CHAPTER 6: STUDIES ON FCC TERNARY ALLOYS
6.1 An X-ray diffraction study of lattice imperfections in cold-worked f.c.c. alloys:
I Copper - Tin - Zinc (α - phase)

6.1.1 Introduction

Most of the prior studies on x-ray line profiles from cold-worked materials are mainly restricted to pure metals and binary alloys and studies with ternary alloy systems are very much lacking (Chapter 4). The state of cold-work in a deformed f.c.c. Cu-Si-Mn alloy was first studied by Welch and Otte (1962) from the x-ray analyses of peak position, peak-shift, lattice parameter change and peak breadth. The same alloy system has recently been investigated by Vasudevan (1969, 1970a,b) using the methods of integral breadth, Fourier analysis and variance and the results obtained from these methods have been compared. Another f.c.c. ternary alloy system, Cu-Ge-Si was studied by Foley, Cahn and Raynor (1963) to evaluate the values of the stacking fault parameter, α, only from the peak-shift measurements at constant solute concentrations. The stacking fault probability, obtained from x-ray diffraction method in some other f.c.c. copper-base ternary alloys, namely Cu-6Al-(Mn, Zn, Ni) has been utilized by Salonen et al (1969) to measure the stacking fault energy of these alloy systems. Warren-Averbach method of Fourier analysis of line shapes (Warren, 1959) has recently been employed to study the x-ray line profiles of cold-worked ternary f.c.c. alloy system, Pd-Ag-Au by Naidu and Houska (1971). The effect of plastic deformation on the position as well as the shape of the line profiles has been analysed, and the observation has been made
for the composition variations of the microstructural parameters.

The present experimental study represents an x-ray investigation of the cold-worked structures of the copper-base f.c.c. ternary system Cu-1 wt % Sn-Zn. The main objectives of the present study are the followings: (i) to make a detailed and more or less complete study of the effect of plastic deformation on the x-ray diffraction profiles of these materials taking into consideration the recent developments in terms of the presence and influence of extrinsic stacking fault (besides intrinsic and twin ones) along with the evaluations of other microstructural parameters namely, long-range residual stresses, lattice parameter changes, coherent domain sizes, microstrains within these domains, dislocation density, fault energy etc. from peak shift, peak asymmetry and Fourier line shape analysis; (ii) to study the effect of solute elements in describing the substructure formed and further to observe the effect of addition of solute tin in the near dilute range (~1 wt %) to the binary α-Cu-Zn system (Wagner and Helion, 1965).

6.1.2 Experimental procedure and methods of analyses

Cu-1 wt % Sn-Zn alloys containing 24.0, 26.5, 29.0, 31.5 and 34.0 wt % of Zn were prepared from spectroscopically pure metals supplied by M/s. Johnson, Matthey & Co. Ltd., London. Accurately weighed quantities of the constituent metals have been sealed in evacuated quartz capsules and melted in a high temperature furnace, the melting being done in the range of 950°C to 1000°C. Thorough
mixing of the materials was achieved by vigorously shaking the capsules. The resultant ingots were then homogenized at about 700°C for 7 to 10 days. The procedure did not lead to any significant weight loss.

The homogenized ingots were then cold-worked by hand-filing at room temperature (30°C ± 1°C). A part of the cold-worked powders after sieving through a 250 mesh was retained in the 'as-filed' condition to represent the cold-worked state and the rest was annealed at about 550°C for 8 hours in evacuated sealed pyrex glass capsules representing the 'standard' for line-shift and line-shape analysis.

Flat diffractometer specimens from both the cold-worked and annealed powders were prepared in the usual manner as adopted earlier (Sen Gupta and Quader, 1966). Nickel filtered CuKα radiation from a Philips stabilized x-ray generator (PW 1010) coupled with a counter diffractometer (PW 1050, 1051) were employed to record the line profiles at room temperature for the reflections 111, 200, 220, 311, 222, 400 by point counting done at intervals of 0.1° in 2θ for the general background decreasing to 0.01° in 2θ near the peak maxima (Chapter 3).

The methods of analyses as regards peak shift, peak asymmetry, peak broadening and also the estimation of dislocation density and stacking fault energy are the same as adopted in Chapter 5, Section 5.1.
6.1.3 Results and discussions

6.1.3.1 Peak shift and asymmetry analysis:

Assuming faulting to be the only cause of peak-shift, the mean values of the stacking fault density \(\alpha(=\alpha'-\alpha'')\) obtained from the peak position measurements of the neighbouring pairs of reflections namely, 111 - 200, 200 - 220, and 220 - 311 using equation (5.2) are shown in Table 6.1. The values of \(\alpha\) are, in general, found to be insensitive to composition variations.

