

THE INFLUENCE OF STRUCTURAL DEFECTS ON THE KINETICS OF PRECIPITATION IN α Fe-N SOLID SOLUTIONS*

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The kinetics of nitrogen precipitation from supersaturated solid solutions in α Fe was investigated by means of potentiometric electrical resistance measurements. The influence of the quenching temperature and of plastic straining by cold rolling and elongation was examined. In samples which were plastically strained by 30 pct, annealed at 632K/1h and quenched in liquid nitrogen the precipitation was a homogeneous one, but no nucleation stage could be observed in the temperature range from 313 to 383K. It was also shown that the precipitation activation energy E is much smaller than the activation energy of nitrogen migration Q : $E = 0.43$ eV, $Q = 0.77$ eV.

1. Introduction

Interstitial solid solutions of carbon and nitrogen in alpha iron and its alloys display, after annealing and quenching from high temperatures an ageing phenomena at room and higher temperatures. This is originated by the precipitation of C or N from supersaturated solutions. The ageing is mainly controlled by the diffusion of interstitial atoms, but there are also other factors, such as: structural defects, internal stresses, the concentration of the solution, the presence of precipitation nuclei. All of these influence the kinetics of precipitation.

In principle, two types precipitations can be distinguished [1]: 1. homogeneous precipitation in the perfect matrix of the solvent, 2. heterogeneous precipitation at structural defects, principally at dislocations. There are two stages in the kinetics of homogeneous precipitation [2, 3]. At the beginning of ageing, nucleation of precipitates occurs; the stage is reversible and originates from the interaction between single interstitial atoms and clusters of these atoms. The second stage is irreversible and corresponds to a steady growth

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of clusters; during this stage interstitial atoms, if attracted by a nucleus, cannot become separate again. The transition time from one stage to the other is determined by the maximum of the precipitation rate, during isothermal ageing [3].

During heterogeneous precipitation no nucleation occurs, because there are regions in the matrix which could act as nuclei.

Regeneration of the physical properties of metallic supersaturated solid solutions, related to precipitation, are frequently described by the introduction of an activation energy E , as a diffusional parameter. A review of literature, devoted to precipitation in interstitial alloys of metals, shows that there are two different approaches to this problem. Some authors consider E to be equal to Q — the activation energy of interstitial migration [4–9], others suppose that E is distinctly smaller than Q [2, 10–16]. There is great confusion among the authors of the second group concerning the reason for the smaller E . In some papers structural defects are assumed to be responsible for $E < Q$ [11, 12], in others the interaction energy between atoms and precipitates is claimed to decrease the activation energy of precipitation [2, 10, 13–16].

In this work, by means of electric resistance measurements, the kinetics of heterogeneous precipitation and the influence of structural defects on homogeneous precipitation were investigated. Until now only a few papers were devoted to precipitation in $\alpha\text{Fe-N}$ [2, 4, 17–19]; in these papers, however, no attention was paid to the influence of defects on homogeneous processes.

2. Samples

The electric resistance of $0.2 \times 5 \times 125$ mm strips was measured. The investigated electrolytic iron (only 0.01% substitutional impurities) was prepared in the Institute of Iron Metallurgy, Gliwice. One series of samples (Fe I) contained 0.012% N and small amounts of C (the magnetic permeability disaccommodation [20] of the carbon Snoek relaxation was 100 times smaller than for nitrogen), in the samples of the second series (Fe II) the amounts of both, N and C, were similar (0.013%).

Before measurements began, all samples were heat treated in a vacuum furnace at 1223 K for 5h and 973 K for 1h, whereafter they were slowly cooled with the furnace. Some samples of series I (Fe I') were annealed at 1223, 1073, 983, 773 and 623 K, respectively, in an argon atmosphere and then quenched in liquid nitrogen (solution heat treatment). The others (Fe I'') were plastically deformed by cold rolling; the deformation amounted to 1, 2, 5, 12.5, 20, 25, 30, 50 and 70 pct. Samples of series II were plastically deformed by elongation at room temperature.

3. Procedure

Isochronal and isothermal ageing curves were determined. The electrical resistance was measured at the temperature of liquid nitrogen. The measurements were performed by means of the potentiometric method, on a precise direct current compensator; the point probe method was applied [16].

