TEMPERATURE DEPENDENCE OF STACKING FAULT ENERGY
IN A Cu-30w/o Zn ALLOY

by
Craig M. Shevlin

A Thesis Submitted to the Faculty of the
DEPARTMENT OF METALLURGICAL ENGINEERING
In Partial Fulfillment of the Requirements
For the Degree of
MASTER OF SCIENCE
WITH A MAJOR IN METALLURGY
In the Graduate College
THE UNIVERSITY OF ARIZONA

1969
STATEMENT BY AUTHOR

This thesis has been submitted in partial fulfillment of requirements for an advanced degree at The University of Arizona and is deposited in the University Library to be made available to borrowers under rules of the Library.

Brief quotations from this thesis are allowable without special permission, provided that accurate acknowledgment of source is made. Requests for permission for extended quotation from or reproduction of this manuscript in whole or in part may be granted by the head of the major department or the Dean of the Graduate College when in his judgment the proposed use of the material is in the interests of scholarship. In all other instances, however, permission must be obtained from the author.

SIGNED: Craig M. Sheehan

APPROVAL BY THESIS DIRECTOR

This thesis has been approved on the date shown below:

L. J. DEMER
Professor of Metallurgical Engineering

17 Jan. 1969
The author wishes to express appreciation to his advisor, Dr. L. J. Demer, for his help and interest throughout this study. His guidance and understanding has made this work possible.

Thanks is also extended to the National Science Foundation for funds which made this project possible.
Dedicated to

Andrew F. Chaloka
# TABLE OF CONTENTS

<table>
<thead>
<tr>
<th>Section</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>LIST OF ILLUSTRATIONS</td>
<td>vi</td>
</tr>
<tr>
<td>LIST OF TABLES</td>
<td>viii</td>
</tr>
<tr>
<td>ABSTRACT</td>
<td>ix</td>
</tr>
<tr>
<td>I. INTRODUCTION</td>
<td>1</td>
</tr>
<tr>
<td>II. THEORETICAL BACKGROUND</td>
<td>5</td>
</tr>
<tr>
<td>2.1 Stacking Faults in Face-Centered Cubic Metals</td>
<td>5</td>
</tr>
<tr>
<td>2.2 Experimental Determination of Stacking Fault Energy</td>
<td>18</td>
</tr>
<tr>
<td>2.3 Stacking Faults Studied by X-Ray Diffraction</td>
<td>20</td>
</tr>
<tr>
<td>2.3.1 Calculation of Faulting Parameters</td>
<td>25</td>
</tr>
<tr>
<td>2.3.2 Calculation of Stacking Fault Energy</td>
<td>33</td>
</tr>
<tr>
<td>2.4 Temperature Dependence of Stacking Fault Energy</td>
<td>38</td>
</tr>
<tr>
<td>III. OBJECTIVES</td>
<td>43</td>
</tr>
<tr>
<td>IV. EXPERIMENTAL PROCEDURE</td>
<td>44</td>
</tr>
<tr>
<td>4.1 Specimen Preparation</td>
<td>44</td>
</tr>
<tr>
<td>4.1.1 Melting and Casting</td>
<td>44</td>
</tr>
<tr>
<td>4.1.2 Chemical Analysis of Cast Alloy</td>
<td>46</td>
</tr>
<tr>
<td>4.1.3 Preparation of Brass Filings</td>
<td>46</td>
</tr>
<tr>
<td>4.2 X-Ray Studies</td>
<td>47</td>
</tr>
<tr>
<td>4.2.1 Depressed Temperature Studies</td>
<td>48</td>
</tr>
<tr>
<td>4.2.2 Elevated Temperature Studies</td>
<td>54</td>
</tr>
<tr>
<td>4.3 Analysis of Data</td>
<td>55</td>
</tr>
<tr>
<td>V. RESULTS AND DISCUSSION</td>
<td>57</td>
</tr>
<tr>
<td>5.1 Low Temperature Results</td>
<td>57</td>
</tr>
<tr>
<td>5.2 Room Temperature Results</td>
<td>59</td>
</tr>
<tr>
<td>5.3 Elevated Temperature Results</td>
<td>60</td>
</tr>
<tr>
<td>5.4 Discussion of Results</td>
<td>61</td>
</tr>
<tr>
<td>VI. CONCLUSIONS</td>
<td>68</td>
</tr>
<tr>
<td>VII. SUGGESTIONS FOR FURTHER WORK</td>
<td>70</td>
</tr>
<tr>
<td>SELECTED BIBLIOGRAPHY</td>
<td>72</td>
</tr>
<tr>
<td>Figure</td>
<td>Description</td>
</tr>
<tr>
<td>--------</td>
<td>------------------------------------------------------------------------------</td>
</tr>
<tr>
<td>1.</td>
<td>Dissociation of a unit edge dislocation into partials in the FCC structure</td>
</tr>
<tr>
<td>2.</td>
<td>Dissociation of a unit screw dislocation into partials in the FCC structure</td>
</tr>
<tr>
<td>3.</td>
<td>Dissociation of a dislocation of mixed orientation into partials in the FCC structure</td>
</tr>
<tr>
<td>4.</td>
<td>Surface tension of faulted area balanced by repulsion force between partial dislocations</td>
</tr>
<tr>
<td>5.</td>
<td>Stacking associated with an intrinsic stacking fault in the FCC structure</td>
</tr>
<tr>
<td>6.</td>
<td>Stacking associated with an extrinsic fault in the FCC structure</td>
</tr>
<tr>
<td>7.</td>
<td>Stacking associated with a twin (growth) fault in the FCC structure</td>
</tr>
<tr>
<td>8.</td>
<td>Tetrahedron describing Thompson’s notation for dislocations in the FCC structure</td>
</tr>
<tr>
<td>9.</td>
<td>Unit dislocation node with Thompson’s notation</td>
</tr>
<tr>
<td>10.</td>
<td>Extended node and stacking fault formed by partial dislocations</td>
</tr>
<tr>
<td>11.</td>
<td>Contracted node and stacking fault formed by partial dislocation</td>
</tr>
<tr>
<td>12.</td>
<td>Typical ln $A_L$ versus $h^2$ curves for first two orders of (111)</td>
</tr>
<tr>
<td>13.</td>
<td>Typical $A_{Pf}$ versus L curve for determining effective particle size</td>
</tr>
<tr>
<td>14.</td>
<td>Typical $a_{(hkl)}^{app}$ versus cosθ cotθ curve for cold-worked filings</td>
</tr>
<tr>
<td>15.</td>
<td>Free energy as a function of temperature for cobalt</td>
</tr>
<tr>
<td>Figure</td>
<td>Page</td>
</tr>
<tr>
<td>--------</td>
<td>------</td>
</tr>
<tr>
<td>16. Sample holder for x-ray studies</td>
<td>50</td>
</tr>
<tr>
<td>17. Sample holder in loading position</td>
<td>51</td>
</tr>
<tr>
<td>18. Sample holder in position on diffractometer</td>
<td>53</td>
</tr>
<tr>
<td>19. Comparison of (111) profiles for 300° K and 373° K</td>
<td>65</td>
</tr>
<tr>
<td>20. Comparison of (111) profiles for 100° K and 300° K</td>
<td>67</td>
</tr>
</tbody>
</table>
# LIST OF TABLES

<table>
<thead>
<tr>
<th>Table</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Detailed Chemical Analysis of High Purity Copper and Zinc</td>
<td>45</td>
</tr>
<tr>
<td>2. Results of Diffraction Analysis</td>
<td>58</td>
</tr>
</tbody>
</table>
ABSTRACT

The stacking fault energy of Cu-30w/o Zn filings deformed at liquid nitrogen temperature was determined as a function of temperature. The x-ray diffraction method was used. Measured values of the stacking fault probability and mean squared strains were used to calculate the stacking fault energy. It was found that the stacking fault energy of this alloy does not change significantly when the temperature is raised from 100° K to 300° K. The measured stacking fault energy agreed closely with published room temperature electron microscopy results. Analysis of the filings at 373° K gave anomalous results.
I. INTRODUCTION

An important area in the field of physical metallurgy is the study of stacking faults in metals and alloys. In recent years emphasis on studies in this area has increased as the effect of stacking faults on the plastic deformation characteristics of metals has become known. Stacking faults affect not only the movement and distribution of dislocations during plastic deformation but also have a strong influence upon electrical properties (Christian and Spreadborough, 1956), texture (Valenzuela, 1965), recovery and recrystallization (Bailey and Hirsch, 1960), and other phenomena as well.

Face-centered cubic (FCC) and hexagonal close-packed (HCP) crystal structures both represent the closest possible packing of atoms. In the FCC structure close-packed layers of atoms are stacked one above the other in such a way that the atoms in the fourth layer are directly above those in the first layer. This results in the stacking sequence ABCABC where A, B, and C represent the positions of the atoms in the respective layers. In the HCP structure the close-packed layers are stacked so that the atoms in the third layer are directly above those in the first. This results in the stacking sequence ABABABA. A stacking fault occurs when there is a region of HCP stacking in a FCC structure or a region of FCC stacking in the HCP structure.

Stacking faults are produced in metals during crystal growth, plastic deformation, and phase transformations as well as through the collapse of vacancy disks. The faulted areas are usually considered to
be analogous to ribbons with widths on the order of 200 Å for faults produced by deformation in the alpha brasses (Warren, 1961). The present study is mainly concerned with faults produced by deformation in the FCC structure.

All stacking faults have a finite energy associated with them called the stacking fault energy. Christian and Swann (1965) define the stacking fault energy as the change in free energy of a crystal per unit area of fault introduced. As will be shown in a later discussion the stacking fault ribbons are actually bounded by partial dislocations which tend to repel each other. The finite energy of the stacking fault supplies a constant attractive force. When this attractive force is sufficient to balance the elastic repulsion of the partials the total energy is minimized, and an equilibrium ribbon width is attained.

It is clear then, that in a metal with a high stacking fault energy such as aluminum or nickel the equilibrium width of the ribbons will be quite narrow, since only a small separation is required to balance the repulsive force exerted by the bounding dislocations. In metals with a low stacking fault energy such as some copper and silver alloys the equilibrium ribbon width is much larger so that the total energy of the faulted area will be sufficient to balance the repulsive force of the partial dislocations.

The significance of the stacking fault energy as a contributing factor in plastic deformation becomes evident. The stacking fault ribbons must come together before a dislocation can leave its slip plane and move on an intersecting slip plane. It follows then that this cross slip is favored by a high stacking fault energy since the narrow ribbons
are more easily forced together by an applied stress. It is for this reason that slip bands observed in aluminum appear wavy while those in an alpha brass with its lower stacking fault energy are straight. This is only one of many observed effects in which the stacking fault energy plays an important role.

In many early treatments of the behavior of FCC metals the stacking fault energy was treated as a constant and was not considered to vary with temperature. In Seeger's model (Diehl, Mader, and Seeger, 1955) cross slip takes place when partial dislocations are forced together over a critical portion of their length and the dislocation expands on the cross slip plane. The stress required to recombine the partials is considered to be supplied by dislocation pile-ups and thermal fluctuations. No effect of temperature on the stacking fault energy is considered. If the stacking fault energy were temperature dependent, however, it would be expected that the partials would be forced together by an increase in the stacking fault energy with increasing temperature. More recently, experimental evidence has shown that in certain alloy systems there is indeed a temperature dependence of stacking fault energy. Using the transmission electron microscope, Swann and Nutting (1960) observed the collapse of stacking fault ribbons in Cu-7 wt% Al upon heating. This indicates an increase in the stacking fault energy with temperature. Using the node method of calculating the stacking fault energy, Ericsson (1966) found an increase in stacking fault energy in some Co-Ni alloys with increasing temperature.
The objective of the present study was to determine if there is a temperature dependence of stacking fault energy in an alloy of cold worked Cu-30\text{wt/oZn}. The x-ray diffraction method was used.