Considering the composite effect of lattice parameter change and faulting on peak-shift, the values of \((\Delta a/a_0)\) and \((\alpha'-\alpha'')\) are also calculated from a least-square analysis of the equation (5.1a) for the same pairs of reflections and are shown in Table 6.2. These values of \((\alpha'-\alpha'')\) are found to be very close to those obtained from the assumption of the shift due to faulting alone (Table 6.2). This shows that the effect of faulting is more prominent in producing the shift of the peaks than the other contributing factors (Wagner and Helion, 1965). This is further supported from the plots of the lattice parameter '\(a_{\text{hkl}}\)' for the cold-worked and annealed specimens against the extrapolation function \(\cos\theta\cot\theta\) (Fig. 6.1), where \(\theta\) corresponds to the peak maxima (Wagner and Helion, 1965; Wagner, 1965). Following the theory of faulting in f.c.c. materials, reference lines were drawn through the points \((2a_{111}+a_{200})/3\), \((2a_{220}+a_{200})/3\) and \((2a_{222}+a_{220})/3\) at \(\{(\cos\theta\cot\theta)_{111}+(\cos\theta\cot\theta)_{200}\}/2\), \(\{(\cos\theta\cot\theta)_{220}+(\cos\theta\cot\theta)_{200}\}/2\), \(\{(\cos\theta\cot\theta)_{222}+(\cos\theta\cot\theta)_{220}\}/2\) respectively for both
the cold-worked and annealed specimens (Fig. 6.1). While the plots
of the $a_{hkl}$ values for different reflections in case of annealed
specimens are very close to their respective 'reference' lines, the
directions and magnitudes of the scatter of the $a_{hkl}$ values for
cold-worked specimens with respect to their 'reference' lines clearly
indicate pronounced influence of stacking faults on peak-shift rather
than that from residual stresses. The magnitude of the scatter
being a function of stacking fault density has also been utilized to
calculate the values $\alpha' = \alpha''$ for the reflections 111, 200 and
220 and the average values of $\alpha$ (Table 6.2) are found to be very
close to those obtained previously from the analysis of peak shift
(Table 6.1). This further supports the conclusion drawn earlier
regarding the influence of faulting on peak shift. Any difference in
these two sets of $\alpha$ values may, however, indicate a possible contribu­
tion from long-range residual stresses and other factors on peak-
shift. The influence of lattice parameter change, $(\Delta a/a_o) \sim 10^{-3}$
(Table 6.2) is also very small and the net difference between $(\Delta a/a_o)$
and $(\Delta a'/a_o)$ (calculated from graphical extrapolation of Fig. 6.1
and shown in Table 6.2) further supports the negligible influence of
stress on peak-shift as the latter quantity includes effect due to
residual stresses. These conclusions are in conformity with those of
Wagner and Helion (1965) in filings of $\alpha$-Cu-Zn and $\alpha$-Cu-Sn,
Chatterjee et al (1975) in filings of Ag-Ga and Naidu and Houska
(1971) in filings of Pd-Ag-Au alloys. The observed difference
between $a_{CW}$ and $a_o$ (Table 6.2) may be associated with the change in
lattice parameter due to segregation of solute atoms and the dislo­
cation structure (Wagner, 1965) and also to destruction of ordering
(Naidu and Houska, 1971).
The asymmetry analysis of the line profiles has been made from the measure of the shift of the centre of gravity of the line profiles from the peak maxima. The values of the parameter \((\beta + 4.5\alpha'')\) were calculated using equation (5.3) and are listed in Table 6.1.

6.1.3.2 Peak broadening analysis:

The Stokes' (1948) corrected normalized Fourier coefficients \(A_L\) (against \(L\)) for the reflections 111, 222, and 200, 400 have been utilised to separate the order-independent particle size coefficients \(A^p\) and order-dependent strain coefficients \(A^D_L\) following the Warren-Averbach's method (Warren, 1959) of analysis of line shapes. Using equation (5.5) and Fig.2, the anisotropic values of the effective particle sizes \(D_e\) and r.m.s. microstrains \(\langle \xi^2 \rangle^{1/2}\) have been evaluated along \([111]\) and \([100]\) directions and are listed in Table 6.1. The variations of these values with composition changes have been found to be insignificantly small. Similar small changes are also found in the values of \(D_{SF}\) (Table 6.1) and \(T_{min}\) (Table 6.1). The average experimental ratio of \(D_e^{111}/D_e^{100}\) and the computed values of \(D_{SF}\) indicate the significant contribution of stacking faults to the particle size broadening. The influences of \(D\) and \(T\) are, however, small as appears from \(T_{min}\) values (Warren, 1961). The values of the compound fault probability \(1.5(\alpha' + \alpha'') + \beta\) as evaluated from equation (5.8) are also shown in Table 6.1. The anisotropy of r.m.s. strain and also its asymptotic decrease with increasing \(L\) (Fig.6.3) are probably related to the dislocation arrangement and also the influence of stress on the dislocations and stacking faults in f.c.c. materials (Sen Gupta and Quader, 1966; Chatterjee, Halder and Sen Gupta, 1975).
6.1.3.3 Stacking fault densities:

