Strain-Ageing in B.C.C. Metals

by

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Most strain-ageing studies have concentrated on the kinetics of the
segregation of interstitial solute during ageing, the rate of depletion
of the matrix concentration being followed by the measurement of
electrical resistance, internal friction or some other suitable physical
property which depends only upon the presence of interstitial atoms in
solution.   The nature and rate of the changes in mechanical properties have
been studied less extensively and in this paper attention is focussed on
this aspect of the problem, but discussion of the relationships between
strain-ageing and fracture properties is excluded.

Classification of Interstitial Solutes

The general properties of interstitial elements in body-centred cubic
lattices can be discussed systematically if a distinction is made between
'soluble' and 'insoluble' elements, the demarcation being arbitrarily
chosen at 1 atomic percent solubility.   The behaviour of insoluble systems,
for example iron-carbon, can be described completely in terms of the hard-
sphere model which assumes that the interstitial atom is not ionised.   The
interstitial diffusion and the interaction of the interstitial with dis-
locations can be explained on the basis of strain-energy considerations
alone and the small solubility is a result of large local strains due to a

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relatively unfavourable size-factor. When considering soluble systems, such as tantalum-oxygen, effects other than those due to strain interactions must be taken into account. Internal friction experiments reveal the existence of stress-induced interstitial atom migration[1] and on increasing the oxygen content of Group 5A metals there is a measurable increase in the lattice parameter of the solvent lattice[2,3,4]. Thus, it must be concluded that strain effects are significant. However, from the lattice parameter measurements, oxygen in tantalum, niobium and vanadium has an atomic diameter of about 2.08Å. Since the diameter of unionised oxygen atom is about 1.4Å and that of the O^{--} ion about 2.64Å, it is suggested that the oxygen in solution in the Group 5A metals is ionised with a single negative charge.

The difference between soluble and insoluble interstitials is revealed clearly by considering the activation energy for interstitial diffusion. The data in Figures 1-3 were obtained either by direct measurements of diffusion couples, measurements of stress-induced ordering (Snoek effect) by internal friction or they were deduced from measurements of blue-brittleness temperatures[5]. It is well known that the activation energy for self-diffusion increases linearly with the melting point T_m of the metal. The rate-determining step is a strain term and, because the elastic constants are related to the melting point, the activation energy increases with T_m. A similar behaviour is expected for any diffusion process in which the jumps are controlled by strain considerations and, in fact, for the interstitial diffusion of insoluble elements the activation energy does increase with the melting point of the solvent. However, for soluble interstitials the variation of activation energy with melting point is either very small or zero (Figures 1-3). This suggests that the diffusion is controlled by electronic effects and a model in these terms which provides a satisfactory qualitative explanation of this effect has been proposed by Stringer and Rosenfield[6].

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Dislocation-Interstitial Binding Energies

According to Friedel[18], the binding energy W_M between a dislocation and an interstitial atom is

$$W_M = G(c) - Go(c) \qquad (1)$$

where $Go(c)$ is the free-energy per interstitial atom dissolved in a perfect lattice and $G(c)$ the corresponding free-energy when the lattice contains dislocations. In general, there are two ways of measuring W_M, either the solubility of the interstitial element may be measured as a function of the temperature and the dislocation density[19], or the yield drop may be measured as a function of temperature for a specimen of known interstitial content and dislocation density. The latter method depends upon the assumption that the equilibrium concentration of interstitial atoms at a dislocation line varies with temperature according to the relation[20]

$$C = C_o \exp(-W_M/kT) \qquad (2)$$

where C_o is the concentration when there is no segregation. Above some temperature T_b, C falls below the saturation value C_m at which the Petch locking parameter k_y is just greater than zero. Thus,

$$W_M = \left[k\, T_b \ln (C_o/C_m) \right]_{k_y = 0} \qquad (3)$$

The value of C_m is somewhat uncertain as it depends on the precise model which is assumed for the unpinning of a dislocation line under the action of an applied shear stress. Estimates vary between 0.07 and 1.0. Fortunately, the value of W_M determined by this method is quite insensitive to variations in the value adopted for C_m.

Usually T_b has been determined by finding the temperature at which the discontinuous yield disappears in tensile tests carried out at successively higher temperatures. However, the conditions existing during tests at elevated temperatures do not satisfy the requirements of equation 3 and the

values of W_M deduced from them are erroneous. Yield points are observed at low temperatures because dislocations can move under the influence of an applied stress faster than interstitial atoms can diffuse. As the testing temperature is raised, the temperature dependence of the dislocation velocity is less than that of the diffusivity and at a particular temperature (and strain rate) the impurity atoms move with about the same velocity as the dislocations, producing the blue-brittleness (serrated yielding) phenomenon. This fact has been used to calculate the activation energy for interstitial diffusion[21]. The discontinuous yield point always disappears at a temperature just above the blue-brittleness temperature because at this temperature the interstitial atoms are sufficiently mobile for the moving dislocations to drag their associated interstitial atoms with them. Thus, in these circumstances the disappearance of a yield drop does not indicate the absence of segregated interstitial atoms. The temperature T_b for iron-carbon alloys is widely quoted as $700^\circ K$[22], but this was obtained from the high temperature tensile tests of Boulanger[15] and consequently the value of W_M deduced from it is incorrect.

To determine T_b it is necessary to carry out all the tensile tests at a temperature below the blue-brittleness temperature. Thus, a series of specimens must be quenched from successively higher annealing temperatures at a rate sufficient to freeze-in the interstitial distribution. The experimental difficulties are considerable since it is necessary to ensure that there is no contamination of the specimen during the annealing and the quench and that either the dislocation density is unaffected by the annealing or that the density corresponding to each annealing temperature is known. However, a number of determinations of W_M for different systems have been reported and these, together with the limited data available from measurements of solubility as a function of dislocation density are given in Table 1. When calculating W_M from T_b determined by the quenching method, the usual practice is followed of taking C_m as 1 atom per atom plane intersecting the dislocation.

