A Study of the Effects of Vacancies on the Angular Correlation of Positron Annihilation Radiation for Iron and Copper

Ronald L. Meade

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A STUDY OF THE EFFECTS OF VACANCIES
ON THE ANGULAR CORRELATION OF POSITRON ANNIHILATION
RADIATION FOR IRON AND COPPER

by

Roland L. Meade

A Thesis
Submitted to the
Faculty of The Graduate College
in partial fulfillment
of the
Degree of Master of Arts

Western Michigan University
Kalamazoo, Michigan
April 1972
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The iron samples used in this study were prepared by Dr. C. Lewis Snead, Jr., Brookhaven National Laboratory. His help is gratefully acknowledged.

Roland L. Meade
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CHAPTER I

INTRODUCTION

Many of the properties of metals depend on the electron states and the distribution of electrons among these states. Angular correlation curves can give information on the momentum spectra of electrons in a sample. This technique has been used in recent years to study the momentum spectra of various metals. Researchers have discovered that defects in metals, particularly vacancies, can cause changes in the angular correlation curve.

It is known that vacancies play a role in determining the bulk physical properties of solids such as electrical resistivity, volume change, and mechanical properties (e.g. elastic modulus, internal friction, and hardening). Experimental methods for studying point defects fall into two classes: direct observation and measurement of a change in some bulk physical property of the solid that is related to the presence of the defects. Most information currently available is based on the second method (2). It appears that angular correlation techniques might prove to be another independent tool for studying point defects, hopefully with a wider range of application than present techniques.

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The purpose of this investigation was to study the changes produced in the angular correlation curve due to the introduction of vacancies into the crystal lattice of the metal samples. Since vacancies are able to migrate at elevated temperatures, the samples were stepwise annealed at elevated temperatures (below the melting point) to determine if vacancy migration and subsequent annihilation with interstitial atoms could restore the angular correlation curve to its original shape.
CHAPTER II

BACKGROUND FOR THE STUDY

Vacancies and Defects

A vacancy is a lattice site in the crystal which is missing an ion. It can be considered as a negative charge due to the absence of the positive ion, and as such it can attract the positively charged positron. The positive ion which is removed may become an interstitial ion.

The formation of vacancies and interstitials in a crystal is temperature dependent. The number of vacant sites is given by

\[ n_v = A N e^{-E_v/kT} \]  

where \( A \) is an entropy term usually taken to be a constant, \( N \) is the total number of atom sites, \( E_v \) is the formation energy (for a vacancy), \( k \) is the Boltzmann constant, and \( T \) is the absolute temperature. The number of interstitials is given by

\[ n_i = A N e^{-E_i/kT} \]  

where \( E_i \) is the formation energy of the interstitial and \( A \) is an integer, usually small, which is the number of equivalent interstitial sites per lattice atom (3).

The formation energy of the interstitial is about four times that of the vacancy hence the formation of vacancies is favored.

Defects can be introduced into a crystal by plastic deformation. However, the simultaneous introduction of large numbers of dislocations, which can interact with point defects as sources,
sinks, and traps, makes the interpretation of experiments difficult. (Point defects include vacancies and interstitial atoms. (2) ) Since the formation energy is higher for interstitials, the vacancy is the primary defect produced (3). The activation energy for migration of the interstitial is lower than that for the vacancy, hence interstitials anneal out at a lower temperature. In copper recovery from deformation due to annealing in stages II and III is thought to be due to the migration of interstitials (the temperature range for stage II is -213° C to -33° C, and for stage III, -33° C to 67° C) (2). Vacancy complexes are also produced by deformation; these include multiple vacancies and dislocation loops.