In isochronal measurements step heatings with average rates of 5 K/15 min. and 5 K/1h were used. The sample was heated for 15 min. or 1h at the ageing temperature, whereafter it was quenched in liquid nitrogen. Then after measuring the resistance, it was aged again at a higher temperature.

During isothermal measurements, the sample was heated at a constant ageing temperature and, at intervals, was quenched in nitrogen for measurement. The current passing through the sample was 0.1 A, but this value did not change its temperature by a measurable amount.

According to Matthiessen's rule the changes in concentration of interstitial solute atoms and in dislocation density can be calculated from the formula

$$\rho = \rho(T) + \rho_i + \rho_d, \quad (1)$$

in which ρ is the resistivity of the alloy, $\rho(T)$ — its thermal part, ρ_i , ρ_d — parts corresponding to interstitial atoms and dislocations, respectively.

For the investigated alloys which are very dilute, ρ_i can be expressed in the form

$$\rho_i = a \cdot c + bc_w, \quad (2)$$

where a and b are proportionality coefficients and c and c_w are the concentrations of N atoms in solid solution form and in precipitates, respectively. For precipitates of dimensions greater than the free path length of conductivity electrons, the coefficient b depends only weakly on their volume and shape and, therefore, could be assumed constant. It is proper to add, that b is much smaller than a , as will be corroborated in chapter 4.2.

At constant temperature and for fixed dislocation density the relative changes in resistance depend on interstitial atom concentrations in solid solution form

$$\frac{R(t) - R_\infty}{R_0 - R_\infty} = \frac{\rho(t) - \rho_\infty}{\rho_0 - \rho_\infty} = \frac{\Delta\rho(t)}{\Delta\rho_0} = \frac{c(t)}{c_0}. \quad (3)$$

In this formula $c(t)$ and c_0 are concentrations at ageing times t and $t = 0$, and $\rho(t)$, ρ_0 , ρ_∞ , are resistivities at t , $t = 0$, $t \rightarrow \infty$, respectively.

Similarly, if all interstitial atoms are either in solid solution form or in precipitates, the resistance changes depend on dislocation densities, only.

In the case of precipitation in $\alpha\text{Fe}-\text{C}(\text{N})$ and $\alpha\text{Fe}-\text{X}-\text{C}(\text{N})$, the isothermal curves are often described by means of a formula given by Wert [4]:

$$c(t) = c_0 \exp\left(-\frac{t}{\tau}\right)^n, \quad (4)$$

where τ obeys the Arrhenius law. Using (4) for homogeneous processes the activation energy was usually found to be equal to the activation energy of migration of interstitial atoms. According to Cottrell and Bilby [21], and to Harper [22] this formula is also valid for heterogeneous precipitation at dislocations. For this precipitation, in [21, 22] $n = \frac{2}{3}$ was obtained. Recently, Humphreys, Plumtree and Bratina [23] have shown experimentally

that, for precipitation at dislocations, two stages can be identified; for the first — $n = \frac{2}{3}$, for the second — $n = \frac{1}{2}$.

In the past most authors used formula (4) to calculate the exponent n ; it was also tried very often to connect the value of n with the shape of homogeneous precipitates. Recently, however, for homogeneous precipitation the model of Damask, Danielson and Dienes [2] is generally accepted. For the case when the sample contains nuclei at $t = 0$ Parisot et de Fouquet gave a more convenient form of the Damask et al. expression [24]

$$c(t) = c_0 \left[\cosh \frac{\gamma}{2} t + \frac{S}{S + Y_\infty} \sinh \frac{\gamma}{2} t \right]^{-\frac{2}{p}}, \quad (5)$$

in which p is the number of atoms in the nucleus, S — the number of nuclei at $t = 0$, Y_∞ — the number of nuclei formed during the whole precipitation process ($t \rightarrow \infty$); γ is a quantity, which can be calculated by means of equations given in [2]. If t is large, (5) becomes:

$$c(t) = c_0 \left(2 \frac{S + Y_\infty}{2S + Y_\infty} \right)^{\frac{2}{p}} \cdot \exp \left(- \frac{\gamma}{p} t \right). \quad (6)$$

Expression (5) describes properly the curves of homogeneous precipitation in the whole time range. As concerns the time dependence, for $n = 1$ formula (4) and (6) are equivalent; in this case (4) is the solution of the first order kinetic equation.