This study is one of a series of investigations into the nature of stacking faults which have been carried out in the Department of Metallurgical Engineering at The University of Arizona. Valenzuela (1965) studied the effect of the stacking fault energy on the texture transition in the alpha brasses. Cotter (1967) studied the stacking fault probability of aluminum alloys of silicon and magnesium as a function of composition. Simmons (1967) determined faulting probabilities in eutectoid steel wire in an effort to correlate this with fracture characteristics. Hinton (1968) determined stacking fault probabilities in electrodeposited copper.
2.1 Stacking Faults in Face-Centered Cubic Metals

The planar surface of separation between two regions of a crystal which have the same orientation but which do not form a continuous lattice is called a stacking fault. In the FCC structure there are three types of stacking faults which can occur. They are the intrinsic fault (deformation fault), the extrinsic fault (double deformation fault), and the twin or growth fault.

The normal FCC stacking is given as ABCABC. It may be expressed by the Frank (1951) notation as $\Delta \Delta \Delta \Delta \Delta \Delta$ where each $\Delta$ represents a transition of the type $A \rightarrow B \rightarrow C \rightarrow A$. HCP stacking is given as ABABABA or $\Delta \triangledown \Delta \triangledown \Delta \triangledown$, where each $\triangledown$ represents a transition of the type $B \rightarrow A \rightarrow C \rightarrow B$. The stacking associated with an intrinsic stacking fault in the FCC structure is ABCABABC or $\Delta \Delta \Delta \Delta \triangledown \Delta \Delta \Delta$. The fault is indicated by the slant bar and is a small region of HCP stacking. This type of fault is also known as a $1\Delta$ fault. It is equivalent to the removal of a portion of one atomic $\{111\}$ plane from the crystal, although it is not formed in this way as will be shown. This type of fault is by far the most important in FCC metals and is the main concern of this study.

The stacking associated with the extrinsic or double deformation fault is ABCABABC or $\Delta \Delta \Delta \Delta \triangledown \triangledown \Delta \Delta \Delta$. Here the fault is equivalent to the removal of a portion of two atomic $\{111\}$ planes or the insertion of a portion of one. This type is also called a $2\Delta$ fault. The growth
twin fault is the third type encountered in the FCC structure. The stacking pattern here is ABCAB/ACBAC or \( \triangle \triangle \triangle \bigcirc \bigcirc \bigcirc \bigcirc \bigcirc \) with the lattice beyond the fault being the twin of the parent lattice. Twin faults form during plastic deformation as well as in the processes of solidification and recrystallization. It should be noted that the insertion of an intrinsic fault at every (111) plane of a FCC structure gives the (111) twin of that structure, while an intrinsic fault introduced at every other plane gives the HCP structure. While more complex fault structures are encountered in the FCC structure they are best regarded as combinations of these basic types. That these faults are the result of certain dislocation interactions will now be shown.

A dislocation whose Burgers vector \((b)\) is a unit lattice distance is called a "complete," "unit," or "perfect" dislocation. When such a dislocation glides on a \( \{111\} \) plane it leaves the crystal structure unchanged. Such a unit slip dislocation in the FCC structure is specified by giving the magnitude and direction of its Burgers vector. Thus \( a/2[110] \) specifies a dislocation whose Burgers vector is one-half of an identity distance in the \([110]\) direction. Such perfect dislocations are not commonly found, however, in FCC metals and alloys. More often it is found that these perfect dislocations undergo a splitting process in which two "partial" dislocations are formed. A typical reaction is: \( a/2[110] = a/6[121] + a/6[211] \). The two product dislocations are the partials and each has a Burgers vector smaller than the perfect dislocation. This reaction is allowed to occur due to a reduction in the total strain energy which is proportional to \( b^2 \) (Frank, 1951).
Dislocations of the type \( a/2 \langle 112 \rangle \) are called Shockley partial dislocations and are glissile since the Burgers vector is \( b \) in the slip plane. Figure 1a shows schematically the dissociation of a unit edge dislocation lying in a \( \{111\} \) plane. The resulting partials are of mixed orientation. Figure 1b shows in vector form that the screw components of the partials are of opposite sign and attract each other. The edge components, however, are of like sign and being larger in magnitude cause a net repulsive force to exist between the partials. This causes the partials to separate in the slip plane. The area separating the partials is a region of intrinsic stacking fault. The entire arrangement of the two partials and the stacking fault is an extended dislocation. The dissociation of dislocations of screw and of mixed orientations are shown in Figs. 2 and 3, respectively. In each of these cases the Shockley partials separate in the slip plane until the repulsive force they exert on each other is balanced by the attraction caused by the energy of the faulted area. Figure 4 shows this relationship in graphical form. As the width of separation increases the repulsive force between the partials decreases and the total faulted area increases. At some width \( w \) the total energy of the faulted area is sufficient to balance the repulsive force. This is the equilibrium separation. In a metal with a low stacking fault energy the partials can separate to a greater width than in a metal with a high stacking fault energy.

A dislocation with its Burgers vector non-parallel to the fault plane is sessile and can move only by climb. The Frank partial is such a dislocation and its Burgers vector is \( a/3 \langle 111 \rangle \). It can be formed by
a. Dissociation of a unit edge dislocation lying in a \(\langle 112 \rangle\) direction into two partial dislocations.

b. Vector form of (a) showing the repulsion of the edge components of the partials.

Fig. 1. Dissociation of a unit edge dislocation into partials in the FCC structure.
a. Dissociation of a unit screw dislocation lying in a \( \langle 112 \rangle \) direction into two partial dislocations.

b. Vector form of (a) illustrating the repulsion of the screw components of the partials.

Fig. 2. Dissociation of a unit screw dislocation into partials in the FCC structure.
a. Dissociation of a unit mixed dislocation lying in a $\langle 110 \rangle$ direction into two partial dislocations.

b. Vector form of (a) showing the repulsion of the edge components of the partials.

Fig. 3. Dissociation of a dislocation of mixed orientation into partials in the FCC structure.
Fig. 4. Surface tension of faulted area balanced by repulsion force between partial dislocations.
the removal or insertion of a portion of a close-packed plane of atoms. This could take place by the coalescence of a platelet of vacancies or interstitials. The former case is shown schematically in Fig. 5 and the stacking fault that has formed is an intrinsic one. If a close-packed plane is inserted as in Fig. 6, the dislocation loop is still of the Frank type but the stacking fault is an extrinsic or $2\Delta$ one. The Frank dislocation has nearly twice the energy of a Shockley dislocation.

Friedel (1964) illustrates the greater distortion of the crystal in the case of a Frank partial. This energy can be reduced, however, by the splitting of a Frank dislocation into a Shockley partial and a stair-rod dislocation. Figure 7 shows the stacking associated with a growth twin. The twin fault is bracketed and is a region of HCP stacking about half as thick as an intrinsic stacking fault.

Thompson (1953) has described a convenient notation for dislocations in the FCC structure. A tetrahedron such as the one shown in Fig. 8 is utilized. The tetrahedron has corners ABCD and faces $\alpha$, $\beta$, $\gamma$, and $\delta$ corresponding to the four sets of $\{111\}$ planes in a FCC crystal. Burgers vectors of unit dislocations are represented by the edges of the tetrahedron while those of Shockley partials are represented by lines such as $A\delta$, $B\delta$, $D\delta$, etc., on the faces of the tetrahedron. $A\delta$ represents the Burgers vector of a Frank dislocation and is normal to a $\{111\}$ plane. This notation best illustrates the vector properties of dislocations and gives a clear picture of the geometry of some dislocation reactions. Thus the reaction $BA = B\delta + \delta A$ is an example of the dissociation of a perfect dislocation into two Shockley partials with fault layer between them. The familiar
Fig. 5. Stacking associated with an intrinsic stacking fault in the FCC structure.

Fig. 6. Stacking associated with an extrinsic fault in the FCC structure.

Fig. 7. Stacking associated with a twin (growth) fault in the FCC structure.
Fig. 8. Tetrahedron describing Thompson's notation for dislocations in the FCC structure.
Lomer-Cottrell lock (Lomer, 1951; Cottrell, 1952) is formed by the reaction $D \beta + \beta C + C \delta + \delta B = D \beta + \beta \delta + \delta B$. Here two dissociated dislocations on intersecting slip planes react at the line of intersection to form a configuration consisting of two Shockley partials with their stacking fault areas and a stair rod dislocation. This arrangement is thought to be a barrier to further slip thus contributing to strain hardening. A dislocation node is formed when three unit dislocations meet as in Fig. 9. When three unit dislocations in the same plane such as $AB$, $BC$, and $CA$ split into the partials $A\delta + \delta B$, $B\delta + \delta C$, and $C\delta + \delta A$, a partial dislocation node may form. The partials then assume one of the configurations shown in Figs. 10 and 11 where the shaded portions represent areas of stacking fault.

An important parameter in the study of stacking faults is the intrinsic stacking fault probability $\alpha$. This quantity is really a measure of the amount of faulting that is present. As it is a probability value it can never exceed unity. A value of the stacking fault probability of 0.05 indicates that out of one thousand $\{111\}$ planes, there is a probability that fifty are completely faulted. A completely faulted plane in a crystal is one whose entire area, to the boundaries of the crystal, is covered by stacking faults. As $\alpha$ approaches unity, a FCC structure becomes completely faulted and reverts to the HCP form. Patterson (1952) has shown that when $\alpha$ is small there is no difference between a few planes completely faulted and many planes only partially faulted. This is important in x-ray studies because the diffraction effects are the same in either case. It is important to emphasize that
Fig. 9. Unit dislocation node with Thompson's notation.

Fig. 10. Extended node and stacking fault formed by partial dislocations.

Fig. 11. Contracted node and stacking fault formed by partial dislocation.
The stacking fault probability is not a fundamental quantity. For a given metal it can vary widely with degree and type of deformation.

The reciprocal stacking fault probability, $\alpha^{-1}$, a quantity used by Warren and Warekois (1955), is also quite useful. It is defined as the average number of unfaulted planes separating the faulted planes. Thus a value of 20 for $\alpha^{-1}$ corresponds to $\alpha$ equal to 0.05 and indicates that on the average every 20th plane is faulted. The probabilities of extrinsic faults and twin faults are designated as $\alpha^1$ and $\beta$, respectively.

The relationship between $\alpha$ and the stacking fault energy $\gamma$ is not as easily interpreted. A high value of $\alpha$ can correspond to either a wide equilibrium spacing of partials or to a large number of closely spaced faults. In the former case the stacking fault energy is low. In the latter case it is high. In order to make assumptions about the stacking fault energy from $\alpha$ it is necessary to know the existing dislocation density and configuration. For a constant dislocation considered to be proportional to $\alpha^{-1}$ (Vassamillet and Massalski, 1963).