Defects may be produced by irradiation with protons, electrons, neutrons or other particles; irradiation of samples reported here was done with electrons. Electrons with an energy of 1 to 3 MeV can create one, or at most a few, vacancy-interstitial pairs for most metals (2). Electrons of this energy, since they do not have sufficient energy to cause large transient temperature changes produce the simplest distribution of vacancies and interstitials.
Angular Correlation Technique

In angular correlation studies the conversion of matter into energy by positron annihilation is utilized to gain information about the substance in which the annihilation takes place. The relationship of mass to energy is given by the Einstein equation \( E = mc^2 \), where \( E \) is the energy, \( m \) the mass, and \( c \) the speed of light. More precisely, \( E_t = 2m_e c^2 + E_k \) (applying conservation of mass-energy), where \( E_t \) is the total energy of the emitted photons, \( m_e \) is the rest mass of the electron (or positron), and \( E_k \) is the kinetic energy of the positron-electron pair which annihilates and produces either two or three photons of energy. In the triplet state the spin directions of the pair are parallel and three gamma annihilation results, with the total angular momentum of the emitted photons equal to \( h/2\pi \), the angular momentum before annihilation. In the other state, with spins anti-parallel, and hence angular momentum zero, annihilation produces two gamma rays; the two-gamma event is favored over the three gamma by a ratio of 373:1 (1). (Other three-gamma states are possible, but their probability of occurrence is very low.) The angular correlation technique is designed to detect radiation produced by the two-gamma events and record the number of events as a function of the momentum of the pair.
If the annihilating particles are at rest, the total momentum of the system is zero and the photons must be emitted in opposite directions, with equal energy. If, however, $E_k$ is not zero, the emitted photons will be separated by some angle other than 180°. The angular correlation experiment consists of measuring the number of photon pairs given off at various angles for the same counting time.

After positrons enter a metal they are slowed to thermal velocities by collisions within the crystal in about $10^{-12}$ seconds (15). Since the time for annihilation is typically about $10^{-10}$ seconds, the positron can be considered to be in its lowest energy state (16). The total momentum of the pair then is $mv$, the product of the mass and the velocity of the electron.

Consider a coordinate system in which the line joining the two gamma-ray detectors and the sample defines the $y$-axis; a line through the sample (more precisely the annihilating pair) midway between the detectors parallel to the detector slits and perpendicular to the $y$-axis, defines the $x$-axis; and a third line through the sample and perpendicular to the $xy$ plane defines the $z$-axis. Such an arrangement is shown in Fig. 1. In this coordinate system the annihilating pair will have three components of momentum, $p_x$, $p_y$, and $p_z$. The momentum due to $p_y$ will cause a Doppler shift of the emitted gamma rays; the electronics can be set to accept a range of energy which will include this...
shifted energy. The component $p_x$ will cause a deviation of the angle between the gamma rays in the xy plane, hence the collimating slits and the detectors are orientated with their long direction in the xy plane to count gamma rays regardless of their $p_x$ component of momentum. The z component of momentum, $p_z$, will cause a deviation in the xz plane.

As the momentum of the electron increases, the angle between the emitted photons will change. If the momentum of the electron is zero, the angle between the emitted photons will be 180°. Theta can be defined as the angle in the xz plane measured above or below this 180° line (see Fig. 1). Theta then is a measure of the momentum of the annihilating electron. It was shown in (17) that the angle $\Theta$ is approximately equal to $p_z/mc$. The movable detector can be set at various values of $\Theta$ to measure the momenta of annihilating electron-positron pairs.

The angular correlation curves for the metals considered are roughly Gaussian in shape. Since the curves are symmetrical they can be folded about the y-axis for comparison. Changes in the curve are caused by plastic deformation or irradiation of the sample. The observed changes are an increase in the maximum value and a narrowing of the curve. This represents a decrease in the high momentum component of the electron spectrum. For comparison the curves are usually normalized to a given area.
Previous Experimental Work

Changes in the angular correlation curve have been noted in studies of the effects of temperature on metal samples, and more recently, in studies involving the introduction of defects into metal samples. Indications are that vacancies play an important role in these changes.

