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LATTICE PARAMETERS OF KAMACITE BY THE KOSSEL TECHNIQUE

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ABSTRACT

Determination of the cell dimension by divergent beam X-ray diffraction was tested on kamacite from six meteorites.

Kamacite grains in enstatite chondrites were studied to test the feasibility of using divergent beam X-ray diffraction (Kossel technique) for determination of lattice parameters of micron-sized grains in ordinary polished sections. In this method, the electron-excited volume acts as a point source of divergent characteristic X rays corresponding to elements present in the sample. Because of low nickel content of kamacite, only FeK_{α_1} ($\lambda = 1.93604$ Å) and FeK_{α_2} ($\lambda = 1.93998$ Å) were of sufficient intensity to be recorded on film in our experiments. For X rays satisfying the Bragg relation, diffraction of divergent X rays from any lattice plane results in enhancement of X-ray intensity in the diffraction direction (ABC in Fig. 1), and in depletion in the transmission direction (ABD in Fig. 1) (for a detailed discussion, see Lonsdale, 1947). Diffraction and deficiency cones, with apices at the point source, are generated, and can be recorded as conic intersections on flat film placed below (transmission mode) or above (back reflection mode) the section. Back reflection conics are recorded as conic intersections (Kossel lines) on flat film positioned above the sample (Fig. 2). Transmission conics were not recorded in the present study due to thickness of the polished section.

Kossel patterns were obtained with an Applied Research Laboratories back reflection camera fitted to an electron microprobe X-ray analyzer. The electron beam, at an accelerating potential of 30 keV, was focused to a one-micron spot on the sample surface. Sample current was 0.31 μA and exposure time approximately five minutes. Lattice parameters obtained are for kamacite grains 50 to 300μm in diameter. Diffraction is, however, actually derived from a much smaller volume within the grain, thus allowing several patterns to be obtained from different areas on an individual micron-sized grain (excited volume in case of kamacite is in the order of 10^3 μm^3).

Kossel patterns were indexed by comparison with a computer-drawn

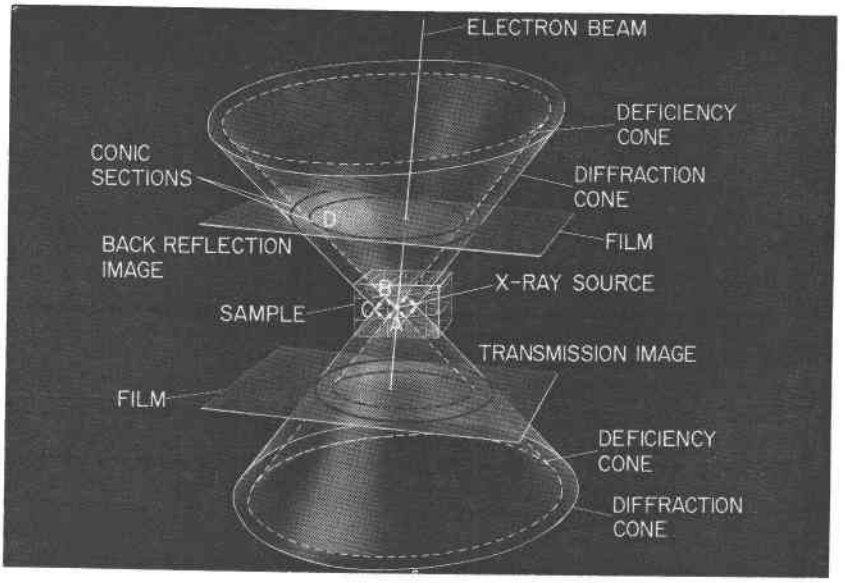

FIG. 1. Schematic representation of divergent beam X-ray diffraction using a focused electron beam to provide a "point" source (A) of divergent characteristic X-rays, whereby the sample itself serves as the X-ray source. Diffraction occurs when Bragg's equation is fulfilled for a given characteristic X-ray line, resulting in enhancement of X-ray intensity in the diffraction direction (ABC) and in depletion in the transmission direction (ABD). Diffraction and deficiency cones, with apices at the "point" source, are generated, and can be recorded as conic intersections on flat film placed below (transmission mode) or above (back reflection mode) the section.

