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MECHANICAL RELAXATION STUDIES OF POINT DEFECTS IN METALS

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Abstract.—This review focusses on three main topics. First, self-interstitial atoms in pure Al (fcc) and pure Mo (bcc). The emphasis lies on a comparison between mechanical relaxation (MR) measurements and diffuse X-ray scattering (DXS) experiments. Second, self-interstitial atom solute-atom complexes in dilute alloys and third, Zener relaxation in Ag-Zn in thermal equilibrium and during irradiation will be discussed.

1. Self-interstitial atoms in Al and Mo: A comparison between MR and DXS.—

The MR of point defects is described by the elastic dipole tensor, \( \bar{\varepsilon} \), which is a measure of the long range displacement field set up in the crystal by the point defects. The dipole tensor is defined as the stresses, \( \sigma_{\alpha\beta} \), induced by the defects per unit volume concentration, \( \rho \), of defects and has the unit of an energy. It has six independent components. There is an equivalent definition which is the strain, \( \varepsilon_{\alpha\beta} \), set up in the crystal by the defects per unit atomic fraction, \( c \), and this is called the \( \varepsilon \)-tensor.

\[
\bar{\varepsilon} = \frac{\partial \sigma_{\alpha\beta}}{\partial \rho} = \begin{pmatrix}
P_1 & P_6 & P_5 \\
P_6 & P_2 & P_4 \\
P_5 & P_4 & P_3
\end{pmatrix}; \quad \lambda = \frac{\partial \varepsilon_{\alpha\beta}}{\partial c}
\]

In cubic crystals there exist three independent elastic deformations which can be chosen as a hydrostatic compression and two pure shear deformations. The paraelastic response of a defect to an externally applied elastic field is described by the modulus changes /1, 2/

\[
\delta \varepsilon = 0
\]

\[
\delta \varepsilon' = \frac{\rho}{2kT} \cdot \varepsilon^{(2)} = \frac{\rho}{2kT} \cdot \frac{1}{6} \cdot \left( (P_1 - P_2)^2 + (P_2 - P_3)^2 + (P_1 - P_3)^2 \right)
\]

\[
\delta \varepsilon = \frac{\rho}{2kT} \cdot \varepsilon^{(3)} = \frac{\rho}{2kT} \cdot \frac{2}{3} \cdot \left( P_4^2 + P_5^2 + P_6^2 \right)
\]

The quantities \( \varepsilon^{(2)} \) and \( \varepsilon^{(3)} \) as defined above determine also the intensity of the DXS, i.e. Huang-scattering \( I_h(k) \): This is due to the fact that the long range strain field is imaged close to the Bragg peaks in the scattering pattern.

\[
I_h(k) = \ldots (j^{(1)} \cdot \varepsilon^{(1)} + j^{(2)} \cdot \varepsilon^{(2)} + j^{(3)} \cdot \varepsilon^{(3)})
\]

where \( j^{(i)} = j^{(i)}(k) \) and \( \varepsilon^{(i)} = \varepsilon^{(i)} \)

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Since Huang-scattering measures the same defect properties as the MR it is an ideal method for comparison and cross checks. This kind of double measurements has worked out well in the case of H and O in Nb and also in the case of electron irradiated Al.

The properties of the single self-interstitial atom in Al determined from MR measurements /3/ and Huang scattering /4/ are

$$E = \begin{pmatrix} A & 0 & 0 \\ 0 & B & 0 \\ 0 & 0 & B \end{pmatrix}; \quad \frac{1}{3} \text{tr} \, P = 15.6 \text{ eV};$$

$$|A-B| = \begin{pmatrix} 1 \pm 0.3 \text{ eV from MR} \\ 3 \pm 3 \text{ eV from DXS} \end{pmatrix}$$

In addition the MR measurements have shown that the migration of these self-interstitial atoms occurs simultaneously with their reorientation with an activation energy of 0.115 eV. All these observations are in agreement with the properties expected for <100>-dumbbells. In fig. 2 the basic jump process of this <100>-dumbbell is shown. One recognizes that simultaneously with the migration jump a rotation by 90° of the dumbbell occurs.