From simultaneous solution of the three parameters involving 
\( \alpha' - \alpha'' \), \( \beta + 4.5 \alpha'' \) and \( 1.5 (\alpha' + \alpha'') + \beta \) from Table 6.1, the individual values of \( \alpha' \), \( \alpha'' \) and \( \beta \) were calculated and are shown in Table 6.1. The result clearly reveals relatively large concentration of intrinsic fault (\( \alpha' \)) along with an appreciable content of extrinsic fault (\( \alpha'' \)). The high negative values for twin fault probability \( \beta \) are physically unrealistic and are due to experimental uncertainties involved in the determinations of respective parameters from different types of analyses. The high negative values for \( \beta \) imply a total absence of deformation twins. As such considering \( \beta \) to be zero (Chatterjee, Halder and Sen Gupta, 1975), a least-squares solution has further been sought for \( \alpha' \) and \( \alpha'' \) and these values of \( \alpha' \) and \( \alpha'' \) are also shown in Table 6.1. The values of \( \alpha' \) thus obtained compare favourably well with the values of \( \alpha' - \alpha'' \) obtained from peak-shift measurements and this indicates relatively small influence of extrinsic fault. The individual concentrations of both intrinsic and extrinsic faults are, however, considerably reduced by this procedure. The values of intrinsic fault probability \( \alpha' \) are nearly halved from the previous ones, the extrinsic fault probability \( \alpha'' \) being in the range of experimental error. From these observations it can be concluded that cold-working in this system produces an appreciable concentration of intrinsic fault along with a negligibly small presence of extrinsic fault but fails to introduce any twin fault. These observations are in conformity with some recent observations in binary copper- and silver-base alloys studied by x-ray diffraction and electron microscopy (Gallagher, 1966; Chatterjee, Halder and Sen Gupta, 1975; Sen Gupta and De, 1970).
6.1.3.4 Dislocation density and stacking fault energy:

The values of the dislocation density, \( \rho \) as calculated from equation (5.10) are shown in Table 6.1 and are also found to vary insignificantly with composition.

As the shear modulus, \( \mu \) for this alloy system is not known, an average value of \( \frac{1}{\mu} \) for this system can be calculated from equation (5.13) substituting the average values of \( \rho \) and \( \alpha \), and is shown in Table 6.1. This parameter \( \frac{1}{\mu} \) is also insensitive to composition variation. As such the value of the stacking fault energy, \( \gamma^s \), for pure copper can be estimated from this parameter by substituting the shear modulus value for pure copper (Simmons and Wang, 1971) and is found to be 15.5 ergs/cm\(^2\) (Table 6.1), which is quite small in comparison to its value \( \sim 72.5 (\pm 14.5) \) ergs/cm\(^2\) obtained from electron microscopy (Jossang and Hirth, 1966). However, diversity in the values of stacking fault energy obtained from different methods of analyses is quite common and has been critically discussed by Gallagher (1969, 1970). The present value of \( \gamma^s \) has, however, been influenced by the assumptions made for the dislocation structure (Williamson and Smallman, 1956). Similar low values for the stacking fault energy from the x-ray fault probability (\( \alpha \)) have been obtained earlier by Vasudevan and Torok (1972) in binary Cu-Ga, Chatterjee et al (1975) in Ag-Ga, Otte and Welch (1964) and Vasudevan (1970) in ternary Cu-Si-Mn and Salonen et al (1969) in ternary Cu-6Al-(Mn,Zn,Ni) alloy systems.
6.1.3.5 Comparison with binary Cu-Zn alloys:

From a comparison of the effect of deformation on faulting in binary $\alpha$-Cu-Zn alloys (Wagner and Helion, 1965) with that in ternary Cu-$1$ wt\% Sn-Zn alloy (Fig. 6.4) it appears that the addition of small amount of tin in Cu-Zn alloys increases, in general, the value of faulting concentration in the ternary system but arrests any further increase in the fault concentration with increasing zinc content, which is normally observed in the binary $\alpha$-Cu-Zn alloys and also in most of the non-transitional binary alloy systems. Similarly, an examination of Table 6.1 further indicates that other micro-structural parameters, namely the effective particle size, non-uniform microstrain, dislocation density etc. are also insensitive to composition variation in this system unlike binary $\alpha$-Cu-Zn system (Wagner and Helion, 1965). Naidu and Houska (1971) have also obtained an identical behaviour of the micro-structural parameters in the ternary Pd-Ag-Au system where the quantities are found to be nearly constant except at the corners of the ternary diagram. In our present case, the addition of solute tin in the dilute range is, however, restricted to a smaller range of zinc composition (i.e., $\sim 24.0$ to $\sim 34.0$ wt\% Zn) and within this range it thus appears that the very small presence of tin can still produce a distinctive effect on the microstructure of the deformed alloy specimens, as compared to the respective binary one.
6.2 An X-ray diffraction study of lattice imperfections in cold-worked f.c.c. alloys:

II. Copper - Nickel-Zinc (α-phase)

6.2.1 Introduction

The present paper deals with the state of cold-work in the deformed f.c.c. Cu-Ni-Zn ternary alloy system employing analyses of the x-ray diffraction line profiles obtained from polycrystalline specimens. This forms a part of a programme undertaken by us to study the micro-structures of a series of ternary alloys in the deformed state by x-ray diffraction as very few of these systems have been investigated so far (Section 6.1). In our previous investigation on Cu-1\% Sn - Zn alloy system (Section 6.1) the effect of plastic deformation was characterized by the microstructural parameters describing the substructure of the deformed lattice namely, intrinsic, extrinsic and twin stacking faults, lattice parameter change, long range residual stresses, coherent domain sizes, microstrain within these domains, dislocation density and stacking fault energy etc. and these parameters were obtained from peak shift, peak asymmetry and Fourier line shape analyses. Apart from these, the effect of the addition of solute elements in the dilute range on these parameters has also been emphasized. The present study with Cu-Ni-Zn alloys, though of the same nature, has been undertaken because one of the solutes (Ni) in this system is a transitional metal whose presence in binary alloys reduces the faulting concentration due to the interaction of the incomplete d-shell electrons of the introduced solute atoms (Goswami et al, 1966).
It will be thus of much interest to see at this stage if similar effect of the transitional solute Ni can also be observed in the case of a ternary alloy system.