stereographic projection of the pattern of kamacite (Frazer and Arrhenius, 1967). Patterns were measured and lattice parameters deduced by methods described in the Appendix. For each conic section on film, the coordinates of many points on the conic were measured in terms of an orthogonal coordinate system. The equation for the intersection of a diffraction conic with the film plane is uniquely determined by the lattice parameters of the crystal, by its orientation, and by the X-ray source-to-film distance (Morris, 1967; for details on the mathematics, see Appendix). By measuring several conics we produce a *highly over-determined* set of equations for these parameters, which are then solved by a computer routine. Thus it is unnecessary in our method to measure either the sample orientation or the source-to-film distance; the equations of the conic sections *determine* these quantities along with the lattice parameters. Moreover, the method provides a statistical estimate of errors from standard statistical techniques based on the degree of inconsistency of the over-determined equations. Range in errors reflects variations in

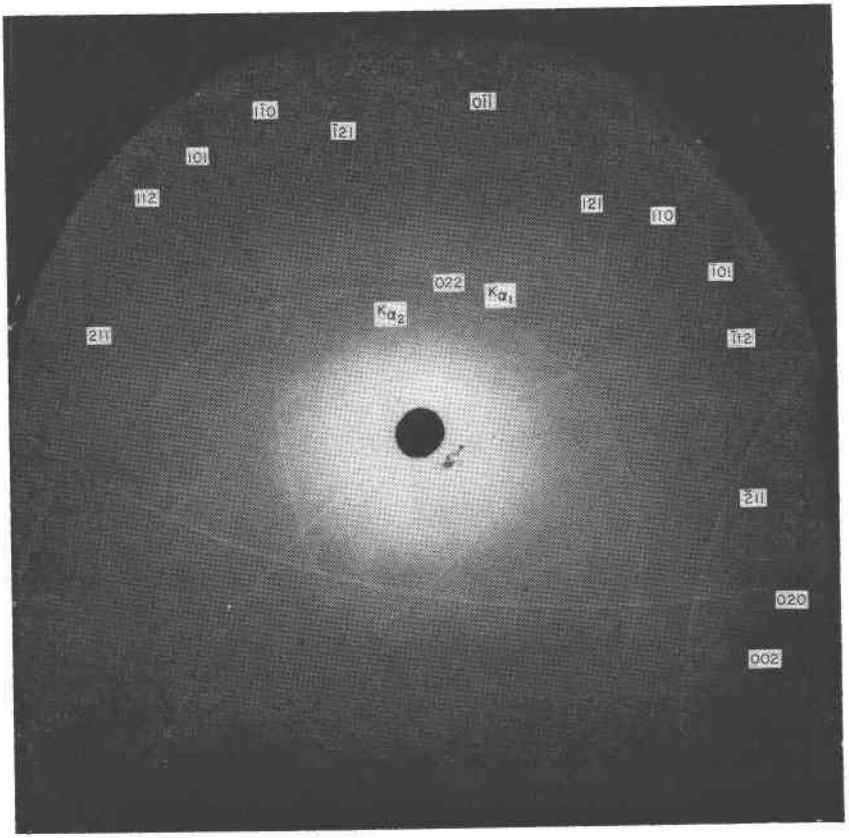

FIG. 2. Kossel pattern (negative) from a micron-sized area on a kamacite grain in a polished section of the Jajh deh Kot Lalu enstatite chondrite.

orientation of grains and in quality of patterns obtained. Kamacite compositions given are average values from electron microprobe analyses of 10–53 grains in each section. Lattice parameters can be calculated from these compositions using data of Owen and Burns (1939) on lattice parameters of kamacite of known nickel content, and of Farquar *et al.* (1945) on pure iron-silicon alloys. To make the calculation it is necessary to assume that relative contraction of the lattice due to presence of silicon is unaffected by presence of up to seven weight percent nickel. Agreement between calculated and measured values is considered satisfactory, as microprobe analyses were not made on the specific grains studied by divergent beam X-ray diffraction.