The self-interstitial atoms in Mo (bcc) are expected to possess the structure of a <110>-dumbbell, which is shown in fig. 3. This figure shows also three characteristic vibrations which the dumbbell can undergo and which possess the largest amplitudes for a given thermal excitation. In calculations using the a-Fe potential of Johnson /7/ the librational vibration in the |110|-plane (librational I) possesses the largest amplitude of the three modes for a given thermal excitation.
The experimental results on Mo can be summarized as follows:

1.) The dipole tensor is according to the DXS /h/:

\[
P = \begin{pmatrix}
20 & 11 & 0 \\
11 & 20 & 0 \\
0 & 0 & 38
\end{pmatrix}
\text{[eV]}
\]

2.) Measurements of the diffuse X-ray scattering /4/, recovery of electrical resistivity /5/ and Mössbauer measurements on Mo containing Fe\textsuperscript{57} /6/ have shown that the migration of the Mo self-interstitial atoms occurs in a temperature regime between 30K and 40K.

3.) If the migration occurs with a simultaneous reorientation or if the dumbbells undergo an in-place reorientation before their migration a relaxation effect is expected for the MR. Since the difference, for instance, between the elements of the diagonal is about 18 eV which is 18 times larger than the corresponding value determined for the \(<100>-dumbbells in Al, a MR-process is expected in Mo which is more than 2 orders of magnitude larger than the corresponding one in Al. Elastic after effect measurements have been performed in the temperature regime from 4K to about 100K on electron irradiated Mo by H. Jacques /8/ and in a similar manner by S. Okuda and J. Mizubayashi /22/ after neutron irradiation in search for this large relaxation effect. The result is shown in fig. 4. The respective upper curves in fig. 4a and 4b are those curves which are expected if dipole tensor components are assumed as determined from DXS. In this scale the actually measured curves are essentially zero for both cases reported.

The expected large relaxation process caused by the \(<110>-dumbbells does not exist in Mo. This means, the \(<110>-dumbbells neither undergo an in-place rotation nor do they reorient during their migration. It could be speculated that the dumbbells migrate one-dimensionally along a \(<110> direction via a pure translational mode. The saddle point for this diffusional jump would be the octahedral center. An interstitial in the octahedral center has however a formation energy which is about 1.1 eV higher than that of a \(<110>-dumbbell /7/. This value would be approximately the migration energy for the translational jump and is much too large in order to account for a migration which takes place at about 30K to 40K. H. Jacques et al /8/ have recently proposed a model which explains the low activation energy as well as the fact that the migration of the \(<110>-dumbbell occurs without reorientation (see fig. 5). The jump
occurs via the librational mode I which is expected to possess the largest amplitude of all resonant vibrations. The estimate of the migration energy is 0.3 eV from the computer calculations /7/. For further details of this jump mechanism see the contribution of H. Jacques et al. /8/ to this conference. It should be pointed out that this jump causes a two-dimensional diffusion of the self-interstitial atoms in a [110]-plane and because of this explains also the peculiar behaviour of resistivity recovery and of the Mössbauer effect in the annealing range of stage I /5,6/.

2. Self-Interstitial Atoms (SIA)-Solute-Atom (SA) - Complexes.-
SIA-SA-complexes which are formed by trapping of migrating SIA at immobile SA play a predominant role in the behaviour of metals during irradiation. Self-interstitial atoms may be preserved in the form of such complexes and act as additional reaction partners at higher temperatures for migrating vacancies. This is the basic mechanism which leads to the reduction of void swelling. The diffusion of SIA-SA-complexes as a unit leads to a preferential mass-transport which is observed in radiation induced segregation and possibly in fast diffusion phenomena. The main points of interest with respect to these phenomena are: the binding energies, $E^B$, the migration energies, $E^M$, and the configurations.