The binding energy W_M has two components, W_{M1} due to strain and W_{M2} due

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to electronic interaction between the dislocation and the interstitial atom. These energies are usually considered to be additive[18]

$$W_M = W_{M1} + W_{M2} \qquad (4)$$

The electronic contribution to the binding energy in the iron-carbon and iron-nitrogen systems is zero because the carbon atom is not ionised, but in systems with appreciable solubility the electronic effect is probably significant. The heterogeneous strain field around an edge dislocation produces an electric dipole[19] because electrons flow from the compressed region above the slip plane to the dilated region below. Thus, a net negative charge is created below the extra plane which attracts positively charged ions but repels ions which carry a negative charge. Thus, it is expected that W_M for tantalum-oxygen alloys will be less than W_{M1} by an amount W_{M2}.

In general, the elastic strain interaction energy is

$$W_{M1} = A/R \qquad (5)$$

where A is an interaction parameter which varies with the elastic constants, the lattice parameter of the solvent and the extent of the dilation of the lattice by the interstitial atom. R is the distance between the interstitial atom and the geometric centre of the dislocation. A can be estimated from the Cottrell-Bilby model[26] or from refinements of this model such as that due to Cochardt, Schoeck and Wiedersich[27].

The dilation caused by a carbon atom in an iron lattice can be estimated from the measured lattice parameter change of the martensite lattice with carbon content[26] and similar calculations can be made from the lattice parameter change with increasing interstitial content for those alloy systems, such as tantalum-oxygen, which do not form martensite but which have appreciable solubility. It is usual to assume that R is one Burgers vector. The calculated values of W_{M1} are compared with the experimental values of W_M in Table 1. In iron-carbon alloys W_M and W_{M1} are identical, confirming the prediction that the interaction in these alloys

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is entirely due to elastic strain. However, in the tantalum-oxygen case W_M is slightly less than W_{M1} requiring W_{M2} to be negative, although the difference is probably only a little greater than the possible experimental error. The sign of W_{M2} is consistent with the concept of a negatively charged oxygen ion in the tantalum lattice and the magnitude of W_{M2} is about that expected from the electronic interaction. According to Friedel[18]

$$W_{M2} = \frac{\Delta E\ ze}{24} \tag{6}$$

where ze is an effective value of charge and ΔE is the width of the conduction band. The value of ze in tantalum alloys depends upon a screening constant which is known only approximately, a reasonable value being 0.3. From the difference $W_M - W_{M1}$ (Table 1), W_{M2} seems to be about 0.1 eV and thus, from equation 6, ΔE is about 7 eV. This is not an unreasonable value for the width of the conduction band in transition metals. Thus, although estimates of this type are inevitably very crude, there appears to be grounds for suggesting that in tantalum-oxygen, and probably in similar alloys, there is a significant electronic contribution to the dislocation-solute interaction energy.

The Variation of the Yield Parameters with Ageing

Until recently it was generally assumed that the return of the yield point could be described completely in terms of Cottrell pinning theory; the interstitial atoms being attracted to the dislocations during ageing under the influence of the elastic strain field interactions and thermal diffusion. The difference $(\Delta\sigma)$ between the lower yield stress (σ_y) of the aged specimen and the stress at which the specimen is unloaded before ageing (σ_p) increases with ageing time and $\Delta\sigma$ was assumed to be a direct measure of the degree of saturation of pinning sites near the dislocation. However, it is now clear that phenomenologically there are two parameters involved in the yield stress; k_y which is a measure of the local stress required to activate a new dislocation source and σ_i the friction stress opposing the propagation of a slip band[28,29]. Although it is probable that in lightly aged specimens dislocation sources are produced by the unpinning of dislocations[30] as visualised by Cottrell[20], it seems that existing dislocations are easily

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completely immobilised so that the discontinuous yield occurs by the generation
of dislocations from other discontinuities such as surfaces or grain boundaries.
Thus, k_y is not necessarily related to unpinning. According to Petch[28]

$$\sigma_y = \sigma_i + k_y \, d^{-\frac{1}{2}} \qquad (7)$$

The relation between stress-strain curves for the prestrained and the aged
specimen is shown schematically in Figure 4,

$$\sigma_y = \sigma_p + \Delta\sigma_f + k_y \, d^{-\frac{1}{2}} \qquad (8)$$

and since, by definition $\Delta\sigma = \sigma_y - \sigma_p$

$$\Delta\sigma = \Delta\sigma_f + k_y \, d^{-\frac{1}{2}} \qquad (9)$$

Thus, the increase in yield stress on ageing has two components. This was
first demonstrated experimentally by Wilson and Russell[31] who, by applying
the Petch method, showed that in iron-carbon alloys strain-ageing is a two
step process; first k_y increases to a maximum value while $\Delta\sigma_f$ remains zero
and subsequently $\Delta\sigma_f$ increases. The Petch method for determining k_y and
$\Delta\sigma_f$, which depends upon measuring the variation of yield stress with grain
size, is valid only if the substructure and distribution of submicroscopic
precipitates are unaffected by the treatments used to establish a range of
grain size; a condition which is satisfied approximately for iron-base alloys
which are annealed through the phase change[32], but which is not usually
achieved experimentally with other body-centred cubic metals. Fortunately,
σ_j (Figure 4) can be determined by a method which does not require the use
of grain size as a controlled variable. The homogeneous strain-hardening
curves AB and CD (Figure 4) can be represented exactly by equations of the
form

$$\sigma = K\mathcal{E}^n \qquad (10)$$

where K is the strength constant, $\mathcal{E}$ the strain and n the strain-hardening
index. So that

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$$\sigma_j = K\varepsilon_p^n$$

K an n are best found from a logarithmic stress versus logarithmic strain plot of CD.

Experimental studies of strain ageing which enable the changes in $\Delta\sigma_f$ and k_y to be distinguished have been reported only in the last few years, and the information available is limited. However, both soluble (tantalum-oxygen[33], vanadium with carbon and nitrogen[34]) and insoluble (iron-carbon[31] and iron-silicon-carbon[35]) systems have been examined and thus it is possible to make some comparisons. On ageing at 261°K mild steel specimens which had been quenched from 700°C and prestrained 4 percent Wilson and Russell[36] found a rapid initial increase in yield stress which they attributed to stress-induced local ordering of the interstitial atoms in the stress field of the dislocation (Snoek effect). This effect is not found in tantalum-oxygen alloys at similar temperatures and strain-rates because the jump time is too long. For carbon in iron the jump time at 293°K is about 0.6 seconds but for oxygen in tantalum it is about 468 minutes.

