THE CHANGE OF YOUNG's MODULUS AFTER DEFORMATION AT LOW TEMPERATURE AND ITS RECOVERY

PROEFSCHRIFT

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CONTENTS

CHAPTER I

INTRODUCTION AND SUMMARY

The past ten years have shown great progress in the understanding of the nature and the properties of physical imperfections in crystals. It was observed that a number of the important properties of the solid are for the greater part controlled by these imperfections, rather than by the properties of the perfect crystal. Limiting ourselves on metals, we can give as example of one of the mechanical properties: the flow stress, which property is in the first place determined by the kind of imperfections, known as dislocations. Imperfections like point defects and impurities can also play a part in this example, but mainly where their interaction with the dislocations is concerned.
A number of excellent reviews on imperfections have appeared in literature, some covering a large field as for example Seitz[1]), Fisher c.s.[2]) and van Bueren[3]), some dealing with a more restricted subject for instance the books by Cottrell[4]), Read[5]) and Friedel[6]) on dislocations. There are also extensive review papers in annual reports like Progress in Metal Physics, Solid State Physics etc.
As imperfections determine many important properties of metals, to which we will limit ourselves, it is important to investigate these imperfections in detail. If we only consider the lattice defects, thus leaving the impurities aside, we can distinguish two methods in which they can be studied. The indirect method, by studying the physical properties of the metal which change due to the presence of the lattice defects. The direct method, for example provided by the electron microscope, which technique made it possible to observe dislocations and stacking faults directly. The method of studying the changes of physical properties has the advantage that they are very often quite simple and accurate, the difficulty however is the interpretation of the results which is always based on more or less plausible suppositions concerning the defect properties themselves. This does not mean though that the example of the direct method given does not meet with interpretation difficulties.
We will limit ourselves to the lattice defects, which are of importance for this work, being dislocations and point defects, the latter we can subdivide in vacancies and interstitials. Dislocations are mainly produced by plastic deformation, their

density increases during deformation due to the multiplicationmechanism of Frank and Read. The moving dislocations can produce point defects, there are different mechanisms possible for this production, thus plastic deformation will produce dislocations and point defects. Measurements on the electrical resistivity, so far the most frequently studied physical property in this field, show an increase of the resistivity after deformation which is attributed to the lattice defects produced. If this is done at low temperature, for example on Cu at 78°K, this increase remains constant as a function of the time after deformation. Isochronal annealing at temperatures above the deformation temperature causes a decrease of this extra resistivity which is called recovery. This recovery occurs in stages, numbered II through V, each related to a certain temperature range running from low to high temperatures. The reason the numbering starts with stage II, is that after introducing point defects by irradiation a stage I below 78°K occurs, which is not observed after plastic deformation. It is generally believed that stages I through IV refer to point defects disappearing by diffusion in their respective recovery stages.
The problem of the identification of a recovery stage with a certain type of point defect is still, in spite of the substantial amount of experimental evidence, not solved. Two other methods of point defect production have partly solved this problem of identification, although it still remains a complicated affair. One of them, the irradiation with fast particles, which has already been mentioned, will mainly produce point defects: vacancies and interstitials in equal number. The other, quenching from high temperature, produces vacancies. Some details of this identification will be presented in chapter II.
Measurements on the elastic properties also provide information on lattice defects, unlike the electrical measurements the most important contribution is supplied by the dislocations in this case. In the next chapter it will be argued that this effect is caused mainly by the movement of the dislocations under influence of the applied stress. The point defect also plays a part though, causing the recovery of the effect, but this is practically only in as far they immobilize the dislocations. The decrease of the elastic moduli after cold work has been observed already by a number of investigators, for example [7-10]).
Also the recovery of these elastic properties after deformation is known for some time[8]). All these measurements were performed at roomtemperature however, where recovery occurs. This makes that the effect and the recovery can not

be separated. Therefore it is important to perform the deformation and the measurement at a temperature where no recovery occurs, thus the effect will not be obscured by recovery phenomena. Measurements of this kind were performed by Crittenden and Dieckamp[11]) and Druyvesteyn, Schannen and Swaving[12]) on the rigidity modulus after deformation at 78° K. In this work the decrease of Young's modulus after deformation at 78°K by elongation, and also the recovery in the temperature range up till 350°K is studied. Part of these results have been published already[13]). The way the measurements were performed is described in chapter III, while the results are given in chapter IV. Chapter V discusses the results on the effect and the recovery. It starts with an extension of the discussion concerning the so called kink model, being one of the theoretical concepts of the behaviour of the dislocations under influence of a stress, given in chapter II. In the following section the results on the effect are discussed.

A saturation of the modulus decrease as a function of the deformation was observed for Cu and Ag, for Au no direct measurement as a function of the deformation was performed. This saturation effect indicates that as the dislocation density increases with proceeding deformation, the free length of dislocation lines that bow out under influence of the stress decreases in length. Two models, one based on the representation of the dislocation by an elastic string the other based on the kink movement, seem to be able to explain this result.

The third section of chapter V discusses the results on the recovery. For Cu two well separated recovery stages were observed, for Ag only the first of these two is observed, for Au the first of the two was only observed if the deformation was larger than about 4%. To gain more information about the recovery, the circumstances were altered in some experiments. For example in some cases part of the recovery was performed with an elastic stress on the specimen, some other experiments were done on specimen which were quenched before the deformation. This enabled us to compare different recovery possibilities and to make a choice between them, giving the recovery stages caused by two different types of point defects as the most likely possibility. The quenching experiment was mainly meant to provide information on the identification of the recovery stages with a certain type of point defect. The results revealed that the second stage, which will be called stage III for reasons of comparison with measurements on other physical properties, has at least something to do with vacancies. Comparing it with the results on other quenching experiments, it seems that we are dealing with double vacancies in this case. Studying the recovery behaviour under different circumstances also revealed that the horizontal part in the recovery curve, which separates the two recovery stages, does not necessarily mean that no recovery occurs in that range.

CHAPTER II

SOME ASPECTS OF LITERATURE AND THEORY

a. INTRODUCTION.

This chapter is intended to give a more or less detailed account of the influence of lattice defects upon the elastic constants, and to discuss some of the main results reported in the literature. No attempt however will be made to give a complete survey of the existing literature, as this is not necessary for the understanding of the results which will be reported later on.
As was remarked in chapter I, the elastic constants are known to decrease by a small amount of cold work, the internal friction shows an increase with increasing deformation. Most work has so far been done on the internal friction, which however can not be divorced from the equally important change in the elastic modulus.
On the internal friction some reviews have been given [14-20]), results in this field will only be discussed so far as needed for a better understanding of the modulus effect. First the influence of the lattice defects on the elastic behaviour will be discussed. There are essentially two ways in which lattice defects can contribute to a change of the elastic constants. The first, to be treated in the next section, is the effect as a result of their mere presence which will be called the bulk effect. The second, and by far the most important one, is the effect due to the contribution of the defects to the non-elastic strain by moving under influence of the stress.
A third section will be devoted to the recovery observed after thermal annealing. Recovery has been studied on a variety of physical properties, of which one can not ignore the results if one wants to draw conclusions from our own measurements on the modulus effect. No general agreement exists on the interpretation of those numerous results, but fortunately some authors [21-23]) have critically examined these results which resulted in some agreement but also in some differences of opinion. Only the most recent ideas will be discussed.

b. BULK EFFECT.

The presence of a lattice defect will change the elastic

constants as the bonds between the atoms will be disturbed. Dienes [24]) has calculated the change of the elastic modulus due to the presence of interstitials and vacancies. He finds an increase of the elastic modulus due to the presence of interstitials and a decrease due to vacancies, predicting a linear dependence of the modulus change upon the number of interstitials respectively vacancies present. As the concentrations of these point defects are usually very low, always being smaller than 10^{-4}, this effect will be small. Thompson and Holmes [25]) observed no effect of this kind after neutron irradiation of Cu single crystals, but as both vacancies and interstitials are present, giving opposite effects, this does not prove that the effect does not exist. Folweiler and Brotzen [26]) did observe an effect after quenching Al, they attribute the observed decrease of the elastic modulus of 0,01% to the vacancies present.
Although their arguments do not seem conclusive, one can conclude that the effect, if present, is very small. To my knowledge the effect of the presence of a dislocation is not calculated. If we regard the dislocation as a row of point defects, knowing very well that this is not a correct approximation at all, the highest dislocation density which is about $10^{12}\,cm^{-2}$ will represent a point defect concentration of 10^{-3}. This will not have a large effect on the elastic modulus. It seems safe to conclude that, as the concentrations of lattice defects are low, their immediate influence on the elastic modulus will be small.

c. NON-ELASTIC EFFECTS.

When movement of defects occurs under influence of an applied stress, these defects can contribute to the strain which contribution we will call the non-elastic strain. Thus for a given stress the strain is larger than it would be if the strain were a pure elastic one, the total strain being the sum of the pure elastic and the non-elastic strain. This total strain being larger than the pure elastic strain, causes the apparent decrease of the elastic modulus. The magnitude of this decrease is $(E-E_o)/E_o = \Delta E/E_o = -\epsilon_{non}/(\epsilon + \epsilon_{non})$ where E_o is the true elastic modulus, E is the observed value due to the presence of a non-elastic contribution to the strain represented by ϵ_{non}, while ϵ is the pure elastic strain. If the stress is a periodic function of time, the component of the non-elastic strain which is in-phase with the applied stress causes the modulus effect, the component out of phase causes the internal friction. The way defects contribute to the non-elastic strain will be reviewed very briefly here. As the contribution of the dislocations is the most important one in relation to our work, their influence will be treated in somewhat more detail.

1) Point defects.

Diffusion of point defects can cause a change of the dimensions of the body, and thus contribute to the non-elastic strain. This diffusion can lead to relaxation phenomena having a relaxation time which decreases exponentially with the temperature.
One can distinguish two processes, one where the diffusion has a preferential direction, the other where only the local ordening changes, usually only needing one atomic jump per defect. The first is of no importance for our purpose, as this only seems interesting for high defect concentrations. An example of the second mechanism is the well known Snoek effect[27]) in a body centered cubic crystal, where an interstitial impurity jumps by preference to the interstitial sites which due to the applied stress provide more room for them, contributing there to the non-elastic strain.
An interstitial atom occupying the center of the cube in a face centered cubic lattice, or a vacancy in this lattice, will not contribute to the elastic strain, as the stress does not provide preferential sites for them. This is different however for the so-called split interstitial, which is according to some authors [28, 29]) the stable interstitial configuration. This split interstitial has a preferential orientation, having its axis in the direction of the applied stress. If a stress is applied the number of split interstitials parallel to the stress will in equilibrium be larger than in other directions. These relaxation effects, where the relaxationtime $\tau = \tau_0 e^{U/kT}$, lead to the damping

$$\delta = \frac{\Delta M}{M} \cdot \frac{\omega\tau}{1 + \omega^2\tau^2}$$

where τ_0 is a constant, U the activation energy, being the energy needed for one jump of the interstitial impurity in the next site for the Snoek effect, and the rotation of the split interstitial in the second example, k is the Boltzmann constant, T the absolute temperature, $\Delta M/M$ is the relaxationstrength being the maximum change of the modulus, ω is the angular frequency of the stress. Seeger c. s.[29]) claim to have observed the damping peak due to the rotation of

these split interstitials in Ni, however there seems to exist some doubt on these results lately.
Hasiguti has made a prolonged attempt to find a peak which could be ascribed to divacancies, which in principle should be able to contribute to the non-elastic strain.
In his latest paper [30]) he expresses his doubt if the peak can be observed at all, the relaxation strength being too low. An additional difficulty is that the energy needed for the rotation of the divacancy will be the same as its migration energy, thus the measurement should be performed at the lowest possible frequency, giving a low peak temperature, otherwise the di-vacancies will disappear.
Concluding it can be remarked that the contribution of the point defects to the non-elastic strain will be small, as their concentrations are small, and will not cause changes in the modulus greater than about 0,1%.

2) Dislocations.

It was pointed out by Read [31]) for the first time, that the small reversible displacement of the dislocations, caused by an applied stress, contribute to the non-elastic strain, thus causing a decrease of the modulus and, as the moving dislocations dissipate energy, an increase of the internal friction. Several models have been proposed since, trying to describe these effects quantitatively. We will start with the most successful development so far and will briefly discuss the other ones afterwards. The models of Koehler [32]), Mott [33]) and Friedel [34]) are based upon the elastic continuum model of the dislocations, treating the dislocation as an elastic string which bows out under influence of an applied stress. Koehler [32]) develops the analogy between the vibration under an alternating stress of a dislocation line segment pinned down at its ends and the problem of the forced damped vibration of a string, and calculated the change of the modulus and the internal friction due to movement of the dislocation. He also considers the increase of the internal friction due to the increase of the length of the dislocation segment if it breaks away from its pinning points, which he assumes to be impurities.
Mott [33]) and Friedel [34]) only consider the modulus decrease due to the bowing out of a dislocation segment, if the crystal contains N segments of length L this gives a decrease of

the shear modulus of $\Delta G/G = \dfrac{NL^3/6}{1 + NL^3/6}$

This relation is valid if the dislocations can move in all directions, thus in their glide plane and perpendicular to it. If the temperature is not high the dislocations will only be able to move in their glide planes; Friedel, Boulanger and Crussard[35]) find a value for Young's modulus:

$\Delta E/E = \dfrac{NL^3/18}{1 + NL^3/18}$ if the glide planes are distributed at random. For a three dimensional dislocation network, assuming that the lengths of the segments are all L, this expression can be written as $\Delta E/E = \dfrac{\Lambda L^2/18}{1 + \Lambda L^2/18}$, where Λ is the dislocation density. Granato and Lücke[36]) extend Koehler's model, they improve the explanation for the amplitude dependent damping by showing that this is due to a mechanical hysteresis instead of a damping increase only due to the increase in looplength, as was proposed by Koehler. This breaking away from the pinning points is illustrated in figure 1, the dislocation is anchored at its extreme ends by the network, a pinning point which, let us assume is an

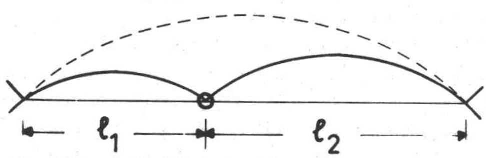

Figure 1. A dislocation line, which is pinned in between its network pinning points, bows out under influence of an applied stress. As the stress increases breakaway from the pinning point occurs and the dislocation bows out as is represented by the dotted curve.

impurity, anchors the dislocation in between the network pinning points. This pinning point exerts a force on the dislocation which has a maximum of about $K_{max} = E_c/b$, where E_c is the interaction energy and b the Burgersvector. The dislocation will, if the force on the dislocation is larger than the pinning force, break away from its pinning point. The force on the dislocation equals $\dfrac{l_1 + l_2}{2} b\sigma$, where l_1 and l_2 are the looplengths on either side of the pinning point, σ is the stress in the direction of the Burgersvector. The dislocation will thus break away if

$\dfrac{l_1 + l_2}{2} b\sigma > \dfrac{E_c}{b}$ and bow out as is represented by the dotted curve in the figure. The mechanical hysteresis is caused by the fact that at a certain stress break away occurs, which means an increase of the strain at this constant stress. If this break away occurs, the round trip in the stress-strain diagram is not reversible, which causes the dissipation of energy. In this break away model the influence of the temperature on the pinning force is not discussed. For low strain amplitudes, where no breakaway occurs, Granato and Lücke find for the modulus decrease $\frac{\Delta E}{E} = \frac{6\Omega}{\pi^2} . \Lambda L^2$ and for the internal friction $\delta = \frac{120\Omega\ B\omega}{\pi^3 . C}\ \Lambda L^4$. In these expressions Ω is an orientation dependent constant, Λ is the dislocation density, L is the average looplength, B is the damping constant, ω the angular frequency and C is the line tension of the dislocation. These expressions have generally been verified by the experiments. For instance, Thompson and Holmes[37]) verified the dependence on the loop length being L^2 and L^4 for the modulus effect and the internal friction respectively. This was done by irradiating high purity Cu, the generated point defects cause a decrease of the average loop length while Λ remains constant, the measurements were performed in the kilocycle range.

