CHAPTER 3

EXPERIMENTAL PROCEDURES FOR LINE PROFILE ANALYSIS
3.1 Specimen preparation

In the present investigation X-ray line profiles have been recorded from spectroscopically pure h.c.p. metals zirconium, titanium, magnesium, zinc, b.c.c. metal molybdenum and from four compositions of f.c.c. silver-gallium and copper-gallium alloys.

Silver and copper base gallium alloys were prepared from the spectroscopically pure silver, copper and gallium metals obtained from Messrs. Johnson, Mathey, and Co. Ltd., London. Quantities of the component metals in accurate weights and sufficient to give 6-8 gms of ingots were sealed in 'VIT' quality quartz capsules under vacuum and melting was done in an induction furnace. Alloys in the f.c.c. phase were prepared for different solute concentrations as per phase diagram and homogenised at different periods at the high temperature region. To account for the loss during preparation and homogenization treatment the actual compositions were calculated from subsequent lattice parameter measurements.

Cold working was achieved by careful hand filing of pure metal rods and prepared ingots in case of alloys by jeweler's file at room temperature (Goswami, Sen-Gupta and Quader, 1966; Sen Gupta, 1967). The drastic form of cold-work can be achieved by this process. The filings were then made free from iron particles if any, by moving a small permanent magnet over the filings for some time. A portion of the filings was always retained in the 'as-filed' condition representing the cold-work state and another was annealed.
under vacuum in sealed quartz capsules at temperatures usually near the temperatures of re-crystallisation for considerable period of time representing the fully recovered state (i.e., free from strains, small domain sizes etc with a negligible concentration of imperfections). To study the recovery processes for h.c.p. metals zirconium and titanium a separate portion of the filings was annealed at some intermediate temperature. The fully annealed samples serve also as a 'standard' for the correction of the instrumental effects. In case of alloys, each composition was treated separately. The filings from both cold-worked and annealed samples were carefully sieved through a 250 mesh screen, and samples of the filings were poured into the rectangular cavity of the specially designed aluminium specimen holder and bonded with a solution of canada balsam in xylene. Care was taken so that the filing surface in holder facing X-ray beam remained uniform and flat. This is a pre-requisite for maintaining the parafocussing diffraction geometry. The filings therefore present accurately flat surface (20 x 12 mm) to the incoming X-ray beam through the soller and divergent slit assembly of the goniometer part of the diffractometer.

3.2 Peak recording and background estimation

The line profile analysis requires a precise record of intensity distribution of diffracting X-rays from particular reflecting planes and this can be best done by the modern counter diffractometer technique which replaces the earlier photographic method.
In the present investigation the standard Philips Geiger counter X-ray diffractometer (Type No. PW 1060, 1051) with Nickel filtered CuKα radiation from the highly stabilised Philips X-ray generator (Type No. PW 1010) operating at 30 KV and 16 mA has been employed throughout, to record the diffraction line profiles from the samples under investigation. The chart recording done initially to locate the peak position and range of scanning, was followed by point counting done manually at intervals of 0.1° in 2θ for the general background decreasing to 0.01° in 2θ near the maxima of the peaks where θ is the Bragg angle. The slit system used for the purpose is a 1° divergence slit, scatter slit of 1° and a 0.1 mm receiving slit.

For the correct estimation of background level which plays an important role in the line profile analysis, the point counting was done to a considerable extent on both sides of the peak maximum position (usually this range extends up to 5° to 6° in 2θ on either side). For overlapping profiles the background level is determined from the average of the recordings to the left of one reflection (i.e., lower 2θ region) and to the right of the other (i.e., higher 2θ region). There is, however, always a possibility for over estimation of the background level due to the overlapping of long tails and also due to the fact that for small particle sizes the tails never reach zero (Warren 1959). As has been pointed out by Warren (1969), the recording from well-annealed sample done exactly under the similar instrumental conditions for the cold-worked sample serves the purpose of locating the actual
background level for the samples under investigation. However, in case of copper samples it was not possible to locate the background level from the annealed pattern since the background level was found to be higher due to excited fluorescent radiation from the sample for copper radiation. In these cases, background level was estimated from individual recordings of the profiles.