After an incubation period, k_y increases with ageing time. On ageing iron-carbon alloys at 60°C a maximum value is reached when the concentration of carbon at the dislocations is 1 or 2 atoms per atom plane intersecting the dislocation line, but the k_y value for tantalum-oxygen aged at the same temperature levels off when less than a tenth of the available dislocation sites are occupied by oxygen atoms. The small k_y which is developed varies very little with testing temperature. Similar results have been found for vanadium-carbon-nitrogen alloys[34]. This contrasts with the iron-carbon alloys in which the k_y is markedly temperature dependent in lightly aged specimens but insensitive to testing temperature when fully aged. It may be that these differences are a result solely of the differences in diffusivities of the interstitial elements and that parallel results to those found in iron-carbon can be found in the tantalum and vanadium alloys when ageing is carried out at higher temperatures. This aspect of the subject has not yet been explored experimentally. A striking feature of strain-ageing is that the value of k_y developed in a fully aged specimen is appreciably smaller than k_y

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for the alloy in the annealed condition. This effect has been attributed to the higher dislocation density in prestrained specimens, but in all the alloys so far examined the concentration of solute is much in excess of that required to saturate the dislocations when segregation is complete and so this explanation is not tenable. Possibly, the effect is controlled by the kinetics of the strain-ageing process. In tantalum the diffusivity of oxygen is very slow and k_y fpr specimens fully aged at 100°C is only about one tenth of the annealed value [33]. The diffusivity of carbon in iron is appreciably faster and about one third of the annealed k_y is developed on ageing for long times at 60°C[31]. On ageing iron-silicon-carbon alloys at 150°C about two-thirds of the annealed k_y value is obtained in fully-aged specimens[35]. Again, this points to the need for extensive systematic study of ageing effects at higher ageing temperatures.

The experimental data show clearly that $\Delta\sigma_f$ and k_y are independent parameters. In alloy systems in which the solubility of the interstitial element is very small there is obviously a tendency for precipitation to occur. In the iron-carbon alloys studied by Wilson and Russell the initial increase in k_y was not accompanied by an increase in $\Delta\sigma_f$, indicating that the Cottrell atmospheres or precipitates formed as a result of the carbon segregation to dislocations in the early stages of strain-ageing do not offer an effective resistance to the motion of unpinned dislocations. However, $\Delta\sigma_f$ does increase after longer ageing time but before any precipitate can be detected by electron-transmission microscopy. This increase has been attributed to carbon clusters or submicroscopic precipitates formed after all the dislocation sites are occupied. The relative changes in k_y and $\Delta\sigma_f$ with time on ageing at 60°C are shown in Figure 5. A similar sequence of changes was found by Smallman and Lindley[34] in impure vanadium. Although carbon and nitrogen have an appreciable solubility in vanadium the alloy clearly contained some insoluble element since precipitates nucleated on dislocation lines were clearly visible by electron-transmission micrography on ageing at 750°C. In alloys containing only soluble interstitial elements no increase in $\Delta\sigma_f$ due to precipitation or preprecipitation effects is likely to occur. Nevertheless, it is observed that $\Delta\sigma_f$ increases with ageing time. In

tantalum-oxygen alloys $\Delta\sigma_f$ starts to increase at the same time as, or even before, any change in k_y can be detected (Figure 6). Effects which have been suggested to account for the increase in $\Delta\sigma_f$ are the pinning of forest dislocations, thus increasing their resistance to the passage of glide dislocations, and the jogging of glide dislocations by the segregation of vacancies to dislocation lines. The observation that, in tantalum-oxygen prestrained at $293^{\circ}K$, aged at $373^{\circ}K$ and tested at $232^{\circ}K$, $\Delta\sigma_f$ increases long before there is any change in k_y seems to eliminate the first suggestion. The vacancy segregation hypothesis has not been tested adequately. Preliminary experiments on tantalum-oxygen alloys indicate that the increase in $\Delta\sigma_f$ with ageing time is much less marked in quench-aged specimens than in prestrained specimens aged at the same temperature and, because the vacancy concentration in the latter specimens is expected to be higher, this suggests that the role of vacancies is important. However, it is not feasible to carry out parallel experiments on quenched and prestrained specimens with the same dislocation density, nor is the change in strain-hardening index compatible with a simple vacancy segregation model, so that for the present this hypothesis must also be considered to be tentative.

The Rate Controlling Step

It is usual to calculate a heat of activation ΔH from experimental data relating the change of some physical property with ageing time to the ageing temperature, assuming an Arrhenius relationship. The values of ΔH measured by changes in the yield stress ($\Delta\sigma$), electrical resistance and internal friction peaks are in surprisingly good agreement. Data which would enable ΔH to be calculated independently for changes in k_y and $\Delta\sigma_f$ are not available. However, from measurements taken during the period when only k_y is changing, it is clear that the rate of diffusion of the most rapidly diffusing interstitial element controls the segregation to dislocations during the early stages of strain ageing. Thus, the fact that reasonably good agreement between ΔH for the change in $\Delta\sigma$ and ΔH from measurements which depend upon the rate of depletion of the matrix interstitial solid-solution is maintained at later stages of ageing when the change in $\Delta\sigma_f$ is significant

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suggests that the controlling activation energy for $\Delta\sigma_f$ is either that for the fastest moving interstitial or for some other process with nearly the same activation energy.

Johnson[37] has claimed that the rate of change of electrical resistivity on ageing niobium can be explained by vacancy migration because the measured activation energy is 0.25-0.30 times the activation energy for self-diffusion. When all the available values of ΔH for strain ageing processes are plotted as a function of the melting point (Tm) of the solvent metal the best fit is given by $\Delta H \cong 0.3$ Tm (Figure 7). Brooks[38] has estimated theoretically that $\Delta H \cong 0.2$ Tm. Thus, either the theoretical estimate of the proportionality constant is incorrect or the rate controlling step is not vacancy migration. Some support for the concept of control by vacancy migration is available from the limited data on the rate of recovery of irradiated materials. ΔH derived from measurements of the isothermal recovery of electrical resistance after irradiation in an atomic pile are listed in Table 2. Considerable ageing occurs in the pile and freshly irradiated specimens have well-developed yield points but, although there is no subsequent change in k_y, on isothermal annealing the flow stress increases, corresponding to a change in $\Delta\sigma_f$. This lattice hardening is usually considered to be the result of vacancy migration. For the metals for which data are available (Nb, Mo, W), the values of ΔH for the recovery of electrical resistance after irradiation agree closely with those for the change in $\Delta\sigma$ on strain ageing.