6.2.2 Experimental procedure and method of analyses

Six different compositions of the f.c.c. Cu-Ni-Zn alloys containing 1.0, 7.0 and 15.0 wt % Ni and 24.0, 34.0 wt % Zn were prepared from spectroscopically pure metals supplied by M/s. Johnson, Matthey & Co. Ltd., London following the same procedure as adopted earlier (Section 6.1), the melting being done in the range 1000 to 1100 °C and the homogenization done at 750 °C. The procedure did not lead to any significant weight loss.

Preparation of the cold-worked and annealed specimens and the recording of the line profiles, 111, 200, 220, 311, 222 and 400 were done in the usual way (Section 6.1) and the methods of analyses are the same as adopted in Chapter 5, Section 5.1.

6.2.3 Results and discussion

6.2.3.1 Peak shift and asymmetry analysis

Assuming faulting to be solely responsible for the observed peak shifts, equation (5.2) has been used to calculate the average values of \( \Delta \alpha' - \Delta \alpha'' \) from the peak shift of the neighbouring pairs of reflections namely 111 - 200, 200 - 220, 220 - 311 and these values of \( \Delta \alpha' - \Delta \alpha'' \) are shown in Table 6.3. It may be seen that for a constant zinc concentration these values of \( \Delta \alpha' - \Delta \alpha'' \) are found to be almost insensitive to the increase of nickel concentration. But for increasing zinc concentration this value of \( \Delta \alpha = \alpha' - \alpha'' \)
increases even when nickel concentration does not increase at all.

Considering peak shift as the composite effects of both lattice parameter change and faulting, the values of \((\Delta a/a_0)\) and \((\alpha'-\alpha'')\) are also calculated from the least square analysis of the equation (5.1a) for the same pairs of reflections and are shown in Table (6.4). The values of \((\Delta a/a_0)\) are very small \((\sim 10^{-3})\) and the values of \((\alpha'-\alpha'')\) are very close to those obtained earlier from the assumption of the shift due to faulting alone (Table 6.3). This shows that the effect of faulting is more prominent in shifting the peaks than the effect of lattice parameter change (Wagner and Helion, 1965). Similarly, the plots of the lattice parameters, \(a_{hkl}\) for both the cold-worked and annealed specimens against the extrapolation function, \(\cos \theta \cot \theta\) (\(\theta\) being the peak maxima)(Fig.6.5) also indicate the pronounced influence of faulting on peak shift rather than that from residual stresses and lattice parameter changes.

Similar behaviours of the lattice parameters were also found earlier in the case of ternary Cu-Sn-Zn alloy system (Section 6.1). The magnitude of the scatter of the lattice parameters calculated from 111, 200, 220 reflections from the 'reference' lines has also been utilized to calculate the values of \(\alpha\) \((\alpha'-\alpha'')\) and the average values (Table 6.4) thus obtained are also found to be very close to those obtained previously (Table 6.3) from measurements of peak shift. This further supports the conclusion drawn earlier regarding the influence of faulting on the observed peak-shift. That the influence of stress on peak shift is negligibly small is also evident from the observed small difference between the values of \((\Delta a/a_0)\) and \((\Delta a'/a_0)\)
(calculated from graphical extrapolation of Fig. 6.5 and shown in Table 6.4) as the latter includes effect due to residual stresses also. The observed difference between $a_{GW}$ and $a_{o}$ (Table 6.4) may be associated with the change in the lattice parameter due to segregation (Suzuki effect) and the dislocation structure (Wagner, 1965) and also due to destruction of order (Naidu and Houska, 1971). All these observations are in conformity with those of Wagner and Helion (1965) in the filings of $\alpha$-Cu-Zn and $\alpha$-Cu-Sn alloys, Chatterjee et al (1975, 1976) in filings of Ag-Ga and Cu-Ga alloys, Naidu and Houska (1971) in the filings of Pd-Ag-Au alloys and Halder et al (1976) in the filings of Cu-1 Sn-2 Zn alloys.

The asymmetry analysis of the 111 and 200 line profiles has been made from the measurements of the centre of gravity of the line profiles from the peak maxima. The values of the relevant parameter $(\frac{\beta}{4} + 4.5 \alpha)$ thus obtained from equation (5.3) are listed in Table 6.3.