The above remarks show, that by means of formula (4) the mechanism of precipitation from supersaturated solid solutions can be determined:

1. for the stage of growth of homogeneous precipitation $n = 1$
2. for heterogeneous — $n = \frac{2}{3}$ or $\frac{1}{2}$.

The activation energy of precipitation can be calculated by means of several methods [25–27]. In the Meechan–Brinkman method isochronal and isothermal ageing curves have to be determined [25]; the activation energy E is given by the straight line (with minus sign):

$$\ln \Delta t_a = \pm \frac{E}{R} \cdot \frac{1}{T_i} + \text{const.} \quad (7)$$

In (7) T_i are heating step temperatures and Δt_a are the times required to get the same concentration changes during isothermal ageing, as at succeeding isochronal steps. In the Parkins et al. method [26] several isothermal curves have to be measured, from which Δt_a can be obtained, corresponding to equal concentration changes; E is given by formula (7) (with plus sign). In the Bell–Sizmann method [27] at least two isochronal curves have to be determined, for different heating rates; as in the case of step heating with equal temperature steps and different heating times Δt_1 , Δt_2 , the activation energy E can be calculated from the expression:

$$E = \frac{RT_1 T_2}{T_1 - T_2} \ln \frac{\Delta t_2}{\Delta t_1}, \quad (8)$$

where T_1 and T_2 are temperatures of homological points in the isochronal curves, which can be obtained from the whole heating range.

The methods described do not depend on the assumed model of precipitation.

4. Results

4.1. Precipitation in samples quenched from different temperatures

The influence of solution heat treatment temperature on precipitation in $\alpha\text{Fe-N}$ (samples Fe I') was investigated. For all temperatures isochronal curves were determined for heating rates of 5 K/15 min., in the range from 273K to 433 K. Fig. 1 presents two

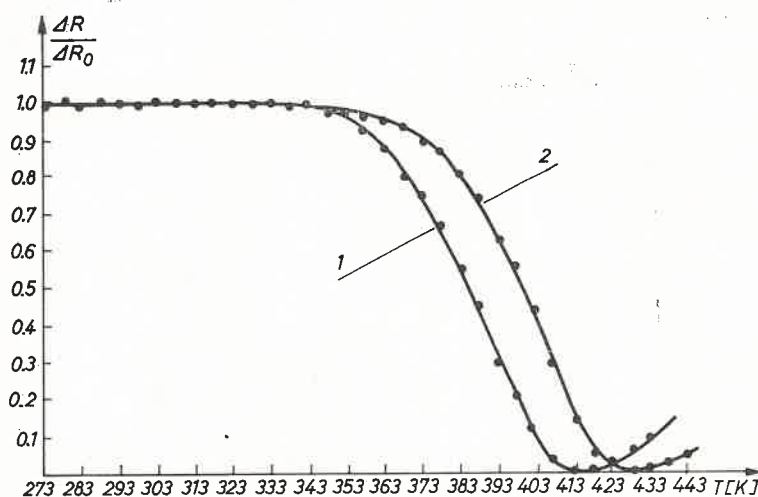


Fig. 1. Isochronal ageing curves of electrical resistance in $\alpha\text{Fe-N}$ samples, annealed at 1223 K/1h; 1 — sample quenched in liquid nitrogen, 2 — sample slowly cooled with the furnace

TABLE I

The temperatures of isochron minima, after solution heat treatment at different temperatures

The temperature of the minimum T_m [K]	The temperature of annealing T_a [K]	Remarks
413	1223	quenched in liquid nitrogen
418	1073	
419	983	
420	873	
421	773	
424	623	
427	1223	slowly cooled with the furnace

isochrons: 1. after heating at 1223 K/1h and quenching in liquid nitrogen, 2. after heating at 1223 K/1h and cooling with the furnace. The curves display distinct minima, the temperatures for which (T_m) depend on the quenching temperature. The minimum in the isochrons is due to the superposition of two effects: 1. precipitation of nitrogen, which depends strongly on the annealing velocity and the number of nuclei, 2. dissolution of precipitates at higher temperatures.