MacKenzie et al. (4) reported increases in the peak heights of angular correlation curves at elevated temperatures for zinc, indium, and cadmium. They interpreted this as an increase in annihilations of positrons with valence electrons. In the same article they reported on small variations in the shape of angular correlation curves with temperature change for lead, tin, aluminum, and bismuth. However these latter changes could be explained by changes in the lattice volume. In their discussion of possible explanations for the changes in the curves for zinc, indium, and cadmium, they rejected the hypothesis that Coulomb repulsion excluded the positron from a volume around the ion cores and enhanced the annihilation of positrons with valence electrons. They cited work by Berko and Plaskett (5) which showed no evidence of structure in the tails for copper (of the angular correlation curve) which the Coulomb repulsion model predicted. Volume changes were ruled out for these metals as a cause for the increases in the peak heights, since a 15%
to 20% increase in volume would be needed to produce the observed effect; the actual volume change was less than 3%.

Dekhytar et al. (6) reported similar changes for plastically deformed Ta, Nb, and Fe-Al in 1966 (7). They interpreted the results in terms of a redistribution of the s and d electrons in the strain field around dislocations produced in the crystal by deformation.

In 1967 Berko and Erskine (8), on the basis of angular correlation curves obtained for aluminum which they estimated had been deformed from 10% to 16%, concluded that the observed narrowing of the angular correlation curve indicated that the positrons were annihilating in regions of lower electron density. (They mentioned also that observed increases in annihilation lifetimes supported this idea.) They suggested that these regions might represent attractive potential sites for the positron; this model could also explain similar results for metals at elevated temperatures.

In the same year MacKenzie et al. (9) reported an increase in the positron mean lifetime in heated metals, in some cases as much as 30%; they studied positron lifetimes in Al, Cd, In, and Zn. They attributed their results to the attraction of positrons by vacancies (interstitials being fewer in number and probably having a different effect on the lifetimes) which had been produced by heating the samples. They pictured the vacancy as an attractive site unable to restrain the positron in the small
volume to form a bound state. The attraction should increase the probability of annihilation with conduction electrons and decrease the probability of annihilation with core electrons. The attractive site should be smaller in size than the ion it replaced because of an inward relaxation of the lattice.

Adamenko et al., in explaining the narrowing of the angular correlation curve and the increase in the peak, pictured the dislocation core as a potential well for thermalized positrons; their results were obtained for samples of Ni-Co, and Ni-Fe alloys in the deformed and annealed states (10).

Brandt and Waung proposed vacancy concentration upon melting as the cause for variances with the predictions of the free electron gas model for positron lifetimes in Na and Ga (11).

In 1969 MacKenzie (12) found that the annihilation line-shape was sensitive to the influence of lattice defects introduced by plastic deformation or heating in Al, Au, and Cu. He suggested that angular correlation studies for deformed Cu might produce greater effects than those observed for Al by Berko and Erskine (8), since his results indicated a more marked effect in Cu than Al. He observed that In was not influenced by defects from cold-working but was influenced by defects resulting from increased temperature. The Ge(Li) spectrometer which he used had resolution adequate to measure changes in the core distribution, but it was insensitive to the small changes in the parabolic part of the momentum distribution.
In 1970 studies were conducted at Western Michigan University by Kusmiss and Esseltine on the effects of deformation and irradiation on Pt samples. Angular correlation curves showed similar changes from both processes. Stepwise annealing was used to determine the effects of recovery of the samples at various annealing temperatures (13).

Huang in 1969 reported a narrowing and an increase in the maximum value of the angular correlation curves for deformed or irradiated Ni and irradiated Fe (14). This paper is a report of the continuation of those studies for deformed or irradiated Fe and for deformed Cu.
CHAPTER III

EXPERIMENTAL PROCEDURE

Equipment Arrangement

The experimental setup is shown in Fig. 2. The sample was placed on a line connecting the two detectors, 50 inches from either detector. A chamber of two-inch-thick lead prevented stray radiation from the source from reaching the detectors. The sample height was adjusted for the maximum number of counts; the height of the source was adjusted to prevent the detectors from "seeing" the source directly. The photon beams from the source chamber passed through a collimating slit four inches long and 0.5 inches high, cut in a lead brick two inches thick and placed twelve inches from the source chamber. The source chamber, a cube eight inches on each side, contained slits on two sides whose long dimension was aligned along the x-axis. Slits in front of the detector, twelve inches long with a 0.05 opening in the plane of the detector (z plane), provided an angular resolution of one milliradian. Additional lead bricks shielded the detectors from stray radiation. One of the detectors was fixed in position, as was the source chamber. The other detector was mounted on a movable platform which could be moved in steps of one milliradian by an electric motor. Adjustable microswitches controlling the motor determined at how many positions on either side of 0° annihilation events were counted.