The background estimation was further examined from a method due to Sato (1962). According to this later method, there should exist a constant ratio between the background levels of the cold-worked and corresponding annealed patterns. The iterative method due to Mitra and Misra (1966) does not, however, yield much better estimation than that obtained from annealed pattern.

3.3 Peak position measurements

There exists some controversies as regards the best description of the position. While certain groups prefer the centroid (c.g.) of a peak as defined in the last chapter to be the real peak position, Warren (1969) and many other workers are of the opinion that the peak maximum (P.M.) position is usually given by extrapolation of the centres of section lines drawn parallel to the background level towards the top part of the peak. Since consideration of c.g. to describe the peak position neglects the asymmetry effects of a profile due to the presence of twin or extrinsic stacking faults in f.c.c. and b.c.c. structures (chapter 2), the latter method due to Warren (1969) appears to be more realistic and hence has been adopted here for peak position measurements.
There also exists a mathematical approach of least-squares fitting of a parabola to the top part of the peak (Wilson, 1965). However, fitting a parabola to three points with intensities $P'(S_k)$ at

$$S_k = S_1 + (k-1)\Delta S$$

where $k = 1, 2, 3$ and $\Delta S = S_2 - S_1 = S_3 - S_2$

($S_k = 2S_{ne}/n$) leads to the following equation for the calculation of $S_0$, the peak maximum position (Wagner, 1965):

$$S_0 = S_1 + \frac{\Delta S/2}{\left[4P'(S_2) - 3P'(S_1) - P'(S_2)\right]/\left[2P'(S_2) - P'(S_1) - P'(S_0)\right]}$$

The peak position obtained from this parabolic method of fitting have been found to be very close (within the range of experimental error) to those determined from the conventional method of mid-point extrapolation of parallel chords. The peak positions have been measured for unresolved profiles and the error in position measurements has been discussed in the Part II.

3.4 Peak centroid (C.G.) measurements

The determination of centroid (C.G.) of a diffraction profile (unresolved) as defined in chapter 2 has been done by the method of numerical integration employing Simpson's rule. As this parameter is range dependent, the ranges have to be chosen properly. The usual practice is to consider the entire diffraction profile with the truncation ranges governed by the Stokes' method of Fourier analysis, discussed in the following section. However, there appears considerable difficulty when the precise truncation limits
can not be chosen due to higher background level of the profiles from cold-worked samples. In those cases, the centroid is calculated for extended ranges of truncation and the determination of actual centroid is made from the position when this does not change appreciably with the range.

The error in centroid calculation due to improper truncation, over estimation of background and other statistical fluctuations has been analysed theoretically by Wilson (1967, 1968).

A simplified and sufficiently accurate method for the determination of centroid within finite truncation limit has been given by Thomsen (1968). According to this method, the true centroid \( C \) is given by:

\[
C = C_1 / (1 - \tau)
\]

where \( C_1 \) is the centroid determined with truncation range 2\( \nu \) given by:

\[
C_1 = \frac{\int_{-\nu}^{\nu} v f(v) dv}{\int_{-\nu}^{\nu} f(v) dv}
\]

and

\[
\tau = \frac{\sqrt{\int_{-\nu}^{\nu} f(v) dv} + \int_{-\nu}^{\nu} f(-v) dv}{\int_{-\nu}^{\nu} f(v) dv}
\]

Physically, this \( \tau \) is the ratio between average height at the end points \( \pm \nu \) to the average height over the entire range \(-\nu \leq v \leq \nu\). This method has, however, been found to yield \( C \approx C_1 \) as \( \tau \) is very small.
The numerical integration has been done using a suitable program on IBM 1130 and the error in centroid measurements has been discussed in Part II.