In Table I the temperatures T_m are gathered.

4.2. The dependence of electrical resistivity on the density of dislocations

For measurements described in chapter 4.2 and 4.3 for $t \rightarrow \infty$, the ageing time t was always taken, as that after which no further changes in ρ or R could be observed during the following 24 hours.

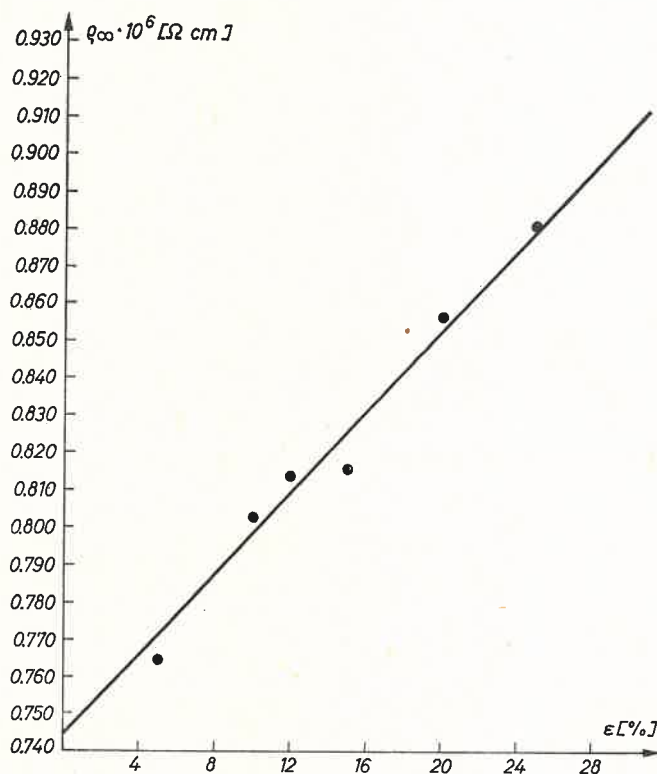


Fig. 2. Dependence of electrical resistivity at the temperature of liquid nitrogen on the degree of plastic straining by rolling; α Fe—N samples aged at 333 K.

Because the next chapter is devoted to the kinetics of nitrogen precipitation in samples with higher dislocation densities, to have a measure of dislocation density, the relation between electrical resistivity (ρ_{∞}) and degree of plastic deformation (ϵ) was determined. Fig. 2 shows the results, obtained for samples FeI''; in order to precipitate interstitial atoms after deformation and to get ρ_{∞} , the samples were additionally aged at 333K.

For the dependence of ϱ_∞ on ε a linear relation was obtained:

$$\varrho_\infty = 0.744 + 0.00545 \cdot \varepsilon \text{ } [\mu\Omega\text{cm}]. \quad (9)$$

The correlation factor of the straight line amounted to $r = 0.99$.

A linear dependence between electrical resistance and the degree of plastic deformation and hence between electrical resistance and dislocation density was obtained for samples containing nitrogen in precipitates, only. For $\varepsilon = 0$ formula (9) gives $\varrho_\infty = 0.744 \mu\Omega\text{cm}$; this value is very close to the result obtained for a high purity Johnson–Matthey iron, not containing N and C: $0.733 \mu\Omega\text{cm}$.

The last remark indicates that in formula (2) $b \cdot c_w$ is very small. Hence, in plastically deformed and aged samples the resistivity (ϱ_∞, R_∞) consists mainly of the thermal and dislocation parts.

Measurements of $\varrho(\varepsilon)$, performed for unaged samples, gave very complicated dependences. In this case, interstitial atoms in solid solution form also influence, in an uncontrolled manner, the results. In [28] a linear relationship for $\varrho(\varepsilon)$ was obtained also. Here, the samples, prepared from very pure iron, contained no interstitial additions.

Formula (9) permitted us to follow the changes of dislocation density in the investigated samples.

4.3. Precipitation kinetics in samples with higher densities of dislocations

4.3.1. Heterogeneous precipitation

In this investigation Fe II samples, deformed plastically by elongation, were used. The method of deformation permitted the observation of heterogeneous precipitation which occurred just after deformation.