The physical origin of the binding of self-interstitial atoms at solute atoms is not yet understood in full detail. A probably very important contribution to this binding can be seen by the aid of the following model (fig. 6). The rather high formation energy of the self-interstitial atom in the fcc lattice is a consequence of the high amount of strain energy associated with the compressed atomic arrangement around the self-interstitial atom. Binding of self-interstitial atoms at solute atoms would occur, if the solute atoms could be incorporated in such a manner that part of this strain energy can be regained again. This is possible for solute atoms with a smaller ionic core than the host atoms, if one of the dumbbell atoms is replaced by the smaller solute atom as shown in fig. 6. In this way a mixed dumbbell is created. On the other hand solute atoms with a larger ionic core can also be incorporated in the neighbourhood of the dumbbell with energy gained as shown in fig. 7. This is due to the fact that the ring of atoms No. 1-4 is opened up by the displacement field surrounding the dumbbell as indicated by the arrows. Since the nearest neighbour No. 5 is located close to

Fig. 5: Jump via librational I mode

Fig. 6: For explanation see text
this ring an oversized solute atom can be accommodated here.

As a measure for the size of the solute atoms it has been proposed to take the relaxation volume of solute atoms in dilute solid solution which can be determined from lattice parameter measurements /7,9/

\[ r_0 = a \cdot \left(1 + \frac{2c_{SA}}{3a} \right) \]

\(a\) is the lattice parameter, and \(c_{SA}\) the atomic fraction of solute atoms. \(r_0\) is usually called the misfit.

If \(r_0\) is negative solute atoms are considered to be undersized with respect to the host atoms and oversized if \(r_0\) is positive.

Detailed computer simulation studies have been shown that undersized solute atoms may be incorporated in different ways as shown in fig. 8. Fig. 8a shows a \(<100>-dumbbell\), where a solute atom replaces a regular host atom in the dumbbell. There are six equivalent positions for the solute atom within the octahedral cell. In each solute atom position the dumbbell has a different orientation. Jumps of the solute atom from one position to another lead to a reorientation of the mixed dumbbell. Since the separation between these positions is rather short, the jumps can be expected to occur with a low activation energy. Therefore, one expects a relaxation effect from these mixed dumbbells which occurs at low temperatures.

Depending on the misfit \(r_0\) or depending on slightest distortions in the interatomic interaction potential, other cages may be possible. An example is shown in fig. 8b. In this case the dumbbell is tilted slightly off the \(<100>-direction and there are now 24 equivalent positions for the solute atom. Two different jump modes may occur with this cage. First, the solute atom may jump with a frequency \(v^1\) within each of the squares. These jumps, however, do not lead to transitions from one square to the other. Such jumps occur via a second mode at frequency \(v^2\). Therefore this cage provides a model for a complex, which gives rise to two internal friction peaks which are separated on a frequency or temperature scale. This is called frozen-free-split phenomena in the literature /1/. The computer calculations show that with increasing misfit \(r_0\) the squares become larger and larger and at the same time the triangles connecting the squares become smaller until finally
the triangles degenerate into one point. Then the configuration shown in fig. 8c is arrived at. This is a cube like cage where there is no mixed dumbbell anymore, but the solute atom possesses three equivalent neighbouring host atoms.

Independent of the particular cage structure, however, all of these cages should give rise to internal friction peaks at low temperatures, i.e. with small activation energies, due to localized diffusion of the solute atom in a narrow cage.

Self-interstitial atoms bound to an oversized impurity atom should also give rise to internal friction peaks due to the fact that there are twelve equivalent positions for the dumbbell bound at the impurity atom as shown in fig. 7 and fig. 9:

Since in this configuration, a regular host atom dumbbell is bound to the solute atom, these dumbbells perform a similar jump as the free dumbbell in order to come from one of the positions to the others only that the motion is restricted to the twelve positions. Therefore, one can expect a mechanical relaxation process at a temperature where also the migration of the free self-interstitial atom dumbbell occurs.

Mechanical relaxation measurements, with the aim to study the vibrational behaviour of SIA-SA-complexes, have been performed in a number of alloys containing undersized as well as oversized solute atoms in solution. These results are shown in fig. 10.