Another verification is the existence of resonance damping. From the string model it would be expected, that a resonance motion of dislocations occurs at a certain frequency, depending on the looplength. The internal friction will show a maximum if this occurs, as the amplitude and thus the velocity of the dislocations will have a maximum, the damping being of a viscous type depends linearly on the velocity. This resonance damping has indeed been observed in the megacycle range [38, 39]), and some important additional facts could be concluded from this. For instance Stern and Granato[39]) show that this maximum consists of two components, which they ascribe to two different types of dislocations, as proposed by Thompson and Paré[40]), each having a different value for C and also a different recovery rate. The assumption that the looplength has an exponential distribution, as suggested by Koehler, gives a better agreement with the experiments than is found by assuming that L has a constant value. The influence of an exponential distribution was also discussed by Granato and Lücke[36]) who calculated this effect. They found that the qualitative results do not change very much, and that a good description of the results is obtained by replacing the average looplength in their relations by an effective looplength which for an exponential distribution is about 3, 3 L.

Comparing the values found for the modulus decrease by

Friedel c.s. [35]) and Granato and Lücke [36]), we see that these are practically the same if we write Λ = NL, and take for Ω the value 1/10 which Granato, Hikata and Lücke [41]) use in their publication, for ΛL^2 small enough.
Other mechanisms for the contribution of dislocations have been proposed by Eshelby [42]) Weertman [43, 44]), Druyvesteyn and Jongenburger [45]) and Jongenburger [46]).
Eshelby regards the dislocations as oscillating in potential wells associated with the Peierls'force [47]). The amplitude of the dislocations will be very small in this case, the pinning seems difficult in this model. The influence of the Peierls' force will be discussed in a later part more extensively.
Weertman assumes that the only restraint on a dislocation in a crystal is the stress field associated with impurity atoms. Although this effect might well have some influence, there are serious objections to a general application of this idea.
Druyvesteyn and Jongenburger calculated the ΔE effect for two models, in both cases the limitation of dislocation movement is caused by the stress field of the other dislocations present. One consists of a uniform distribution of positive and negative edge dislocations as used by Taylor [48]) for the first theory of work hardening, the other of a pile up group as calculated by Eshelby c.s. [49]). Although the calculated effects are of considerable magnitude, the models do not seem to be very realistic. The uniform distribution could possibly be approximated by the situation which is obtained in a single crystal during the initial part of the plastic deformation, known as the easy glide region. The pile up group, which was believed to be responsible for work hardening according to the models of Mott [33]), Friedel [50]) and Seeger c.s. [51]), is seldom observed by electron microscopy. Only in stainless steel, which has a very low stacking fault energy, pile up groups were clearly observed [52]).
Recently Jongenburger [46]) calculated the ΔE effect caused by the change of width of extended dislocations due to an applied stress. For metals with a low stacking fault energy this effect is considerable, it depends linearly on the dislocation density thus is small for small densities. This leads to the conclusion that the effect may exist, but can not be easily reconciled with the experimental facts.
The models discussed so far do not consider the atomic structure of the crystal, treating the dislocation as a string situated in an elastic continuum. In a crystal, the energy of a dislocation depends on its position in the lattice however. Peierls [47]) approximated this influence of the lattice as an opposing shear stress, which varies periodically with the atomic distance as period. The positions of the lowest

potential energy, called Peierls' valleys, run parallel to the close packed directions in the crystal. The magnitude of this Peierls' stress is according to measurements on the Bordoni Peak, which we will discuss afterwards, about 10^{-4} G. This means that, leaving the influence of the temperature out of consideration, a stress of 10^{-4} G in the direction of the Burgers vector would be needed to move a dislocation as a whole from one Peierls' valley to the next. Thus the string model will be approximately correct for stresses which are larger than the Peierls' stress, which is much larger than generally used for internal friction and modulus change experiments. It should be remembered though that the reported value of the Peierls' stress is by no means a generally accepted one, and that the temperature influence has been neglected in this discussion.
Shockley [53]) pointed out that a dislocation which is not parallel to the close packed direction will contain kinks, which are the curved parts of the dislocation as shown in figure 2

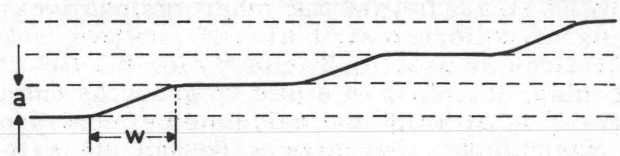

Figure 2. The shape of a dislocation which makes an angle with the Peierls' valleys, represented by the dashed lines, is shown by the drawn line. Straight parts of the dislocation lying in the valleys are connected by the so-called kinks, having a width w.

connecting the straight parts which lie in the Peierls' valleys. These kinks, existing due to the fact that the dislocation makes an angle with the Peierls' valley, will be called geometrical kinks. Under influence of a stress, the kinks can move along the length of the dislocation, such motion causes the dislocation to move normal to itself. For example in figure 2 the dislocation moves up when the kinks move to the left. Only a small stress, supposed to be much lower than the Peierls' stress, is needed to move kinks along the dislocation. This explains how dislocations can move at very low stresses. The length of the dislocation does not change due to this kink motion, thus the increase of dislocation length is not the force opposing the dislocation motion in this case as it was for the string model. Seeger and Schiller [54]) pointed out that the repulsive forces between the parallel kinks, which will be called kinks of the same sign, provide the force opposing the dislocation motion,

thus acting as the substitute of the line tension in this case. Seeger c. s. [54]) calculate the modulus change and the internal friction of a row of kinks vibrating around their equilibrium positions under influence of the applied stress. They report a good agreement between the results of their model and that based upon the string model. In chapter V we will discuss this model somewhat further.
So far only the geometrical kinks, existing due to the fact that the dislocation makes an angle with the Peierls' valley, have been discussed. Shockley pointed out that a dislocation which on the average is parallel to a close packed direction will at moderately high temperatures contain a certain number of kinks as shown in figure 3, which will be called ther-

Figure 3. A pair of kinks in a dislocation line, which on the average is parallel to the Peierls' valley.

mal kinks. From the figure we see, that as the extreme ends of the dislocation remain in their Peierls' valley, for each displacement of a part of the dislocation to the next Peierls' valley 2 kinks of opposite sign have to be formed. Both the entropy and energy are increased by this kink formation, which means that the number of kinks per unit length of dislocation increases with increasing temperature. Seeger [55]) based his model to explain the internal friction peak of the relaxation type known as the Bordoni peak, on the formation of a pair of kinks as shown in figure 3 under influence of the combined action of thermal fluctuations and the applied stress.
This peak was observed for the first time by Bordoni [56]) on Cu at about 80°K, the temperature being dependent on the frequency of course, the peak being of the relaxation type. According to Seeger, this internal friction peak is observed if the frequency of the applied alternating stress is equal to the frequency of formation of these kink pairs. Those pairs with a larger separation than a certain critical distance, will not recombine, but expand outwards under the action of the stress. This expanding outwards of kinks was assumed to be a very fast process, thus not contributing to the internal friction. Recently Seeger c. s. [54]) suggested that this might very well not be true, they conclude that if the expanding outwards of the kinks is a kind of diffusion process it will contribute to the internal friction and might very well be the cause that the Bordoni peak is too wide for a single relaxation peak.

d. RECOVERY.

It was first observed by Köster[8]) on Al, that the internal friction of a crystal decreases and the elastic modulus increases with time after deformation at room temperature.
As was discussed in the previous section, the internal friction and modulus change are due to the oscillations of mobile dislocations under influence of an applied alternating stress. The increase in the damping and decrease in the modulus with deformation, is due to the increase of the number of mobile dislocations.
The subsequent recovery must then be due to the disappearance (annihilation theory) or immobilization of these dislocations. The latter process can be caused either by interaction with other dislocations (rearrangement theory) or with point defects (pinning theory). The first of these mechanisms, the annihilation of the dislocations, was proposed by Smith[9]). The idea is the annihilation of parallel screw dislocations of opposite sign, one lying above the other in a different plane, by the aid of thermal energy, as was proposed by Mott[57]). This explanation however does not agree with the observations made with the electron microscop, revealing that below the recrystallisation temperature the dislocations do not disappear in great number. Also the amplitude dependent internal friction effects seem to indicate, that the dislocations are still present.
Nowick[58]) assumes that redistribution and possibly annihilation of dislocations takes place in the very early stages of annealing. In a later paper[59]) he suggests that the rearrangement of the dislocations may be accomplished by dislocation climb over obstacles, made possible through the agency of point defects generated by plastic flow. Although there may very well be some climb, it is not understood why dislocations only climb to places where they become less mobile. No specific model has been constructed for this rearrangement mechanism, so it can not be tested. We think it will only give a small contribution to the recovery.
The mechanism of the pinning of dislocations by point defects formed during plastic flow, as developed by Granato, Hikata and Lücke[41]) has been quite successful in explaining the observed recovery phenomena. As mentioned in the previous section, Granato and Lücke[36]) derived expressions for the modulus effect and the internal friction due to moving dislocations, being dependent on ΛL^2 and ΛL^4 respectively. If annihilation can be neglected during recovery, L will be the only variable quantity. The looplength L is assumed to decrease with the number of point defects, ac-

ting as pinning points, arriving at the dislocations. Because of the L^2 and L^4 dependence, mainly the earliest stages of precipitation of point defects at dislocations are observed, the situation in which the approximations made in deriving the Cottrell-Bilby[60]) law are most likely to be valid. For, by deriving this $t^{2/3}$ (t = time) dependence for the diffusion of interstitial carbon atoms to the dislocations in iron, they neglect the diffusion current compared to the drift current due to the interaction between the dislocation and the interstitial. Granato c.s. [41]) make the same assumption, as they calculate the change of the looplength as a function of the annealing time, obtaining:

$L = \dfrac{L_o}{1 + (\beta t)^{2/3}}$, where t is the annealing time, L_o is the looplength before annealing, and

$$\beta = \left(\frac{nbWD}{kT}\right)\left(\frac{2\pi C L_o}{b^2}\right)^{\frac{n+2}{2}}$$ [63])

where C is the line tension, n a constant having the value 1 for interstitials, W is the maximum interaction energy, D is the diffusion constant = $D_o \; e^{-E_m/kT}$. According to Friedel [61]) for vacancies, because of their small size effect (misfit) but their large compressibility, the value of n should be 2, and $L = \dfrac{L_o}{1 + (\beta t)^{\frac{1}{2}}}$ for vacancy pinning. Granato c.s. [41]) showed that the agreement between the data on NaCl[62]) and Cu[9]) and their predicted $t^{2/3}$ time dependence of the recovery is satisfactory. Stern [63]) claims to have also observed the $t^{\frac{1}{2}}$ (vacancy migration) dependence at 88° C on Al, although the difference between the two different time dependences is small. A further refinement is the assumption of different values for β for the two dislocation components [39]), this can be understood by realizing that the interaction energy will be different for the two components and also the line tension as was mentioned in the previous section is assumed to be different.

Experimental evidence that point defects immobilize the dislocations is given by quenching and irradiation experiments. Among the different ways of generating lattice defects, as was mentioned in chapter I, quenching or irradiation in the first instance generate point defects. As mobile dislocations will always be present, although in a well annealed crystal their total length and their free looplength will be small, the internal friction will decrease and the modulus increase

after quenching or irradiating the sample, if point defects cause the immobilization. This is indeed observed after quenching for example Al [64]) and Au [65]). As pointed out by Roswell and Nowick [65]) it is interesting to note that pinning can be produced in both Al and Au, although in Au the dislocations will probably be highly extended.
Irradiation with fast particles showed the same effect, many experiments with different particles have been carried out on Cu. The effects produced by neutron irradiation have been studied mainly in Oak Ridge [25, 40, 66-68]), electron irradiation by Sosin and his colleagues [69-72]), while Barnes c.s. [73]) and Stern c.s. [39]) studied effects resulting from γ-irradiation. The exact nature of pinning is not known. As was discussed in the previous section, Granato and Lücke [36]) developed a theory for amplitude dependent damping based on Koehler's [32]) idea that for a high enough stress the dislocation can be torn loose from its pinning points, the pinning point exerting a force which has a maximum of about E_c/b on the dislocation. Values of the interaction energy of E_c have been calculated by several authors. These calculations all have in common, that besides the electrical interaction (which is believed to be small in metals) the interaction is calculated from the interaction of the stress fields around the dislocation and the point defects. The difference is based upon the different approximations used for the stress field around the point defect. The point defect can be approximated as causing a dilatation in the lattice due to its "misfit", analogous to the Cottrell-Bilby [60]) calculation for the interaction of a foreign interstitial atom and an edge dislocation. In this theory only the hydrostatic stress component was considered. Crussard [74]) also considers the shear stress component, obtaining an interaction with both screw-and edge dislocations. Based on this model, van Bueren [75]) calculates at one atomic distance from the dislocation the value of E_c, obtaining for an interstitial 0,4 eV, for a vacancy 0,02 eV. Cochardt, Schoeck and Wiedersich [76]) calculate the interaction for a C-atom in α-iron with tetragonal deformations around it. Essentially in this case is that the dilatation due to the C-atom is not symmetric with respect to the lattice structure. Fleischer [77]) calculates the interaction energy due to the tetragonal distortions around an interstitial atom and a divacancy with a screw dislocation finding 0,44 eV and 0,06 eV respectively. Yoshida and Koehler [78]) find a larger value for the divacancy: interaction with an edge dislocation 0,246 eV, with a screw dislocation 0,094 eV at three atomic distances from the dislocation.
Eshelby [79]) has given the mathematical solution for the in-

teraction energy of an ellipsoidal elastic inhomogenity in the presence of a stress field. Bullough and Newman[80]) apply his results to deduce the interaction energy between a dislocation and a vacancy, the vacancy being the elastic inhomogenity. At a distance of 2/3 b they obtain 0,5 eV with an edge and about half this value with a screw dislocation. Previously Friedel[81]) calculated this effect, but has only taken the hydrostatic stress around the dislocation into account, finding a value of 0,35 eV. From this variety of calculations it can be concluded that the interaction energy for an interstitial will be about 0,5 eV, for the vacancy about half of this value or lower. It seems reasonable to assume that the divacancy will have a higher binding energy than a single vacancy, so Fleischer's value seems rather low. Friedel[82]) reported lately that for an interstitial the value could very well be considerably higher than 0,5 eV.
It should be kept in mind however that the calculations are all based upon the elastic interaction approximation, this is not very likely to hold true for distances between the dislocation and the point defect of the order of an atomic distance. But as nothing is known about the properties of the dislocation core, the elastic approximation can not be replaced by a more realistic model.
The interaction energy has also been estimated for some point defect aggregates. Coulomb and Friedel[83]) suggest that cavities are formed by the precipitation of vacancies along the dislocation. These cavities, supposed to be nearly spherical and having a diameter of about 20 interatomic distances, are calculated to have a large interaction with the dislocation. Also the interaction of a sessile dislocation ring, formed by the condensation of point defects in a (111) plane has been calculated. Kroupa[84]) finds as a value for the maximum interaction energy $E_c = G\, b_o b_1 R/4$ where R is the radius of the sessile dislocation loop, b_o and b_1 are the Burgersvectors of the loop and the dislocation respectively. Kuhlmann Wilsdorf[85]) introduces the superjog, a name for a local deviation of a dislocation line where it bends out of its glide plane and back, formed by the dislocation loop which is formed by condensation of vacancies, in contact with the dislocation. To get an idea of the order of magnitude of the pinning force, the reader is referred to the relation for the breakaway stress of the pinned dislocation given in the previous section on page 15. Considering the situation represented in figure 1, the dislocation loop containing one pinning point, and taking for the looplength and the interaction energy the reasonable values of 10^{-4} cm and 0,5 eV respectively, we obtain for the component of the breakaway stress in the

direction of the Burgersvector a value of $2{,}44.10^7$ dyne $cm^{-2} \approx 5.10^{-5}.G$. The influence of the thermal energy is not considered in this expression, but will certainly lower the value of the breakaway stress considerably. Teutonico, Granato and Lücke [86]) are studying this influence, which results can be expected to appear before long. Their idea is that above a certain value of the stress σ_1, only the unpinned configuration is stable, below a smaller value of the stress σ_2 only the pinned configuration is stable. In the range between σ_2 and σ_1 transitions with the aid of thermal energy are possible [87]).

Proceeding from the assumption that pinning of dislocations by point defects is possible one way or the other, which in view of the experimental results generally known so far seems a reasonable one, the question of identification of the recovery with a certain type of point defect remains. The recovery has been studied on several physical properties, but for the greater part on the electrical resistivity. Reviews on this subject have been given by Broom [88]), Glen [89]) van Bueren [21]) Seeger [22]) and van den Beukel [23]). No attempt will be made here to give a discussion of the annealing behaviour of the electrical resistivity, for this the reader is referred to the reviews mentioned, only the agreement and disagreement on the principal points will be mentioned. The recovery as a function of the annealing temperature, can be roughly divided in five stages, numbered I through V, from low to high temperatures characterised by more or less well defined energies of activation, as was mentioned in chapter I. It has also been remarked in chapter I, that defects can be introduced in several ways. Cold-work will in all probability introduce dislocations, vacancies and interstitials in the lattice. Irradiation produces interstitials and vacancies in equal number, quenching produces only vacancies, neglecting the dislocations which might be introduced by the quenching strains. It is a pity that there is no way of introducing only interstitials, as this would considerably enlighten the task of identifying a certain recovery stage with a certain type of defect. General agreement exists on attributing stages IV and V to vacancy migration and recrystallisation respectively. On the interpretation of stage I, II and III two different conceptions still exist, leaving the details aside.