3.5 Peak broadening analysis: Fourier coefficients

In most of the cases with pure metals and alloys the diffraction profiles from cold-worked samples were so broad and diffuse with long tails that Rachinger's (1948) method of correction for doublets could not be applied with sufficient accuracy. As such the Stokes' method of instrumental broadening correction was applied directly on the unresolved profiles taking into consideration the well-annealed respective specimens as 'standard'. The details of the Stokes' (1948) method have already been described earlier in chapter 2. In order to apply this method the broad profiles from cold-worked samples were normally divided into large number of (usually 150 to 200) equal intervals in $2\theta$ (less than $130^\circ$, so that $\sin \theta \approx \theta$). Care was taken so that the intensity falls to the background level near the truncation limits $2\theta_2$ and $2\theta_1$ on both sides of the peak. This reduced considerably the possibilities of truncation error in Fourier analysis of the line shapes. A total of 151 or 201 ordinates were then tabulated in the form $Y_j$, $Y_j + Y_{-j} (A)$, $Y_j - Y_{-j} (A)$. To evaluate the coefficients $H_r(n)$ and $H_i(n)$ (section 2.4.3) for the profiles of the cold-worked samples, these coordinates then represent the amplitudes $A$ of the functions $A \cos 2\pi n \lambda / 150$ or $A \cos 2\pi n h / 230$ for $H_r(n)$ and $A \sin 2\pi n h / 150$ or $A \sin 2\pi n h / 230$ for $H_i(n)$. Here $n$ corresponds to the harmonic
number varying from 0 to 20 depending on the interval \(a\) to give the column length \(L\) in \(\AA\) and \(\frac{1}{\sqrt{\text{iso}}}\) or \(\frac{1}{\sqrt{\text{iso}}}\) intervals for the ordinates of the cold-worked peak. The values of \(h\) vary from 0 to 75 or 0 to 100 in the respective cases.

To evaluate the coefficients \(G_{P}(n)\) and \(G_{Q}(n)\) for the profiles from annealed samples, a smaller interval usually one-half or one-third of that of the corresponding cold-worked peak was used. Respective Fourier coefficients and the Stokes' corrected \(F_{P}(n)\) and \(F_{Q}(n)\) coefficients were then evaluated using a suitable program of Fourier summations on an IBM 1130 Computer.

It is often advantageous to apply Stokes' correction over the resolved cold-worked peak from the unresolved \(k_{p}k_{q}\) doublet by adopting Rachinger's (1948) method. This is done especially when the interest is in the essentially symmetrical cold-work broadening. Warren (1959), however, has pointed out that it is safer to apply directly the Stokes' correction without applying Rachinger's correction when the interest is in the small peak shift and small peak asymmetry. In Figures 3.1 and 3.2 respectively the 111 diffraction profiles from cold-worked and annealed samples of Cu-15% Ga, are shown with the corresponding truncation ranges and the intervals into which the profiles are divided. In Table 3.1 number of divisions \(j\), \(\gamma_{j} + \gamma_{j-1}\) and \(\gamma_{j} - \gamma_{j-1}\) are given for 111 profile from both cold-worked and annealed samples, Table 3.2 shows the cosine coefficients \(A_{n}\) and sine coefficients \(B_{n}\). This table shows that \(B_{n}\) is negligibly small in comparison to the \(A_{n}\) coefficients, and justifies the reason for neglecting these coefficients.
3.6 Effect of origin, truncation limit and other systematic factors in Fourier analysis

In Fourier analysis the choice of origin is not of much importance so long as it is the same value in sine for the cold-worked and annealed peaks. It is usually preferred that the origin should be chosen close to the centre of gravity of the annealed peak to keep sine coefficients small. In some cases of our present investigations, particularly for the cases with alloys, there was a large displacement of the peak position for cold-worked samples due to an overall expansion or contraction of the lattice or due to the presence of stacking faults and residual stresses. Hence, separate origins for the profiles of the annealed and cold-worked samples have been taken.