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Fig. 1. Coordinate system for the angular correlation. The origin is the location of the annihilating pair.
Fig. 2. Experimental Setup (from 14).
The detectors used were two 2 x 6 inch Harshaw NaI (Tl) crystals. These were optically coupled to RCA 6342A photomultiplier tubes with Dow Corning QC-2-0057 silicone grease. The electrical output of each photomultiplier was fed to a separate Hamner NB-12 preamplifier, through a Hamner NA-12 double-delay-line amplifier, and to a Hamner NC-14A pulse height analyzer. The analyzer was set to pass pulses corresponding to energies in the range $E + \Delta E$, allowing gamma rays to be counted regardless of what their Doppler-shifted $y$-component of momentum was. Pulses passed by the analyzer were counted by a Hamner NR-10 ratemeter. The outputs of both analyzers were fed to a Hamner NR-16 fast ramp coincidence circuit. This circuit would only pass pulses which occurred within thirty nanoseconds of each other. (The resolving time for coincidence was set at thirty nanoseconds: it could be varied from 5 nanoseconds to 150 nanoseconds.) Counting only events which occur in a given time interval from each other allows annihilation events to be distinguished from stray radiation. If the incoming pulses occurred within thirty nanoseconds of each other a pulse was fed to the Ortec-431 scaler which recorded the event.

The events were counted for ten minutes (this time interval was adjustable) at each position of the detector and the count was printed out at the end of the ten minutes by a Teletype. Then the control circuit activated the motor to move the detector through one milliradian and the counting began again. (The scaler
was reset to zero after each print-out.) There were about 32 positions (this was adjustable with the limiting microswitches on the movable platform) so that 320 minutes were required for each run. (For the data considered here the microswitches were set at approximately -14 to +18 milliradians.) Enough runs were completed for each sample to obtain at least 20,000 counts in the peak of the angular correlation curve. Hence the first step in the data reduction consisted of summing the ten-minute observations for each point at the one milliradian intervals. The final results were plotted as a curve, roughly Gaussian in shape with 20,000 or more counts in the peak. Typical count rates in the tail of the curve were about 1,000 giving a maximum uncertainty of \( \sqrt{1,000} = \pm 33 \) or about 3.3% fractional uncertainty.

The positron source emits positrons according to the scheme

\[ ^{11}\text{Na}^{22} \rightarrow ^{10}\text{Ne}^{22} + e^+ + \gamma. \]

\(^{11}\text{Na}^{22}\) has a half life of 2.6 years, so that the decay over the time of the experiment (about four days or less for each sample) can be neglected. The emission of a positron produces a 1.277 MeV gamma ray which is used in positron lifetime experiments to establish the time at which a positron is produced. The strength of our source was about 2.5 millicuries. (For a more complete description of the experimental set-up, see references (14) and (18).)
Treatment of Samples

The plastically-deformed Fe samples were cut from the same sheet to a size of 1 x 1/4 x .02 inches. They were then annealed at 1150° C in a helium atmosphere for two hours. The deformed samples were rolled to a thickness less than their original thickness; for example, a 2% deformed sample would have a thickness 98% of its original thickness. The radiation-damaged Fe sample was irradiated with 2.5 Mev electrons from the Dynamiton accelerator at Brookhaven National Laboratory. The dose was 1.0 x 10^{19} \text{ e/cm}^2, producing an estimated vacancy concentration of 10-20 parts per million (20). Immersion in liquid argon maintained the samples at a temperature of 95°K during irradiation. The sample temperature was monitored by means of a thermocouple attached to the sample; if a large transient temperature change occurred the sample was replaced. The initial preparation of the Fe samples, including cutting to size, initial annealing, and irradiation of deformation, was accomplished at the Brookhaven National Laboratory.

