

Acta Cryst. (1963). **16**, 705

Computer programs for analysis of broadening of diffraction peaks.* By R. J. DE ANGELIS and L. H. SCHWARTZ, *Materials Research Center and Department of Materials Science, The Technological Institute, Northwestern University, Evanston, Illinois, U.S.A.*

(Received 29 November 1962 and in revised form 16 January 1963)

Stokes (1948) has shown that it is possible to eliminate the effects of instrumental broadening from a diffraction profile, by representing the broadened peak and the instrumental profile by Fourier series. Digital computer programs for this purpose are described below; they

have been used successfully for two years on both IBM 650 and 709 machines, and have been used independently by one other laboratory.

The cosine coefficients for the annealed (instrumental) and broadened functions are respectively:

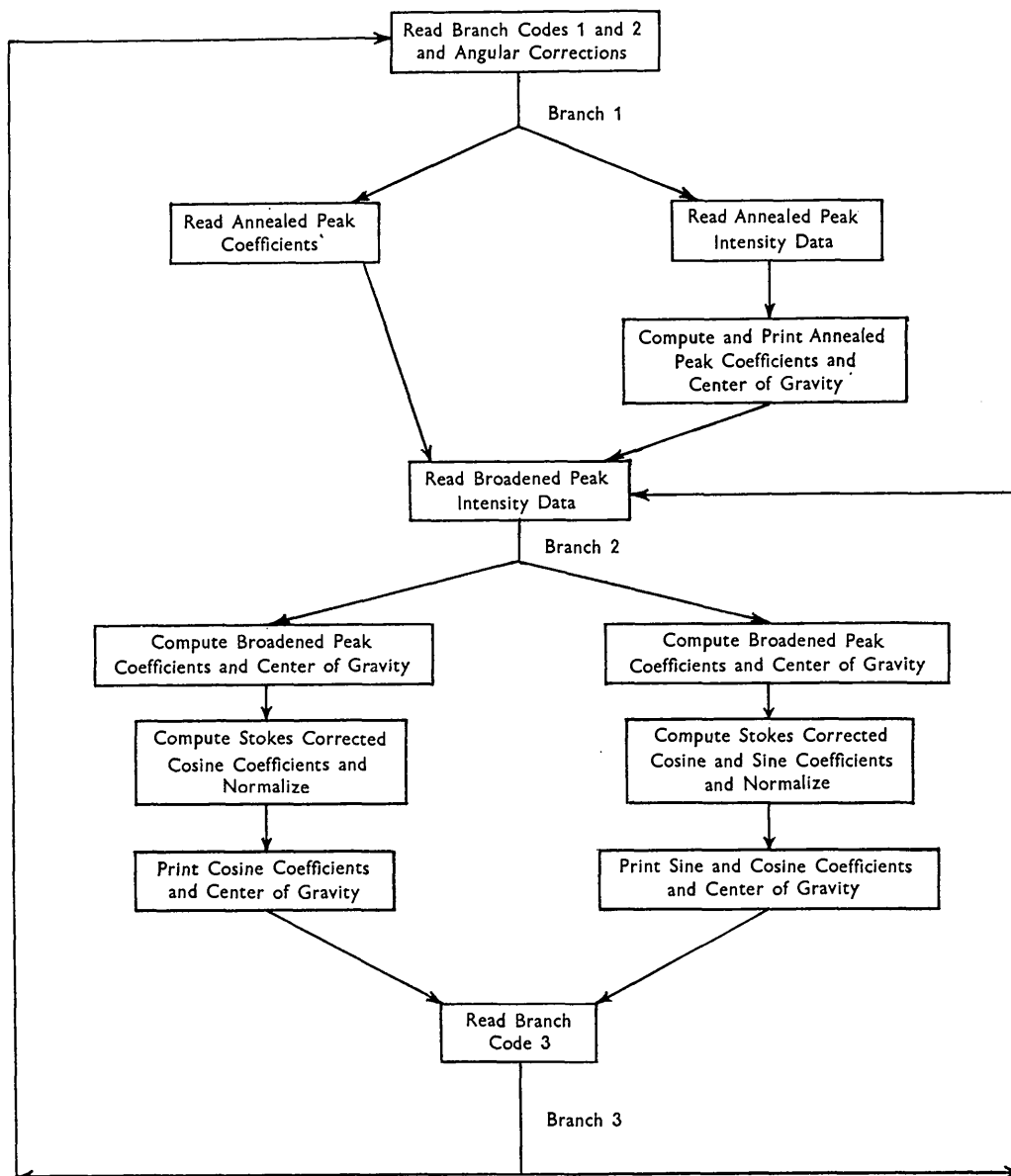


Fig. 1. Flow sheet of program for IBM 709 to determine Fourier coefficients correct for instrumental broadening and compute centers of gravity of X-ray diffraction peaks. All branches are externally controlled by code words read into the computer at the outset of execution. (With the IBM 650 no centers of gravity are calculated.)

$$G_r(n) = \sum_{j=-Y/2}^{Y/2} g(j) \cos 2\pi nj/Y \quad (1)$$

and

$$H_r(m) = \sum_{k=-X/2}^{X/2} h(k) \cos 2\pi mk/X \quad (2)$$

where the annealed peak has been divided into Y intervals, and the broadened peak has been divided into X intervals. Similar expressions can be written for the sine coefficients $G_i(n)$ and $H_i(m)$. $g(j)$ is the observed power of the annealed peak at the angular position corresponding to the j th interval, corrected for factors which vary with angle, and $h(k)$ is the observed power of the broadened peak at the angular position corresponding to the k th interval.