The effect of different errors in the X-ray line profile analysis has been considered by many workers. Among them Bertaut (1952), Estabrook and Wilson (1952), Kukol (1962), Wilkens and Hartman (1963) and Young, Gerdes and Wilson (1967) have considered the effects of background, truncation error, and sampling of observed profile at finite number of points. Young et al (1967) have shown that error in background estimation, or truncation of the observed profile, or the inadvertent truncation error arising due to the over estimation of background level affect the cosine coefficient $A_0$, introducing an oscillatory nature in $A_n$ coefficients with a 'hook effect'. It has been observed that 5% error in truncation range introduces 20% error in the average crystallite size. So extreme care has to be taken for the proper choice of
truncation range and background estimation in this Fourier analysis. The validity of an $A_n$ depends explicitly on the number of points (equally spaced) at which the observed profile is sampled within the observation range. Stokes (1948) showed that the number of sampling intervals must always exceed twice the value of $n$ for the lowest frequency Fourier coefficient which is effectively zero. Doi (1957), Gilli and Borea (1970), and Young et al (1967) have made some valuable studies on the possible error due to improper truncation, sampling range, and background estimation. Young et al (1967), however, showed that if $A_n$'s were transformed back to give observed profile there would be negligible error if and only if $A_n \propto 0$ for $n \gg M/2$ (where $M$ is the number of parts into which observed range is divided).

In a recent study Gilli and Borea (1973) have considered the effect of non-linearity error (NE) in the counting rate in line profile analysis. They, however, have pointed out that the effect is not so important for proportional or scintillation counters, as it is for Geiger counters. The finite value of the time constant gives a retarded response to the variations in the counting rate causing the peak height to be small. In our present study this effect does not come into existence as the experimental observations have been made by manual fixed time point counting method instead of chart recording.
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<td>4.5</td>
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Table 3.2
The values of Fourier cosine coefficients $A_n$ and sine coefficients $B_n$ for 111 reflection of Cu-16.00 Ga (Computer output)*