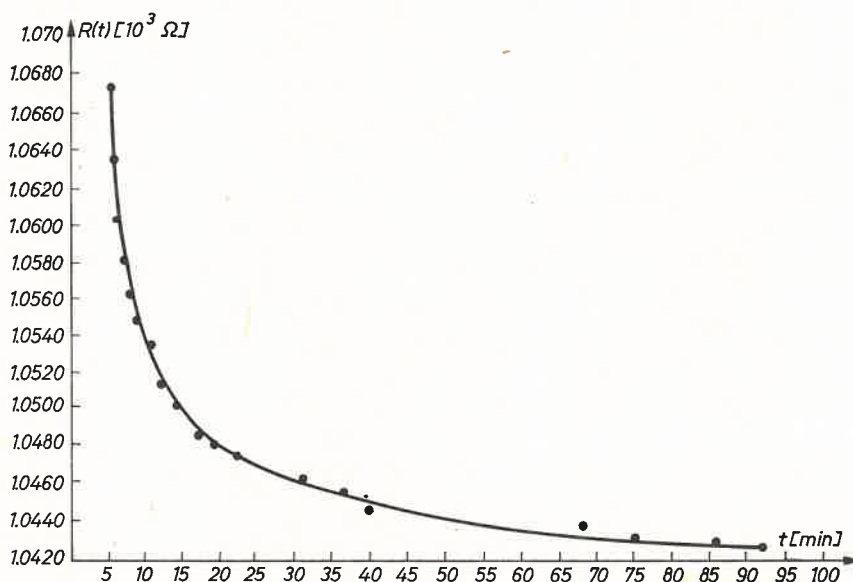


Fig. 3. Isothermal ageing curve in $\alpha\text{Fe-N-C}$, measured at 294.7 K immediately after plastic deformation by elongation ($\varepsilon = 4$ pct)

Fig. 3 presents the isothermal curve, obtained for $\epsilon = 4$ pct at 294.7 K. The curve was analysed by means of formula (4). Fig. 4 shows the dependence of $\ln \left[-\ln \frac{\Delta R(t)}{\Delta R_0} \right]$ on $\ln t$. The slope of the straight line, for t greater than 8 min, gave $n = 0.52 \pm 0.02$; this value confirms that interstitial atoms precipitate heterogeneously at free dislocations.

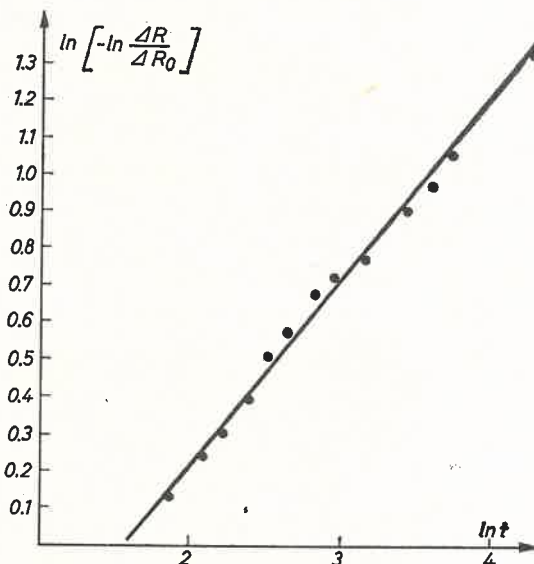


Fig. 4. Analysis of the curve from Fig. 3 by means of formula (4)

4.3.2. Homogeneous precipitation

In this investigation, made on Fe I'' samples, some additional measurements had to be performed. First of all it was shown, that in undeformed Fe I samples the whole nitrogen content could be dissolved at 623 K. It was also ascertained that in samples with a higher density of dislocations, during a single heating at 623 K/1h, a part of the nitrogen was dissolved, whereas the dislocation density did not change at all (it was found that, in agreement with the results obtained in chapter 4.2, the values of R_∞ , determined for aged samples, before and after annealing at 623 K/1h were equal).

After annealing at higher temperatures, dissolution of further amounts of nitrogen occurs. The additional N atoms separate from heterogenous precipitates, as dislocations vanish.

The supersaturation of the solution depends on the dislocation density. On Fig. 5 the dependence of relative resistance changes on ϵ is presented. As the deformation grows, the fraction of nitrogen in solid solution form decreases.