The copper sample was cut to approximately the same size as the other samples from 99.999% pure single-crystal copper. It was then hammered to about 20% deformation (80% as thick as the original). This was done at Western Michigan University.

Some samples were annealed at various temperatures to determine recovery of the samples from the effects of deformation.
or irradiation. The recovery can be determined by comparison of the angular correlation curve after annealing with the angular correlation curve measured for the same sample before annealing, or by comparison with the results for an undamaged sample. The annealing time at a given temperature was one hour for all samples. It should be noted that these times were cumulative. For example, the copper sample for the final run had been annealed three times for an hour each time, at 100° C, 200° C, and 300° C. All samples were annealed in air and were allowed to cool to room temperature before removal from the oven.
CHAPTER IV

ANALYSIS OF DATA

The initial step was to obtain the data totals by summing the runs for each angle. The resulting curve was normalized to an area of 100,000 by a computer program written for this purpose. Since the program discards unpaired points, the normalized area extends from -13 to +13 milliradians. The program used has been listed by Esseltine (18); a revised version has been written (to be used in future work) which is compatible with the new computer at Western Michigan University. The program also calculated the centroid of the curve which was used to calculate the abscissa corresponding to each ordinate.

The normalized values of Y along with the values of X and a weighting factor W (equal to the reciprocal of the Y coordinate) were used to fit the data to an n-th order polynomial by the method of least squares. The program was modified by the W.M.U. computer center from an I.B.M. program and executed on the I.B.M. 1620 computer. The goodness of fit can be estimated from a chi-squared calculation; for the Fe data a typical value of 40 for 20 degrees of freedom was obtained for a 9th order polynomial. For the copper it was necessary to use an 11th order fit to obtain a comparable value of about 30 (for 18 degrees of freedom).
There is no reason to suppose that the angular correlation curve should be an nth order polynomial. However, with a reasonably good fit, integration over values of X allows a comparison of the area under one section of the angular correlation curve to the area under a similar section on another curve.

A value of chi-squared per degree of freedom of 1 to 1.5 indicates a good fit. Most of the fitted curves fell within this range. Normally the number of degrees of freedom is given by N-n (N is the number of points and n is the number of terms in the fitted polynomial). However, normalizing the data to a given area and fixing the centroid of the curve at zero milliradians imposed two additional restrictions on the curves, resulting in a loss of two more degrees of freedom. For an 11th order polynomial with 32 data points, the number of degrees of freedom is 32-(11+1+2) or 18.

After obtaining the equations for the fitted curves the areas under the curves from 0 to 5 milliradians and 5 to 15 milliradians were calculated. The X coordinate 5 milliradians appeared to be a crossover point for all the curves and the value 15 milliradians a point where the curves again came together when plotted on the same graph. The areas were calculated by integrating the polynomial between the desired limits. The areas are given in Tables 1, 2 and 3.

The data in Tables 1, 2 and 3 are based on the fitted curves.
They do not add up to 50,000 (half of the value of 100,000 used for normalization for the total curve) for two reasons, 1) differences in area arising from fitting errors and 2) loss of area past the point $X = 15$ milliradians.
CHAPTER V

RESULTS AND DISCUSSION

Deformed Iron

The results of annealing the 16% deformed iron sample at 400° C are shown in Fig. 3. Table 1 allows a comparison of the results of annealing at several temperatures; some of the changes occurred at the 250° C anneal and some at the 400° C anneal.

Quenching and cold-working experiments in iron have shown stage III recovery (27° C to 127° C) which was attributed to carbon impurities migrating to vacancy sites and the subsequent migration of the carbon-vacancy complex (19). (Recovery stages are temperature ranges over which a given recovery mechanism is thought to operate. See Table 4.) Stage IV recovery (127° C to 247° C) was attributed to single vacancy migration. Recovery in the range from 250° C to 400° C can be attributed to the reduction of small clusters as well as the further removal of single vacancies.