<table>
<thead>
<tr>
<th>n</th>
<th>$E_n$</th>
<th>$H_n$</th>
<th>$G_n$</th>
<th>$A_n$</th>
<th>$B_n$</th>
<th>$A_n/A_o$</th>
<th>L(=na0)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0.71638E+04</td>
<td>0.00000E+00</td>
<td>0.91088E+04</td>
<td>0.00000E+00</td>
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<td>0.00000</td>
<td>1.0000</td>
</tr>
<tr>
<td>1</td>
<td>0.52139E+04</td>
<td>0.19131E+03</td>
<td>0.89403E+04</td>
<td>0.29113E+03</td>
<td>0.5838</td>
<td>-0.0024</td>
<td>0.7438</td>
</tr>
<tr>
<td>2</td>
<td>0.38853E+04</td>
<td>0.94913E+02</td>
<td>0.85569E+04</td>
<td>0.62678E+03</td>
<td>0.4330</td>
<td>-0.0159</td>
<td>0.5482</td>
</tr>
<tr>
<td>3</td>
<td>0.27813E+04</td>
<td>0.17258E+03</td>
<td>0.81531E+04</td>
<td>0.72355E+03</td>
<td>0.3419</td>
<td>-0.0083</td>
<td>0.4538</td>
</tr>
<tr>
<td>4</td>
<td>0.20176E+04</td>
<td>0.88164E+02</td>
<td>0.76299E+04</td>
<td>0.92412E+03</td>
<td>0.2865</td>
<td>-0.0188</td>
<td>0.3261</td>
</tr>
<tr>
<td>5</td>
<td>0.14633E+04</td>
<td>0.10564E+03</td>
<td>0.74689E+04</td>
<td>0.11418E+03</td>
<td>0.1936</td>
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<td>0.20833E+03</td>
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<td>0.18939E+03</td>
<td>0.1436</td>
<td>-0.0127</td>
<td>0.1825</td>
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<tr>
<td>7</td>
<td>0.79047E+03</td>
<td>0.12602E+03</td>
<td>0.55234E+04</td>
<td>0.12019E+03</td>
<td>0.1089</td>
<td>-0.0145</td>
<td>0.1377</td>
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<tr>
<td>8</td>
<td>0.60308E+03</td>
<td>-0.22910E+03</td>
<td>0.61507E+04</td>
<td>0.14440E+03</td>
<td>0.0774</td>
<td>-0.0182</td>
<td>0.0934</td>
</tr>
<tr>
<td>9</td>
<td>0.38928E+03</td>
<td>0.17608E+03</td>
<td>0.57043E+04</td>
<td>0.16810E+03</td>
<td>0.0663</td>
<td>-0.0118</td>
<td>0.0716</td>
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<tr>
<td>10</td>
<td>0.22443E+03</td>
<td>-0.76113E+03</td>
<td>0.52318E+04</td>
<td>0.15852E+03</td>
<td>0.0407</td>
<td>-0.0136</td>
<td>0.0518</td>
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<tr>
<td>11</td>
<td>0.14887E+03</td>
<td>0.14730E+03</td>
<td>0.47737E+04</td>
<td>0.16594E+03</td>
<td>0.0291</td>
<td>-0.0063</td>
<td>0.0370</td>
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<tr>
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<td>0.43402E+04</td>
<td>0.16389E+03</td>
<td>0.0199</td>
<td>-0.0105</td>
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<td>-0.62635E+03</td>
<td>0.39593E+04</td>
<td>0.15170E+03</td>
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<td>0.0166</td>
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<tr>
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<td>0.61092E+02</td>
<td>-0.30457E+03</td>
<td>0.36642E+04</td>
<td>0.14730E+03</td>
<td>0.0092</td>
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<td>0.0117</td>
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<tr>
<td>15</td>
<td>0.37918E+02</td>
<td>-0.17631E+03</td>
<td>0.31875E+04</td>
<td>0.13893E+03</td>
<td>0.0066</td>
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<td>0.0084</td>
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<tr>
<td>16</td>
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<td>-0.28265E+03</td>
<td>0.28455E+04</td>
<td>0.14602E+03</td>
<td>0.0066</td>
<td>-0.0118</td>
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<tr>
<td>17</td>
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<td>-0.69736E+02</td>
<td>0.25850E+04</td>
<td>0.13984E+03</td>
<td>0.0101</td>
<td>-0.0074</td>
<td>0.0168</td>
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<tr>
<td>18</td>
<td>0.40028E+02</td>
<td>-0.17748E+03</td>
<td>0.23303E+04</td>
<td>0.12028E+03</td>
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<td>-0.0149</td>
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<tr>
<td>19</td>
<td>0.38979E+02</td>
<td>0.46314E+02</td>
<td>0.19657E+04</td>
<td>0.87579E+03</td>
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<td>0.0206</td>
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<tr>
<td>20</td>
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<td>-0.12621E+03</td>
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<td>0.0198</td>
<td>-0.0167</td>
<td>0.0282</td>
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* Origin for the annealed profile (composite) was chosen at the $K_\alpha$-peak maximum position.
Table 3.2 (subsidary)*

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<tr>
<th>n</th>
<th>H_L</th>
<th>H_I</th>
<th>G_L</th>
<th>G_I</th>
<th>A_n</th>
<th>P_n</th>
<th>A_n/A_o</th>
<th>L (=neg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0.716188</td>
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<td>0.90328E-04</td>
<td>0.00000E-00</td>
<td>0.7928</td>
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<td>1</td>
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<td>0.88630E-04</td>
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<td>0.5879</td>
<td>0.0294</td>
<td>0.7415</td>
<td>15.90</td>
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<td>0.47011E02</td>
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<td>0.9320</td>
<td>0.0517</td>
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<td>0.17256E03</td>
<td>0.80910E-04</td>
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<td>-0.58165E03</td>
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<td>0.0185</td>
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<td>8</td>
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<td>0.0091</td>
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<tr>
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<td>0.0026</td>
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</table>

* Origin for the annealed profile (composite) was chosen at the mean position of K_L/Z_0.

Note: This alternative origin choice for the annealed profile does not produce any significant change in the values of normalized A_n coefficients for L <200 m (vide Table 3.2), although P_n coeffs. are influenced appreciably. Since the present analysis concerns only A_n coeffs., the origin choice for the annealed profile has been made throughout from previous consideration (Table 3.2).
Cu-15.00 Ga
Il! Annealed.

FIG. 3.2