All of these curves were observed after the samples have been irradiated with electrons at low temperatures and the migration of the free self-interstitial atoms had occurred so that they had the possibility to react with the solute atoms present in the material. The original concentration of solute atoms in these alloys was of the order of 200 atppm and the self-interstitial atom concentrations produced by the electron irradiation was of the order of 40 atppm or less. The most important features for the present concern are the following:
a) In each alloy several different relaxation peaks have been found which grow and decay upon annealing at different temperatures as shown in Fig. 11. If defects causing different internal friction peaks grow or anneal at different temperatures, it means by necessity that they have different configurations. This means that in all of the alloys several different configurations of SIA-SA-complexes are present simultaneously in the sample despite the low self-interstitial atom and solute-atom concentrations. Several reasons may account for this observation. First, a complex containing one self-interstitial atom and one solute-atom may be present in several different meta-stable configurations. Second, several differently sized complexes, i.e. one solute atom with one, two, three and so on self-interstitial atoms bound to it may also account for these different configurations. For the case of Cu-In for instance, Kollers /13/ has shown that the existence of five internal friction peaks is in accordance with the assumption that a poisson distribution of differently sized SIA-SA-complexes is present in the sample.

b) In each of the alloys containing undersized solute atoms low temperature peaks are found. This is in agreement with the picture of localized diffusion in cages. However, low temperature peaks are not unique only to such cages but also occur for trapped di-interstitials involving oversized solute atoms as recently shown by Kollers /13/.

c) The maximum binding energy, $E^B$, can in principle be determined from the annealing temperature, $T_m$, of the complex with the highest $T_m$, provided that the annealing results from complex dissociation and not from its migration or its reaction with another complex diffusing through the lattice. Very helpful in this respect are comparisons with the results of other experiments. Such a comparison is shown for the present 5 alloys in the following table, where the annealing temperatures, $T_m$, are given as determined from mechanical relaxation measurements, from recovery of residual resistivity, from damage rate- and from channeling measurements /14/.

For the case of Al-Fe and Al-Mn there is agreement between the measurements that final detrapping occurs at about 200K, which corresponds to the temperature regime of stage III. It is reasonable to assume that in this case detrapping occurs by annihilation of the SIA-SA-complexes by migrating vacancies. This means that only a lower bound for the binding energy can be estimated which is 0.6 eV. In Al-Zn peak 1
disappears at 150K. In damage rate and channeling studies, however, final detrapping is observed at 200K. This means, that there are in addition to the defects which cause peak 1 in Al-Zn, SIA-SA-complexes must exist in the material which are not seen in the mechanical relaxation studies. Al-Mg and Cu-In show an annealing behaviour which is very similar to that of Cu-Ag and Cu-Au which has been studied in great detail by Cannon and Sosin /15/. Their analysis has shown that the detrapping observed at about 150K is due to a true dissociation process of the complexes. If in analogy the same is assumed for Al-Mg and Cu-In, the binding energy for these complexes in these alloys is about 0.3 eV.

Table 1:

<table>
<thead>
<tr>
<th></th>
<th>( T_m )</th>
<th>Q^{-1}</th>
<th>RRR</th>
<th>DR</th>
<th>Ch</th>
<th>diss. of complex</th>
<th>( E^B ) [eV]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al-Fe</td>
<td>200</td>
<td>200</td>
<td>200</td>
<td></td>
<td></td>
<td>ann. by migr. vac.</td>
<td>( \gtrsim 0.6 )</td>
</tr>
<tr>
<td>Al-Mn</td>
<td>200</td>
<td></td>
<td>200</td>
<td></td>
<td></td>
<td>ann. by migr. vac.</td>
<td>( \gtrsim 0.6 )</td>
</tr>
<tr>
<td>Al-Zn</td>
<td>150 ( ^* )</td>
<td>200 ( ^* )</td>
<td>200 ( ^* )</td>
<td></td>
<td></td>
<td>?</td>
<td>( \gtrsim 0.6 )</td>
</tr>
<tr>
<td>Al-Mg</td>
<td>150</td>
<td>150</td>
<td>150</td>
<td></td>
<td></td>
<td>yes</td>
<td>( \approx 0.3 )</td>
</tr>
<tr>
<td>Cu-In</td>
<td>150</td>
<td>150</td>
<td>150</td>
<td></td>
<td></td>
<td>yes</td>
<td>( \approx 0.3 )</td>
</tr>
</tbody>
</table>

