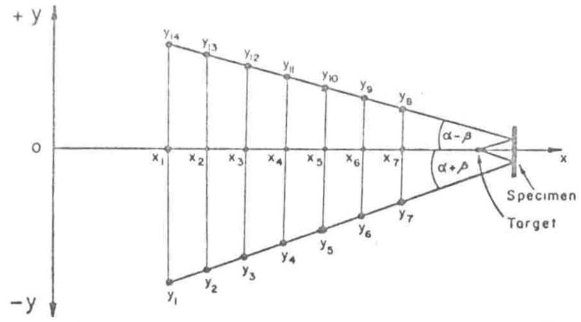

FIG. 3. Schematic representation of multiple exposure technique.

curately determined from measurements of the parameters of the corresponding ellipse.

It was found that the measurements of the film and specimen distance from the x-ray source constitute the principal source of error. These distances are shown in Fig. 1 as a and b, respectively. To eliminate this and other error sources, a number of innovations in experimental and measuring techniques were introduced which will be described presently.

It may be seen from Fig. 1 that the following direct relationship can be established between α, β and the slopes m_1, m_2 of the diffracted rays:

$$m_1 = p/c = \tan(\alpha+\beta), \tag{1}$$

$$m_2 = q/c = \tan(\alpha-\beta), \tag{2}$$

where α is the semiapex angle of the incident x-ray cone equal to $\pi/2-\theta$, θ being the Bragg angle, and β is the angle subtended by the normal of the reflecting (hkl) plane and the axis of the x-ray tube. $c=x_1-x_2$ is the distance between two consecutive film positions (Fig. 3).

If the slope parameters m_1 and m_2 of an (hkl) reflection are experimentally determined, one obtains the value of α and therefore the corresponding value of the Bragg angle θ from the solution of Eqs. (1) and (2). Subsequent substitution in the Bragg equation yields the corresponding d value.

The determination of the d spacing of an (hkl) set of planes is thus independent of the troublesome a and b parameters if the slopes m_1 and m_2 of the diffracted rays can be obtained. The principal innovation consists, therefore, of a precision determination of the slopes by a method of least squares employing a multiple exposure technique and exact measurements of distances between consecutive film positions. This is accomplished through the use of precision spacers. Thus in this method the film, after being exposed once, is moved a known distance (x_1-x_2) and a second exposure is taken. After repeating this procedure seven or eight times the film is processed in the usual way. Consequently, instead of a single ellipse, one obtains a pattern consisting of a family of seven or eight ellipses corresponding to one (hkl) reflection. Figure 2 represents such a multiple exposure diagram of an undeformed, zone-refined tungsten crystal in which the elliptical patterns have been recorded at eight different film positions (x_1 to x_8).

Figure 3 shows schematically the multiple exposure method. The points y_8 and y_7 are the intersections of the major axis with the ellipse produced during the first exposure and x_7 is the distance of the film from a fixed origin 0. Similarly, y_9 and y_6 are the intersections with the second ellipse and x_6 is the corresponding film distance, and so on.

If the equation to the line y_1y_7 is

$$y = m_1x + B_1,$$

one obtains by means of the least-squares method (see Appendix A)

$$m_1 = \sum y_i(x_i-\bar{x})/\sum(x_i-\bar{x})^2, \quad i=1, 2, 3\cdots 7.$$

But the slope of this line is also $\tan(\alpha+\beta)$, and

$$Km_1 = \tan(\alpha+\beta), \tag{3}$$

where K is the film shrinkage factor.

Similarly for the line $y_{14}y_8$,

$$Km_2 = \tan(\alpha-\beta). \tag{4}$$

By combining Eqs. (3) and (4),

$$\alpha = \tfrac{1}{2}[\arctan(Km_1)+\arctan(Km_2)]. \tag{5}$$

Also $\theta=\pi/2-\alpha$. Therefore

$$d = \lambda/2\cos\alpha. \tag{6}$$

3. EXPERIMENTAL TECHNIQUE

The precision measurements of the d spacings and lattice parameters by the divergent beam method depend on a number of factors which must be closely controlled and which will be presently discussed.

a. Film Measurements

It has been shown that the precision of the d values is greatly dependent on the accuracy of the slope parameters m_1 and m_2, which in turn depend on the accuracy of measurements of the y_n ordinates and x_n abscissas of the multiple exposure diagram (Fig. 3).

The y_n coordinates are measured along the major axis of the ellipse corresponding to a specific (hkl) reflection, this axis being extended through the entire family of ellipses generated by the multiple exposure technique. Before the major axis can be constructed it is necessary to determine the center of the film. If the points of intersection of two families of ellipses p_1, p_2 etc. are connected, a line is produced which extrapolates through the center of the film (Fig. 2). If this process is repeated for a number of intersections, it is possible to locate this center accurately. Once it has been located, a major axis is constructed on the film with the aid of precision dividers. The actual readings of the y_n