## STRAIN AND PRECISION LATTICE PARAMETER MEASUREMENTS 3367

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ong the major pecific (hkl) resugh the entire altiple exposure e constructed it the film. If the of ellipses  $p_1$ , fich extrapolates If this process it is possible to as been located with the aid of ings of the ye

and inates are carried out with the aid of an automatic ording microphotometer (Rigaku-Denki MP-3). The reprofiles of the various ellipses of a family are traced talong the major axis and the peak-to-peak distances the profiles, measured with an accuracy of  $\pm 5 \mu$ , dethe end-points of the corresponding  $y_n$  coordinates. The  $x_n$  coordinates are controlled by fixed precision eacers which define the discrete film positions and hich span the range of specimen-to-film distance from s num down to virtually zero. The size of the ellipses shich can be recorded is limited by the dimensions of the film and the existence of a hole at its center. Therethe selection of the discrete film positions will be werned by considerations pertaining to the completion the elliptical patterns. For large values of  $y_n$  some octions of the ellipses may fall outside the film area and ensequently the selection of  $x_n$  will depend principally crystal orientation, wavelength used, and lattice cucing of planes which are being measured.

To insure accurate determinations of the  $x_n$  coordiites it is necessary to satisfy two requirements: (1) the im should be perfectly flat and (2) the film surface build be maintained normal to the direction of its disicement. The first requirement was fulfilled by ichining grooves in the backplate of the film cassette that a vacuum could be applied. Only when the film id been flatly pressed to the backplate through the oplication of the vacuum was the clamping frame ditened. The second requirement is satisfied by aciate machining of the cassette. Special care must be iden in mating the cassette base with the spacers to the errors in the determination of the discrete film without  $x_n$ .

It was observed that the precision of measurements of bed spacings increases with decreasing  $\alpha$ , that is, with breasing  $\theta$  values. Consequently, it is highly desirable bread a great many complete ellipses in the vicinity the center of the film. To minimize the interception the diffracted x rays at small  $\alpha$  values by the x-ray de itself, a long, tapered tip was fitted to the tube. This tip may be described as a truncated cone having a chapex angle of 7°, a circular tip surface of 0.16-cm 146-in.) diameter and a height of 4.45 cm (1.75 in.)

## b. Photographic Technique

A number of precautions in photographic processing are to be taken to achieve a high degree of precision. Is, for example, absolutely imperative that the film takage be uniform (isotropic) if the differences in pacings between various (hkl) reflections of a single are to be utilized as a basis for a subsequent strain disis After long experimentation it was found that Pont Cronar base, single-coated, graphic arts film X12 in.) satisfied the requirements for isotropic film base. Correction for film shrinkage is made by possing a standard scale on the exposed film and buring it after photographic processing. In order to minimize film shrinkage and background scattering, and thereby produce maximum contrast, the following photographic processing practice was adopted. All the films were developed for 2 min in Kodak HC110 developer diluted 5:1, followed by fixing in Kodak x-ray fixer for 6 min. Washing for six minutes and then immersion in Photoflo solution for 30 sec was followed by natural drying of the film. In this way it was possible to reduce film shrinkage to a minimum and also to insure that any dimensional change was uniform over the whole film.

## c. Computer Programming

A computer program was written to expedite the repeated computation of d spacings. The input to this program was: coordinates  $(x_i, y_i)$ , shrinkage factor, and wavelength used. The output was: d spacings and their corresponding standard errors.

In addition, a program was also written for the computation of the lattice parameter based on the method of weighted least squares. The input to this program was: d spacings, standard error of d spacings, and Miller indices of planes. The output was: the parameters of the weighted least-squares line and their associated standard errors. Mathematical details for the computation of the d spacings, lattice parameter, and respective errors are given in Appendix A.

The output of *d*-spacing computations was used for the computation of the stress-strain configuration of the strained crystals.<sup>1,2</sup> Indeed, it is primarily for reasons of attaining the highest degree of precision in the stress-strain analysis of crystals that the precision measurements by the divergent beam method were developed here in such detail.

## 4. EFFECT OF HOMOGENEOUS AND IN-HOMOGENEOUS STRAINS ON THE PSEUDO-KOSSEL PATTERN

If the crystal is subjected to long-range elastic strains or to homogeneous internal strains (residual strains), the shape of the pseudo-Kossel lines will be significantly altered. The homogeneous strains are then manifested by changes in the length of the major axis of the elliptical patterns. These changes in turn affect the slope parameters  $m_1$  and  $m_2$  and consequently the strain fluctuation can be recorded in terms of the changes of the d spacings of various (hkl) planes. Thus, if the changes of d spacings of more than six independent (*hkl*) planes are recorded, the complete strain distribution of the crystal can be obtained.<sup>1,2</sup> However, if the strains are inhomogeneous, then in addition to dimensional changes of the elliptical pattern local line broadening, kinking or displacement of line segments will occur. The effect of such strains on the pseudo-Kossel lines is shown in Fig. 4. From the schematic drawing of Fig. 1 it may be seen that adjacent areas on the specimen surface give rise to adjacent segments of .