\( Q^{-1} \): Internal Friction

RRR: rec. of resistivity

DR: damage rate

Ch: channeling

\( \dagger \) The determination of the SIA-SA-complex configurations so far results mainly from channeling measurements /23/. In order to achieve these measurements self-interstitial-atom as well as solute-atom concentrations of the order of 1000 atppm and more had to be used. Due to the particular sensitivity of MR-measurements, the SIA-SA-complex configurations can be determined from the orientation dependence of MR-processes at much lower levels of the order of 50 atppm and less. In Al-Fe clear evidence for the existence of a cage motion has been found. This cage has definitely not the \(<100>\)-tetragonal symmetry required for the \(<100>\)-mixed dumbbell. This result has recently been corroborated by the analysis of the Mössbauer spectra of an irradiated Al-Co alloy /24/. A cage model which accounts for all properties observed in the mechanical relaxation studies and in the Mössbauer studies on Al-Fe has however not yet been developed /16/. In Al-Zn the ultrasonic studies show an orientation dependence which is in agreement with the \(<100>\) symmetry of the mixed dumbbell. In this alloy there is also evidence that at low temperatures the reorientation of this mixed dumbbell occurs via tunneling modes /17/.

3. Zener relaxation in Ag-Zn alloys in thermal equilibrium and under irradiation. Berry and Orehotsky /18/ have shown many years ago that Zener relaxation measurements in concentrated Ag-Zn alloys are a powerful tool in order to study the ordering phenomena observed in these alloys. Halbwachs, Hillairet and others /19-21/ have carried further this method and applied it in particular to alloys under irradiation in order to study defect production and defect mobilities under
irradiation in these alloys. The basic experimental observation is shown in fig. 12. In this figure normalized relaxation curves are shown as a function of time in a logarithmic time scale. One recognises that the relaxation in thermal equilibrium is enhanced by one or two orders of magnitude, respectively, during γ- or electron irradiation.

The relaxation rate, \( \tau^{-1} \), is related to the ordering rates, \( \nu^* \), by equation (21):

\[
\tau^{-1} = a_V c_V \nu^* + a_{SIA} c_{SIA} \nu^*.
\]

\( a_V \) and \( a_{SIA} \) are effectiveness factors of the order of 1, \( c_V \) and \( c_{SIA} \) are the atomic fraction of vacancies and self-interstitial atoms, respectively. In thermal equilibrium only vacancies are present with concentration

\[
c_V = C_V^0 \exp (-E^F/kT)
\]

where \( E^F \) is the vacancy formation energy, \( k \) the Boltzmann factor and \( T \) the temperature. For the material during irradiation, the time dependent concentrations of vacancies, \( c_V \), and of the self-interstitial atoms, \( c_{SIA} \), can be calculated from the following rate equations (21):

\[
\frac{dc_V}{dt} = P \phi - R c_V c_{SIA} (\nu_{SIA} + \nu_V) - \rho_V c_V \nu_V
\]

\[
\frac{dc_{SIA}}{dt} = P \phi - R c_V c_{SIA} (\nu_{SIA} + \nu_V) - \rho_{SIA} c_{SIA} \nu_{SIA}
\]

where \( P \) is the production rate, \( \phi \) the irradiation flux, \( R \) the recombination term, \( \rho_V \) and \( \rho_{SIA} \) the effective sink densities for vacancies and SIA and \( \nu_V \) and \( \nu_{SIA} \) the jump rates of vacancies and SIA, respectively.

For low sink concentrations the time evolution is as shown in fig. 13. At the beginning of the irradiation defects are produced and stored in the lattice. As their concentration increases, mutual recombination sets in and leads to a quasistationary level after a certain time. In the long run the faster diffusing defects arrive at a larger rate at the sinks and therefore their concentration goes down again. At the same time the concentration of the slower defects increases.

For the relaxation rate, \( \tau^{-1} \), this implies a dependence as shown in the lower part of fig. 13. In the time regime where recombination dominates the relaxation rate goes through a quasistationary plateau, \( \tau^{-1}_{qst} \), and for long times it goes down again to a stationary value, \( \tau^{-1}_{st} \).

Fig. 12: Relaxation curves at 100°C. The fraction of normalized total relaxation was plotted versus time:

a) with no flux
b) under a gamma flux of 3 Mrad/h
c) under an electron beam of 1.6x10^11 e^-/cm^2/sec.