The other possibility is that all stages of strain-ageing are controlled by the diffusion of the fastest-moving interstitial element. As shown by the data collected in Table 2, in general there is good agreement between ΔH derived from measurements of the rate of change of $\Delta\sigma$ and ΔH for diffusion of the appropriate interstitial.

Thus, it must be concluded that the available data are insufficient to enable an unambiguous distinction to be made between interstitial element diffusion and vacancy migration. It is possible that the rate controlling step varies with the alloy system and with the different stages of strain ageing. However, before these questions can be answered it is clear that much more precise data on the activation energies for interstitial diffusion,

vacancy migration and the change in mechanical properties during strain a겡eing are required. It is particularly important that in future work experiments are designed which separate effects producing changes in k_y from those affecting $\Delta \sigma_f$.

Table 1

Measured and Calculated Binding Energies

System	Reference	W_M Measured eV	W_{M1} Calculated eV	W_{M2} Calculated eV	A_4 Calculated $\frac{dynes}{cm^2}$
Fe-C ⎫	23	0.80	0.76	0	3.0×10^{-20}
Fe-N ⎬	24	0.73			
	19	0.71			
Ta-O	25	0.54	0.64	-0.10	2.9×10^{-20}

Table 2

Measured Activation Energies for Strain-Ageing Processes, Interstitial Diffusion and Recovery of Radiation Damage in B.C.C. Metals

Material	Method	Reference	Ageing Temperature °C	ΔH for strain ageing eV/atom	ΔH for Interstitial Diffusion Element	eV/atom	ΔH for Recovery after Irradiation eV/atom
α-Fe	Change in $\Delta\sigma$.Internal Friction	23	25–60	0.78 0.89			
					N	0.81	
					C	0.89	
V	Change in $\Delta\sigma$	2	125–200	1.20			
	Change in $\Delta\sigma$	34	100	1.09			
					C	1.19	
Cr	Internal Friction	39	30–330	~1–1.5			
					C or N	1.26	
Nb	Change in $\Delta\sigma$	40	100–162	1.17			
	Electrical Resistance	41	100–180	1.22			
					O	1.17	
	Electrical Resistance	41	100–180				1.22
Mo	Electrical Resistance	42	100–130	1.25			
	Internal Friction	43	300–400	1.3–1.5			
	Change in $\Delta\sigma$	44	575–700	1.58			
	Electrical Resistance	45	~150	1.3			
					C	1.45	
	Electrical Resistance	41	140–200				1.25

Table 2 (continued)