The energy of a stacking fault originates from the forcing of electrons into higher energy states in the region of HCP stacking. A clue to the stacking fault energy is obtained from studying the nearest and next nearest neighbor relationships. Weertman and Weertman (1964) point out that to a first approximation the stacking fault energy is zero since an atom in a faulted region, either intrinsic or extrinsic, has the same number of nearest neighbors as an atom in a perfect lattice. The energy of the fault compared to an unfaulted structure arises in the difference in the number of next nearest neighbors. Here
the extrinsic and intrinsic fault energies appear to be equal. When next-next nearest neighbors are counted however, it is found that the extrinsic fault has the higher energy. Since an intrinsic fault can be considered to consist of two coherent twin faults lying on adjacent planes, the energy of a twin fault should be about half that of an intrinsic stacking fault (Fullman, 1951). In fact this relationship has been used to arrive at experimental values of stacking fault energy by a method which will be described in a later section.

2.2 Experimental Determination of Stacking Fault Energy

There are several methods of measuring the stacking fault energies of metals and alloys. These will be described here briefly. The x-ray method used in this study will then be described in more detail. Christian and Swann (1965) give a complete review of each of these methods.

The node method of measuring stacking fault energies was first described by Whelan (1959). In this method the stacking fault energy is determined from the curvature of partial dislocations at extended nodes in lightly deformed material. Although the node radius is easily measured using the transmission electron microscope, it is more difficult to use this information to calculate the stacking fault energy. The equations have undergone continual refinement however, and this method does seem to yield the most reliable values of stacking fault energy.

The ribbon method utilizes the electron microscope to measure the spacing between partial dislocations. If it is assumed that the
equilibrium separation is determined only by the energy of the fault and the elastic repulsion between the partials then the stacking fault energy can be calculated. This method can only be used if the stacking fault energy is sufficiently low that the spacing between the partials can be measured. The method is described in detail by Amelinckx and Delavignette (1963).

The twin boundary method has as its basis the relationship between the twin boundary energy \( J_t \) and the stacking fault energy \( J \). The stacking fault energy is considered to be equal to approximately twice \( J_t \) since an intrinsic stacking fault is really equivalent to two coherent twin boundaries on adjacent planes. The ratio of the twin boundary energy to the grain boundary energy can be determined by measuring angles of intersection between twin boundaries and grain boundaries. Since the grain boundary energy can be determined independently (Inman and Tipler, 1963), the twin boundary energy is known. Unfortunately the relation \( J = 2J_t \) is now thought to be incorrect (Thornton, Mitchell, and Hirsch, 1962) and the values of stacking fault energy from this method appear to be too low.

The onset of the third stage of plastic deformation in FCC single crystals is thought to be associated with slip from the primary slip plane onto the cross slip plane (Diehl et al., 1955). The stress \( \gamma_{III} \) necessary to bring on cross slip is dependent upon temperature and strain rate. The stacking fault energy of FCC metals can be determined by an analysis of the dependence of \( \gamma_{III} \) on the temperature and strain rate (Haasen, 1958). The stacking fault energies of pure metals
measured by the $\gamma_{\text{III}}$ method are normally higher than the results from node measurements. It is not certain where this difference arises.

Stacking fault energy can also be determined by a method requiring a knowledge of the free energy of a tilt boundary of dissociated dislocations. Li and Chalmers (1963) have used this method to arrive at a value of stacking fault energy for pure copper of 25 erg/cm$^2$. This value, however, is considered to be unreasonably low compared to results from node measurements.

Friedel (1964) shows that it may be favorable for a Frank dislocation to split into a stair rod dislocation and a Shockley dislocation by a reaction of the type: $\alpha' = \alpha + \beta\gamma$, using Thompson's notation. In a Frank loop this splitting often takes place on three of the $\langle 110 \rangle$ type directions and continues until the Shockley dislocations meet and react to form stair rod dislocations along the intersections of their glide planes. The result is a tetrahedron with stair rod dislocations along its edges and stacking fault on its faces. These stacking fault tetrahedrons are very stable. Czjzek, Seeger, and Mader (1962) have developed a method for calculating the stacking fault energy knowing the maximum stable size of a tetrahedron. It is difficult, however, to determine if a tetrahedron has been able to reach its maximum size.

2.3 Stacking Faults Studied by X-Ray Diffraction

Plastic deformation of a FCC metal brings about several important changes in the internal structure. Among these are the formation of inhomogeneous residual strains, small coherent domains and intrinsic
stacking faults. Each of these has an effect on the x-ray diffraction pattern of the metal. In order to effectively study stacking faults by means of x-rays it is important to consider all of the effects of cold work on the diffraction pattern.

When a metal is uniformly stressed it has its interplanar spacings altered to new values. These changes can be measured by the displacement of Debye rings in the powder diffraction pattern. In the case of inhomogeneous residual strains resulting from residual micro-stresses the interplanar spacings take on a range of values with the result that a diffraction line is broadened into a corresponding range of Bragg angles. The mean interplanar spacing is the same as in the unstrained condition. The reciprocal lattice for such a non-uniformly strained crystal is compressed where the crystal is stretched and expanded where the crystal is compressed. If the stress varied in magnitude but was constant in direction each reciprocal lattice point would be stretched into a line. In any given reciprocal lattice row the length of the lines would be proportional to the distance from the origin. This is true because points farthest from the origin correspond to planes with the smallest d spacing. Thus a stress will cause \( \Delta d \) for these planes to be a larger percentage of d. Normally, of course, the stresses vary in direction as well as magnitude and the reciprocal lattice points elongate into bundles of non-parallel lines filling a small volume. The sizes of these volumes is also proportional to the distance from the origin in any radial row. Since a radial row in reciprocal space consists of the different orders of a particular (hkl) reflection, the broadening of a diffraction line due to inhomogeneous
residual strains is dependent upon the order of the reflection. The broadening is greater for higher order reflections.

During plastic deformation the size of the regions that diffract as coherent crystallites is reduced. This is a consequence of the general disruption of the lattice and the increase in dislocation density. The particle sizes in cold-worked filings are often on the order of 200 Å (Warren, 1959). The deformation has not formed discrete particles of this size but has produced some type of domain structure within the filings. When the size of the coherently diffracting domains becomes less than about 1000 Å the diffraction lines become broadened symmetrically. The reason for this is that some of the rays scattered at non-Bragg angles from the top portion of a particle do not undergo destructive interference. The particle or domain is so small that the planes which would diffract the cancelling rays simply do not exist.

The broadening of a diffraction line is described by the Scherrer (1920) formula: \( t = k \lambda / B \cos \theta_B \). Where \( t \) is the particle size in cm., \( \lambda \) is the x-ray wavelength, \( B \) is the line broadening in radians at one-half the peak maximum, \( k \) is a constant, and \( \theta_B \) is the Bragg angle.

In considering the reciprocal lattice for a very small crystal it is found that the points become enlarged into volumes but that the volumes are of equal size for points in any radial row. Thus \( \Delta r^* \), where \( |r^*| \) is the length of the reciprocal lattice vector, is constant for different orders of hkl. Unlike strain broadening then, broadening due to small particle size is the same for different orders of a reflection.
Intrinsic and extrinsic stacking faults and twin faults are also produced during plastic deformation and produce changes in the x-ray diffraction pattern. Wagner (1957) gives a complete treatment of these effects. Much of the original theory was developed by Patterson (1952). Wagner, Tetelman, and Otte (1962) point out that the theory of diffraction effects from faulted structures rests on four assumptions:

1. The faulted layer extends across the entire coherently diffracting domain.

2. Where the faulting plane is one of several variants (such as \{111\} in FCC crystals) faulting occurs on only one variant.

3. In the case of alloys the scattering power of all \{111\} planes is the same.

4. Faults occur at random. There is no clustering of faults.

Warren (1961) found that assumption (1) is valid for a number of FCC metals and alloys including alpha brass. It is doubtful whether faulting in FCC metals is restricted to only one set of \{111\} planes. In cases where faults due to cold work are distributed on all sets of \{111\} planes Otte (1954) and Warren and Warekois (1955) show that the effect on the x-ray pattern is simply due to the sum of the faulting effects on all sets of planes. Thus \(d\) can be considered to be the sum of the fault probabilities for the four sets of \{111\} planes. Assumption (3) is thought to be valid in the case of alloys deformed at low temperatures (Wagner et al., 1962). There is evidence (Otte, 1957) that faults do tend to form in clusters. However, Wilkens (1960) shows that
any non-randomness of faulting is not likely to produce any effect more serious than a percentage error in the measured faulting probability.

In the FCC structure intrinsic stacking faults cause a broadening and a shift of certain \((hkl)\) reflections (Patterson, 1952). Those reflections which have \(h + k + l = 3n\) where \(n\) is an integer are not affected by stacking faults. Reflections with \(h + k + l = 3n - 1\) are those that have had their structure factor, \(F_{hkl}\), altered by the faulting. In the reciprocal lattice all points in a given class \([i.e., (111), (222), (333)]\) are elongated equally due to the faulting. The broadening due to stacking faults then is the same for different orders as is also the case with crystallite size broadening. The displacement of a diffraction peak as a consequence of faulting can be used to calculate the faulting probability \(\alpha\) as will be shown.

The effect of twin faults is to asymmetrically broaden a diffraction line and produce negligibly small peak shifts (Warren, 1959). There have been several measurements of the twin fault probability \(\beta\) from peak asymmetry measurements such as the displacement of the peak maximum from the center of gravity. Willis (1959) has shown, however, that the principal effect of segregation in FCC alloys is to make reflections asymmetrical. Wagner et al. (1962) point out that it is difficult to separate the component of asymmetry resulting from twin faults and that caused by segregation. Therefore the twin fault probability has not been reported in this study.

Extrinsic faults also cause a shift and broadening of diffraction lines. Warren (1959) has shown that the shift is equal in magnitude but opposite in direction to the shift caused by intrinsic faults.
The measured fault probability should therefore be equal to $\alpha - \alpha^1$. Wagner and Helion (1965) conclude that there is no evidence of extrinsic faults in alpha copper-zinc filings. Barrett and Massalski (1966) state that extrinsic faults are rare but have been found by Otte and Chessin (1966) in cerium. Extrinsic faults are therefore not considered to be of importance in this study and the faulting probability is taken to be due to intrinsic faults only. The broadening contributed by extrinsic faults is an asymmetric broadening unlike that caused by intrinsic faults. In the absence of extrinsic faulting this is of course neglected.

2.3.1 Calculation of Faulting Parameters

The broadening of powder pattern peaks is a result of faulting, small coherent domains, and microstrains. Wagner, Boisseau, and Aqua (1965) have shown that the profile of a diffraction peak can be represented in terms of a Fourier series:

$$ P_{(2\theta)} = K \sum_{L=-\infty}^{+\infty} \left\{ A_L \cos \left[ 4\pi L (\sin \theta - \sin \theta_0) / \lambda \right] + B_L \sin \left[ 4\pi L (\sin \theta - \sin \theta_0) / \lambda \right] \right\} $$

where $K$ is a constant given by Warren (1959) which depends on the experimental arrangement. $L$ is a distance normal to the reflecting planes. $\theta$ is from the intensity location $\theta$ in $P(2\theta)$. $\theta_0$ is the position of the observed peak maximum. $A_L$ and $B_L$ are the Fourier cosine and sine coefficients, respectively. Since the functions dealt with here
are symmetrical functions, the sine coefficients are zero and we are left with a Fourier cosine series:

\[ P_{(2\theta)} = K \sum_{L=-\infty}^{+\infty} A_L \left[ \cos \frac{4\pi L (\sin \theta - \sin \theta_0)}{\lambda} \right] . \]  

Warren and Averbach (1952) show that the coefficients \( A_L \) which describe the broadening of the peak are really the product of two coefficients. Thus \( A_L = A^{PF}_L \cdot A^D_L \), where \( A^{PF}_L \) is the part related to broadening from small particle size (i.e., small coherent domains) and faulting, \( A^D_L \) is the part due to inhomogeneous residual micro-strains. Instrumental broadening is removed through the use of an annealed standard in which the only line broadening is due to instrumental errors. These would include any displacement of the sample from the diffractometer axis and the use of a flat sample rather than a curved one. No broadening due to faults, lattice strains, or particle size exists in the annealed sample.