In work conducted at Western Michigan University, it was shown that there were still differences between the angular correlation curves for the deformed iron after annealing to 400° C and a sample which had not been deformed (20). The 400° C anneal had evidently not been sufficient to anneal out all defects which affect the angular correlation.
Fig. 4 shows the difference between the angular correlation curves for 2% deformed iron and 16% deformed iron. Table 2 indicates that the greatest change occurred up to 8% deformation. The changes from 8% to 16% are smaller, suggesting that the concentration of defects has reached a maximum between 8% and 16% deformation.

Irradiated Iron

Fig. 6, showing the results for an iron sample which had been subjected to a high dose of irradiation by electrons \((1.0 \times 10^{19} \text{ e}/\text{cm}^2)\), compares the angular correlation curves after annealing at 100° C and after annealing at 250° C. The results of the annealing at 100° C showed almost complete recovery when compared to an unirradiated sample (20). Since vacancy migration is thought to occur at approximately 200° C (19) the recovery at 100° C may be attributed to the diffusion of carbon interstitial impurities to vacancy sites at a temperature of about 50° C (20). The results of the anneal at 250° C (Fig. 6) would seem to support this idea; no additional recovery can be noted.

Two explanations can possibly account for the lack of similar recovery after annealing to 100° C in the deformed (16%) iron sample. First, the effects of the carbon impurities may be masked by the large numbers of vacancies remaining in the saturated sample. Second, the carbon atoms can become trapped at
clusters and dislocations in the deformed sample, which are not present in the irradiated sample, and prevent the carbon atoms from migrating to vacancy sites.

Deformed Copper

A comparison of the results for a deformed copper sample with the results for a pure undeformed sample (not the same sample) in Fig. 7 shows a large change upon deformation. Most of the recovery occurred after annealing at 300° C (Table 3). The 100° C anneal falls in the stage IV recovery range (see Table 4). Damask and Dienes place 200° C in stage V (2), but van Bueren (21), defines stage V as greater than 227° C. Both authors place 300° C in stage V. It would seem that there is a lack of precise definition of these two stages. Both authors attribute stage IV recovery to vacancy migration; our results would tend to indicate that this process might be active at 300° C as well.

The large differences between the results for the undeformed sample and the annealed sample (Fig. 5) would suggest that only part of the defects have been removed. Unless other recovery processes are activated at higher temperatures, further recovery should be noted by allowing a longer annealing time at 300° C.

The apparent role of interstitial defects has been noted in resistivity experiments in copper (2). Stage II and III recovery was attributed to impurities since vacancies are not
thought to be mobile until stage IV. Stage II recovery did not appear in very pure copper, but was always observable when impurities were present. Copper doped with 0.02% cadmium exhibited a large stage II recovery.
Fig. 3. Comparison of angular correlation curves for 16% plastically deformed iron with the same sample after annealing at 400°C for 1 hr.

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Fig. 4. Comparison of angular correlation curves for 16% plastically deformed iron with 2% plastically deformed iron.
Fig. 5. Comparison of angular correlation curves for plastically deformed copper (approx. 20%) with same sample after annealing at 300°C for 1 hr.
Fig. 6. Angular correlation curves for an irradiated iron sample after annealing at 100°C 1 hr, and after annealing at 250°C 1 hr. Irradiation dose was $1.0 \times 10^{19} \text{e/cm}^2$ at 90°K followed by an anneal to room temperature.

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Fig. 7. Comparison of angular correlation curve for pure copper with a plastically deformed sample.
### Table 1

Fe 16% deformed and annealed.

<table>
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<th>Treatment</th>
<th>Area Under Curve</th>
<th>Difference from Unannealed</th>
</tr>
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<tbody>
<tr>
<td></td>
<td>0-5 mrad 5-15 mrad</td>
<td>0-5 mrad 5-15 mrad</td>
</tr>
<tr>
<td>Unannealed</td>
<td>34,262 15,927</td>
<td>----- -----</td>
</tr>
<tr>
<td>100°C</td>
<td>34,203 16,139</td>
<td>59 -212</td>
</tr>
<tr>
<td>200°C</td>
<td>33,521 16,212</td>
<td>714 -285</td>
</tr>
<tr>
<td>400°C</td>
<td>33,548 16,957</td>
<td>741 -1030</td>
</tr>
</tbody>
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### Table 2

Fe deformed.