Mo	Electrical Resistance	45	150				1.30
Ta	Electrical Resistance	45	120-180	1.25			
					O	1.11	
W	Electrical Resistance						
	'Pure'	46	325-650	1.7			
	'Doped'	46	325-650	2.3			
	Electrical Resistance	47	~350	1.7			
					C	1.72	
	Electrical Resistance	48	330-450				1.7
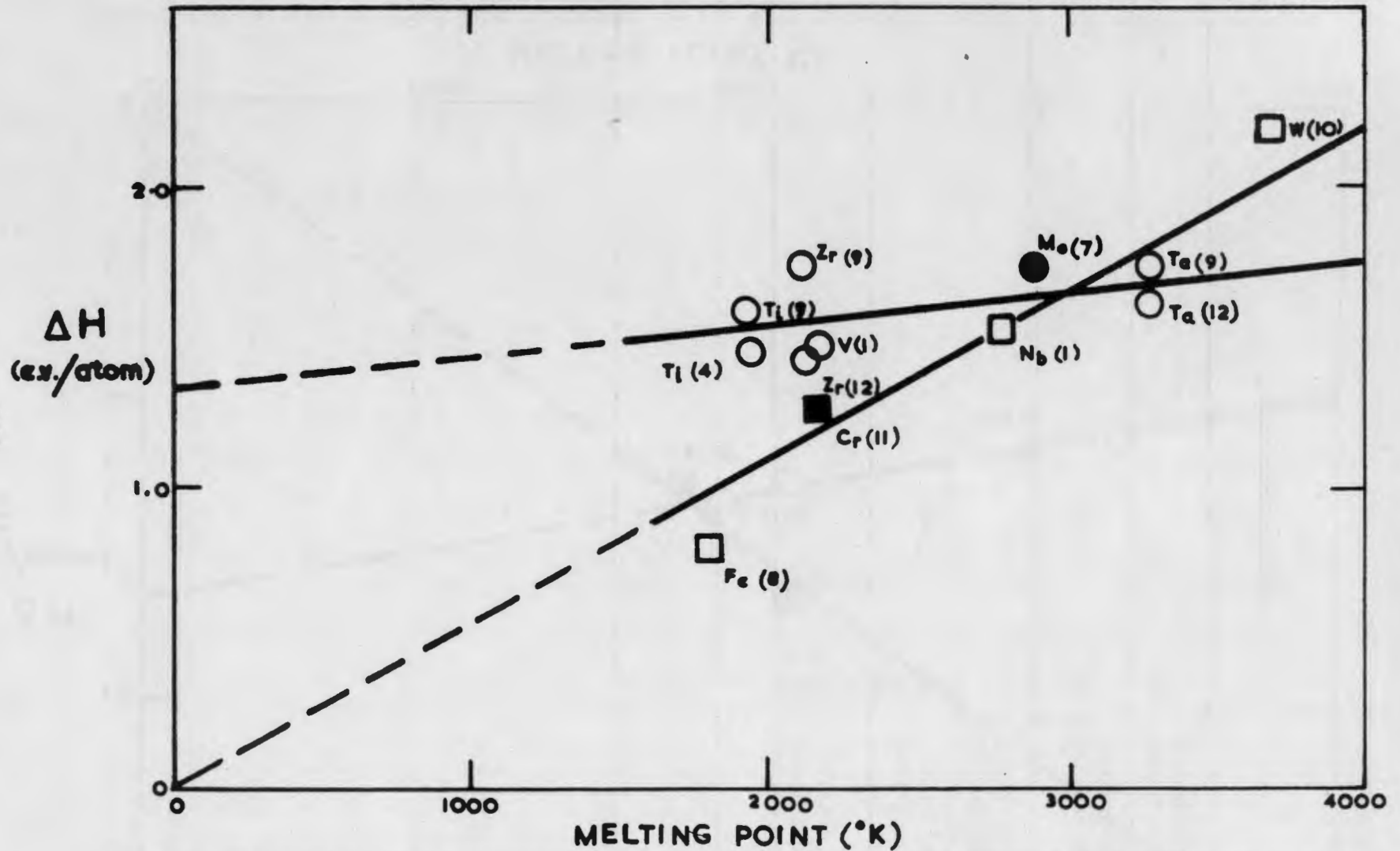
Figure 1 The variation of activation energy for the diffusion of nitrogen with the melting point of the solvent. Circles, high