Stage I exists only after irradiation, it is therefore obvious to ascribe this stage to recombination of interstitials and vacancies which are formed in pairs during irradiation. However according to Brinkman [90]) and Seeger [22]) the interstitial will be mobile in stage III, which forces them to ascribe stage I to a different interstitial configuration the so called crowdion. Their recovery theory does not provide for stage II, although Seeger[22]) suggests that a vacancy aggregate, for example a trivacancy, might be mobile in this stage.

The other concept starts with mobile interstitials in stage I. Stage II after irradiation is different from this stage observed after plastic deformation. After irradiation stage II is strongly dependent on the impurity concentration, being practically absent in very pure Cu. After deformation a recovery in a more narrow temperature range is observed in this stage. These facts are explained by a release of interstitials trapped at impurities after irradiation[91]) and dissociation of interstitial pairs formed by deformation[23]) respectively.

The observation that the presence of dislocations cause a decrease of stage I and an increase of stage III after irradiation[90]) could according to Sosin[92]) be explained by the trapping of interstitials by dislocations in stage I and their release in stage III. It does not seem very probable that stage III recovery as a whole can be explained by this mechanism. There are for instance the quenching experiments, which show that probably divacancies are mobile in this range[93-95]).

Very important in this respect are the results of de Jong and Koehler[95]), who find that the activation energy of migration which is observed, is dependent on the concentration of, and the binding energy between, the vacancies. This should be a warning against too much confidence in argumentations which are entirely based upon the measured activation energies in the recovery stages. Korevaar[96]) observes an increase of ordening in stage III after plastic deformation in Au with a small percentage of Cu added. This seems to indicate that also after plastic deformation probably double vacancies are mobile in stage III. If double vacancies could be expected after electron irradiation, the influence of the dislocations on the size of stage III could be explained by the diffusion of divacancies in this stage. According to Lomer[97]) divacancies could very well be formed by electron irradiation, according to de Jong[98]) they will be formed during the recovery, again depending on the concentration and binding energy. Recapitulating it can be concluded that if the interstitial is mobile in the temperature range of stage I, stage II can very well be explained by the dissociation of interstitial pairs or the release of interstitials from impurity traps. If after electron irradiation divacancies can be expected to exist, stage III can be explained in terms of divacancies. On the other hand if stage

III is caused by interstitials and stage I by crowdions, stage II still remains to be explained, as it does not seem very probable that a trivacancy will move with a lower activation energy than a divacancy.

CHAPTER III

EXPERIMENTAL DETAILS

a. MATERIAL.

The copper and silver used, were supplied by Johnson and Matthey, with a stated purity of 99,999%, in the form of rolled sheet with a thickness of 1 mm. The gold was supplied by Drijfhout, with a stated purity of 99,99%, also in the form of rolled sheet with a thickness of 1 mm.
From these sheets, strips of dimensions approximately 65 x 10 x 1 mm were cut, having their length in the direction perpendicular to the direction of rolling. To check if the direction of cutting does have an influence, one of the experiments was repeated with a strip having its length parallel to the direction of rolling. This was observed to have no noticeable effect on the result. The strips were subsequently hard soldered in small blocks of copper (10 x 10 x 10 mm), an example is shown in figure 4. This was done to obtain a

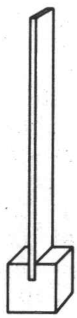

Figure 4. Strip soldered in a block of copper.

reproducible fixing point at the clamped end of the strip. In some cases however, for reasons which will be given later on in chapter IV section f, instead of the copper block a steel block consisting of two parts was attached to the strip by means of two bolts passing through the strip.
Before the measurement the strips were annealed in vacuum at 550° C for about $1\frac{1}{2}$ hours. After this annealing the grain size of the copper strip was about 0,4 mm, the silver strip 0,2 mm and the gold strip 0,3 mm.

b. APPARATUS.

As has been explained in chapter I, the deformation has to be performed, and Young's modulus measured, at low temperature. Besides, a possibility to heat the specimen to produce recovery must exist. Young's modulus was measured dynamically, by determining the natural frequencies of a strip clamped at one end, using the relation:

$$E = \frac{48\pi^2\rho\, l^4 f^2}{d^2 m^4}$$

where f is the frequency in cycles per second, l the length of the strip, d the thickness, ρ the density and m a constant, where $m = \frac{\pi}{2}$ (1,1194; 2,988; 5,000; 7,000;....) for respectively the first, second, etc. natural frequency[99]). A dynamic method of measuring Young's modulus was chosen, because the precision is high as the resonance peak is sharp, giving an accuracy in determining f better then 0,1%.

The accuracy is of course much lower if we compare the values obtained before and after deformation, as the dimensions of the specimen change in this case, the accuracy of l^4/d^2 being about 0,5%. For all the measurements after deformation however, thus all recovery measurements, the only variation is that of the frequency. Another reason why a dynamic method is chosen is the very low strain amplitude at which measurements can be performed. This is very important in view of the amplitude dependent effects mentioned in chapter II. In view of the fact that the deformation must occur at low temperature, it appeared easier to have the specimen already clamped at one end. These considerations have led to the apparatus shown in figure 5.

The apparatus is made of stainless steel, to prevent a large heat conduction from the outside, and consists of a pot in which the drawbar D, to which the strip S is connected, is placed. It will be reported later on in this section that for some measurements this drawbar was replaced by one showing a slightly different construction. A gripping apparatus G, containing a movable wedge W, is found at the bottom of the pot. To extend the strip plastically, the drawbar is pushed down, so that the strip is pushed in the gripping apparatus, as can be seen in the figure. By turning screwnut N afterwards, the drawbar is pulled upwards, while

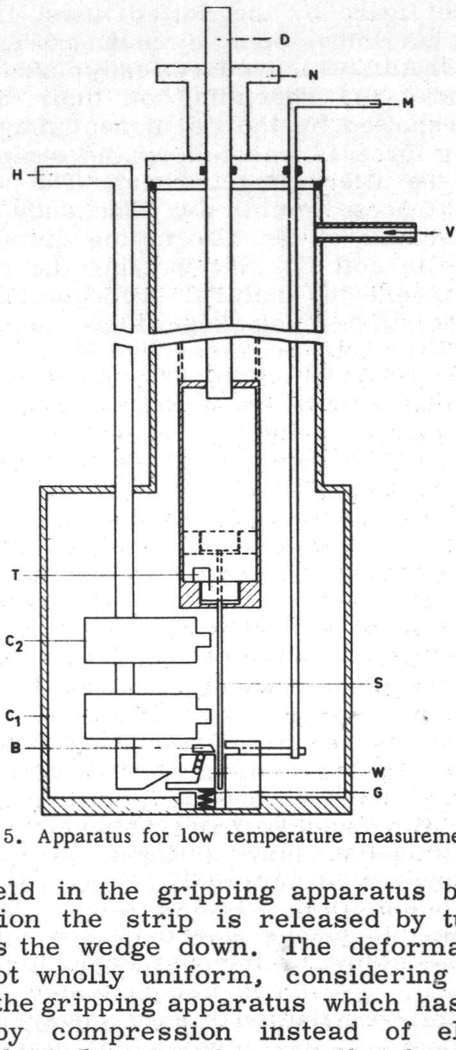

Figure 5. Apparatus for low temperature measurements.

the strip is held in the gripping apparatus by the wedge. After deformation the strip is released by turning handle H, which pushes the wedge down. The deformation obtained this way, is not wholly uniform, considering the part that has been held in the gripping apparatus which has been deformed slightly by compression instead of elongation. To check a possible influence, the part that has been held in the gripping apparatus was cut off by shear blade B, which is moved by turning handle M. This influence appeared to be small, and therefore the cutting off was not repeated. Considering the fact that the bending moment will be very small near the free end of the strip, this is not surprising. To measure the modulus, the strip is put opposite the two coils C_1 and C_2, both containing a permanent magnet. This measuring position, where the drawbar is pulled upwards, wervel stromen

is shown in the figure by the dotted lines. Through coil C_1 an alternating current is sent, by means of an RC oscillator (Peekel, type 31 ASP) which induces eddy currents in the strip. These currents are, depending on their direction either attracted or repelled by the permanent magnet in coil C_1. Thus a varying force is exerted on the strip with the same frequency as the alternating current. The amplitude of the vibrations is traced by coil C_2. The eddy currents in the strip arise because of the alternating distance to the permanent magnet in coil C_2. By varying the frequency of the alternating current the natural frequencies can be found, as the amplitude will be much larger there causing a maximum in the tension over coil C_2, measured by an electronic voltmeter (Philips, type GM 6012). The frequency is measured by an electronic counter (Beckman, model 7350 H).
The construction, as described, where the strip is attached to the drawbar, is inadequate to do measurements on gold strips. The resonance peak is obscured, probably due to vibrations of the drawbar, as Au is heavier and has a lower value for E, thus a lower natural frequency than Cu and Ag. Therefore a different drawbar was constructed, where the strip is placed with its clamped end at the bottom of the pot, while the gripping apparatus is attached to the drawbar finding itself above the strip. Compared to the other construction, shown in the figure, the strip and gripping apparatus have changed places. The construction of the gripping apparatus was slightly altered, by equipping it with a double wedge. The only disadvantage of this construction compared to the other is that the position of the strip with respect to the two coils is fixed. Therefore it can only be used in a smaller deformation range, as for larger deformations the top of the strip will rise too high above coil C_2. The deformation is carried out at about 78°K, while the whole assembly is put in a bath of liquid nitrogen.
The temperature of the strip is measured by thermocouple T (copper-constantan). To prevent ice formation on the strip, the pot is evacuated through V during the experiment. For recovery measurements, the pot is heated by a heating spiral, which is mounted around the pot. The heating current is regulated in order to obtain a temperature which increases linearly as a function of time. For this purpose a platinum resistance thermometer, which is part of the servomechanism that controls the heating current, is attached to the pot. The temperature of the strip was checked to vary, apart from a starting time, linearly as a function of time. During the recovery measurement, the whole assembly is put in a dewar vessel.

c. EXPERIMENTAL PROCEDURE.

The change of Young's modulus E, after a single deformation, is found by measuring one of the natural frequencies before and after deformation. The length and thickness of the specimen are measured before and after the experiment, the change in density is taken to be very small and will be neglected. If we now look at the relation for E, given in the preceding section, we can regard E as $E = Cf^2l^4/d^2$, where C is a constant, depending only through m on the frequency considered. Usually the second and third natural frequency were measured, giving for example for a copper strip having a length of 66,85 mm and a thickness of 0,97 mm values of 817,6- and 2289,6 cycles/second for the second and third natural frequency respectively. Both frequency values give a value of $12,39.10^{11}$ dynes/mm^2 for Young's modulus, which is a very reasonable one. Otherwise only the value for f^2l^4/d^2 is calculated for the second natural frequency, multiplying it with the factor $(m_2/m_3)^4$ if the third natural frequency was measured.
For measuring the effect as a function of deformation, the deformation is increased stepwise, and after each step the frequency is measured. The difficulty here is to measure the length after each deformation step, as the measurement is done at low temperature, thus the strip can not be seen or removed. The length was measured in this case, by putting the shearblade B in front of the gripping apparatus after the strip has been released from the gripping apparatus, and pushing the strip down until it touches the blade. The length increase compared to the previous value is then measured outside the apparatus on top of the drawbar by an attached micrometer. The values for the thickness were calculated starting from the assumption that the volume of the strip remains constant before and after deformation. This assumption seems reasonable for small deformations. The change of E is expressed by $\Delta E/E_o = (E-E_o)/E_o$, where E is the measured value after deformation, E_o the one before deformation. So $\Delta E/E_o$ will be negative for a decrease of the modulus.
The recovery as a function of the temperature, was measured in the following way: E was measured as a function of temperature, before and after deformation, while the temperature increased at a certain constant rate. The warming up rate, for the construction where the strip is attached to the drawbar, was 3/4° K/minute, for the construction where the strip was placed at the bottom of the pot 1°K/minute. An example of a result of a measurement on Cu is shown in figure 6.

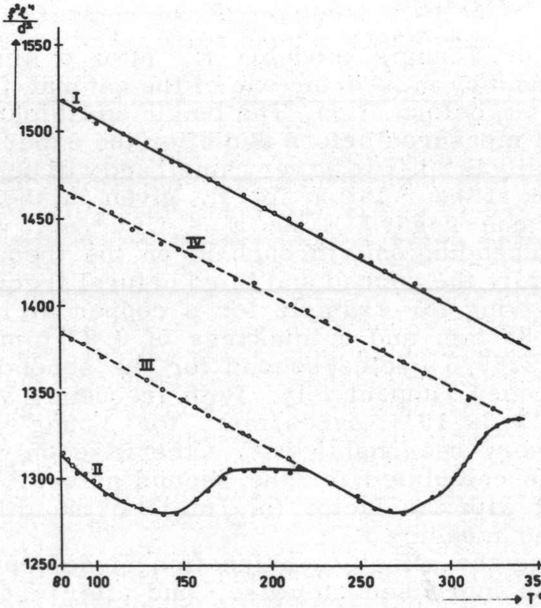

Figure 6. E as a function of the temperature for Cu, before deformation (I), after deformation at 78°K (II), (III) and (IV), while warming up.

Curve I represents E before deformation as a function of the temperature, curve II after deformation at 78° K while warming up. The difference between the two curves gives us ΔE, which divided by the corresponding E_o value gives us $\Delta E/E_o$. The recovery curve obtained this way is shown in figure 7. To ensure that the effect is an irreversible

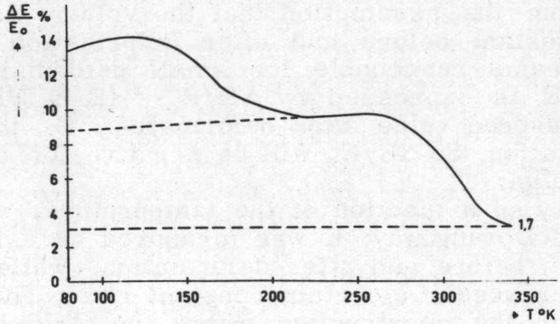

Figure 7. The recovery curve obtained from the difference of curves I and II in figure 6. The dotted lines are the differences between I and III, and I and IV.

one, and to be able to compare these measurements with the kind of measurements where after recovery at a temperature T during a certain constant time the measurement is repeated at 78 °K, the specimen was cooled down again three times to 78°K. This gives us the value for ΔE after a certain recovery measured at 78°K, which then can be compared with the value measured at the annealing temperature. The dotted curves III and IV represent the value of E, after recovery up till a certain temperature below this temperature, obtained by cooling down after this recovery and subsequent warming up. In this way we can separate the reversible and irreversible change of ΔE, about which more will be said in the next chapter. This way of measuring the recovery was chosen in the first place for the practical reason that it is difficult to produce quick changes in temperature of the strip, needed if the specimen is to be held at a certain temperature during a well defined constant time. A disadvantage of the method used is, that most recovery measurements are done by this pulse heating. But as most of these measurements are on the electrical resistivity, that method is also chosen for a practical reason: as to get a good precision the electrical resistivity should be measured at the lowest possible temperature. An advantage of the method used is that the recovery is measured continuously, enabling one to take as many measuring points as wanted. In addition to deformation, lattice defects were also produced by quenching. For Au and part of the Ag strips, the heating before quenching took place in the atmosphere. The Cu and the remaining Ag strips were heated in vacuum, which was performed in a quartz tube which at one side is connected with the vacuum pump and closed at the other side by a plug to which the strip by means of a thin copper wire was attached. The quartz tube was put in an oven. To be able to remove the strip to be quenched in iced water, argon (to suppress oxydation) was passed into the tube to enable one to remove the plug. Some experiments were performed, where recovery took place to a certain temperature, while the strip was under influence of an elastic stress. After this the specimen was cooled down again to 78°K, where the elastic stress was removed, subsequently the recovery was measured in the normal way. The elastic stress was measured by putting a spring under screwnut N (figure 5), the distance across which the spring is pushed in, measured by a micrometer, is a measure for the stress.

CHAPTER IV

EXPERIMENTAL RESULTS

a. INTRODUCTION.