The kinetics of precipitation was investigated in detail for samples strained plastically by 30 pct and then annealed at 623 K/1h. After quenching, isothermal ageing curves between 313 K and 383 K were determined. Fig. 6 depicts e.g. the curve obtained at 313 K;

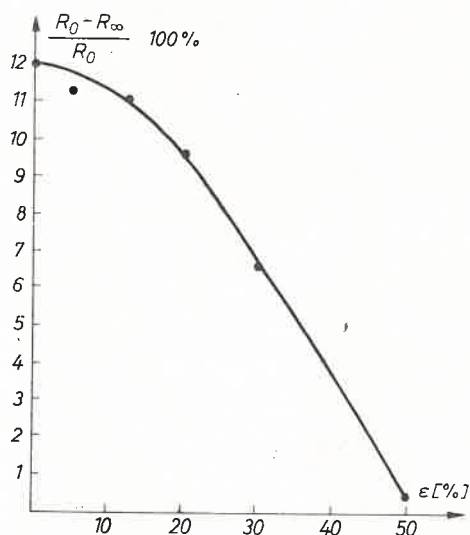


Fig. 5. Dependence of relative changes of electrical resistance on the degree of strain in α Fe—N samples annealed at 623 K/1h; R_0 was measured immediately after quenching R_∞ after ageing at 323 K

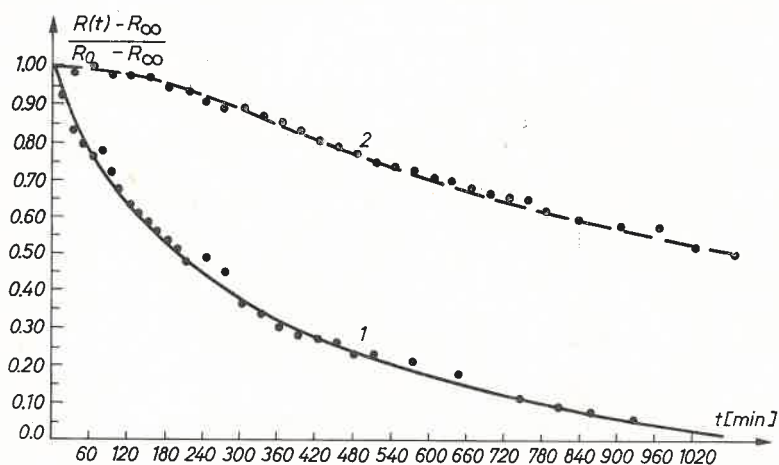


Fig. 6. Isothermal ageing curves of α Fe—N samples; 1 — sample strained by 30 pct and annealed at 623 K/1h, ageing at 313 K, 2 — undeformed sample annealed at 623 K/1h, ageing at 353 K

for comparison the isotherm of ageing at 353 K, measured for an undeformed sample annealed at 623 K/1h, is presented.

Fig. 7 presented the dependence of $\ln [R(t) - R_\infty]$ on time, calculated for the isotherm at 313 K (Fig. 6). A straight line fits the points evidently well; hence, according to formula (6) and (4) with $n = 1$ the precipitation is a homogeneous process. Similar results were obtained for samples with smaller straining.

The results of this chapter, discussed until now, show that during the annealing of deformed samples at 623 K/1h the dislocation density does not change at all and that the

kinetics of precipitation comply with expressions (6) and (4) with $n = 1$. Hence, it seems to be reasonable to assume that during annealing only atoms from homogeneous precipitations dissolve in the matrix.