solubility; squares low solubility. Full points evaluated from Portevin-le Chatelier effect. Stringer and Rosenfield (6).
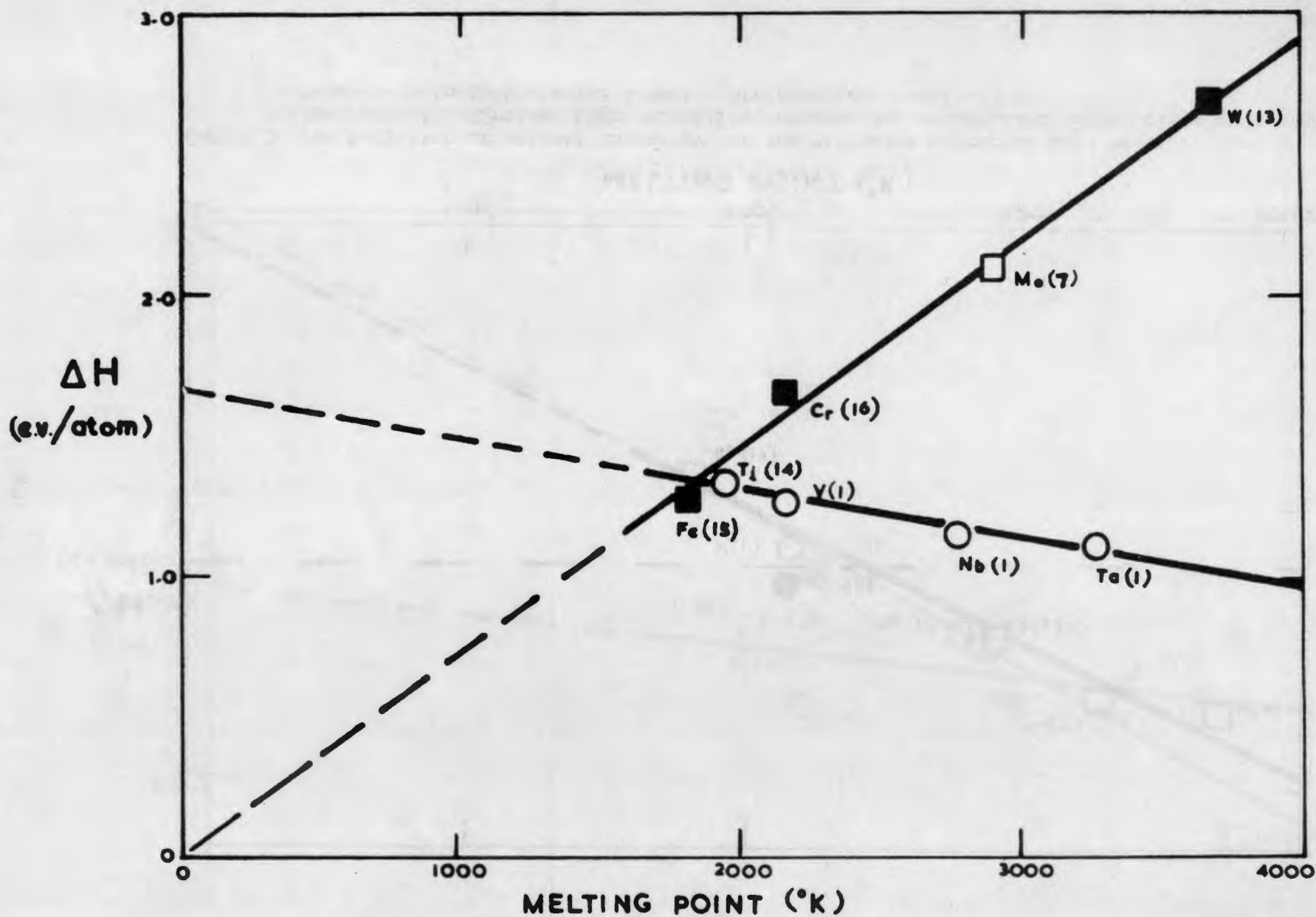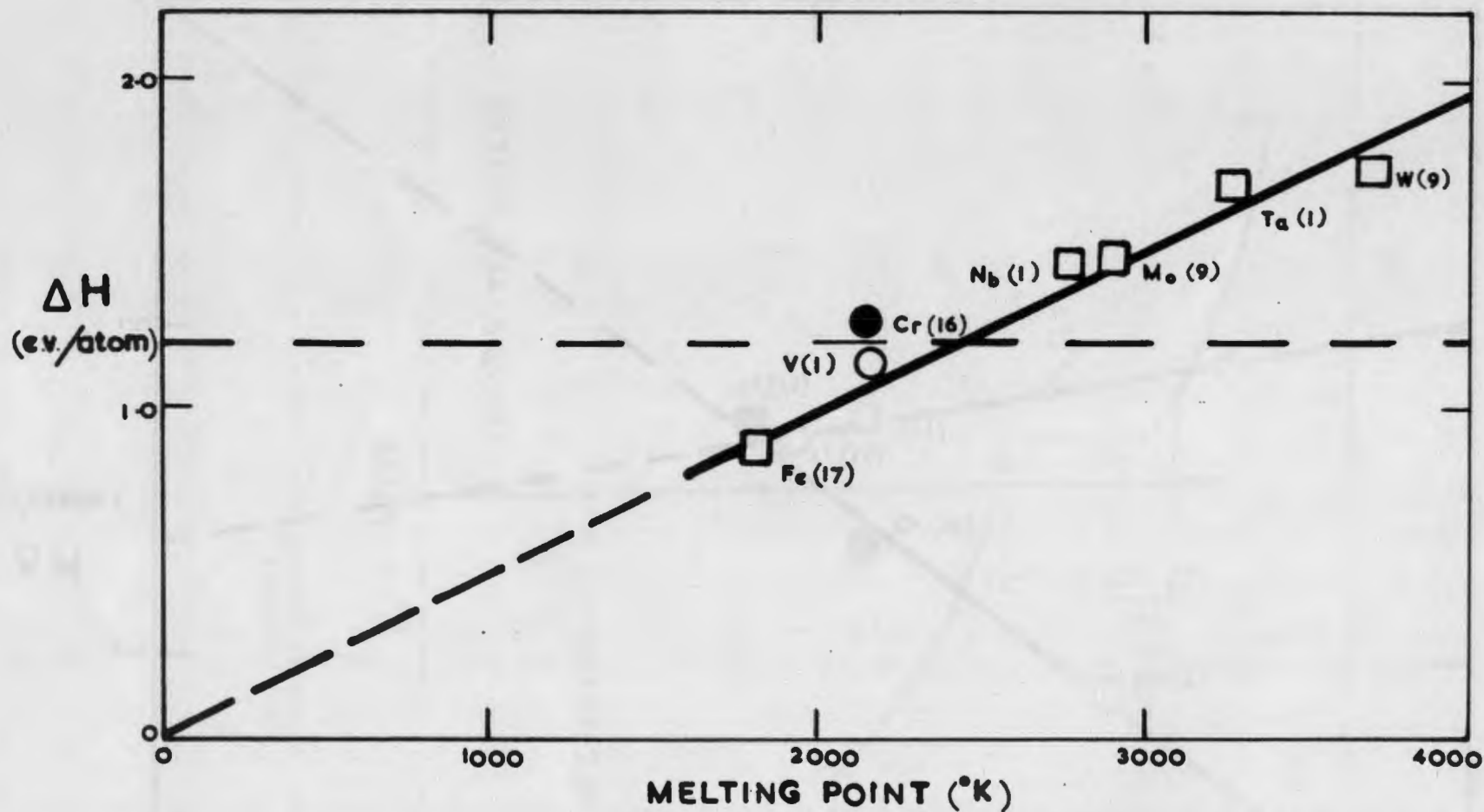
Figure 2 The variation of activation energy for the diffusion of oxygen with the melting point of the solvent. Circles, high solubility; squares, low solubility. Full points evaluated from Portevin-le Chatelier effect. Stringer and Rosenfield (6).