<table>
<thead>
<tr>
<th>Treatment</th>
<th>Area Under Curve</th>
<th>Difference from 2%</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0-5 mrad 5-15 mrad</td>
<td>0-5 mrad 5-15 mrad</td>
</tr>
<tr>
<td>2%</td>
<td>32,846 17,435</td>
<td>----- -----</td>
</tr>
<tr>
<td>4%</td>
<td>33,266 16,946</td>
<td>-420 489</td>
</tr>
<tr>
<td>8%</td>
<td>33,865 16,370</td>
<td>-1019 1065</td>
</tr>
<tr>
<td>16%</td>
<td>34,262 15,927</td>
<td>-1416 1508</td>
</tr>
</tbody>
</table>

### Table 3

Cu deformed.

<table>
<thead>
<tr>
<th>Treatment</th>
<th>Area Under Curve</th>
<th>Difference from Unannealed</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0-5 mrad 5-15 mrad</td>
<td>0-5 mrad 5-15 mrad</td>
</tr>
<tr>
<td>Deformed</td>
<td>34,459 15,665</td>
<td>----- -----</td>
</tr>
<tr>
<td>100°C</td>
<td>34,341 15,841</td>
<td>118 -176</td>
</tr>
<tr>
<td>200°C</td>
<td>34,340 15,914</td>
<td>119 -249</td>
</tr>
<tr>
<td>300°C</td>
<td>33,914 16,172</td>
<td>545 -507</td>
</tr>
</tbody>
</table>
Table 4

Definition of temperature ranges for recovery stages.

<table>
<thead>
<tr>
<th>Metal</th>
<th>Stage</th>
<th>Temperature Range in °C</th>
<th>Reference</th>
<th>Possible Mechanism</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>I</td>
<td>-273 to -213</td>
<td>(2)</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>-243 to -233</td>
<td>(21)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>II</td>
<td>-213 to -33</td>
<td>(2)</td>
<td>Interstitial Migration</td>
</tr>
<tr>
<td></td>
<td></td>
<td>-183 to -73</td>
<td>(21)</td>
<td></td>
</tr>
<tr>
<td>Copper</td>
<td>III</td>
<td>-33 to 67</td>
<td>(2)</td>
<td>Interstitial Migration</td>
</tr>
<tr>
<td></td>
<td></td>
<td>-63 to 47</td>
<td>(21)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>IV</td>
<td>67 to 177</td>
<td>(2)</td>
<td>Vacancy Migration</td>
</tr>
<tr>
<td></td>
<td></td>
<td>approx. 127</td>
<td>(21)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>V</td>
<td>over 177</td>
<td>(2)</td>
<td>Divacancy Migration, Complex Loops, etc.</td>
</tr>
<tr>
<td></td>
<td></td>
<td>over 227</td>
<td>(21)</td>
<td></td>
</tr>
<tr>
<td>Iron</td>
<td>III</td>
<td>27 to 127</td>
<td>(19)</td>
<td>Carbon Impurity Migration to Vacancy Sites</td>
</tr>
<tr>
<td></td>
<td>IV</td>
<td>127 to 247</td>
<td>(19)</td>
<td>Single Vacancy Migration</td>
</tr>
</tbody>
</table>
CHAPTER VI

CONCLUSIONS

1. The effects of plastic deformation in iron increases with increasing deformation, with saturation of the effects noted between 8% and 16% deformation.

2. In iron the movement of interstitial carbon atoms removes changes in the angular correlation curves caused by the production of vacancies by electron irradiation. This seems to occur at about 50° C. The effect can be seen at the lower concentrations of vacancies produced by electron irradiation. At the higher vacancy concentrations produced by plastic deformation the effect of the migration of the carbon atoms is probably masked.

3. After being annealed to 400° C deformed iron still contains complexes of defects which affect the angular correlation curve. (All anneals were of one hour duration; longer anneals might allow additional recovery.)

4. The recovery due to annealing seems to be most pronounced in copper after annealing at 300° C.
REFERENCES