6.2.3.2 Peak broadening analysis:

Following Warren - Averbach's method (Warren, 1959) of Fourier analysis of line shapes the Stokes' corrected normalised Fourier coefficients $A_L$ have been employed to separate the anisotropic values of the effective particle sizes, $D_e$ (Fig. 6.6) and the r.m.s. microstrains, $\langle \xi^2 \rangle^{1/2}$ along the [111] and [100] directions. These values are also shown in Table 6.3. A close examination of the Table 6.3 indicates that the variations of these parameters with increasing nickel concentration is insignificantly small for fixed
zinc concentration. But with the increase of zinc concentration these parameters are found to vary considerably (effective particle sizes decrease and r.m.s. strains increase or decrease slightly). The values of other parameters such as $D_{GF}$ (calculated from equations 5.6 and 5.7) and $T_{min}$ (calculated from equation 5.9) (Table 6.3) and also the average experimental ratio of $(D_e)_{111}/(D_e)_{100}$ indicate significant contribution of stacking faults to the particle size broadening. The influence of $D$ and $T$ are however small (Warren, 1961). The values of the compound fault probability $[1.5(\alpha' + \alpha'' + \beta)]$ calculated from equation (5.8) are also shown in the Table 6.3. The anisotropy of r.m.s. strain and its asymptotic decrease with increasing $L$ (Fig. 6.7) are probably related to the dislocation arrangement and also the influence of stress on the dislocation and stacking fault in f.c.c. materials (Chatterjee et al, 1975, 1976).

6.2.3.3 Stacking Fault Concentrations

From simultaneous solution of the three parameters involving $(\alpha' - \alpha'')$, $(4.5\alpha'' + \beta)$ and $[1.5(\alpha' + \alpha'') + \beta]$ the values of $\alpha'$, $\alpha''$ and $\beta$ are calculated and are shown in the Table 6.3. The concentrations of intrinsic stacking fault, $(\alpha')$ are found to be considerably higher than those for extrinsic stacking fault $(\alpha'')$. The concentrations of twin fault $(\beta)$ are found to be negative which is physically unrealistic and is probably due to the errors involved in the measurement of different related parameters. These low negative values of $\beta$ are, however, within the range of experimental error which implies total absence of deformation twins. As such
considering $\beta$ to be zero, a least-square solution has been sought for $\alpha'$ and $\alpha''$ from the above three parameters. These values of $\alpha'$ and $\alpha''$ (Table 6.3), thus obtained, are found to be slightly lower than the previous ones and the agreement is found to be fairly satisfactory. Moreover, the values of $\alpha'$ compare well with the values of $(\alpha' - \alpha'')$ (Table 6.3), obtained previously from peak shift measurements and the values of $\alpha''$ are close to the range of experimental error. This signifies that cold-working in this system produces an appreciable amount of intrinsic stacking fault along with insignificantly low concentration of extrinsic stacking fault but fails to introduce any deformation twin fault. Similar observations have, however, been made in the previous investigation with ternary Cu-1 wt% Sn-Zn alloys (Section 6.1) besides some binary copper- and silver-base alloys studied by x-ray diffraction and electron microscopy (Gallagher, 1966; Chatterjee et al., 1975; Sen Gupta and De, 1970).

6.2.3.4 Dislocation density and stacking fault energy:

The values of the dislocation density $\rho$ as calculated from equation (5.10) are also shown in Table 6.3. The average values of $\rho$ and the values of $\alpha$ ($\alpha' - \alpha''$) have been substituted in equation (5.13) to evaluate the values of $(\gamma/\mu)$ for this system (Table 6.3). As the values of shear modulus, $\mu$ for this alloy system are not known, the value of $\mu$ for pure copper (Simmons and Wang, 1971) has been substituted in the $(\gamma/\mu)_{av}$ to obtain the approximate value of stacking fault energy, $\frac{1}{\rho}$ for pure copper (Table 6.3). The approximation is justified as the variation of $(\gamma/\mu)$ for this
system with the change in composition is not appreciable. This value of $\gamma_0 \approx 11.63 \text{ ergs/cm}^2$ thus obtained for pure copper is, however, small in comparison to its value ($\approx 72.5 \pm 14.5 \text{ ergs/cm}^2$) obtained from electron-microscopy (Jossang and Hirth, 1966). Similar low value of $\gamma_0$ for pure copper, obtained from x-ray fault probability method, has earlier been obtained by a number of investigators (Section 6.1) and is probably due to the assumptions made in the dislocation structure (Williamson and Smallman, 1956).

6.2.3.5 **Effect of solutes on the microstructural parameters:**

At this stage of development the following tentative conclusions can be made from the observation of the dependence of the different microstructural parameters (Table 6.3 and 6.4) which are the manifestation of the cold-worked state, on the two solutes Ni and Zn of the present alloy system:

(i) The effect of addition of the solute Ni in the dilute range ($\approx 1 \text{ wt }\%$) on faulting can be compared with that of adding solute Sn in the dilute range done earlier with Cu-1 wt $\%$ Sn - Zn alloys (Section 6.1), where it was observed that the addition of small amount of Sn ($\approx 1 \text{ wt }\%$) increases slightly faulting concentration in the ternary system when compared with the binary Cu - Zn system (Wagner and Helion, 1965). But the nature of the faulting parameters in Cu-Sn-Zn system remains almost unchanged with increasing Zn concentration (from 24 to 34 wt $\%$) signifying the role of dilute solute Sn. In the present case, however, the addition of 1 wt $\%$ Ni has least influence in altering the faulting concentration and other microstructural parameters of the ternary system.
This shows that the presence of the transitional solute nickel in the dilute range in this ternary system does not produce any distinctive effect on the microstructural parameters of the deformed specimens when compared with the respective binary one.

