

16.5-2 TRUNCATION CORRECTION IN THE FOURIER (WARREN-AVERBACH) ANALYSIS OF DIFFRACTION LINE PROFILES FOR THE DETERMINATION OF ACCURATE CRYSTALLITE SIZE AND MICROSTRAINS. J.I.Langford, *University of Birmingham, Birmingham B15 2TT, U.K.*, R. Delhez, Th.H de Keijser & E.J. Mittemeijer, *Delft University of Technology, 2628 AL Delft, The Netherlands*, and D. Louër, *University of Rennes, 35042 Rennes, France*.

Several authors have drawn attention to large differences between estimates of crystallite size obtained by the Fourier (Warren-Averbach) and the variance methods. A main source of error is the unavoidable truncation of the broadened profile, for which a correction to the variance has recently been devised. As was pointed out by Young, Gerdes and Wilson, typically truncation can result in estimates of size which are too large by as much as 20-25% in the Fourier method and the values of strain can also be seriously affected.

Truncation influences the Fourier cosine coefficients in two ways. The omission of part of the tails of a profile increases and diminishes alternate coefficients slightly and the finite maximum range in real space gives rise to a sampling interval in Fourier space. A few procedures have been proposed in the past to "correct" the Fourier coefficients for truncation - "Hook-effect" corrections. In this work a systematic truncation correction in Warren-Averbach analysis has been developed. In the variance method the truncation correction is based on the approximate s^{-2} variation of intensity in the tails of a profile, where s is the distance, in reciprocal units, from the centroid. The same assumption leads to a method for correcting the observed Fourier coefficients to take into account the unrecorded part of the profile tails. The correction contains a constant that can be found from the measured intensities.

The truncation correction has been applied to four cases: (a) the theoretical profile due to small tetrahedral crystallites, with 6% of the integrated intensity lost by truncation, (b) data for the 103 line, truncated at 4%, for a sample of ZnO powder which exhibits only small-crystallite broadening, (c) the 200 and 400 lines, truncated at 2% and 4%, for cold-worked nickel filings and (d) the 110 and 220 lines, truncated at 2% and 4%, for an iron catalyst powder. Experimental data were used for (b) to (d); (c) and (d) include both size and strain broadening. General conclusions are: (i) In cases (a) and (b), with pure size broadening, after correcting for truncation the Warren-Averbach estimates of size are equal to the sizes obtained from the variance method, and in (a) are equal to the true size. Differences between size values before and after correction amount to as much as 30%. (ii) In cases (c) and (d), with both size and strain broadening present, it is difficult to make a direct comparison between the results of the two methods, as the variance analysis is normally applied to a single line. However, if in the latter approach the strain is assumed to be entirely due to dislocations, then the sizes from the two methods are in reasonable agreement.

The proposed correction procedure thus removes a large systematic error in the Warren-Averbach method for line-broadening analysis, which then gives estimates of crystallite size which are comparable in accuracy with those obtained from the corrected variance. It is recommended that the truncation correction is applied routinely when using the Warren-Averbach method.

16.5-3 A TWIN COUNTER X-RAY GUINIER DIFFRACTOMETER FOR STRUCTURE RESEARCH BETWEEN 12 AND 700 KELVIN. By J. Ihringer and W. Appel, *Institut für Kristallographie der Universität Tübingen, West-Germany*.

A previous version of the diffractometer, capable of cooling the sample from 300 to 12 K with a single counter, was described elsewhere (J. Ihringer, *J.Appl.Cryst.* (1982) 15, 1). By the use of an additional heater inside the cryostat a temperature range from 12 to 700 K is available; measuring time is reduced by a second counter.

Figure 1 shows the heating/cooling device. A two-stage closed-cycle helium refrigerator is used for cooling. The first stage is attached to a 70 K radiation shield. Within the range of 12-300 K temperature is controlled by heater 1 only. At higher temperatures the sample is heated by a small furnace in front of it (heater 2); the surroundings still remain at room temperature. The energy of heater 2 is absorbed by the cooling engine to avoid heating of the whole cryostat.

A heating/cooling rate in the low temperature range from 0.5 to 160 K/h and 0.5 to 600 K/h in the high temperature range can be achieved. Under any conditions movement of the sample is possible. The sample chamber can be evacuated or filled with a gas (pressure up to 0.2 MPa).

Measurements can be made by the twin counter or a film lift device. The counters are driven by a single stepper motor. A θ -range from 0 to 45 degrees is available with a smallest step-width of 0.0025 degrees. The difference of angles between the counters is adjustable within 12 to 45 degrees.

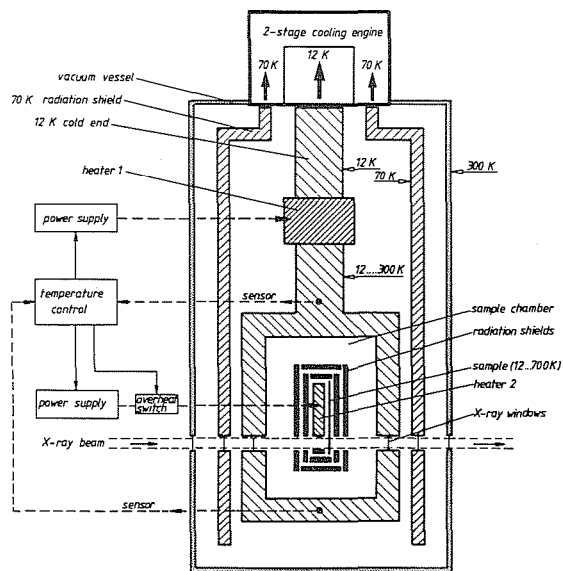


Fig.1 Schematic drawing of the heating/cooling device (Working range: 12...700 K)