Figure 3   The variation of activation energy for the diffusion of carbon with melting point of
the solvent.   Circles, high solubility; squares low solubility.   Full points evaluated
from Portevin-le Chatelier effect.   Stringer and Rosenfield (6).
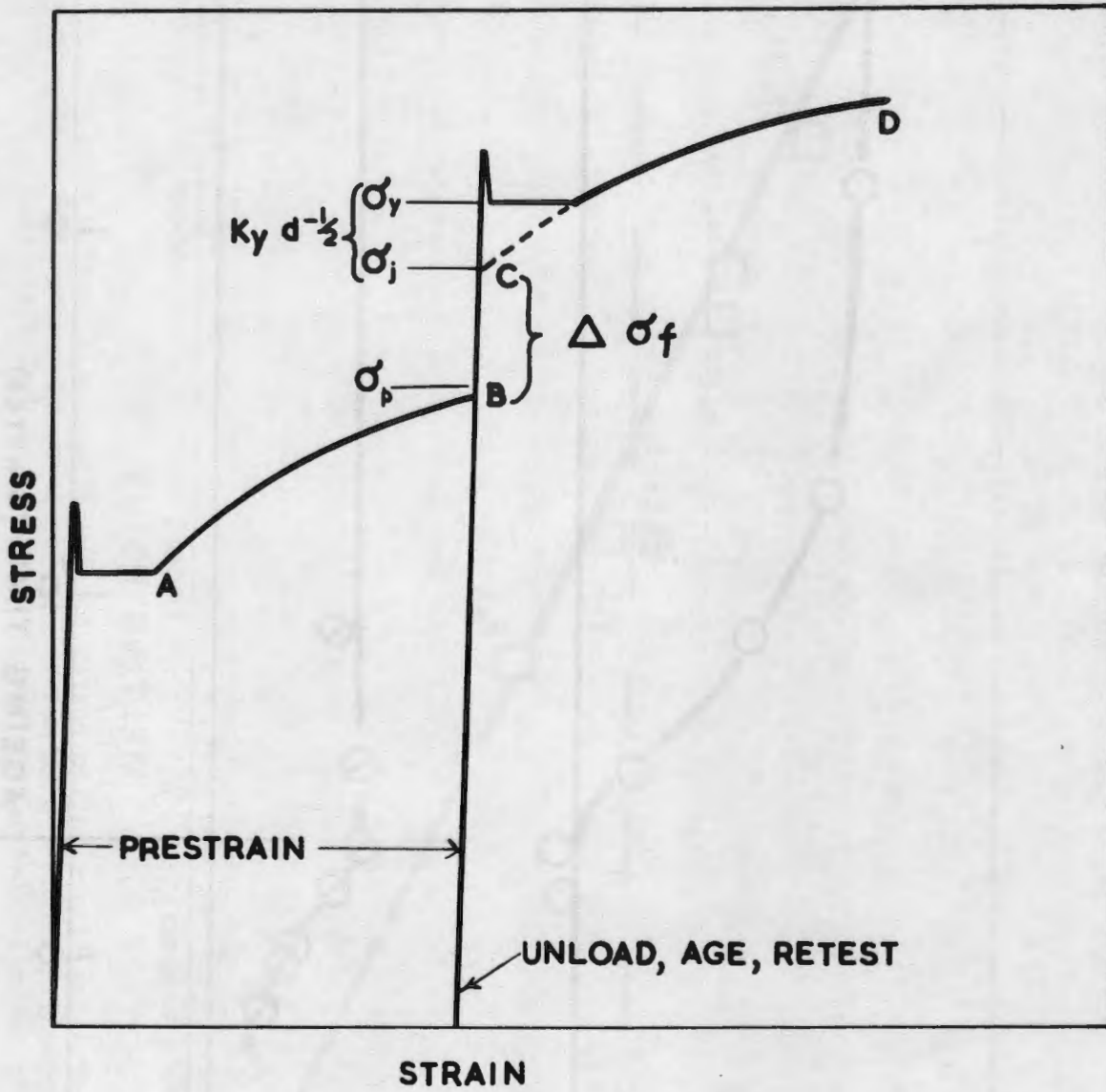
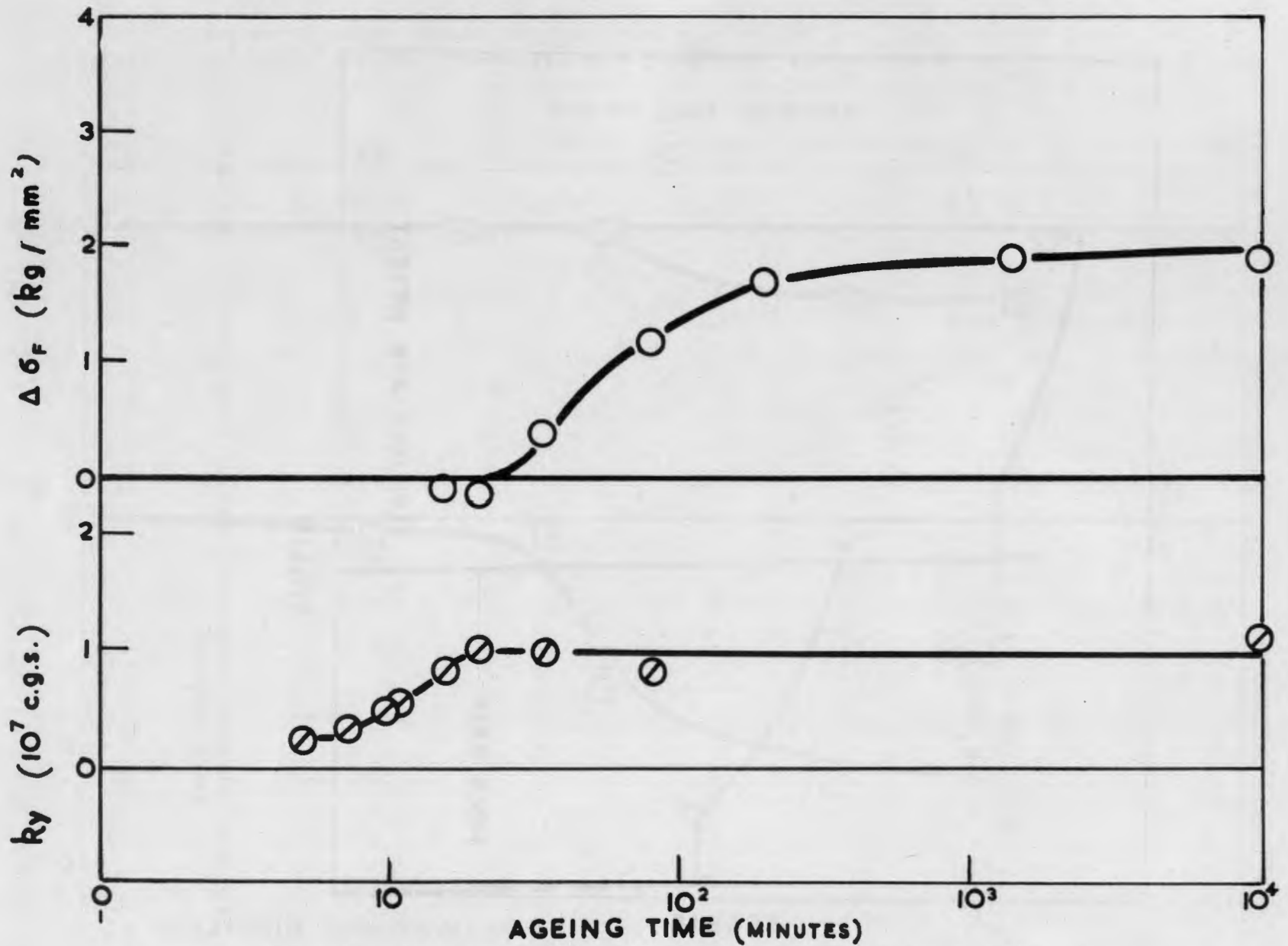Figure 4   Schematic stress-strain curve for a prestrain, age and retest sequence.