(ii) The effect of increased addition of nickel content from 1 wt % to 15 wt % for both 24 and 34 wt % Zn alloys is also not very prominent as regards its influence on the microstructural parameters (Table 6.3). This sort of behaviour of the transitional solute appears to be similar to that observed in binary Cu-Ni alloys (Goswami et al., 1966) where the interactions of incomplete 3d-shells become more important.

(iii) The faulting concentration and other microstructural parameters, however, vary gradually with the increase of Zn concentration from 24 to 34 wt % for a fixed amount of nickel concentration. This indicates the typical behaviour of the non-transitional solute zinc\textsuperscript{\textit{\textbar}} influenced by the nickel content. This is quite similar to the observations made earlier for binary Cu-Zn alloys (Wagner and Helion, 1965).
6.3 General Observations

From the detailed analysis of the peak-shift, peak-asymmetry and peak-broadening performed on several cold-worked f.c.c. ternary Cu-1 \% Sn-Zn and Cu-Ni-Zn alloys (Section 6.1 and 6.2 respectively), the following significant observations regarding the micro-structure of the deformed lattice are apparent.

1. As observed earlier in binary alloys (Chapter 5), net stacking fault density plays the most important role in shifting the positions of line profiles than the other contributing factors namely, lattice parameter change and long-range residual stresses. In the case of Cu-1 \% Sn-Zn alloys, the presence of small amount of tin almost in the dilute range increases the net stacking fault concentration but arrests any further increase with increasing zinc concentration. For Cu-Ni-Zn system, the fault concentration increases gradually with the increase of zinc concentration, but remains more or less uniform with the increase of nickel concentration thereby indicating influences of transitional and non-transitional solutes in the ternary system.

2. Cold-working in both the systems produces an appreciable amount of intrinsic stacking fault with a very small probability of occurrence of extrinsic stacking fault, and there is a total absence of deformation twin fault. This observation is thus quite identical to that observed in binary alloys (Chapter 5).
3. The presence of dilute amount of tin in Cu-1% Sn-Zn system and the transitional solute Ni in Cu-Ni-Zn system has least influence in altering the microstructural parameters namely, stacking fault density, coherent domain size, r.m.s. strain, dislocation density and fault energy etc.

4. The comparable values of $D_e$ and $D_{PF}$ indicate the significant contribution of stacking faults to the particle size broadening for both the systems.

5. For both the cases considered, the estimated value of stacking fault energy for pure copper is apparently low in comparison to that obtained from other method namely, electron microscopy.
Table 6.1: Results from line profile analysis

<table>
<thead>
<tr>
<th>Compositions (wt. %)</th>
<th>$\langle k^2 \rangle^{1/2}$</th>
<th>$D_0$ in Å</th>
<th>$D_{SP}$ in Å</th>
<th>$\phi_{min}$ in Å</th>
<th>$\rho \times 10^{-11}$ (cm/cm$^3$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cu-1 Sn-24.0 Zn</td>
<td>116</td>
<td>68</td>
<td>1.28</td>
<td>1.13</td>
<td>94</td>
</tr>
<tr>
<td>Cu-1 Sn-26.5 Zn</td>
<td>94</td>
<td>63</td>
<td>1.01</td>
<td>1.02</td>
<td>262</td>
</tr>
<tr>
<td>Cu-1 Sn-29.0 Zn</td>
<td>84</td>
<td>60</td>
<td>1.02</td>
<td>1.36</td>
<td>262</td>
</tr>
<tr>
<td>Cu-1 Sn-31.5 Zn</td>
<td>86</td>
<td>57</td>
<td>1.45</td>
<td>1.44</td>
<td>219</td>
</tr>
<tr>
<td>Cu-1 Sn-34.0 Zn</td>
<td>86</td>
<td>55</td>
<td>0.95</td>
<td>1.38</td>
<td>219</td>
</tr>
</tbody>
</table>

Table 6.1 (contd.)
Table 6.1 (contd.)

<table>
<thead>
<tr>
<th>Compositions (wt %)</th>
<th>$(\alpha' - \alpha'') \times 10^3$ error limits $\pm 2.0$ to $\pm 4.0$ (eqn. 5.2)</th>
<th>$(4.5\alpha' + \beta) \times 10^3$ error limits $\pm 2.0$ to $\pm 10.0$</th>
<th>$[1.5(\alpha' + \beta)] \times 10^3$ error limits $\pm 3.0$ to $\pm 7.0$</th>
<th>$\alpha' \times 10^3$ error limits $\pm 3.0$ to $\pm 8.0$</th>
<th>$\beta \times 10^3$ error limits $\pm 3.0$ to $\pm 20.0$</th>
<th>Considering $(\frac{j}{\mu})<em>{av}$ and $J</em>{c}$ for copper (ergs/cm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cu-1 Sn- 24.0 Zn</td>
<td>27.3</td>
<td>29.8</td>
<td>38.9</td>
<td>48.5</td>
<td>21.2</td>
<td>-65.6</td>
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<tr>
<td>Cu-1 Sn- 26.5 Zn</td>
<td>22.1</td>
<td>35.0</td>
<td>32.4</td>
<td>45.9</td>
<td>23.8</td>
<td>-72.1</td>
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<tr>
<td>Cu-1 Sn- 29.0 Zn</td>
<td>23.9</td>
<td>28.7</td>
<td>32.4</td>
<td>45.3</td>
<td>21.4</td>
<td>-67.6</td>
</tr>
<tr>
<td>Cu-1 Sn- 31.5 Zn</td>
<td>25.5</td>
<td>35.2</td>
<td>38.9</td>
<td>48.5</td>
<td>23.0</td>
<td>-68.3</td>
</tr>
<tr>
<td>Cu-1 Sn- 34.0 Zn</td>
<td>25.8</td>
<td>28.7</td>
<td>38.9</td>
<td>44.8</td>
<td>19.0</td>
<td>-56.8</td>
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</table>
Table 6.2. Results from lattice parameter measurements