With cubic minerals such as kamacite it is relatively easy to obtain

TABLE 1. LATTICE PARAMETERS (a) OF KAMACITE IN ENSTATITE CHONDRITES

Meteorite	Measured a (Å)	Error (Å)	Calculated a (Å)	Composition, in wt. % (Average)	
				Ni	Si
Hvittis	2.86756	0.00061	2.8669	6.1	1.1
Khairpur	2.86759	0.00015	2.8670	6.8	1.2
Atlanta	2.86803	0.00080	2.8667	6.1	1.2
Jajh deh Kot Lalu	2.86717	0.00017	2.8666	6.2	1.3
Jajh deh Kot Lalu	2.86627	0.00056	2.8666	6.2	1.3
Indarch	2.86322	0.00071	2.8637	7.1	3.5
Indarch	2.86334	0.00045	2.8637	7.1	3.5
St. Marks	2.8614	0.0015	2.8632	6.0	3.6

useful Kossel patterns: indexing and measuring of films, and computerized data reduction to deduce lattice parameters can be done rapidly yet give results with adequate precision for most purposes (between about 0.0002–0.002 Å). For example, measuring a single pattern took about one hour and lattice parameter calculation involved about 25 seconds of computer time. Attempts are presently being made to apply the method to noncubic minerals and to define its limitations.

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Appendix:

Interpretation of Kossel Patterns

The first step in the method, calculating the interplanar spacing for each conic, was developed by Morris (1968). Using this approach we set up a coordinate system with the origin at the X-ray source and the z axis normal to the plane of the film. If t is the film-to-source distance and (x, y) a point on a conic section, then the equation of the conic section on the film is of the form

$$L_1x + L_2y + L_3t = \frac{\lambda}{2d}\sqrt{x^2 + y^2 + t^2} \tag{1}$$

where L_1, L_2 and L_3 are the direction cosines of the cone axis. The equation can be written

$$\xi_1x + \xi_2y + \xi_3t - \sqrt{x^2 + y^2 + t^2} = 0 \tag{2}$$

where $\xi_o = 2dL_j/\lambda$. For n points (x_i, y_i) on a given conic there are n simultaneous equations, and if $n > 3$ the set of equations is overdetermined. A least-squares solution is sought for the ξ's, minimizing the quantity

$$\chi^2 = \sum_{i=1}^{n} E_i^2 \tag{3}$$

where

$$E_i = \xi_1x_i + \xi_2y_i + \xi_3t - \sqrt{x_i^2 + y_i^2 + t^2} \tag{4}$$

Differentiating with respect to ξ_1, ξ_2, and ξ_3 the conditions for a minimum are

$$\sum_{k=1}^{3} H_{jk}\xi_k = \eta_j \tag{5}$$

where

$$\begin{aligned} H_{11} &= \Sigma x_i^2 & H_{12} &= H_{21} = \Sigma x_i y_i \\ H_{22} &= \Sigma y_i^2 & H_{23} &= H_{32} = t\Sigma y_i \\ H_{33} &= nt^2 & H_{13} &= H_{31} = t\Sigma x_i \end{aligned} \tag{6}$$

$$\eta_1 = \sum_{i=1}^{n} x_i\sqrt{x_i^2 + y_i^2 + t^2}, \qquad \eta_2 = \sum_{i=1}^{n} y_i\sqrt{x_i^2 + y_i^2 + t^2}, \qquad \eta_3 = t\sum_{i=1}^{n}\sqrt{x_i^2 + y_i^2 + t^2}$$

The computer solves for the ξ's and calculates the direction cosines and interplanar spacings for each conic section by formulae

$$d = \frac{\lambda}{2}\Big/\sqrt{\sum_{j=1}^{3}\xi_j^2} \tag{7}$$

$$L_j = \xi_j\Big/\sqrt{\sum_{j=1}^{3}\xi_j^2} \tag{8}$$

Morris' treatment can be extended by calculating the error in d for each conic section by the standard statistical formula (see, for example, Orear, 1958).

$$(\Delta d)^2 = \frac{\chi^2}{n-3}\cdot\frac{\sum_{i,j=1}^{3}(H^{-1})_{ij}\xi_i\xi_j}{\sum_{i=1}^{3}\xi_i^2} \tag{9}$$

A weight is then assigned to each conic on the basis

$$W = \frac{1}{(\Delta d)^2(h^2 + k^2 + l^2)} \tag{10}$$

For cubic crystals each conic section gives a value of the lattice parameter a from the formula $a^2 = d^2/(h^2+k^2+l^2)$.

The computer then determines t by a search routine which minimizes the variance in the values of a found from each conic section. The weighted average of these a values is the most probable value of a, and their standard deviation is the error in a.