Warren and Averbach (1952) show that the coefficients \( A_L \) which describe the broadening of the peak are really the product of two coefficients. Thus \( A_L = A^{PF}_L \cdot A^D_L \), where \( A^{PF}_L \) is the part related to broadening from small particle size (i.e., small coherent domains) and faulting, \( A^D_L \) is the part due to inhomogeneous residual micro-strains. Instrumental broadening is removed through the use of an annealed standard in which the only line broadening is due to instrumental errors. These would include any displacement of the sample from the diffractometer axis and the use of a flat sample rather than a curved one. No broadening due to faults, lattice strains, or particle size exists in the annealed sample.

The Fourier coefficients of the annealed peak, \( A_L(\text{ann}) \), are used to remove the instrumental broadening from the cold-worked sample using the Stokes (1948) relation, \( A_L = A_L(\text{cw})/A_L(\text{ann}) \). \( A_L \) are the cold-worked coefficients corrected for instrumental broadening.

Wagner and Helion (1965) show that when the peak maximum is taken as the origin of the Fourier transformation the coefficients \( A^{PF}_L \) and \( A^D_L \) are given by:

\[ A^{PF}_L = 1 - L \left\{ \frac{1}{D_{(hk1)}} \frac{\langle \cos \varphi \rangle}{T} \left[ 1.5(\Delta + \xi_1) + \beta \right] \right\} \]  

\[ A^D_L = 1 - \left\{ \frac{2\pi L \sigma^2}{a_o} \right\} \langle \xi^2 \rangle \]  

Wagner and Helion (1965) show that when the peak maximum is taken as the origin of the Fourier transformation the coefficients \( A^{PF}_L \) and \( A^D_L \) are given by:

\[ A^{PF}_L = 1 - L \left\{ \frac{1}{D_{(hk1)}} \frac{\langle \cos \varphi \rangle}{T} \left[ 1.5(\Delta + \xi_1) + \beta \right] \right\} \]  

\[ A^D_L = 1 - \left\{ \frac{2\pi L \sigma^2}{a_o} \right\} \langle \xi^2 \rangle \]
where $D_{(hkl)}$ is the size of the coherently diffracting domains normal to the reflecting planes. $T$ is the width of the domains in the $\{111\}$ planes (i.e., the average width of the faulted region). $\cos \phi$ is the average value of the cosine of the complement of the angle between the $\langle hkl \rangle$ direction and the $\langle 111 \rangle$ direction. $\alpha$, $\alpha^1$, and $\beta$ are the probabilities previously discussed. $a_o$ is the lattice parameter and $V$ is a constant given by Warren (1959). $h_o^2 = h^2 + k^2 + l^2$ for the reflection being considered. $\langle \epsilon_L^2 \rangle$ is a component of the residual strain which is normal to the reflecting planes. It is an average value over the length $L$, squared, and averaged over all regions of the sample. Warren (1959) points out that while this is only one component of the strain it is often multiplied by three for use in computing a mean strain energy. The relation is: $V = 3E \langle \epsilon_L^2 \rangle / 2$ (Smallman and Westmacott, 1957). $E$ is Young's modulus.

Warren and Averbach (1952) have shown how to separate the cosine coefficients $A_L$ into their two components $A_L^{PF}$ and $A_L^D$. From reciprocal lattice considerations, as discussed previously, the broadening due to the residual strains varies with the order of the reflection. The quantity $h_o^2$ in Eq. (4) shows this dependence upon the order. It was also noted that the broadening due to small particle size and faulting does not depend upon the order of the reflection. Equation (3) bears this out. Writing the expression for $A_L$ in logarithmic form:

$$\ln A_L = \ln A_L^D + \ln A_L^{PF}$$

(5)
and replacing $A_L^D$ (strain term) by (4) gives:

\[
\ln A_L = \ln A_L^{PF} + \ln \left[ \frac{1 - 2 \pi^2 L^2 h_0^2}{a_0^2} \langle \xi_L^2 \rangle \right].
\]

Cotter (1967) shows the steps for the reduction of Eq. (6) into workable form using a Taylor series expansion. The final form of (6) is:

\[
\ln A_L = \ln A_L^{PF} - \frac{2 \pi^2 L^2 h_0^2}{a_0^2} \langle \xi_L^2 \rangle.
\]

$\ln A_L$ is then plotted versus $h_0^2$ for multiple orders of (hkl). The curves are plotted for values of $L$ in 10 Å increments. Typical curves are shown in Fig. 12. Examination of Eq. (7) shows that since $A_L^D$ is dependent upon the order of the reflection, extrapolation of these curves to $h_0^2 = 0$ gives values of $\ln A_L^{PF}$. If these values are non-zero then part of the broadening must be due to particle size and faulting.

The first derivative of Eq. (7) is:

\[
\frac{d(\ln A_L)}{d h_0^2} = \frac{-2 \pi^2 L^2}{a_0^2} \langle \xi_L^2 \rangle
\]

which upon rearrangement yields the mean squared strains knowing the slope of the plot of $\ln A_L$ versus $h_0^2$. Thus:

\[
\langle \xi_L^2 \rangle \text{(hkl)} = \left\{ \frac{a_0^2}{2 \pi^2 L^2} \left[ \frac{-d(\ln A_L)}{d h_0^2} \right] \right\}^{\frac{1}{2}}.
\]

The knowledge of the mean squared strains is necessary to the final calculation of the stacking fault energy.
Fig. 12. Typical $\ln A_L$ versus $h_o^2$ curves for first two orders of (111).
Warren and Averbach (1952) then plot the $A_{L}^{PF}$ values obtained from the intercept at $h_{o}^{2} = 0$ against their corresponding $L$ values. This is shown in Fig. 13. $L$ is the previously mentioned lattice distance in angstroms perpendicular to the reflecting planes. The intercept of this line with the $L$ axis gives a value for the effective particle size normal to the reflecting planes. This is an effective particle size since it includes the effect of faulting. If the true particle size is desired this contribution due to faulting must be removed.

The density of stacking faults (stacking fault probability) can be calculated directly from peak displacements in a cold-worked powder sample. It is for this reason that cold-worked filings are normally used in x-ray stacking fault studies. Wagner et al. (1965) point out that cold-worked bulk samples contain residual elastic strains that may not sum to zero, as well as possible preferred orientation. This net elastic strain results in peak displacements independent of any faulting effects. In randomly oriented filings however, residual macrostresses sum to zero and peak displacements are due to faulting alone.

The peak positions from the x-ray pattern of a well-annealed powder sample (e.g., without stacking faults) can be converted to lattice parameter values. If these are plotted against $\cos \theta \cot \theta$, all the $a_{(hkl)}$ values fall on a straight line. $\cos \theta \cot \theta$ is an extrapolation function, $f(\theta)$, suitable for the geometry of the diffractometer. Extrapolation to $\cos \theta \cot \theta = 0$ yields the true lattice parameter. When the material contains stacking faults the lattice parameters calculated from the cold-worked peak positions, $a_{(hkl)}$, deviate in a systematic way from the straight line. Four values of $a_{(hkl)}$ are normally
Effective particle size

Fig. 13. Typical $A_{\frac{PF}{L}}$ versus L curve for determining effective particle size.
calculated using the positions of the first and second order (111) and
(200) peaks. The theory of faulting in FCC metals (Warren, 1959) shows
that the deviation of the $a_{(hkl)}$ values from the straight line is
according to the values of the constant $G$. These values are given by
Wagner and Helion (1965) and these values are: $G_{(111)} = -3.45 \times 10^{-2}$,
$G_{(200)} = 6.89 \times 10^{-2}$, $G_{(222)} = 1.73 \times 10^{-2}$, $G_{(400)} = -3.45 \times 10^{-2}$.
Since $G_{(111)} \approx -1/2 G_{(200)}$ and $G_{(222)} \approx -1/2 G_{(400)}$ the straight line in
a plot of $a_{(hkl)}$ versus $\cos \theta \cot \theta$ can be drawn through the points
$a' = \left[\frac{2a_{(111)} + a_{(200)}}{3}\right]$ and $a'' = \left[\frac{2a_{(222)} + a_{(400)}}{3}\right]$ at
$f(\theta)' = \left[\frac{f(\theta)_{111} + f(\theta)_{200}}{2}\right]$ and $f(\theta)'' = \left[\frac{f(\theta)_{222} + f(\theta)_{400}}{2}\right]$,
respectively. As shown by Wagner et al. (1962) the stacking fault
probability $Q$ can be calculated without the use of an annealed standard
from the equation:

$$\frac{\Delta a_{(hkl)}}{a_{(hkl)}} = G_{(hkl)} Q$$

where

$$\Delta a_{(hkl)} = a_{(hkl)}^{\text{app}} - a_{(hkl)}$$

$\Delta a_{(hkl)}$ is thus the deviation of the lattice parameter $a_{(hkl)}^{\text{app}}$, (calculated
from the experimentally observed peak position) from the straight
line passing through the points $a'$ and $a''$. The point $a_{(hkl)}$ on the
line is given by:

$$a_{(hkl)} = \frac{a_0 + m \cos^2 \theta}{\sin \theta}$$
where $a_o$ is the extrapolated lattice parameter. The line given by Eq. (12) and the points $a_{(hkl)}^{app}$, $a_{(hkl)}$, and $a_o$ are shown in Fig. 14. A value for $\alpha$ is calculated for each of the experimentally measured peak positions using Eq. (10). It is important to emphasize that the theoretically derived constants $G_{(hkl)}$ make it possible to calculate $\alpha$ from this method. Without them it would be necessary to calculate $\alpha$ from peak position differences between an annealed standard and the cold-worked sample. This requires that the extrapolated lattice parameters for the annealed and cold-worked samples be identical. This is a very difficult condition to satisfy experimentally.