In the application of Stokes's method of correction for instrumental broadening, coefficients of the annealed and broadened peaks must be compared for equal values of the true length normal to the diffracting planes, L — i.e. for $na_3 = L \langle \text{annealed} \rangle \equiv L \langle \text{broadened} \rangle = (ma_3)$. The coefficients corrected for instrumental broadening are:

$$F_r(L) = \frac{G_r(L)H_r(L) + G_i(L)H_i(L)}{G_r(L)^2 + G_i(L)^2} \quad (3)$$

$$F_i(L) = \frac{G_r(L)H_i(L) - G_i(L)H_r(L)}{G_r(L)^2 + G_i(L)^2}$$

a_3 (or a'_3) is defined as follows: Let θ_0 be the angular position of the origin of the peak ($j=0$) and θ_2 be the angular position of one of the extremes of the peak ($j=Y/2$). Then a_3 is given by:

$$\frac{2a_3}{\lambda} (\sin \theta_2 - \sin \theta_0) = \frac{1}{2} \quad (4)$$

where λ is the wavelength of the radiation used. Peaks of the same hkl from several samples may be analyzed at once, with the same instrumental function. The value of a_3 is determined for the broadest peak and this is divided into $X=60$ intervals. (Simple changes in the program would enable X to be changed to any value.) The same value of a_3 is used for all other broadened peaks (and the annealed standard); however for these peaks the number of intervals X (or Y) is taken to be any even number ≥ 60 such that $h(k)=h(-k)=0$ at $k \geq 30$ ($g(j)=g(-j)=0$ at $j \geq 30$). For the less broad peaks the data points are thus taken at closer spaced intervals, more adequately describing the details of these.

* This research was sponsored by ONR, and by the Advanced Projects Agency of the Department of Defense under Contract SD-67 with the Northwestern University. One of the authors (L.H.S.) also acknowledges a fellowship by the Standard Oil Foundation.

Letting $b=Y/X$, equation (1) becomes:

$$G_r(n) = \sum_{j=-bX/2}^{bX/2} g(j) \cos 2\pi n(j/b)/X \quad (5)$$

Comparing equations (2) and (5), one sees that:

(a) Although the summation index (k or j) runs only from -30 to $+30$ in the computer, the sums are complete, since $h(k)$ and $g(j)$ are zero for $|k|$ and $|j| \geq 30$.

(b) The harmonic numbers m and n are now equal. Thus the Stokes correction can be applied to coefficients of equal harmonic number, this being equivalent to equal L values.

As shown in the flow diagram (Fig. 1), several externally controlled branches have been included. The programs correct for angular factors affecting the power variation (f^2e^{-2m} and Lorentz polarization) with a linear approximation between the value of the product of these terms at the peak's center and at the positions of the tails. The three values calculated for angular positions taken from the broadest peak in an hkl set are input data, with the actual observed powers for each interval for each peak. The running times to compute the corrected coefficients of one peak on the 650 and 709 are 8 and 0.15 minutes respectively. No separate wiring boards are required. Subroutines could be easily added at the beginning of the program to take printed or punched data and accomplish a background correction in the machine rather than by hand, or at the end of the program to synthesize the corrected peaks or to carry out analysis of multiple orders for strain and particle size if the broadening is due to deformation (Warren, 1959). The centers of gravity may be used to calculate twin fault probabilities (Cohen & Wagner, 1962) or for lattice parameters.

The authors know of other programs for this problem written by B. Roessler, and E. R. Boyko & G. J. Mohn (Westinghouse Research Laboratories), S. Allen (Instrumentation Laboratory, M.I.T.), W. P. Evans (Caterpillar Tractor Co.) and G. R. Mallett (Dow Chemical Co.).

Detailed instructions and/or a completed program in FORTRAN or machine language can be obtained from Prof. J. B. Cohen, Department of Materials Science, Northwestern University.

The authors acknowledge the assistance of the Northwestern Computing Center.

References

- COHEN, J. B. & WAGNER, C. N. J. (1962). *J. Appl. Phys.* **33**, 2073.
 STOKES, A. R. (1948). *Proc. Phys. Soc.* **61**, 382.
 WARREN, B. E. (1959). *Prog. Metal Phys.* **8**.