(D. Beretz, M. Halbwachs, J. Hillairet)
Under simplifying conditions, \( (v^* \alpha \nu \text{ and } p_{\text{SIA}} = \nu) \) the relaxation rates in the quasistationary regime, \( \tau_{\text{qst}}^{-1} \), and in the stationary regime, \( \tau_{\text{st}}^{-1} \), are given by the relations \( /21/ \)

\[
\tau_{\text{qst}}^{-1} = a_f \left( \frac{v_f}{R} \right)^{1/2} \quad \text{and} \\
\tau_{\text{st}}^{-1} = (a_s + a_f) \left( \frac{v_f}{R} \right)^{1/2}
\]

where \( v_f \) and \( v_s \) are the jump rates of the faster and slower moving species. These formula demonstrate the power inherent in this method: The mobilities of the faster and the slower component can be determined independently from each other and in principle also the production- and recombination-rates inherent in the system.

By doping the material with the faster or the slower component it is also possible to determine what species is faster and what species is slower, vacancy or interstitial. As shown in the lower part of fig. 13 by curve \((x)\) doping with the faster component leads to an enhancement of ordering and thus to an enhancement of the relaxation rate. Doping with the slower component curve \((xx)\) enhances recombination and therefore reduces the relaxation rate. The result of such a doping experiment is shown in fig. 14. Here the relaxation rate is shown as a function of irradiation time for a well annealed sample of Ag-Zn and a sample which was doped with vacancies before irradiation. It is seen that vacancy doping leads to a considerable increase in the relaxation rate. According to fig. 13 this means that the vacancies are the fast species and thus faster moving than the interstitial atoms. Once this identification is done it is now possible on the basis of preceding equations to determine the activation energy for the migration of the vacancies and of the interstitial atoms from the temperature dependence of the quasistationary relaxation rate and the stationary relaxation rate. The result is shown in fig. 15.

Activation energies of 0.56 eV and 0.82 eV for the diffusional jumps of vacancies and SIA, respectively, are determined. Both figures show also the curve observed in thermal equilibrium. The value of 1.44 eV determined from the slope of these curves corresponds to the activation energy for self diffusion in Ag-Zn.
The production rates with which free migrating vacancies and self-interstitial atoms are produced under irradiation by photons, electrons, neutrons, ... can also be determined from these Zener relaxation studies. The evaluation requires a more detailed analysis of the original rate equations which is beyond the scope of the present review. The reader is referred to the original paper /21/ and to the contribution by D. Beretz et al. to this conference.

Conclusions.
1. Self-interstitial atoms: In Al, the self-interstitial atoms have the configuration of \(<100>-dumbbells\) and their dipole tensor is only slightly anisotropic. Simultaneously with their diffusional jump they reorient with an activation energy of 0.115 eV. This migration occurs in stage I. In Mo, the DXS measurements show that self-interstitial atoms have the form of \(<110>-dumbbells\). From the absence of a corresponding relaxation effect it has been concluded that these dumbbells migrate without reorientation. A model has been proposed which implies that the dumbbells migrate two dimensionally in \([110]-planes\).

2. SIA-SA-complexes: The mechanical relaxation processes observed in irradiated dilute alloys support the qualitative view that undersized solute atoms are incorporated as a member of the dumbbell, and that their motion is confined to a cage (localized diffusion). Oversized solute atoms bind with a regular dumbbell at a nearest neighbour position and this dumbbell may jump around the solute atom like on a leash. Even at low concentrations of self-interstitial atoms and solute atoms of the order of 50 atppm many different configurations of SIA-SA-complexes are observed. This contradicts the interpretation of channeling measurements /23/ where one configuration is assumed for the data evaluation.

3. Zener relaxation: Ordering in Zener alloys can be considerably enhanced by irradiation. Vacancies and self-interstitial atoms contribute both to ordering. Vacancies in Ag-Zn are faster moving than self-interstitial atoms. Such measurements are a valuable tool in order to determine the mobilities of vacancies or self-interstitial atoms in thermal equilibrium as well as during irradiation.
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