2.3.2 Calculation of Stacking Fault Energy

Data obtained from x-ray line broadening and peak shift measurements can be used to calculate the stacking fault energy. In studies by Smallman and Westmacott (1957) and Otte and Welch (1964) the stacking fault energy $\gamma$ was obtained from the relationship

$$\gamma = \left[ K_{(hkl)} \right] \frac{G a_o}{12 \kappa \sqrt{3}} \left\langle \varepsilon_{L}^2 \right\rangle_{(hkl)} / \alpha$$

(13)

where $K_{(hkl)}$ is a constant such that $K_{(111)} = 4K_{(400)} \approx 80$ and is independent of the properties of the material. $G$ is the shear modulus and $a_o$ the lattice parameter. $\left\langle \varepsilon_{L}^2 \right\rangle_{(hkl)}$ are the mean squared strains in the (hkl) direction determined from line broadening and $\alpha$ is the stacking fault probability from peak displacements. Equation (13) shows that for a given alloy $\gamma$ should be directly proportional to $\left\langle \varepsilon_{L}^2 \right\rangle_{(hkl)} / \alpha$ regardless of the type of deformation, since $G$, $a_o$, and $K_{(hkl)}$ are constant. Adler and Otte (1966) show that $\left\langle \varepsilon_{L}^2 \right\rangle_{(hkl)} / \alpha$ can
Fig. 14. Typical $a_{\text{app}}^{(hkl)}$ versus $\cos \theta \cot \theta$ curve for cold-worked filings.
vary with the mode of deformation. Thus they find that $\langle \varepsilon_L^2 \rangle_{(hkl)} / \alpha$ for filings is different by a factor of about three than the same ratio for drawn wires. From Eq. (13) then it could be concluded that $\gamma$ also varies with the type of deformation. Adler and Otte (1966) regard $\gamma$ as a more fundamental property, however, and one that helps determine the dislocation configuration rather than one that is determined by it. They therefore modify Eq. (13) so that $\gamma$ is the same regardless of the mode of deformation. They use the relationship

$$\gamma = \left[ \frac{K_{(hkl)}}{a_0 \sqrt{3}} \right] g \left[ \frac{\langle \varepsilon_L^2 \rangle_{(hkl)}}{\alpha} \right]$$

(14)

where $g$ is a proportionality constant between $\gamma^{-1}$ and the width $\omega$ of the extended dislocation. The value of $g$ depends upon the interaction of the leading partials at slip plane intersections and the barriers formed. With different modes of deformation there are different stress distributions and different dislocation configurations. Consequently different types of barriers can be expected to form at slip plane intersections. It is the factor $g$ which takes into account the mode of deformation. Adler and Otte (1966) give a complete derivation of Eq. (14).

Jøssang, Hirth, and Hartley (1965) treat all of the extended dislocation barrier configurations that occur in the FCC structure. These are, using Thompson's notation:

<table>
<thead>
<tr>
<th>Reaction</th>
<th>Barrier</th>
<th>$g$ (dynes)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. $B \delta (d) + \delta A (d) + D \delta (a) + \alpha B (a) = \delta A + D \delta + \alpha \delta$</td>
<td>$B (5.61 \times 10^{-6}$</td>
<td></td>
</tr>
<tr>
<td>2. $B \delta (d) + \delta A (d) + D \delta (a) + \alpha C (a) = \delta B + \alpha C + \delta D / \alpha \delta$</td>
<td>$12.54$</td>
<td></td>
</tr>
<tr>
<td>3. $B \delta (d) + \delta A (d) + C \delta (a) + \alpha D (a) = \delta A + \alpha D + \beta C / \alpha \delta$</td>
<td>$17.65$</td>
<td></td>
</tr>
</tbody>
</table>
4. \[ \beta \delta (d) + \delta A(d) + D \alpha (a) + \alpha B(a) = \alpha B + \beta \delta + \delta D/\delta \alpha \]

5. \[ \beta \delta (d) + \delta A(d) + D \alpha (a) + \alpha C(a) = \delta A + \alpha C + BD/\delta \alpha \] \quad 11.03 \text{ (average)}

6. \[ \beta \delta (d) + \delta A(d) + B J(c) + J D(c) = B \delta + B J + \delta J/AD \] \quad 18.44 \text{ (average)}

The small letters in parentheses refer to the plane in which the partial lies. Thus (d) refers to the plane opposite corner D of the tetrahedron. Reactions 1-4 form symmetric barriers with barrier (1) being the Lomer-Cottrell barrier mentioned previously. Reactions 5 and 6 form asymmetric barriers.

Using anisotropic elasticity theory they have calculated the \( \gamma \) values for each of these barriers for some alloys including some Cu-Zn alloys of from 4.1 to 22.7 per cent Zn. The value of \( \gamma \) is in units of dynes and is interpreted as the sum of the glide forces exerted by two arms of an extended barrier on the third arm. Adler and Otte (1966) extrapolated the values of \( \gamma \) for the copper-zinc alloys studied by Jøssang et al. (1965) to 30 per cent zinc. These extrapolated values for the various barriers for Cu-30w/o Zn are given with the above list of barriers.

For Cu-30w/o Zn the value of \( \gamma = 14 \text{erg/cm}^2 \) has been measured by Howie and Swann (1961) using the node method. Using this as their anchoring value for \( \gamma \) and the values for \( \left< \varepsilon^2 \right>_{(hkl)} \) and \( \alpha \) measured using the x-ray method by Warren and Warekois (1955) for Cu-30w/o Zn filings produced at room temperature, Adler and Otte (1966) used Eq. (14) to calculate a value for \( \gamma \) for Cu-30w/o Zn filings. They arrived at a value of \( 22.8 \times 10^{-6} \) dynes. Comparing this value with the extrapolated values given for this alloy they conclude that filing
produces both symmetric barriers of Type 3 and asymmetric barriers of Type 6. Using x-ray data for tensile loaded wire and the same \( J \) value they find that tensile deformation produces barriers of Types 2 and 3 primarily.

In discussing the application of Eq. (14) to x-ray diffraction data, these authors point out that when the stacking fault energy is low, dislocation pile-ups tend to form and Eq. (14) can be modified to take this into account. They modify Eq. (14) as follows:

\[
J = \frac{K(hkl)}{a_o \sqrt{3}} \cdot \frac{\xi}{\lambda} \frac{n}{F} \left[ \langle \xi_2 \rangle_{hkl} / \lambda \right]
\]

where \( n \) is the number of extended dislocations per pile-up and \( F \) is a factor which takes into account that there may be an interaction between the strain fields of the dislocations. \( F \) is given by

\[
F = \left[ \ln \left( \frac{R_o}{r_o} \right) + 0.776 n \right] / \ln \left( \frac{R_o}{r_o} \right)
\]

Where \( R_o \) is the outer cutoff radius and \( r_o \) is the core radius. \( F \) is generally in the range of 2 to 5. Adler and Otte (1966) assume that the dislocations piled up behind the barriers are dissociated screw dislocations and use the room temperature value of \( \xi = 4.2 \times 10^{-6} \) dynes which they extrapolate from the data of Jøssang et al. (1965). Using this value of \( \xi \) and the values of \( \langle \xi_2 \rangle_{hkl} \), \( \xi \), and \( J \) given previously for deformed filings, Adler and Otte (1966) obtain \( n/F = 6 \) using Eq. (15). Using \( F = 4 \), \( n \) is then in the range of 20 to 25 dislocations per pile-up.
In his review of the x-ray method of measuring the stacking fault energy, Otte (1967) uses a value for $\mathcal{G}$ for Cu–30w/o Zn filings of $19 \times 10^{-6}$ dynes as a compromise between Type 3 and Type 6 barriers. He takes $K_{(111)}$ to be 80. The value of $K_{(hkl)}$ is considered to be a critical quantity in making the x-ray method more valuable. Only estimates for $K_{(hkl)}$ are now available.

In calculating the quantity $\mathcal{G}$ and determining the types of barriers formed, Otte (1967) notes that the value of $\gamma$ from transmission electron microscopy is only thought to be accurate to within $\pm 10\%$. Since this was the anchoring value it affects the value of $\mathcal{G}$ as well. The error in $\gamma$ from Eq. (14) is thought to be greater than that of the anchoring value, probably about $\pm 20\%$.

Although the transmission electron microscope is far superior to the x-ray method for studying the details of dislocation structure, the x-ray method is capable of measuring the effects of very high dislocation densities (such as in deformed filings) in much greater volumes of material. The x-ray method takes into account the effect of large numbers of stacking faults while the electron microscope can examine only a few at a time. X-ray data are thus more meaningful in a statistical sense and correlate well with mechanical measurements.

2.4 Temperature Dependence of Stacking Fault Energy

Many authors have shown that the stacking fault energy in alloy systems is dependent upon composition. Vassamillet and Massalski (1963) found a decrease in stacking fault energy with increasing solute concentration in some binary inter-noble metal alloys between Au, Cu, and
Ag. In their x-ray studies they assumed that the stacking fault energy is proportional to $\lambda^{-1}$ and that this is valid for a constant dislocation density and configuration. The results of x-ray measurements by Warren and Warekois (1955) on Cu-Zn alloys show this same decrease of stacking fault energy with increasing solute concentration.

In a metal such as cobalt which has a HCP $\rightarrow$ FCC transition the stacking fault energy is clearly temperature dependent. This can be seen with reference to Fig. 15 which is a plot of the free energies of the two allotropic forms of cobalt as a function of temperature. Since the free energy of the hexagonal phase is lower below the transformation temperature this phase predominates. The FCC phase predominates above the transformation temperature. The stacking fault energy at any temperature is equivalent to the difference between the free energies of the two phases. The stacking fault energy is expected to be zero at the transformation temperature.

Working with Co-Ni alloys, Ericsson (1966) studied the temperature dependence of stacking fault energy between $20^\circ$ C and $710^\circ$ C using the electron microscope. His node measurements, made at the elevated temperatures showed that the stacking fault energy decreased with increasing temperature in the HCP phase and increased with temperature in the FCC phase. Tisone (1967) also reports an increase in stacking fault energy with temperature in a Cu-14w/o Al alloy. His node measurements were made on samples quenched from various temperatures. The node radius existing at the elevated temperature was assumed to be retained by the quench.
Fig. 15. Free energy as a function of temperature for cobalt.
Smallman and Westmacott (1957) have made x-ray studies of low temperature deformation in several copper and nickel alloys. They noted about a threefold increase in the stacking fault probability in pure copper when filing and x-ray analysis was done at liquid nitrogen temperature rather than at room temperature. This effect was apparently due to the non-equilibrium spacing of the partial dislocations and the presence of pile-ups. When samples were allowed to warm to room temperature the stacking faults annealed out (as measured by the stacking fault probability) along with a major portion of the line broadening. As the time at room temperature increased to 5000 minutes faults continued to anneal out and the stacking fault probability showed a steady decrease. At the same time the measured strains $\langle \varepsilon_L^2 \rangle_{(111)}$ showed a decrease as well, although the only values given are for the extremes of 0 and 5000 minutes at room temperature. Their data do not show the change in $\langle \varepsilon_L^2 \rangle_{(111)}/\sigma$ with temperature but as Eq. (14) shows, a change in this ratio would indicate a change in the stacking fault energy. The data of these authors do show however that the final recovered line breadth of the samples deformed at liquid nitrogen temperature is smaller than that of a sample deformed at room temperature, although greater than that of a well-annealed sample. They note that since the dislocation configuration in copper filed at room temperature is observed to be piled-up, the barriers that cause pile-ups of dislocations at liquid nitrogen temperature must be dispersed when the sample warms to room temperature. Thus in calculating the stacking fault energy in a sample filed at liquid nitrogen temperature and allowed to warm to room temperature, a change in the value of $\mathcal{G}$ in Eq. (14) from those for deformed filings, might be
necessary. This would compensate for changes with temperature in the types of barriers and the number of piled-up dislocations.