<table>
<thead>
<tr>
<th>Compositions (wt %)</th>
<th>((\Delta a/a_o)) x 10^3</th>
<th>((\Delta a'/a_o)) x 10^3</th>
<th>(a_o) in Å</th>
<th>(a_c) calc.</th>
<th>(a_c) extrapol.</th>
<th>((\alpha' - \alpha'')) x 10^3</th>
<th>((\alpha' - \alpha'')) x 10^3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cu-1 Sn- 24.0 Zn</td>
<td>0.699</td>
<td>1.142</td>
<td>3.6790</td>
<td>3.6816</td>
<td>3.6832</td>
<td>25.19</td>
<td>34.14</td>
</tr>
<tr>
<td>Cu-1 Sn- 26.5 Zn</td>
<td>0.543</td>
<td>0.136</td>
<td>3.6785</td>
<td>3.6805</td>
<td>3.6790</td>
<td>21.52</td>
<td>17.05</td>
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<tr>
<td>Cu-1 Sn- 29.0 Zn</td>
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<td>0.380</td>
<td>3.6848</td>
<td>3.6867</td>
<td>3.6862</td>
<td>23.40</td>
<td>23.12</td>
</tr>
<tr>
<td>Cu-1 Sn- 31.5 Zn</td>
<td>0.724</td>
<td>0.163</td>
<td>3.6901</td>
<td>3.6928</td>
<td>3.6907</td>
<td>24.60</td>
<td>21.42</td>
</tr>
<tr>
<td>Cu-1 Sn- 34.0 Zn</td>
<td>0.483</td>
<td>0.217</td>
<td>3.6960</td>
<td>3.6978</td>
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<td>25.37</td>
<td>25.38</td>
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Table 6.3: Results from line profile analysis

<table>
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<tr>
<th>Composition (wt %)</th>
<th>De in Å</th>
<th>$\langle \xi_{L=5;A}^2 \rangle^{\frac{1}{2}}$ D in Å</th>
<th>T_{min} in Å</th>
<th>$(\alpha'^2) \times 10^3 (4.5 \times 10^{-3}) \times 10^3$</th>
<th>( \xi_S (\alpha'^2 \times 10^3) ) ( \times 10^3 ) limit</th>
<th>( \times 10^3 ) limit</th>
<th>( \times 10^3 ) limit</th>
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</thead>
<tbody>
<tr>
<td>Cu-1 Ni-24 Zn</td>
<td>130</td>
<td>68 3.33 3.62 187 81</td>
<td>273</td>
<td>17.85 30.12 45.26</td>
<td>±4.0 to</td>
<td>±10.0 to</td>
<td>±5.0</td>
</tr>
<tr>
<td>Cu-7 Ni-24 Zn</td>
<td>142</td>
<td>76 3.14 3.58 214 93</td>
<td>266</td>
<td>19.42 26.03 39.35</td>
<td>±2.0 to</td>
<td>±3.0 to</td>
<td>±4.0</td>
</tr>
<tr>
<td>Cu-15 Ni-24 Zn</td>
<td>153</td>
<td>94 2.80 5.44 320 138</td>
<td>182</td>
<td>18.08 8.99 28.30</td>
<td>±2.0 to</td>
<td>±3.0 to</td>
<td>±4.0</td>
</tr>
<tr>
<td>Cu-1 Ni-34 Zn</td>
<td>100</td>
<td>54 3.16 2.76 154 67</td>
<td>178</td>
<td>22.02 28.36 55.32</td>
<td>±2.0 to</td>
<td>±3.0 to</td>
<td>±4.0</td>
</tr>
<tr>
<td>Cu-7 Ni-34 Zn</td>
<td>75</td>
<td>41 2.71 3.92 119 51</td>
<td>128</td>
<td>30.68 40.06 71.59</td>
<td>±2.0 to</td>
<td>±3.0 to</td>
<td>±4.0</td>
</tr>
<tr>
<td>Cu-15 Ni-34 Zn</td>
<td>118</td>
<td>58 4.75 4.31 150 65</td>
<td>342</td>
<td>29.48 22.73 56.54</td>
<td>±2.0 to</td>
<td>±3.0 to</td>
<td>±4.0</td>
</tr>
<tr>
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Table 6.3 (contd.)