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Figure 5 $\Delta\sigma_f$ and k_y for iron-carbon as a function of ageing time at 60°C. Prestrained and tested at 293°K. Wilson and Russell(36).
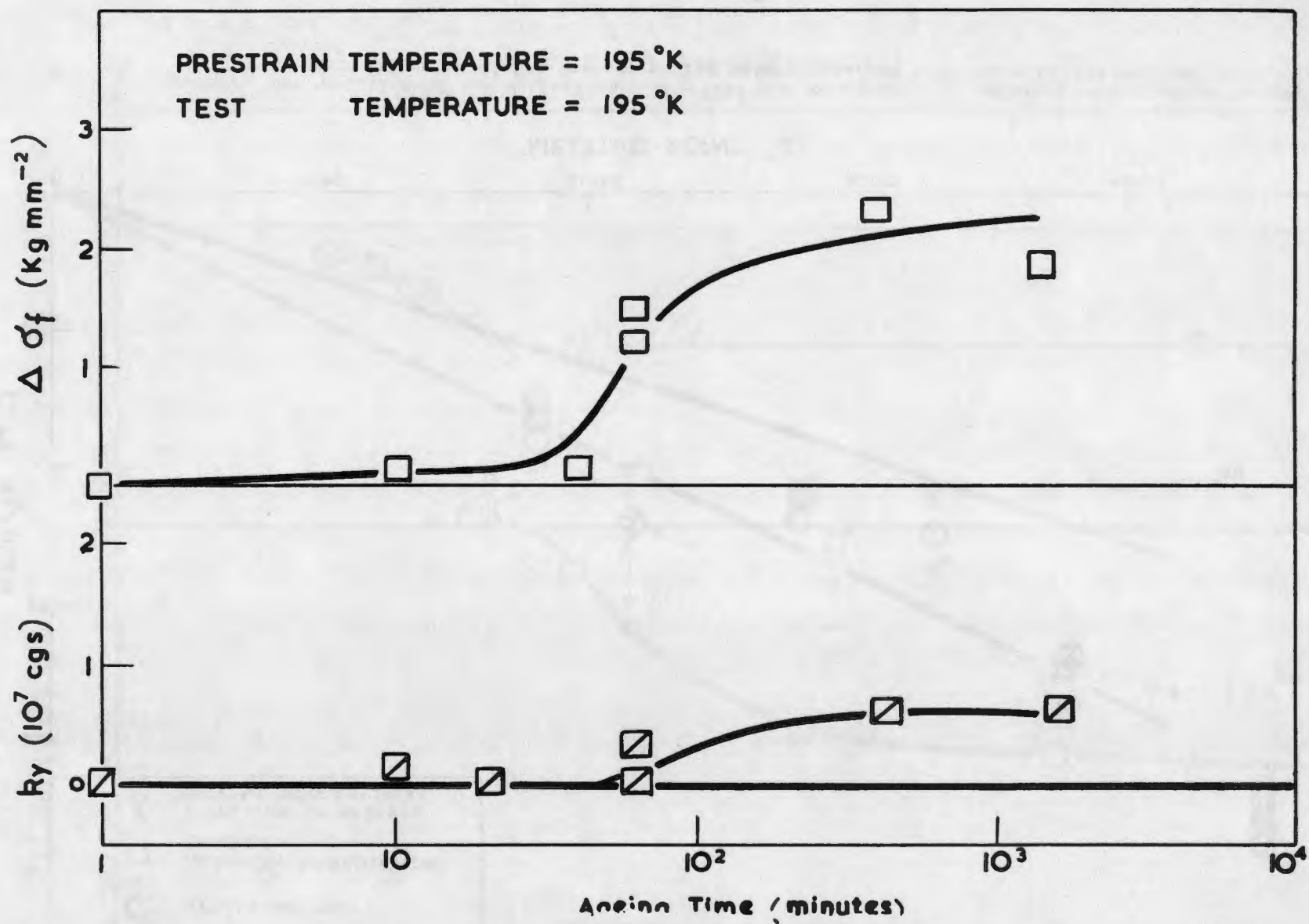
Figure 6 $\Delta \sigma_f$ and k_y for tantalum-oxygen as a function of ageing time at 100°C. Prestrained and tested at 195°K. Rosenfield and Owen[33].
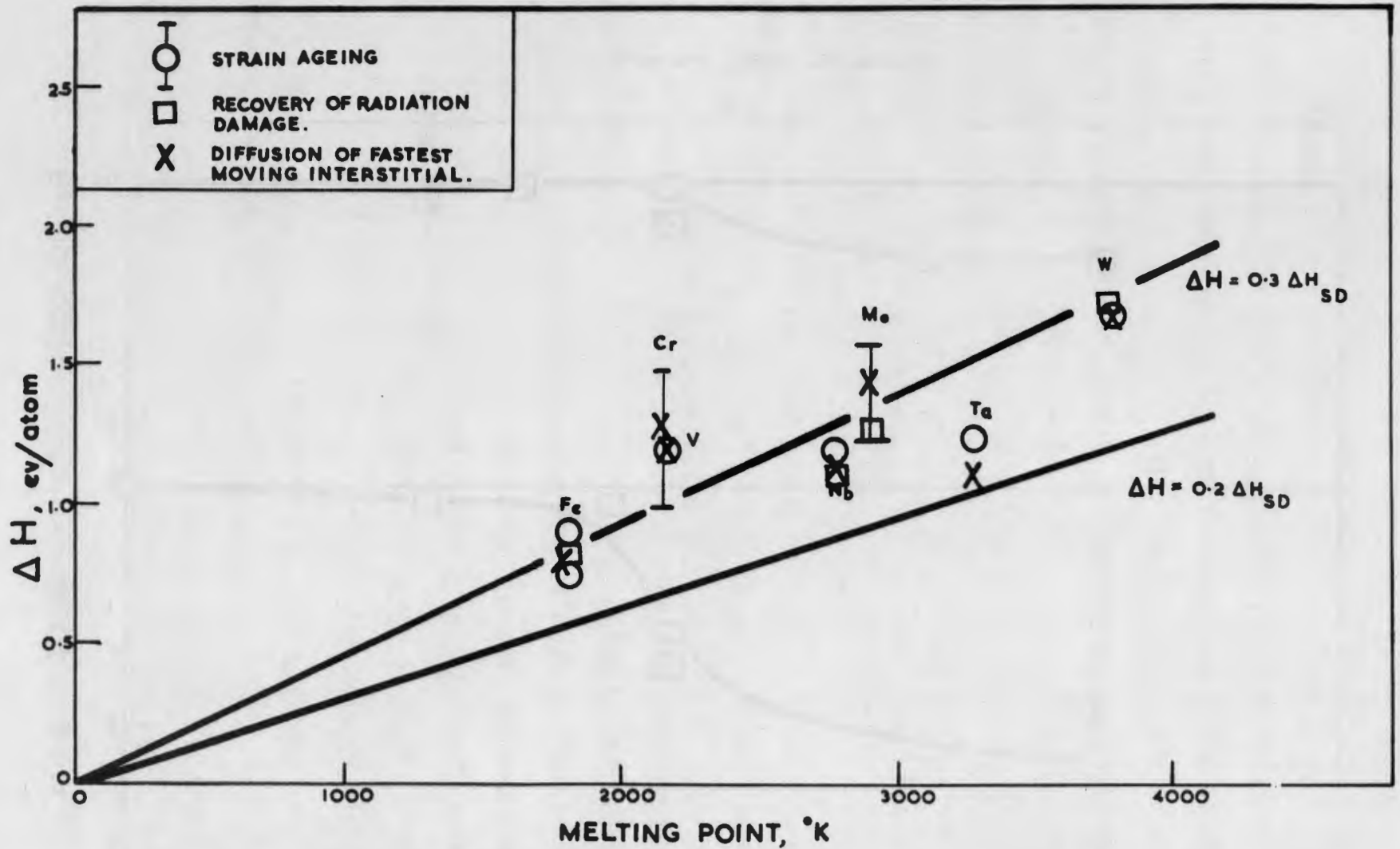
Figure 7 The variation of the activation energy for strain ageing, for recovery of radiation damage, and for diffusion of the fastest moving interstitial as a function of the melting point of the solvent.

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