The way the measurements were performed, has been described for the greater part in chapter III. The results can be roughly divided into two groups: those giving information about the effect itself, and those about the recovery of the effect. The former will be treated in section b, the latter in sections c through f. The recovery was measured in different circumstances, each treated in a separate section, in order to get some idea on the mechanism of the recovery. The existing theories about the recovery phenomena are briefly discussed in chapter II, to which the reader is referred.
The discussion of the results will mainly be postponed to chapter V, and will thus be restricted in this chapter as far as is required to make the results clearer.

b. THE EFFECT.

After deformation at room temperature, recovery is taking place during and after the deformation. This is illustrated in figure 8, showing a measurement on Cu. For $\Delta E/E_0$ a value of nearly 9% was found about 1 minute after deformation, while E is increasing (which means $\Delta E/E_0$ is decreasing) as a function of time. Both the deformation and recovery temperature were 293 °K in this case, similar results have been observed by others[9]).
To obtain results of the effect itself, not obscured by recovery phenomena, the deformation has to be performed at a temperature where no recovery occurs. For Cu, Ag and Au no recovery was observed after deformation at 78°K, while keeping the specimen for 24 hours at that temperature. Measurements in the temperature range of 4,2° to 78°K after deformation at 4,2°K, carried out by Druyvesteyn and Blaisse[100]), show that in accordance with electrical resistivity measurements, only a slight recovery occurs in that temperature range. The effect as a function of the deformation for Cu and Ag is shown in figures 9 and 10. As was described in chapter III-c, the deformation was in-

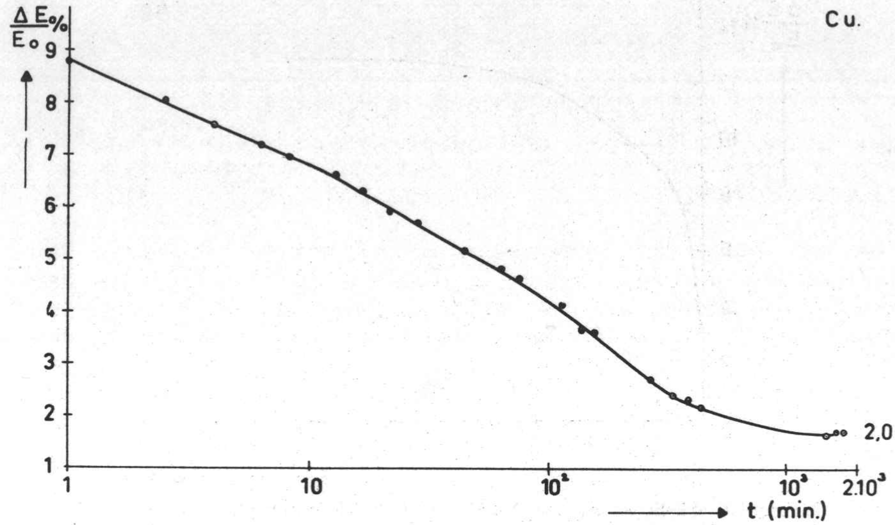

Figure 8. Recovery curve for Cu as a function of time after an elongation of 2% at 293°K.

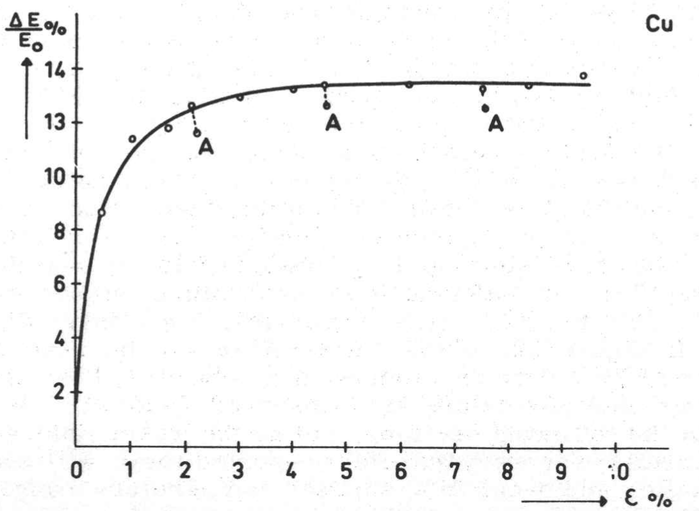

Figure 9. $\Delta E/E_0$ of Cu as a function of the deformation at 78°K.

creased stepwise in this case. It appears that a saturation value for $\Delta E/E$ of about 13 to 14% is reached in both cases. A similar effect was observed on the torsion modulus by Druyvesteyn c.s.[12]). It seems that the saturation value is reached later in the case of Ag. An interesting detail is

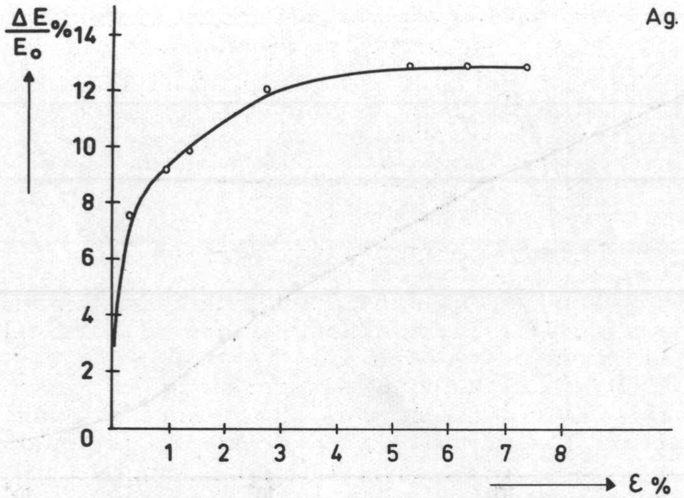

Figure 10. $\Delta E/E_0$ of Ag as a function of the deformation at 78°K.

that if one of the steps of the stepwise deformation was chosen to be small (an extra elongation of about 0,1%) a small increase of the modulus was found. This is shown by the points A in figure 9, and has also been observed, although not represented in the figure, on Ag. The saturation effect was also observed at 4,2°K and 20°K [100]). As these results are of great importance for the effect as a whole, the figures belonging to them will be represented here. In figure 11 $\Delta E/E_o$ as a function of the deformation at 4,2° and 20°K is shown. The saturation value reached is lower as the temperature is lower. This decrease appears to be reversible and is observed to be practically independent of the deformation temperature in the range of from 4,2° to 78°K. The reversible behaviour is even clearer in figure 12, where curve II shows the reversible change of $\Delta E/E_o$ after an elongation of 6% at 4,2°K. Above 78°K reversible effects are also observed, and will be discussed in the following sections, but as the reversible effect is decreased by recovery the effect reported there will usually be smaller and occur in a smaller temperature range for Cu and Ag. No direct measurement of the effect as a function of the deformation was made on Au. As was mentioned in chapter III-b a different construction of the apparatus was used for Au, which did not have the facilities needed for length measurements in between the deformation steps. Two measurements on Au are shown in section c, figure 15. Recovery starts at a higher temperature, the reversible ef-

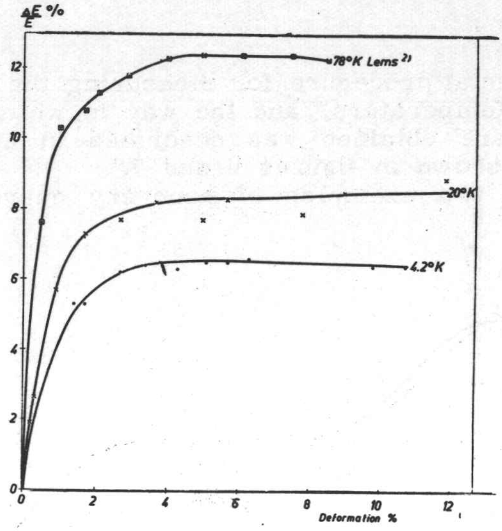

Figure 11. $\Delta E/E_0$ of Cu as a function of the deformation at 4,2°K and 20°K, r ported by Druyvesteyn and Blaisse [100]).

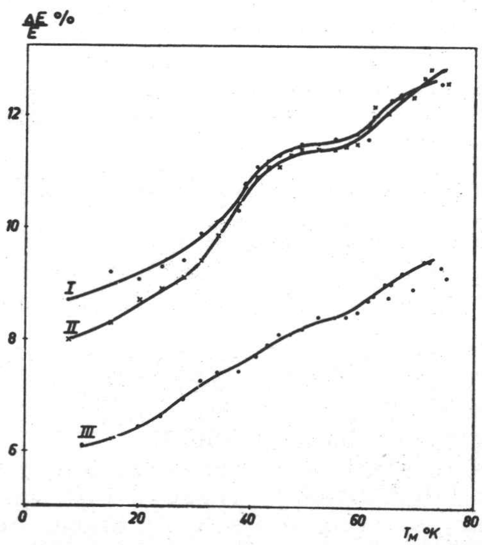

Figure 12. The reversible change of $\Delta E/E_0$ as a function of the temperature is represented by curve II [100]).

fect is clearly visible. The effect increases for increasing deformation, it can be concluded from the results that if a saturation value is reached, it is reached more slowly than for Cu and Ag.

c. RECOVERY.

The experimental procedure for measuring the recovery as a function of temperature, and the way in which the recovery curves are obtained was described in chapter III-c, respectively shown in figures 6 and 7.
In figure 13 two examples of recovery curves measured

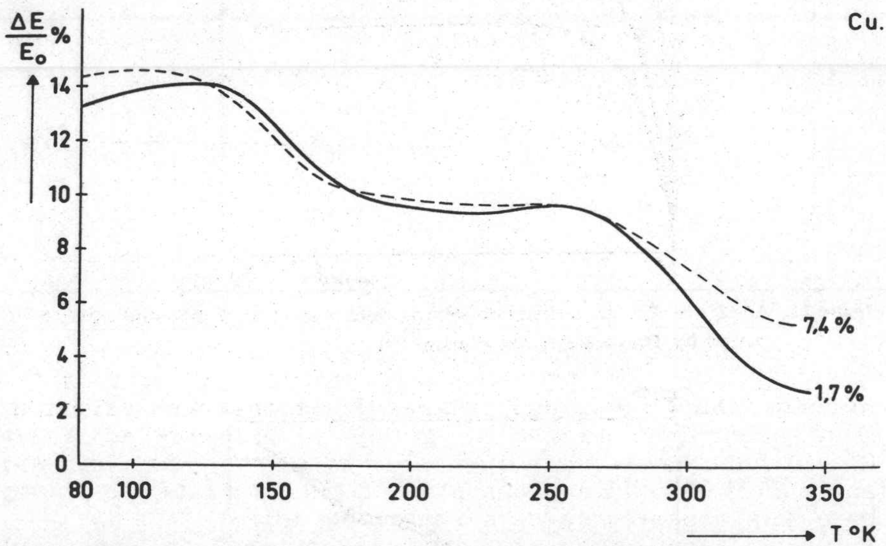

Figure 13. Recovery curves for Cu after elongations of 1,7% and 7,4%.

on Cu, for two different degrees of deformation, are shown. Recovery occurs in two stages, the first for reasons mentioned in chapter I we will call stage II, the second will be called stage III.
Stage II, giving a recovery of about 5% extends from about 110° to approximately 190°K, stage III giving a recovery that depends on the degree of deformation is in the range of from 250° to approximately 350°K.
Stage II appears to start at a somewhat lower temperature as the degree of deformation increases. For smaller deformations, recovery starting at a slightly higher temperature, the last part of the reversible effect discussed in the previous section can be seen below stage II. A very small reversible effect is noticeable between 220° and 250°K. This can be concluded from the difference in slope of curves III and I in figure 6 (chapter III-c).
Figure 14 shows two examples of recovery curves obtained

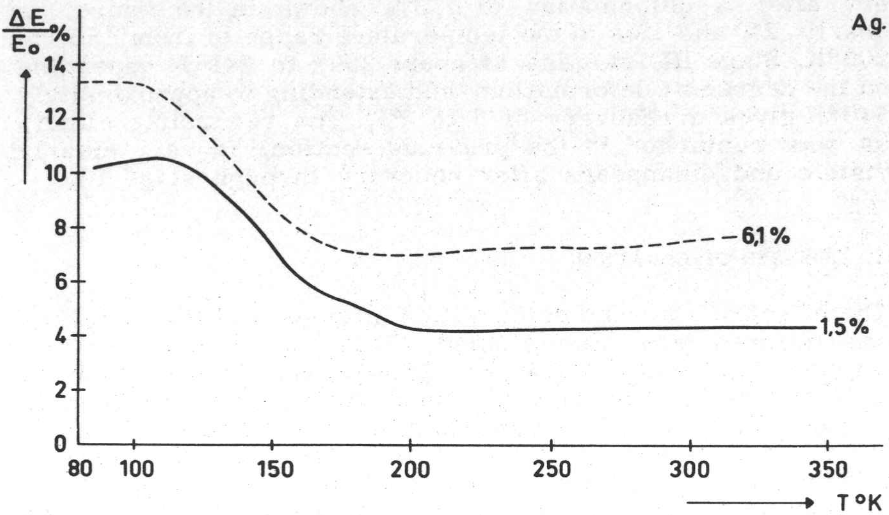

Figure 14. Recovery curves for Ag after elongations of 1,5% and 6,1%.

for Ag. Only one stage, stage II, is observed extending from about 100° to 200° K, giving a recovery of about 6%. In some cases a small increase of $\Delta E/E_0$ was observed above 270° K, but in most cases as for the two examples shown here this appeared to be a reversible effect.

For Au, represented in figure 15, stage II is found only

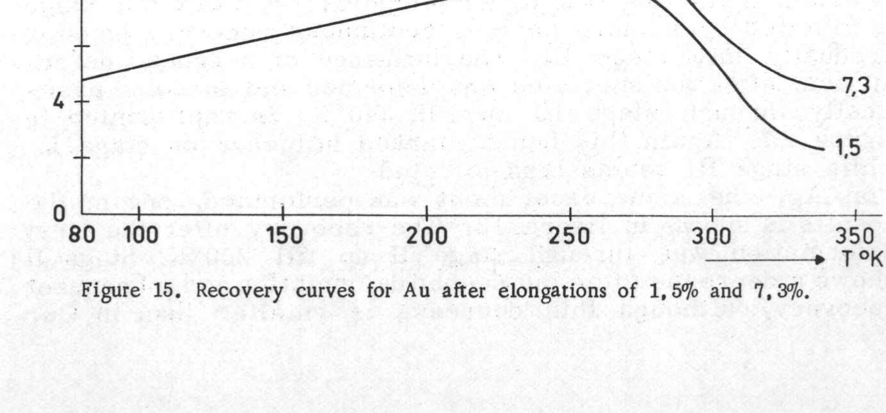

Figure 15. Recovery curves for Au after elongations of 1,5% and 7,3%.

for deformations larger than about 4%. The stage II recovery after a deformation of 7,3%, shown in the figure, is nearly 2% and lies in the temperature range of from 150° to 200°K. Stage III, starting at about 230° to 240°K depending on the degree of deformation, and extending to approximately 340°K gives a recovery of 6 to 7%. The reversible effect, as was remarked in the previous section, is very clearly visible and disappears after recovery through stage III.

d. REPEATED DEFORMATION.

The effect of a second deformation after part of the recovery has occured was also studied. This experiment, done on Cu and Ag, was intended to provide some more evidence concerning the recovery process. As will be explained in chapter V, we will assume that the recovery in stages II and III is caused by different point defects. The idea of this experiment is, that if for example in the case of Cu, the specimen has after the first deformation been annealed through stage II, the greater part of the type of point defect which causes stage II recovery, will have disappeared. If the specimen is cooled down again, a subsequent deformation at 78°K will together with the first one represent a total deformation that will be different from a deformation of the same value, which was not subject to a recovery treatment. As the effect was most clear in cases where the second deformation was small compared to the first, while no fundamental difference was found if both deformations were about the same value, only the former case will be represented in the figures.
Figure 16 shows a measurement on Cu, after the first deformation of about 3% recovery was measured up till 215°K, the specimen was cooled down and again deformed (0, 7%) after which the recovery up till 340°K was measured. It shows us that for the second run a much smaller stage II is obtained starting at a higher temperature, while this stage is followed by a more or less continuous recovery passing gradually into stage III. The influence of a second deformation, after the specimen was deformed and annealed practically through stage III up till 340°K, is represented in figure 17. Again this has a marked influence on stage II, while stage III seems less affected.
For Ag, the same experiment was performed, one of the results is shown in figure 18. The recovery after the first deformation was through stage II up till 200°K. Stage II shows a decrease after the second deformation and subsequent recovery, although this decrease is smaller than in Cu.