Christian and Swann (1965) state that in measuring the effect of temperature on stacking fault energy the x-ray method gives only a change in stacking fault probability which is related more to the recovery kinetics on unequilibrium stacking fault widths than to any change of $\gamma$ with temperature. This would be true if $\alpha$ was the only parameter measured. The measurement of both $\alpha$ and $\langle \mathcal{E}_{L}^{2} \rangle_{(hkl)}$, however, would seem to permit the calculation of $\gamma$ at various temperatures using Eq. (14).
III. OBJECTIVES

The objectives of this investigation were the following:

1. The determination of $\Delta$ and $\langle \xi_L^2 \rangle_{(hkl)}$ for Cu-30w/o Zn filed at liquid nitrogen temperature, using the x-ray diffraction method.

2. The determination of these same parameters in the sample at room temperature and at 100° C.

3. The calculation of the stacking fault energy of the alloy at the different temperatures.
IV. EXPERIMENTAL PROCEDURE

4.1 Specimen Preparation

4.1.1 Melting and Casting

The brass used in this investigation was prepared from 99.998% Cu donated by the American Smelting and Refining Company and 99.999% Zn provided by the Consolidated Smelting and Refining Company of Canada. The chemical analysis of these materials is given in Table 1. The component metals were chemically cleaned and sealed in an evacuated Vycor tube in the approximate ratio of Cu-30w/o Zn. Melting was accomplished by heating the tube in a muffle furnace at 1100° C for one hour. The molten charge was thoroughly mixed by shaking the Vycor tube. It was then allowed to cool in air. The tube was then replaced in the furnace and the alloy was homogenized for 72 hours at 800° C. Total ingot weight was approximately 550 grams.

4.1.2 Chemical Analysis of Cast Alloy

The brass alloy was analyzed using the x-ray fluorescence method. A General Electric XRD-5 spectrometer was used. The analysis required the preparation of five standard solutions of known weight per cent zinc. These solutions were prepared by first making separate solutions of Zn and Cu in nitric acid to a concentration of 0.01 g/cc. A standard of 30w/o Zn was then prepared by adding 30 cc. of Zn solution to 70 cc. of Cu solution. In this way standards of 28, 29, 30, 31, and 32w/o Zn were prepared. The ratio of the Cu K-alpha intensity to the Zn K-alpha
Table 1

Detailed Chemical Analysis of High Purity Copper and Zinc

<table>
<thead>
<tr>
<th>Material</th>
<th>Sb</th>
<th>Pb</th>
<th>Sn</th>
<th>Ni</th>
<th>As</th>
<th>Te</th>
<th>Se</th>
<th>S</th>
<th>Others</th>
</tr>
</thead>
<tbody>
<tr>
<td>99.999 + Cu</td>
<td>0.0001</td>
<td>0.0001</td>
<td>0.0001</td>
<td>0.0001</td>
<td>0.0002</td>
<td>0.0002</td>
<td>0.0001</td>
<td>0.0001</td>
<td>0.00017</td>
</tr>
<tr>
<td>99.999 + Zn</td>
<td>0.1</td>
<td>1.0</td>
<td>0.3</td>
<td>0.1</td>
<td>1.0</td>
<td>0.1</td>
<td>0.1</td>
<td>0.3</td>
<td>0.8</td>
</tr>
</tbody>
</table>

*Reported by suppliers.
intensity for these solutions was then determined using the x-ray spectrometer. This established a standard curve of the intensity ratio Cu K-alpha/Zn K-alpha versus weight per cent Zn. The weight per cent Zn in an unknown sample of the brass alloy, dissolved in nitric acid, could then be found by determining the Cu K-alpha/Zn K-alpha intensity relation and comparing this with the standard curve. This was done for samples taken from the top, middle, and bottom of the ingot. The results were as follows:

<p>| | | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Top</td>
<td>30.99 w/o Zn</td>
<td></td>
</tr>
<tr>
<td>Middle</td>
<td>30.68 w/o Zn</td>
<td></td>
</tr>
<tr>
<td>Bottom</td>
<td>30.77 w/o Zn</td>
<td></td>
</tr>
<tr>
<td>Average</td>
<td>30.81 w/o Zn</td>
<td></td>
</tr>
</tbody>
</table>

The actual filings used in the stacking fault study were also analyzed in this way and were found to be 30.79 w/o Zn. Diffraction conditions for the fluorescence analysis are given below.

- **KVP**: 50
- **Receiving Slit**: 0.01 in.
- **MA**: 6
- **Counting Time**: 1000 sec.
- **Tube**: Platinum
- **Scintillation Counter Voltage**: 1400
- **Analyzing Crystal**: LiF

Valenzuela (1965) has discussed quantitative analysis using x-ray fluorescence in detail.

### 4.1.3 Preparation of Brass Filings

The cold worked Cu-30w/o Zn powder was produced by filing the ingot while at liquid nitrogen temperature. The ingot was clamped in a
holder inside a stainless steel container. The container was then filled with liquid nitrogen until the ingot was submerged. A Nicholson mill-smooth file adapted for use in a sabre-saw was used for the filing operation. Filing for approximately 20 minutes produced enough filings for x-ray analysis.

The annealed standard required for the Stokes (1948) correction for instrumental broadening was produced in much the same way. Here, however, the filings were produced at room temperature and annealed under a hydrogen atmosphere for one hour at 450° C as suggested by Warren (1959). The annealed powder was stored in an evacuated dessicator until used in the diffraction analysis.

4.2 X-Ray Studies

The General Electric XRD-5 x-ray unit was aligned prior to the x-ray studies as outlined in the manufacturer's manual. The absolute zero of the instrument was set using a powdered sample of α quartz. The diffraction conditions used with both the cold-worked and annealed samples were as follows:

<table>
<thead>
<tr>
<th>Radiation</th>
<th>Co K-alpha</th>
<th>Counter Tube</th>
<th>Proportional</th>
</tr>
</thead>
<tbody>
<tr>
<td>KVP</td>
<td>50</td>
<td>Voltage</td>
<td>1805 volts</td>
</tr>
<tr>
<td>MA</td>
<td>12</td>
<td>Gain</td>
<td>9</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Take-off Angle</td>
<td>3°</td>
</tr>
<tr>
<td>Incident Beam Slit</td>
<td>3° MR</td>
<td>E out</td>
<td>5 volts</td>
</tr>
<tr>
<td>Diffracted Beam Slit</td>
<td>.010 in.</td>
<td></td>
<td>0.12°/min.</td>
</tr>
<tr>
<td>Scanning Speed</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Diffracted Beam Monochromator—LiF crystal, 0.010 in. slit
Intensity values were recorded by a digital printer which printed each 0.02° of 2θ.

It is important in studies of this type to maximize the peak-to-background ratio. This enables more accurate determination of the peak maximum positions resulting in better experimental values of d. It was found that Co K-alpha radiation minimizes fluorescence from the sample and thus improves the peak-to-background ratio. Copper radiation was found to be unsuitable due to the large amount of fluorescence produced in the sample by the short wavelength portion of the continuous spectrum. A diffracted beam monochromator using a doubly curved lithium fluoride crystal was utilized as well. This also helped to increase the peak to background ratio. A slow scanning speed, as recommended by Warren (1959) was used to minimize errors. The take-off angle of 3° is recommended by the manufacturer as providing good resolution as well as high intensity.

4.2.1 Depressed Temperature Studies

The cold-worked filings were kept at liquid nitrogen temperature throughout the diffraction analysis. The filings were left in the filing container until only a small amount of liquid nitrogen remained. They were then poured through a 35 mesh screen which was submerged in liquid nitrogen. This screening served to remove particles of ice which formed during filing. A small horseshoe magnet was used to remove particles of steel from the brass powder. These particles formed as the steel file deteriorated during filing. The filings were then transferred to the sample holder for the diffraction analysis. The sample holder,
shown in Fig. 16, was used for both the liquid nitrogen temperature and higher temperature studies. The tank which held the liquid nitrogen was made of 1/16-in. brass sheet surrounded by 3/8-in. thick styrofoam for insulating purposes. Tank capacity was approximately 400 cc. The front face of the tank was 1/8 in. stainless steel provided with a 1-in. wide opening for placement of the sample. A 0.020 in. thick section of copper sheet was soldered into this opening in such a way as to leave a depression 1/16 in. deep into which the sample could be packed. The copper sheet was thus between the powder sample and the liquid nitrogen in the tank, providing a high rate of heat abstraction from the sample. The sample holder was equipped with a micrometer adjustment which permitted the front face to be moved in or out with respect to the diffractometer axis. This made it possible to keep the face of the sample aligned with the diffractometer axis in spite of the expansion of the sample holder with temperature. Rotational adjustment of the sample holder about the diffractometer axis was also provided.

In order to load the filings into the sample cavity the entire sample holder was first immersed in a large tank of liquid nitrogen with the face of the sample holder in a horizontal position. This is shown in Fig. 17. The level of the liquid nitrogen was made just high enough to cover the sample cavity. The mixture of liquid nitrogen and cold-worked filings was then poured carefully into the sample cavity. A flat steel blade, cooled to liquid nitrogen temperature, served to press the filings into the sample cavity. The surface of the powder sample was then made completely flat and coincident with the stainless steel face of the sample holder. An extremely flat sample surface is critically
Fig. 16. Sample holder for x-ray studies.
Fig. 17. Sample holder in loading position.
important in assuring reliable results. When the sample holder was raised to the upright position with liquid nitrogen in its tank, the sample remained packed in the sample cavity. The sample holder was then mounted on the diffractometer and covered with a plastic envelope which was sealed to the base of the sample holder. This arrangement is pictured in Fig. 18. The purpose of the plastic covering was to prevent the formation of frost on the sample by maintaining a dry nitrogen atmosphere. The dry nitrogen was fed in through the base of the sample holder and emerged through the small section of copper tubing visible in Fig. 18. The dry nitrogen was cooled by passing it through coils cooled by liquid nitrogen as shown in Fig. 18. The nitrogen stream was directed at a point about 1 in. below the face of the sample.

In order to prevent attenuation of the x-ray beam by the relatively thick (0.004 in.) plastic envelope, a window of very thin (0.0005 in.) plastic was used. Both the incident and diffracted beams passed through such windows. Any decrease in intensity was not measurable. The outside of the plastic envelope and window was kept free of frost and moisture with streams of warm air from electric blowers. Liquid nitrogen from a pressurized flask was used to replenish the supply in the sample holder tank. This entered the tank at the top through a copper tube. Once the sample holder was in position on the diffractometer, approximately a half an hour was allowed for the temperature to stabilize. This minimized any chance of expansion or contraction occurring during the diffraction scan. This is an important point since any shift of the sample holder face causes a shift in the 2θ position of
Fig. 18. Sample holder in position on diffractometer.
the diffraction peaks. This would in turn affect the calculated value of $\alpha$.

4.2.2 Elevated Temperature Studies

When the diffraction scans of the first and second order (111) and (200) peaks had been obtained the liquid nitrogen in the tank was allowed to boil away. After a period of about two hours the sample was considered to have reached room temperature and the dry nitrogen flow was stopped and the plastic envelope removed. The diffraction scan at room temperature was then carried out. The sample was not removed or disturbed in any way. Except for the change in temperature the experimental conditions were unchanged.

The analysis at 100° C followed. This temperature was achieved by the simple expedient of boiling water in the sample holder tank. An immersion type electric heating coil was used with water added to the tank at the rate of about 10 ml/min. The water was allowed to boil for a half-hour before the diffraction scan was begun. This allowed the entire sample holder to adjust to the new temperature thus minimizing positioning errors.

