressure assembly and shows the split die halves in place between the pistons. A fifty ton ram provides for force between the pistons and this force will produce over 100 kilobars pressure using 0.150 inch pistons and a 0.200 inch die bore. Since the assembly weights less than 100 lbs it can be placed on the top of a conventional microfocus X-ray source. Alignment is obtained by means of a positioning table on which it sits. This table provides lateral and vertical motion and rotation about the front of the entrance groove.

The unique feature of the design is the split die shown in cut-away in Figure 2. The inner piece is hardened tool steel (Carpenter Hampton RC 60), while the outer binding ring is high strength steel (Vasojet 1000, RC 52). After the two pieces are assembled and the mating surface is ground flat, the grooves and fans are ground into this surface. The entrance-exit groove is accurately placed on a diameter line of the die. Therefore, when the X-ray beam is detected emerging from the exit groove and its intensity maximized, the system is aligned. The sample is located on the axis of the die and since it is rigidly fixed to the die and the die geometry, misalignment will be caused only by lateral sample movement within the high pressure region. This type of movement is small because of the axial symmetry of the high pressure geometry.