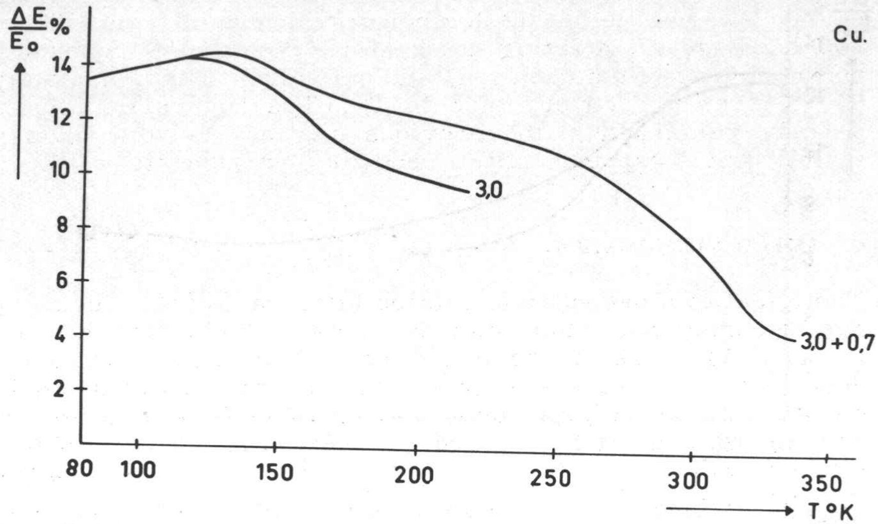

Figure 16. The influence of a repeated deformation of 0,7%, after a previous deformation of 3%, and subsequent recovery up till 215°K, on the recovery of Cu.

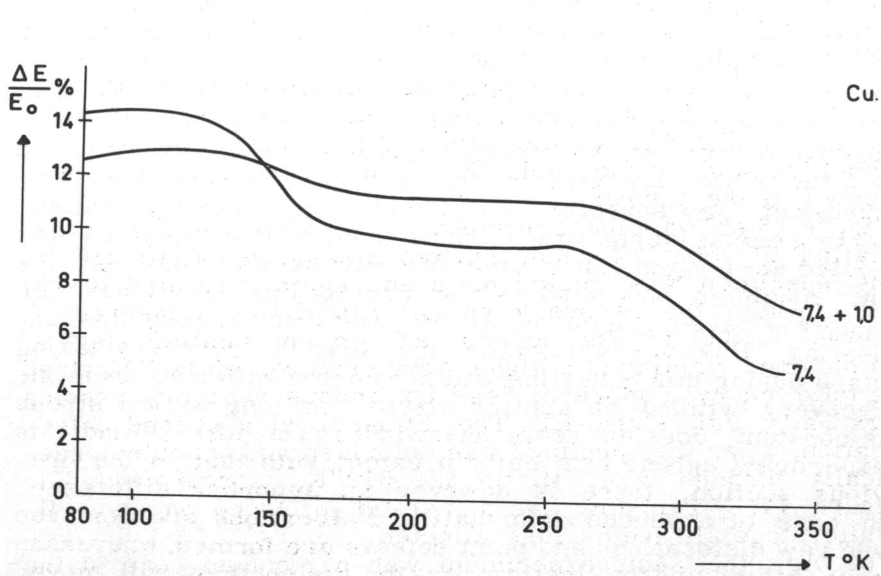

Figure 17. The influence of a repeated deformation of 1%, after a previous deformation of 7,4% and subsequent recovery up till 340°K, on the recovery of Cu.

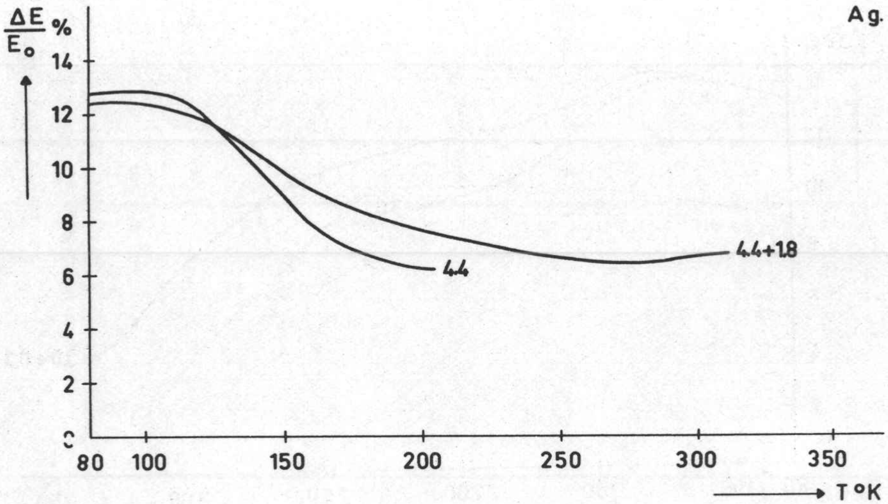

Figure 18. The influence of a repeated deformation of 1,8%, after a previous deformation of 4,4% and subsequent recovery up till 200°K, on the recovery of Ag.

e. INFLUENCE OF AN ELASTIC STRESS.

In the following experiments an elastic stress was kept on the specimen during part of the recovery. After deformation a part of the stress, being in the range of 0,1 to 0,5 times the flow stress, was kept on the specimen. The specimen was annealed, with the stress constantly applied, up to a certain temperature. Subsequently the specimen was cooled down again, after which the stress was removed. As the specimen was clamped in during this treatment, no recovery measurement could be performed during this annealing. However the assumption that the recovery taking place during this annealing will not be too different from the recovery without an applied stress, as long as the stress is constant, does not seem hazardous. The idea behind this experiment agrees to a certain extent with that in the previous section, there is however an important difference. In case of a second deformation dislocations are removed and new dislocations and point defects are formed, whereas in case of removing the elastic stress the dislocations will move, but no new dislocations or point defects will in principle be formed. The removal of the elastic stress will however

be a large enough change of the stress to free the dislocations from a great part of possible anchoring or pinning points, provided by point defects.
In figure 19, two measurements on Cu are represented,

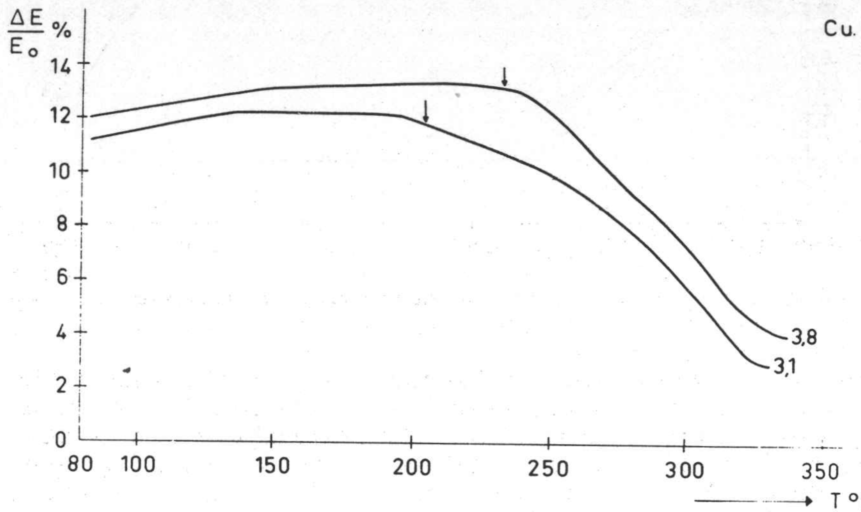

Figure 19. The recovery curves of Cu observed after the removal of an elastic stress which was kept on the specimen during a first annealing run up till the temperature indicated by the respective arrows.

the arrows indicating the temperature to which the specimen was annealed during the first run, the temperature was chosen in between stage II and III in this case. Upon removing the stress at 78°K, a value of $\Delta E/E_0$ which is only slightly less than normally obtained immediately after deformation, is found. Thus it appears that the ΔE effect returns almost entirely upon removing the stress. The subsequent annealing behaviour is entirely different compared to the normal recovery found immediately after deformation. Stage II has completely disappeared, leaving the reversible effect visible up till 140°K.
Recovery starts at the temperature where the first annealing run was stopped. From that temperature recovery is taking place continuously up till the temperature where stage III normally ends. The total amount of recovery is about the same as is found after a normal annealing run, which is the sum of stage II and III recovery. The amount of the applied stress during recovery, in the range considered, did not have any observable influence on the result. Figure 20 represents a specimen annealed up till 330°K, thus near

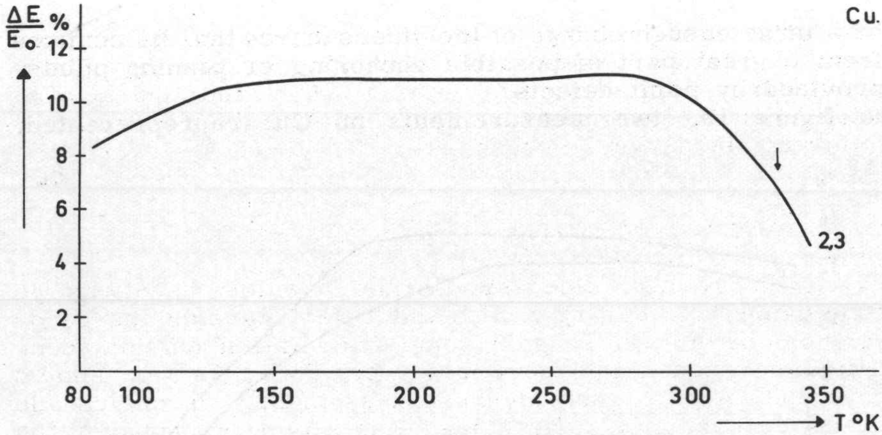

Figure 20. The recovery curve of Cu after the removal of an elastic stress which was kept on up till 330°K.

the end of stage III, during the first run. The ΔE effect measured upon unloading seems to be smaller than normally observed after deformation. The reversible effect is very clearly visible. Recovery starts below the annealing temperature of the first run, but at a temperature which is about 20° higher than where stage III normally starts. The recovery is not yet completed at the temperature where the measurement is stopped.
The same experiment was done on Ag, and is shown in figure 21.

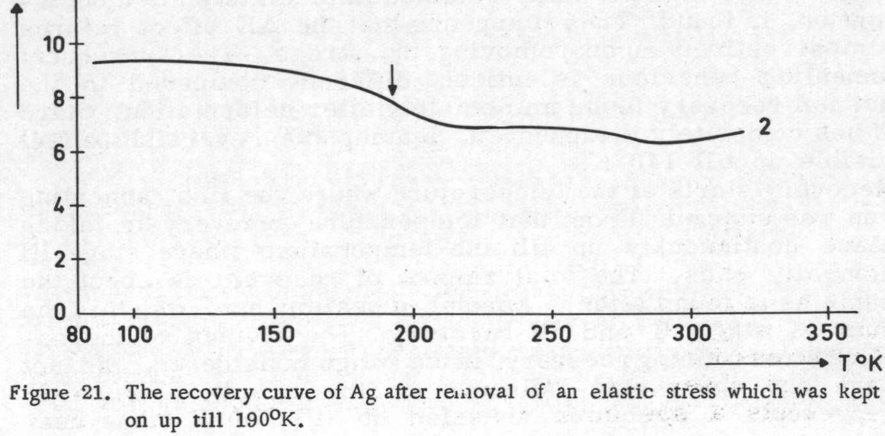

Figure 21. The recovery curve of Ag after removal of an elastic stress which was kept on up till 190°K.

The effect returns for the greater part, stage II is much smaller but not absent as in Cu. There appears to be a small amount of recovery between 260° and 290°K. The total amount of recovery is much smaller than found after following the normal procedure.

f. DEFORMATION AFTER QUENCHING.

From the results discussed in the previous sections no conclusions on the type of point defect causing the recovery can be drawn. To gain some information on this identification, quenching experiments were done. As was pointed out in chapter I, quenching produces mainly vacancies and eventually vacancy aggregates. The effect of quenching on the electrical resistivity, flow stress and internal friction has been studied by several authors and some of the results have been discussed in chapter II.
The influence of quenching on the modulus of Ag, as a function of the time after quenching, at room temperature, is shown in figure 22. The quenching in this case was per-

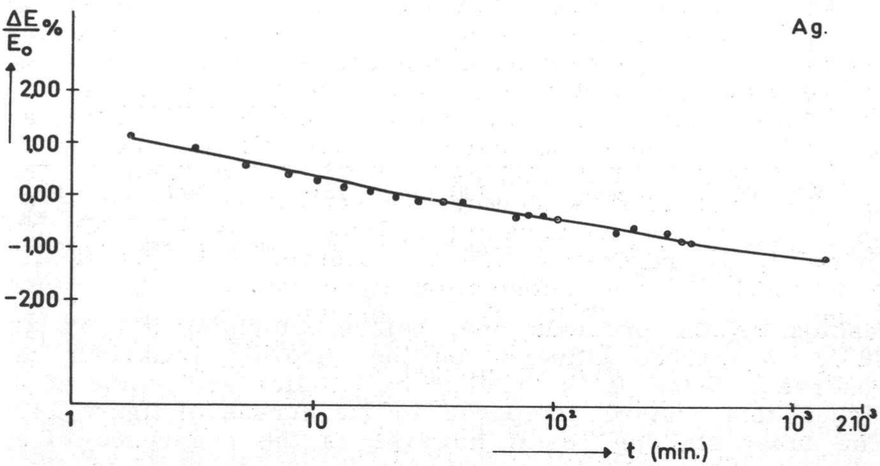

Figure 22. The recovery of Ag as a function of time at room temperature after quenching from 550°C.

formed by heating the specimen in the atmosphere and dropping it from 550°C in iced water. The concentration of vacancies will have a value in the range of 10^{-6} to 10^{-5}, which is somewhat larger than the estimated concentration due to a deformation of 1%, which lies in the range of 10^{-7}

to 10 $^{-6}$. About 2 minutes after quenching, a small decrease of the modulus, compared with the slowly cooled specimen, was observed. An increase as a function of time is observed, to a value of the modulus which is greater than the modulus before quenching. From figure 22 it appears that the point defects do not disappear very quickly after quenching, as the recovery is still not finished after 1000 minutes. Therefore it seems justified to assume that if the strip is put in the apparatus immediately after quenching and cooled down to 78°K, there will still be an excess of vacancies left. Figure 23 represents a curve of a strip of Ag quenched in the same

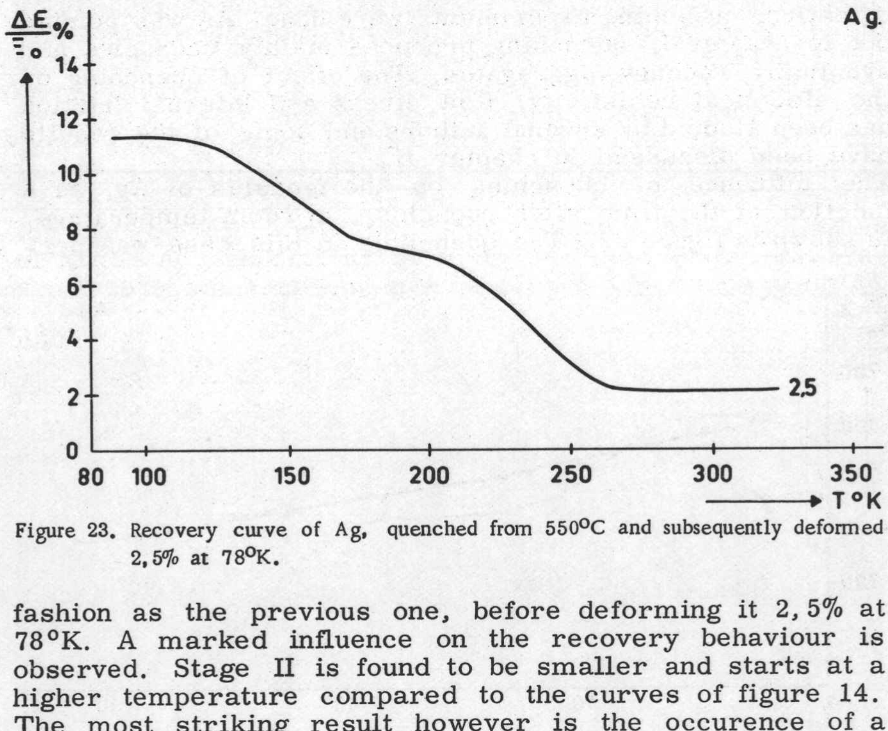

Figure 23. Recovery curve of Ag, quenched from 550°C and subsequently deformed 2,5% at 78°K.

fashion as the previous one, before deforming it 2,5% at 78°K. A marked influence on the recovery behaviour is observed. Stage II is found to be smaller and starts at a higher temperature compared to the curves of figure 14. The most striking result however is the occurence of a following recovery stage comparable to stage III in Cu. To check this result, the specimen was afterwards annealed at 550°C and again deformed at 78°K. The same curve as is shown in figure 14, consisting of only stage II was observed in this case. This makes it improbable that this second recovery stage, observed after quenching the specimen before deformation, is caused by some impurity introduced by heating the strip in the atmosphere.