Later a second sample was filed at liquid nitrogen temperature and then allowed to warm to room temperature for two hours. A diffraction scan of the (111), (200), (222), and (400) peaks was made. A diffraction scan of this sample was also made at 100° C. This second sample was used as a check on the data for room temperature and 100° C obtained from the analysis of the first sample. A screen analysis was carried out on both samples when the diffraction scans were complete.
In order to use the Stokes (1948) method of removing the instrumental broadening it was necessary to complete a diffraction scan of the annealed standard at each of the three different temperatures. Identical diffraction conditions were used for the cold-worked and annealed samples. The same sample loading procedure was not necessary for the annealed standard. The only requirement is that both the annealed and cold-worked samples be studied at the same temperature and under identical experimental conditions.

4.3 Analysis of Data

The Fourier cosine coefficients $A_L$ were obtained using the Stokes (1948) relationship. The actual computation was accomplished using a program compiled for the CDC 6400 computer. This program was written by Aqua (1964). The program removes the background intensity and the K-alpha-2 component of the K-alpha doublet by the Rachinger (1948) method. The positions of the peak maximums are located by a three-point parabola fitting routine. Simmons (1967) presents this program in detail. The Fourier coefficients were recorded on standard IBM cards for use in a second computer program.

This second program, written by Cotter (1967) calculates the mean squared strains $\langle c_L^2 \rangle_{(hkl)}$, and the stacking fault probability $\alpha$, from the Fourier coefficients and the peak positions, respectively. The program plots Eq. (7) analytically giving values for the intercepts at $h_o^2 = 0$ which are $A^\text{PF}_L$. The slopes of the curves for $\ln A_L$ versus $h_o^2$ are also computed and correspond to $\langle c_L^2 \rangle_{(hkl)}$. The effective particle
sizes can be determined by extrapolating the linear portions of the $A_{PF}^L$ versus $L$ curves to their intercepts on the $L$ axis as shown in Fig. 13.

The stacking fault probability $\alpha$ was calculated using Eq. (10). Initially the experimental peak positions were converted to $a_{\text{app}}^{(hkl)}$ values which were plotted versus $\cos\theta \cot\theta$. Then a straight line was fitted to the points $a'$ and $a''$ at $f'(\theta)$ and $f''(\theta)$, respectively. This gave values of $a_{(hkl)}$, which were then used in Eq. (11) for the determination of $\Delta a_{(hkl)}$.

The stacking fault energy calculation was not included in the computer program. The stacking fault energy was calculated for the three different temperatures using Eq. (14). The value of $\left< \epsilon_{L}^2 \right>^{(hkl)}$ used was $\left< \epsilon_{50}^2 \right>^{(111)}$. The $\left< \epsilon_{50}^2 \right>^{(100)}$ value does not give reliable results because of the extremely broad (400) peak normally found in cold-worked materials as pointed out by Otte (1967). The value for $L = 50 \, \text{Å}$ was used because this is the value commonly reported and used in the literature. The value of $\alpha$ used in Eq. (14) was the average value $\left[ \alpha_{(111)} + \alpha_{(200)} \right] / 2$. The values of $\alpha$ obtained from the (222) and (400) peak positions are less accurate as pointed out by Warren and Warekois (1955), since these peaks are extremely broad and there is considerable uncertainty in the location of the peak maximum position. The values of the constants $\theta$, $K$, $a_0$, and $F$ which were used in the actual calculations are reported in a later section.
V. RESULTS AND DISCUSSION

The values of the stacking fault probability, $\alpha$, were calculated using Eq. (10) and the experimentally determined peak positions. The rms strains were calculated from the slopes of the $\ln A_L$ vs. $h_0^2$ lines at $L = 50$ Å using Eq. (9). The stacking fault energy was obtained from Eq. (14) for each of the three temperatures. The number $n$ of dislocations in a pile-up was obtained using Eq. (15). These results, as well as values for $\bar{\epsilon}$ and $a_0$ used in calculating the stacking fault energy, are presented in Table 2. The room temperature results of Warren and Warekois (1955) and Wagner and Helion (1965) for $\alpha$ and $\langle \epsilon_5^2 \rangle_{\langle 111 \rangle}$ are shown also for purposes of comparison. The value shown as $\bar{\epsilon}$ is equal to $\frac{[a_{\langle 111 \rangle} + a_{\langle 200 \rangle}]}{2}$. The lattice parameter $a_0$ is the value determined from the individual $a_{\text{app}}^{(hkl)}$ values for the four reflections studied.

5.1 Low Temperature Results

The stacking fault energy of the cold-worked filings at 100° K was found to be 16.8 erg/cm$^2$. This is in good agreement with the room temperature electron microscopy results of Howie and Swann (1961) of 16.2 erg/cm$^2$. In calculating the stacking fault energy the value of $K(n) = 80$ was used as recommended by Otte (1967). Since $\bar{\epsilon}$ varies directly with the shear modulus $G$, the value of $\bar{\epsilon} = 19 \times 10^6$ dynes recommended by Otte (1967) for room temperature calculations, was adjusted for the change in $G$ with temperature. The measurements of the
<table>
<thead>
<tr>
<th>Sample</th>
<th>$\alpha_{(111)}$</th>
<th>$\alpha_{(200)}$</th>
<th>$\overline{\alpha}$</th>
<th>$\langle \varepsilon_{50}^2 \rangle_{(111)} \times 10^6$</th>
<th>$a_o$ (Å)</th>
<th>$\overline{\gamma}$ (dynes $\times 10^6$)</th>
<th>$\gamma$ (erg/cm$^2$)</th>
<th>n</th>
</tr>
</thead>
<tbody>
<tr>
<td>100° K</td>
<td>0.0240</td>
<td>0.0243</td>
<td>0.024</td>
<td>15.4</td>
<td>3.686</td>
<td>20.3</td>
<td>16.7</td>
<td>18</td>
</tr>
<tr>
<td>300° K #1</td>
<td>0.0180</td>
<td>0.0183</td>
<td>0.018</td>
<td>13.7</td>
<td>3.677</td>
<td>19.0</td>
<td>17.9</td>
<td>18</td>
</tr>
<tr>
<td>300° K #2</td>
<td>0.0144</td>
<td>0.0142</td>
<td>0.0143</td>
<td>12.9</td>
<td>3.676</td>
<td>19.0</td>
<td>21.5</td>
<td>18</td>
</tr>
<tr>
<td>373° K #1</td>
<td>0.0304</td>
<td>0.0306</td>
<td>0.0305</td>
<td>0.4 $\langle \varepsilon_{50}^2 \rangle_{(100)}$</td>
<td>3.685</td>
<td>17.9</td>
<td>0.3</td>
<td></td>
</tr>
<tr>
<td>373° K #2</td>
<td>0.0122</td>
<td>0.0122</td>
<td>0.0122</td>
<td>16.3</td>
<td>3.677</td>
<td>17.9</td>
<td>32.4</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Warren and Warekois (1955)</th>
<th>Wagner and Helion (1965)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\alpha = 0.023$</td>
<td>$\alpha = 0.022$</td>
</tr>
<tr>
<td>$\langle \varepsilon_{50}^2 \rangle_{(111)} = 12.96 \times 10^{-6}$</td>
<td>$\langle \varepsilon_{50}^2 \rangle_{(111)} = 12.96 \times 10^{-6}$</td>
</tr>
</tbody>
</table>
low temperature elastic constants of some alpha brasses made by Rayne (1959), and extrapolated to 30\% Zn were used. The value of $\mathcal{G} = 19 \times 10^{-6}$ dynes was then multiplied by $G(100)\,^\circ K/G(300)\,^\circ K$ to obtain $\mathcal{G} = 20.3 \times 10^{-6}$ dynes for the low temperature calculations. Both $\langle \varepsilon_{50}^2 \rangle_{(111)}/\mathcal{G}$ and the individual $\mathcal{A}$ and $\langle \varepsilon_{50}^2 \rangle_{(111)}$ values agreed closely with the room temperature x-ray results of Warren and Warekois (1955) and Wagner and Helion (1965). It seems somewhat surprising that the individual values of $\mathcal{A}$ and $\langle \varepsilon_{50}^2 \rangle_{(111)}$ are not considerably higher than those reported by these authors since the deformation here was at liquid nitrogen temperature. As noted previously, Smallman and Westmacott (1957) found that $\mathcal{G}$ and $\langle \varepsilon_{50}^2 \rangle_{(hkl)}$ increased with low temperature deformation. This effect can be explained, however, on the basis of the particle size of the filings and is discussed in a later section.

The value of $n \approx 18$ dislocations per pile-up was calculated using $\mathcal{G} = 16.8 \text{ erg/cm}^2$ and $\mathcal{G} = 4.4 \times 10^{-6}$ dynes for a dissociated screw dislocation. $F$ was taken to be 4 as suggested by Otte (1967). He points out that $F$ is usually in the range of 2 to 5.

5.2 Room Temperature Results

The average value of the stacking fault energy from the two room temperature determinations was $\bar{\gamma} = 19.7 \text{ erg/cm}^2$, only slightly higher than the low temperature value. This indicates little or no change in the stacking fault energy between 100° K and 300 ° K. The room temperature values are also in close agreement with the room temperature electron microscopy results of Howie and Swann (1961). The values of
$\alpha$ and $\langle \epsilon_{50}^2 \rangle_{(111)}$ for the two room temperature measurements were in close agreement with each other indicating good reproducibility at this temperature. The measured values of both $\alpha$ and $\langle \epsilon_{50}^2 \rangle_{(111)}$ showed a decrease when the sample was warmed to room temperature in agreement with the results of Smallman and Westmacott (1957). The ratio $\langle \epsilon_{50}^2 \rangle_{(111)}/\alpha$ showed a slight increase in both of the room temperature samples. In the calculation of $f$, the value of $f$ was adjusted to compensate for the change in the shear modulus in going from 100° K to room temperature. The value of $n$ determined from Eq. (15) using $\theta = 4.2 \times 10^{-6}$ dynes for dissociated screw dislocations did not change in going from 100° K to 300° K.

5.3 Elevated Temperature Results

No value for $\langle \epsilon_{50}^2 \rangle_{(111)}$ for the first elevated temperature sample is given since the measured value was less than the minimum value of $10^{-10}$ allowed by the computer program. A considerable decrease from the room temperature value would be expected, however, due to the stress relieving effect of the elevated temperature. The value for the $[100]$ direction was $40 \times 10^{-8}$ and is the one given in Table 2.

The measured value for $\alpha$ for this sample showed an increase in the faulting probability over both the 100° K and 300° K measurements. This is a questionable result since other studies (Wagner, 1957; Smallman and Westmacott, 1957) have shown that faults produced at liquid nitrogen temperature anneal out at or slightly above room temperature. A further decrease in $\alpha$ from the room temperature value is to be expected.
The second sample was analyzed at 100° C and again the results appear anomalous. While the faulting probability in this sample does show a decrease from the room temperature value, the measured rms strains were found to be slightly higher than those at the other temperatures rather than lower. For the first sample, \( \alpha \) seems unreasonably high, while for the second, \( \langle \varepsilon_{50}^2 \rangle_{(111)} \) appears to be unreasonably high. This situation makes the validity of any calculated value for the stacking fault energy at this temperature very questionable.