<table>
<thead>
<tr>
<th>Composition (wt %)</th>
<th>$\alpha' \times 10^3$</th>
<th>$\alpha'' \times 10^3$</th>
<th>$\beta \times 10^3$</th>
<th>L.S. calculation considering $\beta = 0$</th>
<th>$\rho \times 10^{-11}$</th>
<th>$\rho_{o}$ $\times 10^{-11}$</th>
<th>$(\frac{1}{\mu})_{o}$ $\times 10^{11}$</th>
<th>$(\frac{1}{\mu})_{o \omega}$ $\times 10^{11}$</th>
<th>$\gamma_{0}$ $\times 10^{3}$</th>
<th>$\gamma_{0}$ $\times 10^{3}$ for Cu</th>
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<tbody>
<tr>
<td>Cu-1 Ni-24 Zn</td>
<td>25.61</td>
<td>7.76</td>
<td>-4.80</td>
<td>23.83</td>
<td>6.63</td>
<td>7.43</td>
<td>15.43</td>
<td>11.43</td>
<td>2.42</td>
<td>2.31</td>
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<tr>
<td>Cu-7 Ni-24 Zn</td>
<td>29.96</td>
<td>10.54</td>
<td>-21.40</td>
<td>22.02</td>
<td>5.50</td>
<td>6.43</td>
<td>13.71</td>
<td>10.07</td>
<td>1.94</td>
<td>11.63</td>
</tr>
<tr>
<td>Cu-15 Ni-24 Zn</td>
<td>24.62</td>
<td>6.54</td>
<td>-21.44</td>
<td>17.04</td>
<td>1.72</td>
<td>5.34</td>
<td>16.90</td>
<td>11.12</td>
<td>2.28</td>
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<tr>
<td>Cu-1 Ni-34 Zn</td>
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<td>-16.85</td>
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<td>6.08</td>
<td>9.12</td>
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<td>1.64</td>
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<tr>
<td>Cu-7 Ni-34 Zn</td>
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<td>27.67</td>
<td>19.06</td>
<td>2.37</td>
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<td>Cu-15 Ni-34 Zn</td>
<td>36.42</td>
<td>6.94</td>
<td>-2.50</td>
<td>33.27</td>
<td>4.94</td>
<td>11.67</td>
<td>21.57</td>
<td>16.62</td>
<td>2.12</td>
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Table 6.4: Results from lattice parameter measurements

<table>
<thead>
<tr>
<th>Composition</th>
<th>(Δa/ao) x10^3</th>
<th>(Δa'/ao) x10^3</th>
<th>a_0</th>
<th>a_{calc.} (Å)</th>
<th>a_{extrapol.} (Å)</th>
<th>(α' - α'') x10^3</th>
<th>(α' - α'') x10^3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cu - 1 Ni - 24 Zn</td>
<td>0.9028</td>
<td>0.2455</td>
<td>3.6664</td>
<td>3.6697</td>
<td>3.6670</td>
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<tr>
<td>Cu - 7 Ni - 24 Zn</td>
<td>0.8808</td>
<td>0.1915</td>
<td>3.6557</td>
<td>3.6589</td>
<td>3.6557</td>
<td>18.52</td>
<td>24.87</td>
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<tr>
<td>Cu - 15 Ni - 24 Zn</td>
<td>1.1137</td>
<td>0.5764</td>
<td>3.6429</td>
<td>3.6470</td>
<td>3.6452</td>
<td>16.89</td>
<td>14.47</td>
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<tr>
<td>Cu - 1 Ni - 34 Zn</td>
<td>0.5649</td>
<td>0.2168</td>
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<td>3.6916</td>
<td>3.6900</td>
<td>27.53</td>
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<td>Cu - 7 Ni - 34 Zn</td>
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<td>0.1631</td>
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<td>3.6794</td>
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<td>34.45</td>
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<tr>
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<td>0.4639</td>
<td>3.6646</td>
<td>3.6699</td>
<td>3.6660</td>
<td>28.11</td>
<td>35.87</td>
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</tbody>
</table>
Fig. 6.1 The plots of lattice parameter $a_{hk1}$ vs the extrapolation function for Cu -1 wt % Sn-Zn alloys.
Fig. 6.2 The plots of the particle size co-efficients $A_s^L$ vs $L$ in Å for the (a) Cu - 1 wt% Sn - 24 wt% Zn and (c) Cu - 1 wt% Sn - 29 wt% Zn alloys.
Fig. 6.3 The plots of the r.m.s. strain vs \( L \) in Å for Cu-1 wt% Sn-29.0 wt% Zn alloy.
Fig. 6.4 The plots of stacking fault probability vs wt% Zn in the ternary Cu-1 wt% Sn-Zn and binary Cu-Zn alloys.
Fig. 6.6 The plots of lattice parameter $a_{hkl}$ vs the extrapolation function for Cu-Ni-Zn alloys.
Fig. 6.6 The plots of the particle size coefficients $A_L$ vs $L$ in Å for Cu-15Ni-24Zn alloy.
Fig. 6.7 The plots of the r.m.s. strain vs L in Å for Cu-15Ni-24Zn alloy.
Fig. 6.8 The plots of stacking fault density vs wt% Zn in the ternary Cu-Ni-Zn and binary Cu-Zn alloys.

- △ - Cu-1% Sn-Zn (Haider, De and Sen Gupta, 1975)
- ○ - Cu-Zn (Wagner and Helion, 1965)
- □ - Cu-(1,7,15)av. Ni-Zn (Present Work)