As was mentioned in chapter III-a, in some cases a steel block attached to the strip by means of bolts was used instead of the soldered copper block. This was done to be able to quench from higher temperatures, where the soldered block would come off the strip. A further reason was to obtain higher quenching rates, as the soldered block will certainly lower this rate while the steel block is attached to the strip after quenching. The results agreed with the one given in figure 23, stage II being a little smaller though and stage III somewhat larger, but these differences were not important enough to need an extra figure. Varying the quenching temperature in the range of from 550° to 600°C did not influence the results very much. For quenching temperatures around 700°C the results showed principally the same picture, they did not however reproduce very well. By quenching out of the vacuum oven, in the way described in chapter II-c, also essentially the same results were found. Cu was quenched out of the vacuum oven, as heating in the normal atmosphere would cause oxydation. In some cases we succeeded in getting the Cu specimen quenched without a trace of surface oxydation, but in a good many cases the surface was more or less oxydated. However, this did not have any observable influence on the results. A result is shown in figure 24, the Cu specimen with a soldered block

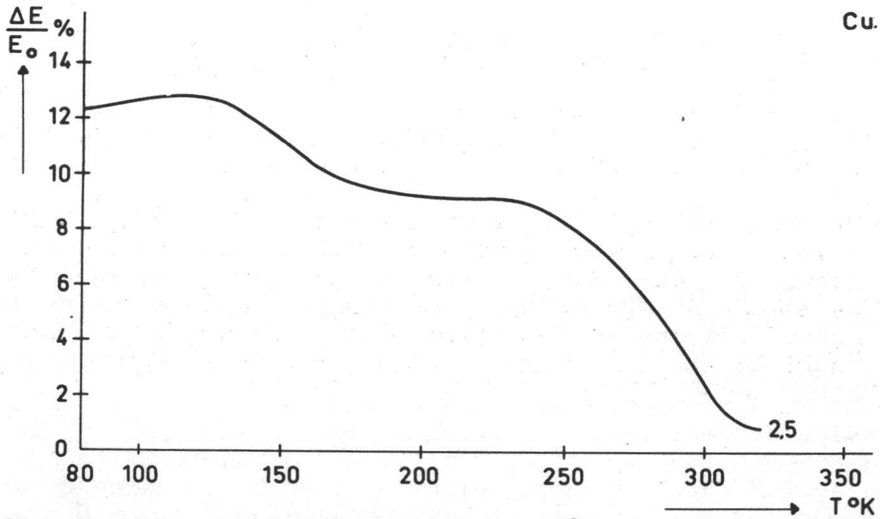

Figure 24. Recovery curve of Cu, quenched from 550°C and subsequently deformed 2,5% at 78°K.

was quenched from 550°C before deformation (2,5%) at 78°K.

Stage II is a little smaller compared with the recovery curve in figure 13, stage III is considerably larger and starts at a lower temperature. The same results were obtained on Cu strips with the steel block attached to them after quenching from temperatures in the range of from 550°C to 650°C. In some cases however continuous recovery was observed between stage II and III.
The Au strips were heated in the atmosphere, as there seems to be need in this case to do it in vacuum. A result is shown in figure 25, the quenching temperature was 550°C

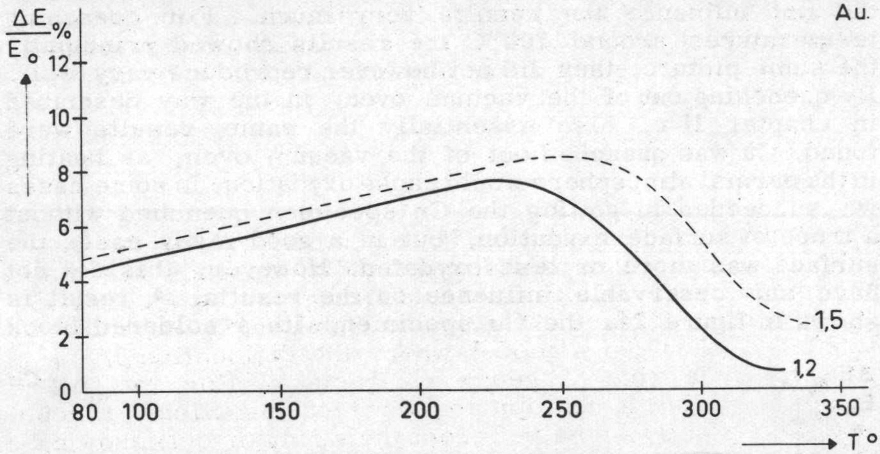

Figure 25. Recovery curve of Au, quenched from 550°C and subsequently deformed 1,2% at 78°K. The dotted curve represents the normal recovery after a deformation of 1,5% at 78°K, as shown in figure 15.

the deformation 1,2%. Stage III starts and is completed at a lower temperature compared with the normal recovery curve, of which the example for a deformation of 1,5%, as shown in figure 15, is represented here by the dotted curve. The size of this stage is only slightly larger which could be due to the fact that the recovery is practically completed.

CHAPTER V

DISCUSSION AND INTERPRETATION OF THE RESULTS

In this chapter we will try to explain the experimental results, and discuss some details from chapter II a little more extensively. We will start with an extension of the kink-model, which was discussed in chapter II, section a. This is treated before the sections of the results, as the results of this model are needed for the discussion of the results on the ΔE effect, given in section b. Section c is devoted to the recovery of the effect.

a. THE MODEL.

As was discussed in chapter II, the model where the dislocation is represented by a string fixed at its ends has met with considerable success in explaining the experimental results known so far. Seeger and Schiller [54]) have calculated the effect on a model where the dislocation is represented by a row of geometrical kinks. For low frequencies they find a relation for the modulus effect, which is about the same as obtained from the string model, showing also the L^2 dependence on the looplength. This does not seem very surprising however, as they treat this row of kinks analogous to a row of mass points of an elastic string. We have tried a different way to calculate this effect. Consider a dislocation containing so many kinks, that the distance between them becomes so small that although the applied stress wants to move the kinks to the right, the kink at the extreme left side does not move because the repulsive force of its neighbours is larger than the force due to the applied stress. For this repulsive force we will take the relation used by Seeger:

$$K = \frac{Gb^2a^2}{8\pi d^2(1-\nu)}\left[(1+\nu)\cos^2\phi + (1-2\nu)\sin^2\phi\right]$$

where G and b have their usual meaning, ν is the Poisson constant, a is the distance between the Peierls' valleys = $\frac{1}{2}b\sqrt{3}$, d is the distance between the kinks and ϕ is the angle between the Burgersvector and the average direction of the dislocation. If the applied stress exerts a force to the right on the kinks, we will get a kind of pile up of the kinks against the kink at the extreme right, shown in figure 26. The equilibrium distances between the kinks of a dis-

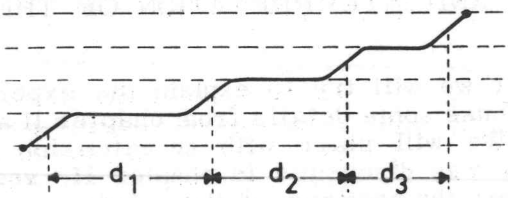

Figure 26. The applied stress moves the kinks to the right, the distances between the kinks decrease from left to right.

location, containing n kinks, under influence of a stress: $d_1, d_2, \ldots, d_{n-1}$, will thus decrease in this sequence. This pile up is different from the pile up of parallel infinitely long dislocations, as considered by Eshelby, Frank and Nabarro [49]), the repulsive force varying with $1/d^2$ for the kinks, while this varies with $1/d$ for the parallel dislocations.
For a certain value σ of the component of the stress in the direction of the Burgers vector, the equilibrium positions of the kinks will be given by the following relation for the distances between them, considering the interactions with the nearest neighbours only:

$$\frac{1}{d_p^2} - \frac{1}{d_{p-1}^2} = \frac{\sigma bw \; 8\pi(1-\nu)}{Gb^2a^2\left[(1+\nu)\cos^2\phi + (1-2\nu)\sin^2\phi\right]} = C$$

where σbw is the force on a kink, which has a width w, due to the applied stress. This relation will be equal to a constant C if σ is constant. From the relation we see that:

$$d_p/d_1 = 1/\sqrt{1 + (p-1)Cd_1^2}$$

For a dislocation of length L, we get as condition:

$$\sum_{p=1}^{n-1} d_p = d_1 \sum_{p=1}^{n-1} \frac{1}{\sqrt{1 + (p-1)Cd_1^2}} = L - w \approx L \quad (\text{as } w \ll L) \ldots \quad (1)$$

The movement of the kinks means a movement of the dislocation, which gives a contribution to the elastic strain in direct proportion to the area of displacement. This area will be equal to:

$$A = a(d_1 - d_0) + a(d_1 + d_2 - 2d_0) + \ldots + a\left[d_1 + \ldots + d_{n-2} - (n-2)d_0\right]$$

where d_o is the distance between the kinks if $\sigma = 0$, thus $d_o = L/(n-1)$. Expressing d_2, d_3 etc. in terms of d_1, leads to:

$$A = ad_1 \sum_{p=1}^{n-2} \frac{n-p-1}{\sqrt{1 + (p-1)Cd_1^2}} - \frac{a(n-2)}{2} L \ldots \qquad (2)$$

If n is large enough and Cd_1^2 small enough, the first term of the serie will be small compared with the sum, so we can write:

$$A = ad_1 \int_1^{n-1} \frac{n-x-1}{\sqrt{1 + (x-1)Cd_1^2}} \, dx - \tfrac{1}{2}a(n-2)L$$

while we can write relation (1) as:

$$d_1 \int_1^n \frac{dx}{\sqrt{1 + (x-1)Cd_1^2}} = \frac{2}{Cd_1} \left(\sqrt{1 + (n-1)Cd_1^2} - 1\right) = L$$

which gives us:

$$d_1 = \frac{4L}{4(n-1) - CL^2} \qquad {}^{*)}$$

For A we get:

$$A = \frac{2ad_1}{3C^2 d_1^4} \left[2\left\{1 + (n-2)Cd_1^2\right\}^{\frac{3}{2}} - 3(n-2)Cd_1^2 - 2\right] - \tfrac{1}{2}a(n-2)L$$

*) We can conclude that the condition n is large enough and Cd^2, is small enough means that CL^2 should be small enough compared with 4 n. It depends of course of the precision wanted which value of C, thus of σ, we can allow. Assuming that n has a value of about 30, if L = 1000 b, which seems close enough in the neighbourhood of the maximum number which the dislocation can contain if Seeger s value W = 30 b is correct, the value of σ should be smaller than 10^{-7} G.

Substituting the value for d_1, and remembering the condition that n should be large, we get:

$$A = \frac{aCL^3}{24}$$

This same value is obtained for very small stresses, where if $nCd_1^2 \ll 1$ we can write:

$$1/\sqrt{1 + (p-1)Cd_1^2} = 1 - \tfrac{1}{2}(p-1)Cd_1^2$$

Substitution of C gives:

$$A = \frac{8\pi w\ (1-\nu)}{24a\left[(1+\nu)\cos^2\phi + (1-2\nu)\sin^2\phi\right]} \cdot \frac{\sigma}{Gb} \cdot L^3$$

According to Seeger $w \approx 30$ b, we thus obtain for a screw dislocation ($\phi = 0$) taking $\nu = 1/3$:

$$A_s = 5,8\pi \frac{\sigma}{Gb} L^3$$

For an edge dislocation:

$$A_e = 23,2\pi \frac{\sigma}{Gb} L^3$$

The string model gives for the area of displacement:

$$A_s = \frac{1}{24} \frac{\sigma}{Gb} L^3 \quad \text{and} \quad A_e = \frac{1}{6} \frac{\sigma}{Gb} L^3 ,$$

if we take for the line tension the values given by Stern c.s.[39]) for a face centered cubic crystal: $C_s \approx 2Gb^2$ and $C_e \approx \frac{1}{2}Gb^2$. The area of displacement for the kink model is thus about 440 times larger than for the string model. Substituting this larger area in the relation for $\Delta E/E$ derived by Friedel c.s.[35]) for a random distribution of the glide planes, as given in chapter II, we obtain:

$$-\frac{\Delta E}{E} = \frac{440\Lambda L^2/18}{1 + 440\Lambda L^2/18} \approx \frac{24\Lambda L^2}{1 + 24\Lambda L^2}$$

If $\Lambda L^2 = 3$, a value usually taken for a regular three dimensional network, the value for $\Delta E/E$ is nearly seven times larger than for the string model. In reality this value will be lower, as for instance dislocations lying in one Peierls' valley will not contribute. The value of the area of displacement also depends on the uncertain value of w (according to Lothe [101]) $w \approx 10b$) therefore the order of magnitude of the effect is very uncertain.

The most important result however, is that the relation based on the kink model shows the same dependence on the looplength and the dislocation density as the relation based on the string model. In both cases the recovery can be described as a shortening of the looplength L, due to the pinning by point defects. In the string model the dislocation line is anchored at a certain point by the pinning point, thus bowing out less under influence of the stress. In the kink model the pinning point immobilizes a kink, or forms an obstacle against the movement of kinks along the dislocation line. An advantage of the kink model seems the fact that the displacement of the kinks is large compared with the displacement of the dislocation represented by a string, under influence of the same stress. This gets round the difficulty of how to visualize pinning for very low stresses, where the dislocation string moves over distances which are much smaller than an atomic distance and thus much smaller than the size of the pinning point.
From the difference in the relations of the two models, it can be concluded that the value for $\Delta E/E$ should depend on the amplitude of the strain, if the kink model is a good approximation for low stresses and the string model a good one for higher stresses. It should also be noted that if the condition σ small enough is not fulfilled, the area of displacement of the kink model will not increase in direct proportion with σ, thus also causing an amplitude dependence of $\Delta E/E$.

b. THE EFFECT.

From figures 9 and 10 in chapter IV, we see that the decrease of Young's modulus reaches a saturation value of about 14% as a function of the deformation.
The theory based on the string model, discussed in chapter II, predicts a value for $\Delta E/E$ equal to about $\Lambda L^2/19$. The fact that as a function of the deformation a saturation value is reached, seems to indicate that ΛL^2 has a constant value after a certain amount of deformation. This does not seem

very surprising, as Λ will increase and L decrease on proceeding deformation. For a regular three dimensional network ΛL^2 will be about equal to 3. Substituting for ΛL^2 this value in the relation for $\Delta E/E$, we obtain a value of nearly 16% for the decrease of the modulus. This seems to be in excellent agreement with our experimental value of 14%, the more so as this experimental value is the decrease of the modulus compared with the annealed specimen, which will undoubted contain some partly mobile dislocations and thus show a modulus effect itself. Assuming that the saturation value of the modulus increase of about 2%, observed after irradiating an annealed crystal, reported by several authors as was discussed in chapter II, gives the right order of magnitude for the modulus effect of the annealed crystal, this 2% should be added to the 14% observed.

This agreement between our experimental results, and the relation for $\Delta E/E$ based on the string model, does not prove however that this relation is correct.

For instance the dislocations formed by deformation will in all probability not form a regular three dimensional network. Observations by the electron microscope reveal that the dislocations formed by deformation in pure f.c.c. metals form a kind of cell structure consisting of relatively dislocation free areas separated by bounderies with a high dislocation density. This dislocation arrangement will probably have a different value for ΛL^2 than the regular three dimensional network.

The experimental fact that $\Delta E/E$ at low temperature, changes reversibly with the temperature, makes it difficult to speak of a definite value for $\Delta E/E$.

There is strong experimental evidence that the dependence on ΛL^2 is correct, as was discussed in chapter II. However the kink model, discussed in section a) also shows this dependence, but it gives a larger value for $\Delta E/E$ compared with the string model. Although at first sight the string model seems to give the best agreement with the experimental facts, we cannot conclude in favour of one of the two models.

Comparing the value for $\Delta G/G$ of about 17% observed by Druyvesteyn c.s. [12]) after plastic deformation by torsion at 78°K with our value for $\Delta E/E$ of 14%, we see that they are of the same order of magnitude. This could be expected as their deformation and measurement are both performed by torsion, while ours both by elongation.