Calculations based upon \( \langle \varepsilon_{50}^2 \rangle_{(100)}/\alpha \), using \( \langle \varepsilon_{50}^2 \rangle_{(100)} = 40 \times 10^{-8} \) for the [100] direction, for the first sample show \( \gamma = 0.29 \) erg/cm\(^2\). The measured value of \( \langle \varepsilon_{50}^2 \rangle_{(111)}/\alpha \) for sample No. 2 yields \( \gamma = 32.4 \) erg/cm\(^2\). It should be noted that since the \( \gamma \) value for the first sample is based upon \( \langle \varepsilon_{50}^2 \rangle_{(100)} \) rather than \( \langle \varepsilon_{50}^2 \rangle_{(111)} \) it is not strictly comparable to the other measurements. The two \( \gamma \) values at this temperature differ greatly, however, and it is felt that neither can be considered completely reliable. Since \( n \), the number of dislocations in a pile-up is calculated using these values for \( \gamma \), its reliability is affected as well.

### 5.4 Discussion of Results

As mentioned previously, Smallman and Westmacott (1957) found that low temperature deformation of pure copper produced a much higher faulting probability \( \alpha \) and \( \langle \varepsilon_{L}^2 \rangle_{(hkl)} \) value than did room temperature deformation. Filings formed at room temperature had a faulting
The fact that the stacking fault probability for the sample filed at liquid nitrogen temperature in this study was not considerably higher than that measured by Wagner and Helion (1965) who filed at room temperature, may be explained on the basis of the particle size of the sample. Anantharaman (1961) found that as the size of filed particles increases the stacking fault probability and the x-ray line breadth decrease. He concludes that the larger particles have undergone less deformation and thus have lower stacking fault densities than the smaller particles. For Cu-33w/o Zn filed at room temperature he found that $\mathcal{Q} = 0.039$ when the filings were all less than 0.06 mm. For a sample with particles all greater than 0.22 mm the stacking fault probability decreased to 0.029. At the same time the line breadth also decreased. Since residual lattice strains contribute to the breadth of x-ray reflections it is expected that $\langle \xi^2 \rangle_{(hk\ell)}$ decreased as well.

A screen analysis of the samples used in this study showed that while only 2.4w/o of the sample had particles greater than 0.208 mm, 11.4w/o was greater than 0.147 mm and 57.1w/o was between 0.053 mm and 0.147 mm; 31.5w/o was less than 0.053 mm. Since the sample was loaded while the sample holder was in the horizontal position it would be expected that the smaller particles would settle toward the back of the sample cavity leaving the larger particles nearer to the surface. Thus the small particles would contribute less to the total diffracted beam than the larger particles nearer to the sample surface. The data of Wagner and Helion (1965) were based upon a sample with particles less
than 0.06 mm. For these reasons the stacking fault probability and rms strains for the sample at 100° K are not considered to be inordinately low.

The value for $q$ at 300° K was adjusted for the change in the shear modulus in going to room temperature but not for any change in the nature of the barriers present. Although the collapse of some of the weaker barriers with increasing temperature is expected as pointed out by Smallman and Westmacott (1957), and Otte (1967), the relative strengths of the various barriers are not well known. Also since the $q$ value used corresponds to barriers of Type 3 and Type 6, and is virtually the largest that can occur, it does not seem that the collapse of some of the barriers would significantly affect the value of $q$. Barriers with higher $q$ values have been postulated (Teutonico, 1964) but have not been treated in detail. Thus it seems unwarranted to make any assumptions regarding a change in $q$ with possible barrier collapse.

The decrease in $\sigma$ and $\langle \varepsilon_{50}^2 \rangle_{(111)}$ which occurred with warming to room temperature is in agreement with the results of Smallman and Westmacott (1957) and Wagner (1957). The decrease in $\langle \varepsilon_{50}^2 \rangle_{(111)}$ with increasing temperature is associated with some barrier collapse at the head of piled-up groups as well as the rearrangement of non-equilibrium dislocation configurations such as tangles into arrangements with lower values of residual stress. The partial recombination of dissociated dislocations is responsible for the decrease in $\sigma$ with increasing temperature. A decrease in the width of stacking faults results in less total faulted area and a lower faulting probability. The important point is that the decrease in the faulting probability obtained in this
study seems to be due to thermally activated processes rather than to any increase in the stacking fault energy with temperature. The stacking fault energy values obtained for 100° K and 300° K differ only slightly and this difference is not considered significant. The driving force for the decrease in the widths of faults is supplied by the internal stress. As the temperature is increased the internal stress is thought to be sufficient to overcome the resistance to the motion of dislocations provided by jogs and forrest dislocations. The decrease in the faulting probability thus seems to be associated with the amount of deformation that can be introduced and retained at depressed temperatures. The increased temperature allows the partials to assume a narrower spacing.

It is unfortunate that the anomolous results for $\alpha$ and $\langle \varepsilon^2_{50} \rangle$ in the first and second elevated temperature samples do not permit the calculation of reliable values for the stacking fault energy. The increase in $\alpha$ for the first sample at 100° C may have been caused by a slight shifting of the face of the sample during the analysis. Although great care was taken to insure that this did not occur, it seems to be the only reasonable explanation.

An indication of the decrease in the value of $\langle \varepsilon^2_{50} \rangle$ from 300° K to 373° K is found in Fig. 19. Here an average line has been drawn through the actual diffractometer trace for the (111) reflection from the sample at 300° K and 373° K. The two (111) profiles were then superimposed. The breadth of the peak for 100° C is not as great as that for the sample at room temperature. This decrease in breadth is due mainly to the large decrease in the lattice strains, $\langle \varepsilon^2 \rangle_{(hkl)}$. 

Fig. 19. Comparison of (111) profiles for 300° K and 373° K.
Figure 20 compares the peak breadths for the same reflection, of the sample at 100° K and room temperature. While the room temperature peak is slightly narrower, the data show that the rms strains did not decrease nearly as much going from 100° K to room temperature as from room temperature to 373° K. Since small domain size and faulting effects also contribute to line broadening, as discussed previously, it is probable that an increase in the domain size and decreased faulting account for most of the decrease in line breadth at room temperature.

The extremely high value for \( \langle \varepsilon^2_{50} \rangle_{(111)} \) for the second sample at 100° C seems to be due to some experimental error. It is not known exactly what caused the rms strains to be so large but any change in the contour of the sample face could have contributed to this error. The large decrease in \( \langle \varepsilon^2_{50} \rangle_{(hkl)} \) noted in the first sample at 100° C is thought to reflect the true situation. An increase in \( \langle \varepsilon^2_{50} \rangle_{(hkl)} \) with temperature must be due to errors. Time limitations precluded a further investigation of the 100° C temperature.
Fig. 20. Comparison of (111) profiles for 100° K and 300° K.
VI. CONCLUSIONS

The results of this study permit the following conclusions to be drawn.

1. The stacking fault energy of Cu-30w/o Zn deformed at liquid nitrogen temperature does not appear to change significantly when the temperature is raised from 100° K to 300° K. The decreased stacking fault probability and recovery effects noted with the increased temperature are considered to be caused by the unpiling of some piled-up groups, the collapse of some weak dislocation barriers, and an increase in the size of the coherently diffracting domains. The narrowing of stacking fault ribbons as a result of the thermally activated processes described, and not an increase in the stacking fault energy, also contributes to the recovery effects.

2. The recovery effects noted when the temperature was raised to room temperature are associated mainly with decreased faulting and an increase in the size of the coherently diffracting domains. Raising the temperature from 300° K to 373° K brought about the decrease in the rms.strains previously noted.

Otte's (1967) analysis of the x-ray method for determining the stacking fault energy has been used as a basis for calculations of the stacking fault energy in this study. Prior to the publications by Adler
and Otte (1966) and Otte (1967) the mode of deformation and the types of barriers formed were not considered in stacking fault studies.

The present study was conducted using Cu-30w/o Zn in order to confirm the reliability of the method as there is considerable published data for this alloy. The low temperature sample holder developed for this study permits an extremely flat sample face to be formed while the sample is at liquid nitrogen temperature. Another advantage is that no window is required to cover the sample face. This results in a more intense incident and diffracted beam. In its present state the sample holder can be used to attain temperatures up to about 170° C utilizing boiling liquids. Soldered connections in the tank portion will fail above this temperature. This sample holder should be very useful in further stacking fault studies.

The x-ray method of measuring the stacking fault energy seems to be the best technique for measuring this parameter at depressed temperatures. It would be difficult indeed to introduce low temperature cold work into a thin foil specimen for the electron microscope and then transfer and maintain the sample at the depressed temperature. Furthermore there is no low temperature tilting stage available for the electron microscope thus making the examination of such a specimen very difficult. The x-ray method also permits the measurement of the stacking fault probability, a parameter unique to this method. This is a meaningful parameter and gives important information as to the density of stacking faults in a particular sample.
It should be useful to study the reproducibility of data obtained from some common FCC metals and alloys filed at room temperature. Prior to the present study values of $\alpha$ and $\langle \varepsilon^2 \rangle_{50}$ were obtained for 325 mesh Cu-30w/o Zn filings formed at room temperature. These values correspond very closely to those of Wagner and Helion (1965) for the same alloy. Room temperature filing and X-ray examination eliminates the tedious and delicate experimental work required for studies at liquid nitrogen temperature.

A further investigation into the effects of elevated temperatures upon cold work introduced at liquid nitrogen temperature should be carried out. Specifically the 100°C temperature should be studied in an effort to determine the source of the errors encountered in this study for that temperature.

A study of the effect of particle size on the stacking fault probability and rms strains should be made. While Anantharaman (1961) has investigated this, further studies should be made. His measurements were of $\alpha$ and the line breadth and $\langle \varepsilon^2 \rangle_{L}$ for (hkl) was thus only measured indirectly. Since small domains also contribute to line broadening it should be interesting to study the effect of the particle size upon both residual strains and the size of the coherently diffracting domains.

It was found to be practical to screen a sample under liquid nitrogen. Although only a 35 mesh screen was used in this study, finer...
screens could also be used. Thus the particle size could be made at liquid nitrogen temperature. This has never been done before.

In order to utilize Eq. (14) for the calculation of the stacking fault energy from x-ray data, it is necessary to have values for the force factor $\gamma$. The values of Jøssang et al. (1965) were used in this study. For other FCC metals and alloys, it would be necessary to calculate values of $\gamma$ for the various barriers using anisotropic elasticity theory. This could be done for an alloy that has not been studied before. Subsequent x-ray analysis would yield measured values for $\gamma$ and $\langle\xi_L^2\rangle_{(hkl)}$ which could be used to calculate the stacking fault energy. The electron microscope could be used as a check on such measurements.
SELECTED BIBLIOGRAPHY

Amelinckx, S., and Delavignette, P., Electron Microscopy and Strength of
Aqua, E. N., Computer Applications in Metallurgical Engineering,
American Society for Metals (1964), p. 27.
Barrett, C. S., and Massalski, T. B., Structure of Metals (third
Christian, J. W., and Swann, P. R., in Massalski, T. B. (ed.), Alloying
Behavior and Effects in Concentrated Solid Solutions, Gordon
and Breach (1965), pp. 105-269.
Cotter, K. H., Stacking Fault Probability in Aluminum Alloys, M. S.
Cottrell, A. H., Phil. Mag., 43, 645 (1952).
(1962).
Frank, F. C., Phil. Mag., 42, 809 (1951).

Howie, A., and Swann, P. R., Phil. Mag., 6, 1215 (1961).


Lomer, W. M., Phil. Mag., 43, 645 (1951).