The reversible $\Delta E/E$ effect cannot be understood from the expression given, unless it is assumed that L changes as a function of the temperature. Gordon [19]) suggests this to happen by thermal unpinning, and thus tries to explain the reversible ΔE effect measured on rocksalt by Bauer [102]). This does not seem a very likely explanation in our case though, the temperature dependence would presumably not be approximately linear, but much stronger. Also the relaxation process that causes the Bordoni peak does not seem to be able to explain the effect. As was explained in chapter II, the elastic modulus decreases from the unrelaxed to the relaxed value going from the temperature below to one above the relaxation peak. The modulus change due to this relaxation mechanism is about 0,3% if the Bordoni peak consists of a single relaxation peak, and somewhat higher if the peak consists of more relaxation processes. This is much too low to explain the reversible effect. The Bordoni peak is also restricted to a much smaller temperature range than the reversible effect. In addition, the annealing behaviour of the Bordoni peak, as has been studied after deformation at low temperature by Okuda [103]) and Bruner and Mecs [104]), is different from the annealing behaviour of the reversible effect.

The fact that the recovery of the reversible effect acts in concert with the recovery of the effect itself, seems to indicate that it is a property of the effect itself. Nothing in the relation for $\Delta E/E$ except may be the change of L through thermal unpinning as has been mentioned, appears to be temperature dependent. However by deriving this relation, the line tension of the dislocation is taken to be $\frac{1}{2}Gb^2$. The temperature dependence of the line tension in this case is the same as the temperature dependence of G. As ΔG is divided by G (respectively ΔE by E) the relation thus obtained shows no temperature dependence.

If however the temperature dependence of the line tension is different from that of G, increasing more strongly with decreasing temperature for instance, a reversible effect would be expected. Stern and Granato [39]) calculate values for the line tension of a screw and an edge dislocation from experimental values at room temperature and at 4,2°K for Cu. They find that the line tension of the screw dislocation is nearly temperature independent, while the line tension of the edge dislocation increases at decreasing temperature by about 30% over its room temperature value. This means that the contribution of the edge dislocations to the non elastic strain decreases by 23%, which means a change of ΔE of 23%. Actually this value will be less as the dislocations will certainly not all be of the edge type. It should be pointed out though that the contribution of the edge dislocations to the ΔE effect will be the largest, as their line

tension is smaller than that of a screw dislocation, as the values in previous section show. This is also the case in the kink model, where the repulsive force between the kinks, as can be seen in section a), is larger for a screw than for an edge dislocation. The difference in temperature dependence does not follow from the kink model, but again it should be remembered that this model represents the dislocation only very schematically. For instance it could very well be that the kink width is temperature dependent, or that the repulsive force between the kinks should contain a temperature dependent factor.
From the experiments of Druyvesteyn and Blaisse [100]) it appears that ΔE changes about 40%, which is larger than would be expected from the value mentioned above.
This change is also found in a smaller temperature range than the change of the line tension. It should be pointed out though that the approximations used to calculate the line tension are still far from being perfect. Therefore the possibility that the reversible effect is caused by the temperature dependence of the line tension can not be ruled out.
The degree of deformation in which the saturation value is reached seems to increase in the sequence Cu, Ag and Au. This is probably the same sequence in which the stacking fault energy of these metals decreases. According to Howie [105]) the results from electron microscope observations show that as the stacking fault energy is increased from a low value there is a steadily increasing tendency for dislocations to leave their slip planes as the deformation proceeds. If the formation of a three dimensional network, leaving the nature of this network aside, is attained at a lower degree of deformation the higher the stacking fault energy is, it could be that ΛL^2 reaches its constant value sooner in a metal with a high stacking fault energy than in a metal with a low one. It seems questionable though that dislocations leaving their slip plane should occur at these small deformations at 78°K, neither is it known how far this process is needed to form a three dimensional network.
From figure 9 there appears a small effect which depends on the size of the steps of the stepwise deformation.
If the step was chosen to be small a decrease of ΔE, indicated by the points A in figure 9, was observed.
This effect could probably be due to the same mechanism that causes the small yield point effect during an interrupted tensile test, observed for example by Westwood and Broom [106]) and Haasen and Kelly [107]). This effect is illustrated in figure 27, a part of the stress-strain curve is drawn showing the small yield point that is observed after unloading

and subsequent reloading the specimen. Upon proceeding the deformation beyond this yield point, the same stress-

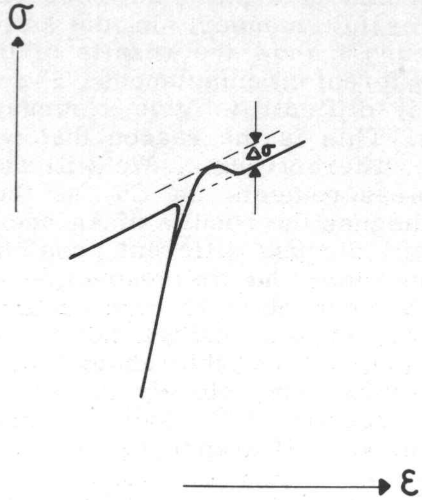

Figure 27. The yield point effect as observed by Haasen and Kelly [107]).

strain curve is obtained as if no unloading had taken place. This means that if the deformation steps are chosen large enough in our experiment, the effect of the previous unloading will probably not be felt. If this step is small though, we might well find ourselves in this yield point, where the same mechanism causing this effect might cause the increase of the modulus. It depends on the exact nature of the yield point however, if this explanation is correct. Figure 27 represents the figure as given by Haasen and Kelly [107]), we see that the maximum after subsequent reloading is reached after some additional deformation. Makin [108]) makes it appear in his figure, as if the maximum of $\Delta\sigma$ is obtained immediately upon reloading, thus showing an upper yield point as is observed in α-iron. This means that the effect is produced by the unloading alone, while for the yield point as shown in figure 27 the upper yield point seems to be produced by the small subsequent deformation. It is clear that if Makin's representation is the right one, our effect has to be caused by something else, as the specimen is always unloaded before measuring. If however figure 27 represents the true situation, it could very well be that our small modulus increase could be due to the same mechanism causing the yield point, whatever this may be.

c. RECOVERY.

As was discussed in chapter II, pinning of dislocations by point defects seems to explain the recovery phenomena in the most successful manner. In the preceding chapter, sections c through f show the results of the recovery behaviour under different circumstances. The variety of information makes it difficult to give a surveyable discussion of these results. This is the reason that we will subdivide this section in different parts. We will also first discuss the recovery measurements on Cu, as they are the most extensive, and discuss the results of Ag and Au afterwards. In part 1) we will discuss different possibilities to explain the two recovery stages in the recovery curve of Cu. The results of the other experiments on Cu will be used for the argumentation to make a choice between the alternative mechanisms. Starting from this choice we will proceed by examining the results more closely in part 2). Part 3) will be devoted to the results on Ag and Au, mainly by pointing out the agreement and difference between these results and those on Cu.

1) Looking at the recovery curve of Cu, we could imagine the two recovery stages to be caused by one of the following four mechanisms:

1° The two stages are caused by two different types of dislocations. In stage II the type that has the greatest interaction energy with the point defects will be pinned, in stage III the other type is pinned.

2° The two stages, or one of them, are caused by impurities which move to the dislocations and act as pinning points.

3° One type of point defect causes the recovery. The point defects in the immediate neighbourhood of the dislocations are attracted by them, thus moving by a drift current to the dislocations, causing stage II. Stage III is then caused by the same type of point defect which is at a greater distance from the dislocations, thus reaching the dislocations mainly by random diffusion.

4° The two stages are caused by at least two different types of point defects.

We will successively examine each one of the possibilities in view of the different experimental facts.

Mechanism 1° does not seem to agree with the results reported in chapter IV section e. The applied stress, which was kept on during the first annealing run, was removed after cooling down again. Upon this unloading practically the same value for $\Delta E/E$ was observed, as was normally found immediately after deformation. This suggests that the

dislocations are mobile again, being torn loose from the pinning points. The fact that stage II is absent upon re-annealing, would in terms of mechanism 1° mean that the type of dislocation belonging to this stage has disappeared or converted itself into the other type. It seems very unlikely that unloading the specimen could cause either one of these effects.
Mechanism 2° also meets with serious difficulties.
As the concentration of impurities will not change with the deformation, the size of the stages should decrease quite strongly with increasing deformation, which is not observed. Also the absence of stage II after the unloading experiment of section e), and the decrease of stage II after the repeated deformation shown in section d) of chapter IV can hardly be explained by impurities. There is also the influence of quenching the specimen before deformation, which causes stage II to decrease by a small amount and stage III to increase as shown in chapter IV section f, which does not add much in favour of mechanism 2° either. Finally the results of Druyvesteyn c.s.[12]) on the recovery of $\Delta G/G$ do not show much dependence on the purity.
Mechanism 3° can not make plausible the fact that the two recovery stages are separated by a horizontal part.
One would expect the recovery to start with point defects which were originally lying very close to the dislocations, while as the annealing proceeds point defects which come from a greater distance arrive at the dislocations. There does not seem to be any reason why this recovery would not be a continuous process. Also the effect of quenching seems to reject this mechanism.
Mechanism 4°: at least two different types of point defects, remains as the most likely concept. This does not mean that all the results can easily be fitted into this picture, and that effects as mentioned in the other three mechanisms can not possibly occur, but it will be shown that to explain the results as a whole, mechanism 4° is by far the most successful. Also the correlation with other kinds of experiments as the electrical resistivity and stored energy measurements for instance, however different from our experiments they may be, support this choice. We will from now on proceed from this assumption and examine the results more in detail.

2) The recovery of the ΔE effect is not directly proportional to the number of point defects disappearing, as is approximately the case for the recovery of the electrical resistivity and the release of stored energy.

Considering the relation for $\Delta E/E$ given in chapter II, we can write this relation as a constant A times ΛL^2 if $\Delta E/E$ is not too large. In this relation, $\Delta E/E = A.\Lambda L^2$ we see that only L changes during the recovery if the dislocation density remains constant, which most likely will be the case in the temperature range considered. The change of L after P pinning points have arrived on the dislocation network per unit volume is expressed by $\Lambda/L = P + \Lambda/L_o$ where Λ/L_o is the original number of pinning points.

Hence is follows that $L = \dfrac{L_o}{I + PL_o/\Lambda}$ thus

$$\Delta E/E = \frac{A\Lambda . L_o^2}{(I + PL_o/\Lambda)^2}$$

after the arrival of P pinning points on the dislocation network. This relation is shown in figure 28 where $\Delta E/E$

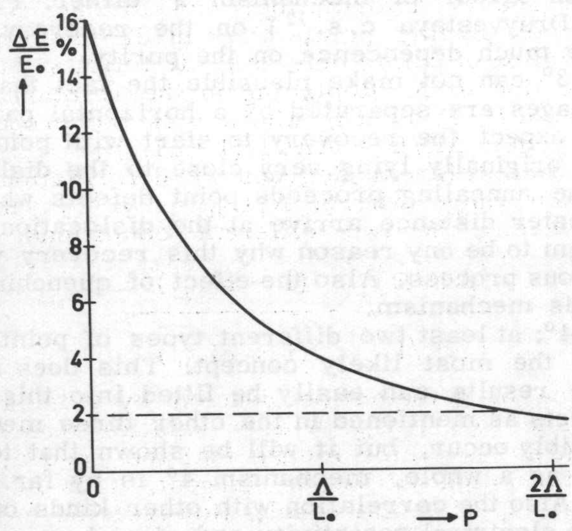

Figure 28. The change of $\Delta E/E_o$ as a function of the number of pinning points P.

is plotted against P. For the value of $\Delta E/E$ immediately after deformation 16% is taken, correcting for the mobile dislocations present in the annealed specimen, as was discussed in section b. The zero line of our experiments is shown by the dotted line. Comparing this figure with figure 13, showing the recovery of Cu, two conclusions can be drawn: 1°. The number of pinning points P is small compared to

the estimated number of points defects generated during deformation. The value for P is the order of magnitude of Λ/L_o, taking for Λ the reasonable value of $10^9 cm^{-2}$ and for L_o a value of about 10^{-4} cm, we find for P a value of $10^{13} cm^{-3}$, which is equal to a concentration of approximately 10^{-9}. According to estimates based on electrical resistivity measurements, the point defect concentration in the deformation range between 1% and 10% will be of the order of magnitude of 10^{-6}. Thus the number of pinning points is only about 0,1% of the number of point defects. It cannot be concluded from this that only 0,1% of the point defects reach the dislocations, as they could disappear shortly after their arrival, neither do we know the number of point defects needed to form one pinning point. It also appears that the total recovery, being the total of stage II and III recovery, decreases with increasing deformation. If we assume that the dislocation density Λ and the point defect concentration c increases in direct proportion to the increasing degree of deformation, and further assume that the number of pinning points P is in direct proportion to the concentration of points defects c, it follows that P is in direct proportion to Λ. Replacing in the relation for $\Delta E/E$ as a function of P, P by $\alpha\Lambda$, where α is the proportionality constant, we get: $\Delta E/E = A\Lambda L_0^2/(1+\alpha L_o)^2$.
Comparing two different degrees of deformation, both in the saturation range where: $\Lambda_1 L_{01}^2 = \Lambda_2 L_{02}^2$, the indices 1 and 2 denoting the two different conditions, we find:

$$\frac{(\Delta E/E)_1}{(\Delta E/E)_2} = \frac{(1+\alpha L_{02})^2}{(1+\alpha L_{01})^2}$$

If the degree of deformation denoted by 2 is larger than the one denoted by 1, L_{02} will be smaller than L_{01}, thus $(\Delta E/E)_1 < (\Delta E/E)_2$ which means that the recovery for the smallest deformation will be the largest. This is in agreement with the experimental result. However the assumptions made, from which it followed that P is directly proportional to Λ could very well be wrong, thus not too much attention should be paid to this reasoning. If however the relation between P and Λ is written as $P = \alpha\Lambda^n$, $n \geqslant 3/2$ would mean that $(\Delta E/E)_1 \geqslant (\Delta E/E)_2$, thus giving the opposite effect, being inconsistent with the experimental evidence. Thus if the relation for $\Delta E/E$ is correct, it follows that if $P = \alpha\Lambda^n$, n should be smaller than 3/2.
2°. The other conclusion from comparing the recovery curve of Cu with figure 28, is that stage II is related to a smal-

ler number of pinning points than stage III, being 1/5 of the total number of pinning points for the deformation of 1,7% shown in the figure and 1/3 for the deformation of 7,4%. Also the size of stage II depends differently on the deformation, remaining practically constant while stage III decreases as a function of the deformation. This means, as is also shown by the example given above, that the fraction of the stage II pinning points of the total number increases with increasing deformation. It could be that the stage II point defect, to which we will refer as the type II defect from now on, is produced at a faster rate as a function of the deformation than the type III defect. However as we are not sure that the size of the stages is in direct proportion with the number of related defects created, this conclusion is by no means indisputable. We will see later on, that there are also other factors determining the size of the recovery stages.
From the normal recovery behaviour of Cu, the only conclusion that can be drawn is the fact that only a small fraction of the point defects act as pinning points, provided that there are not a larger number of point defects needed to form one pinning point, and some rather dubious conclusions based on the size of the recovery stages. It is therefore important to gain information from recovery experiments performed under different circumstances. Figures 16 and 17, 19 and 20 show us the results obtained from a repeated deformation and unloading after a first annealing run with applied stress respectively. To learn the details of these experiments, the reader is referred to the respective sections in chapter IV, only the results will be discussed here. From figures 16 and 17 we see that stage II is smaller after a repeated deformation, as could be expected if a great number of the type II defects have disappeared. The behaviour of stage III is different, from figures 17 and 20 we see that although the number of type III defects has decreased, there still seems an appreciable number left after the first recovery. One of the most important results however can be seen from figures 16 and 19. If the first annealing run was stopped in between the two recovery stages, as shown in figure 13, so the type II defects have practically all disappeared, recovery is observed in the range which separated both stages and where it was assumed that no recovery occured. The absence, or the decrease of stage II, while the type III defects are still present for the greater part, reveals this recovery which gradually passes into stage III, and which we will consider to be part of this stage. The total amount of re-

covery is practically unchanged in both cases. This leads to the conclusion that the horizontal part of the recovery curve shown in figure 13 does not necessarily mean that no recovery occurs in this range. It could very well be that two opposite effects balance each other in this range. These two effects could be the unpinning due to the disappearance of the type II pinning points, and the pinning by the type III pinning points. This unpinning process could be visualized by either one of the following processes. If the type II defects move along the dislocations after their arrival, pinning occurs as long as the number arriving at the dislocations is larger than the number disappearing by diffusion along the dislocations, the so called pipe diffusion. The other possibility is that the migration energy of the type II defects along a dislocation is higher than the migration energy in the lattice, thus the pinning points become mobile along the dislocations at a higher temperature. The fact that in figures 17 and 20 the recovery in this range is not observed, is probably due to the fact that only a small number of stage III defects is present. There are other experimental facts which support this view. Electrical resistivity measurements for instance show that the stages II and III are not separated by a large temperature range as figure 13 suggests. Birnbaum and Tuler[109]) explain their results on the hardening, observed after annealing in stage II subsequent to deformation, where they observed an overaging effect: the hardening decreasing again after the initial increase at a certain temperature as a function of time, by pinning and subsequent disappearance of the pinning points along the dislocation.

Hasiguti and Okuda[110]) observe a large internal friction peak of relaxation type, in Cu at 153°K at a frequency of 0,115 cps, and in Au at 193°K (4 cps), after deformation at 78°K and measuring the internal friction with increasing temperature. This peak grows and decays at about the peak temperature, and has disappeared at about 250°K. The behaviour of the modulus was also studied, and was observed to increase to go through a maximum and decrease afterwards, while the specimen was held at 192°K, as a function of time. Leaving the explanation of the internal friction peak aside, it seems safe to conclude that the increase of the modulus and the growth of the peak are both caused by the arrival of type II point defects at the dislocation. Its subsequent decrease would than very likely be due to the disappearance of these defects.

Although more information on the behaviour of the type II and III defects is obtained, there is still no evidence to identify these types with a certain type of point defect.

Information of this kind is obtained from the quenching experiments. The influence on the recovery by quenching the specimen before deformation is shown in figure 24. From this result it can be concluded that the stage which we have called stage III has increased in size and thus probably vacancies or complexes of vacancies play a role in this stage. Experimental evidence that stage III after deformation has something to do with vacancies is supplied by the measurements of Korevaar [96]), who observed an ordering effect on gold-copper alloys in this stage. Assuming that vacancies and interstitials are generated by deformation, this result would mean that stage II could be due to interstitials or the dissociation of complexes of interstitials. The decrease of the size of stage II in the quenched specimen supports this view. Figure 24 also shows us that stage III starts at a lower temperature than in figure 13, this again seems to support the concept of two processes balancing each other in the horizontal part of the recovery curve. The discussion on the identification of the recovery stages will be extended in the next part on the results of Ag and Au.

3) If we now look at the results of Ag and Au, we see that some results are in conformity with the results on Cu, but also some marked differences are observed. Especially Ag shows a different behaviour. Figure 14 shows us that the normal recovery of Ag consists of only one stage: stage II. This can mean that either the type III defects are not formed, that they produce no pinning, their pinning balances the possible unpinning of type II defects, or that for some reason the type III defects do not arrive at the dislocations.

As the normal recovery can not give any more information, justifying a choice between these possibilities, we have to look at the other results on Ag. Figures 18 and 21 show the behaviour of respectively the repeated deformation and the applied elastic stress experiment. The behaviour is different from Cu, the decrease of stage II after the repeated deformation is much smaller than in the corresponding experiment on Cu. The elastic stress experiment reveals that stage II is not absent. It therefore seems that there still were some type II defects present after recovery through stage II. They also seem to provide strong pinning points as the ΔE effect upon unloading is smaller than is observed immediately after deformation. There is only a slight evidence of recovery after stage II in both cases, indicating that if type III defects are present only a very small number arrive at the dislocations, or that they produce no pinning. Of course we cannot reject the possibility that these defects

are so mobile along the dislocations that they disappear immediately after their arrival. It seems unlikely that for Ag the horizontal part in the recovery curve, after stage II, is caused by a pinning and unpinning effect balancing each other.
The fact that the pinning points seem to be strong, seems to indicate that the type II defects do not all disappear by pipe diffusion which would cause unpinning, but possibly tend to form clusters which are not mobile and provide stronger pinning points. If the type II defects have not disappeared after stage II, but are still present in the lattice, the fact that no additional pinning occurs would than mean a further growing of these clusters.
The result on quenched Ag, shown in figure 23, is rather surprising. A second stage, which we will call stage III, is clearly visible now. We again can assume that this stage is caused by vacancies or vacancy complexes. Assuming that the type III defects after deformation are the same as those after quenching, the possibility that these effects can not produce pinning appears to be rejected by the result in figure 23. There still remain the two explanations put at the beginning: the type III defects are not formed by deformation, or for some reason the type III defects do not arrive at the dislocations. The first of these two does not seem very likely considering the electrical resistivity experiments, where a stage III is observed after deformation. Thus there remains the second one: for some reason the defects generated by the deformation do not arrive at the dislocations, for their immediate disappearance along the dislocations seems very unlikely as this is not observed in the quenched specimen.
What could be the difference between the type III defects produced by deformation and quenching, assuming that these are produced by both deformation and quenching? After quenching we can assume a more or less homogeneous distribution of the vacancies, while after deformation this could very well not be the case as the point defects could for example be generated in rows. It appears from the experiments of Doyama and Koehler[93]), that the binding energy between vacancies in Ag is large, being about 0, 38 eV, larger than in Au for example, where the binding energy is about 0, 1 eV according to de Jong and Koehler[95]). Considering this large binding energy, it could be that the vacancies generated in rows do not dissociate in mobile single or double vacancies but tend to form a cluster instead. There are two observations on the electrical resistivity which might support this idea. Stage III is observed to be

smaller in Ag than in Cu and Au [111,112]), it is to be expected that clustering of point defects gives a smaller recovery than their disappearance. The measurements on the magneto resistivity [113]) indicate a difference in annealing behaviour between Ag on the one side and Cu and Au on the other hand. It appears that the anisotropic scattering increases in stage II and III in Ag, while this remains constant in Cu and Au. This could be due to the clustering of the point defects in Ag. Although the explanation given could well be correct, or partly correct, the experiments do not give enough information to prove this reasoning, it thus should be taken to be a more or less worked out hypothesis.

The measurements on Au reveal a smaller difference in behaviour compared with Cu than those on Ag. From figure 15 we see that in Au stage II is deformation dependent existing only for deformations larger than about 4%. This same experimental fact is observed on the electrical resistivity by Dawson [112]), who also observes stage II to be absent in Au after a very small deformation. This indicates that this is probably due to the absence of the type II defects after a small deformation, rather than to the fact that they are present but do not arrive at the dislocations for some reason. The quenching experiment on Au is probably the safest to draw conclusions from, because no impurities as for instance oxygen seem to be soluble in this case. As we can see from figure 25, quenching has again an influence on stage III, the stage becoming slightly larger and starting at a lower temperature. From the lowering of the temperature at which the recovery starts, we can try to calculate the ratio between the point defects produced by quenching and those produced by deformation. Assuming that in both cases the same number of jumps of the point is needed to start the recovery, we find:

$$(C_{def} + C_{q})/C_{def} = e^{-\frac{E_m}{k}(1/T_1 - 1/T_2)}$$

where c_{def} is the concentration produced by deformation, c_q is the concentration produced by quenching, E_m the activation energy of migration of the point defect, T_1 and T_2 are the temperatures at which the recovery starts, after deformation only, and after deformation of the quenched specimen respectively. Taking for T_1 and T_2 the values obtained from figure 25, and for E_m the migration energy of a divacancy, which is according to de Jong c.s. [95]) 0,66 eV, we find $C_q/C_{def} \approx 20$. If we take for E_m the value of a single vacancy, 0,83 eV, we find $C_q/C_{def} \approx 40$. These values seem to be in agreement with the estimates given in chapter IV. However this way of comparing the concentrations is not correct if only point defects which are in the immediate neighbourhood of the dislocations provide the pinning points. It should also be pointed out that if the point defects which produce the pinning are divacancies, their number is not directly proportional to the concentration of vacancies, as the fraction of divacancies of the total number of vacancies increases if the total number increases. In this case the ratio will thus be smaller than the calculated value of 20.

The only positive fact for the identification in all three metals seems to be that stage III is at least partly caused by vacancies or vacancy complexes. The only experiments that indicate that stage III after deformation is also due to these point defects are the experiments of Korevaar[96]) who observed an ordering effect in an Au-Cu alloy in stage III after deformation. As there are good reasons to reserve stage IV for single vacancies, and as the quenching experiments of for example Cuddy and Machlin[94]) on Ag and de Jong and Koehler[95]) on Au show recovery in the same temperature range as our stage III, which they attribute on sound grounds to divacancies, we think that our stage III is caused by the pinning of dislocations by divacancies.

It is also felt that, as stage II also seems to be due to pinning of the dislocations by point defects, this stage is to be attributed to the pinning by interstitials. Van den Beukel's idea that the interstitials are dissociated from rows or pairs in this stage seems reasonable. His idea to attribute stage III for the greater part to the disappearance of the interstitials which were pinning the dislocations, seems less realistic. From our experiments it seems that such a process occurs in Cu, but the number disappearing this way seems rather small, as the number of pinning points is small. In Ag this disappearance was not observed to happen, while stage III is not absent in the other experiments like the electrical resistivity and the stored energy measurements. It therefore seems likely that this disappearance might explain a small part of this stage, but it should for the greater part be attributed to another effect of which the diffusion of divacancies seems the most likely one.

SAMENVATTING

De afname van de elasticiteitsmodulus E werd gemeten na plastische vervorming aan Cu, Ag en Au. E werd bepaald door van een aan één uiteinde ingeklemd plat staafje de eigenfrequenties van de buigingstrillingen te bepalen. Daar de afname van E, aangeduid door ΔE, door optredend herstel afneemt, werden de meting en de vervorming verricht bij lage temperatuur (78°K), waar geen herstel optreedt.
De waarde van de afname, uitgedrukt door $\Delta E/E$, als functie van de vervormingsgraad werd aan Cu en Ag gemeten. Het blijkt dat $\Delta E/E$ aanvankelijk met de vervorming toeneemt, waarna bij een rek van enkele procenten een verzadigingswaarde van ongeveer 14% bereikt wordt. Dit verschijnsel blijkt op bevredigende wijze met behulp van het model van een driedimensionaal netwerk van dislocaties, die onder invloed van een spanning uitbuigen, verklaard te kunnen worden. Op ~~Over~~ de wijze waarop de dislocatie uitbuigt wordt nader ingegaan. Er werden ook reversibele veranderingen van $\Delta E/E$ als functie van de temperatuur waargenomen.
Het herstel dat bij temperaturen boven 78°K optreedt vertoont bij Cu, als functie van de hersteltemperatuur, twee herstelstappen. Deze stappen kunnen op verschillende manieren verklaard worden, indien men ervan uitgaat dat het herstel plaats vindt doordat puntfouten die naar de dislocaties bewegen deze op bepaalde plaatsen vastzetten: de z.g. "pinning". Deze pinning berust op de interactiekracht die tussen de dislocatie en de puntfout heerst; op het pinningsmechanisme wordt niet verder ingegaan.
Om tussen de diverse herstelmogelijkheden een keuze te kunnen doen, werden de omstandigheden waaronder het herstel plaatsvindt gewijzigd. Hiertoe werd bijvoorbeeld nadat een gedeelte van het herstel had plaatsgevonden opnieuw bij 78°K vervormd.
Ook werd in enkele gevallen een gedeelte van het herstel doorlopen, waarbij het staafje onder een elastische spanning gehouden werd, die na wederom afkoelen tot 78°K weggenomen werd. De resultaten uit deze proeven wijzen erop dat het herstel in de beide herstelstappen waarschijnlijk door verschillende typen puntfouten veroorzaakt wordt. Ter identificatie van de herstelstappen met een bepaald type puntfout werden metingen aan afgeschrikte staafjes verricht, de invloed van een overmaat aan vacatures of vacaturecomplexen werd op de herstelstappen na plastische vervorming nagegaan. Hierbij blijkt de tweede herstelstap, aangeduid

als stap III, groter te zijn indien aan afgeschrikt metaal wordt gemeten. Hieruit kan geconcludeerd worden dat deze stap iets met vacatures of vacaturecomplexen te maken moet hebben.
Uit de proeven blijkt verder dat indien de omstandigheden gewijzigd worden, herstel waargenomen wordt in het horizontale gebied dat bij het normale herstel de beide stappen scheidt. Dit zou erop kunnen duiden dat dit horizontale gedeelte niet betekent dat er geen herstel plaatsvindt, maar alleen dat gemiddeld het aantal "pinning points" niet verandert. Dit zou bijvoorbeeld op kunnen treden indien er tegelijkertijd "pinning" en "ont-pinning" verschijnselen optreden.
Behalve diverse overeenkomsten, tonen de resultaten van Ag en Au ook opvallende verschillen met die van Cu. Deze verschillen worden besproken, mogelijke verklaringen worden genoemd.

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STELLINGEN

1. Het maximum dat door Cook, Richards en Bidmead in de afname van de elasticiteitsmodulus van koper als functie van de plastische rek bij een rek van ongeveer 3% wordt waargenomen, wordt waarschijnlijk door herstel veroorzaakt.

   M. Cook, T. L. Richards, G. F. Bidmead,
   J. Inst. Met. 83, 41 1954 - 1955.

2. De afleiding die Shibuya van de elasticiteitsmodulus van geordende legeringen geeft, alsmede de hieruit volgende verhoudingen van de elasticiteitsmoduli in de verschillende kristallografische richtingen, is onjuist.

   Y. Shibuya, Sci. Rep. RITU, A - 1, 23, 1949.

3. Bij het beschouwen van de door Ramsteiner, Schüle en Seeger gepubliceerde isochrone herstelkromme van zilver, ontkomt men niet aan de indruk dat herstelstap IV kunstmatig door het grote aantal meetpunten ter plaatse geïntroduceerd is.

   F. Ramsteiner, W. Schüle, A. Seeger,
   Phys. Stat. Solid. 2, 1005, 1962.

4. De juistheid van de veronderstelling van Takamura, dat bij afschrikken van dikke (1,5 - 3 mm) draden een groot deel van de vacatures door de deformatie ten gevolge van het afschrikken gevormd wordt, kan getoetst worden door in plaats van ronde draden platte strookjes te nemen.

   J. Takamura, Acta Met. 9, 547, 1961.

5. De gedachte van Kuhlmann-Wilsdorf, dat indien dislocaties goede verdwijnplaatsen voor vacatures zijn, tijdens afschrikken vrijwel alle vacatures verdwijnen, is als argument voor de verdeling van de dislocaties in oude - en nieuwe - niet houdbaar.

   D. Kuhlmann - Wilsdorf,
   A.S.M. Seminar Philadelphia 1960.

6. De afleiding die Hordon, Lement en Averbach geven voor het verband tussen de verandering van de elasticiteitsmodulus en van de uitzettingscoëfficiënt na plastische vervorming, is onjuist.

   M. J. Hordon, B. S. Lement en B. L. Averbach,
   Acta Met. 6, 446, 1958.

7. De opmerking van Paré en Thompson, dat zeer beweeglijke puntfouten niet stabiel genoeg zijn om dislocaties vast te zetten is niet algemeen geldig; doch slechts in zoverre het de beweeglijkheid langs de dislocatie betreft.

V.K.Paré en D.O.Thompson,
Acta Met. 10, 382, 1962.

8. Het is zeer wel mogelijk dat de kleine afname van de elasticiteitsmodulus door Folweiler en Brotzen na het afschrikken van aluminium gevonden, door een klein aantal door het afschrikken gevormde dislocaties veroorzaakt wordt en niet door de in het rooster aanwezige vacatures.

R.C.Folweiler en F.R.Brotzen,
Acta Met. 7, 716, 1959.

9. Het bezwaar dat van den Beukel tegen eventuele herrangschikking van dislocaties in stap II aanvoert, namelijk dat de hoeveelheid warmte die in deze stap vrijkomt van dezelfde orde van grootte is als die tijdens rekristallisatie vrijkomt, is niet juist indien deze herrangschikking door het klimmen van dislocaties veroorzaakt wordt. De puntfouten die bij dit klimmen verdwijnen bepalen dan voor een groot deel deze vrijkomende warmte.

A. van den Beukel,
dissertatie Delft 1962, blz. 72.

10. De eventuele vrijlating van de vier oorlogsmisdadigers in Breda, verschaft de voorstanders van de doodstraf een krachtig argument.

11. Indien technisch wetenschappelijk onderwijs een wezenlijk bestanddeel van de opleiding aan een instelling voor militair hoger onderwijs gaat vormen, ligt hier gezien de historische ontwikkeling de kiem voor een nieuwe technische hogeschool.