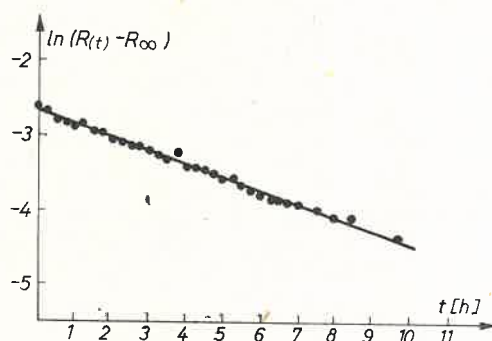


Fig. 7. Analysis of the curve 1 presented in Fig. 6, by means of formula (4)

Data from literature corroborate the last conclusion. The binding energy of N, even in Fe_4N , amounts to 0.35 eV [21]. In Fe_8N , which probably is the precipitate observed in this investigation, the binding is certainly smaller (the same relation occurs between

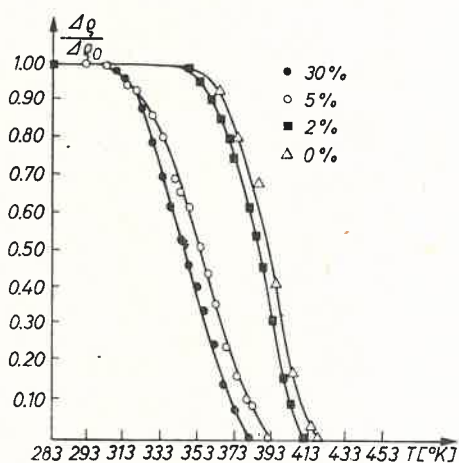


Fig. 8. Normalized isochronal curves for heating rate 5 K/15 min; $\alpha\text{Fe-N}$ samples after different plastic deformations

ε — carbide and cementite: 0.27 eV and 0.5 eV, respectively). Whereas at dislocations nitrogen is bound with an energy of 0.45 eV [29].

Fig. 8 shows isochronal curves determined for samples of series Fe I'' after different straining; deformed samples were annealed at 623 K/1h, quenched in liquid nitrogen and then aged at a rate of 5 K/15 min., in the range from 273 K to 433 K. As the deformation grows, the curves shift to lower temperatures.

4.4. Activation energy of the precipitation process

The activation energy of precipitation was determined for 30 pct strained samples. In calculations the methods described briefly in chapter 3 were applied.

Fig. 9 presents $\ln \Delta t_a$ vs T^{-1} dependence. A straight line fits well the experimental points. The slope of the line gave $E = (0.44 \pm 0.02)$ eV by the Meechan-Brinkman method

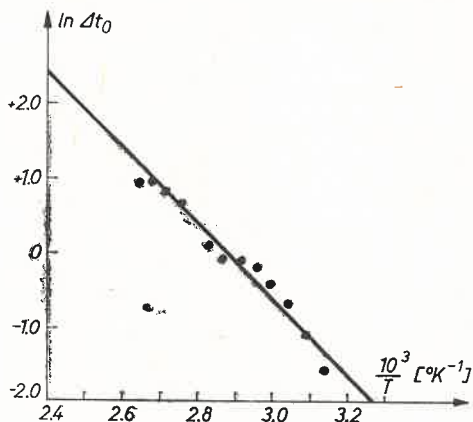


Fig. 9. The $\ln \Delta t_a$ vs. T^{-1} dependence, used in the Meechan-Brinkman method

[25]. The activation energy of precipitation was also calculated by the methods of Parkins et al. and Bell-Sizmann: $E = (0.42 \pm 0.03)$ eV and $E = (0.43 \pm 0.03)$ eV were obtained, respectively.

5. Discussion

The ageing processes, occurring in $\alpha\text{Fe-N}$ alloys in the temperature range 273 K—473 K, originate from the precipitation of nitrogen from the supersaturated solid solution. Fig. 1 and 8 show, that the kinetics of precipitation depends markedly on the presence of structural defects in the matrix.

The higher the temperature of annealing before quenching, the shorter the time necessary for precipitation, and therefore the greater the shift of the isochronal curve to lower temperatures (Table I, Fig. 1). This effect seems to be mainly related to the dependence of vacancy concentration c_v on quenching temperature. And so, for iron $c_v = 5 \times 10^{-13}$ pct at 623 K and $c_v = 1 \times 10^{-4}$ pct at 1223 K (values obtained from vacancy formation energy 1.5 eV [11, 12]).

The effect of shorter precipitation times and of isochronal ageing curves shifting was also observed in $\alpha\text{Fe-N(C)}$ samples irradiated with elementary particles [30-33]. It seems that high vacancy concentration creates many additional regions of nucleation and thus enhances the precipitation process.

Nevertheless, the influence of annealing temperature, observed in this work, seems to be too weak. At higher temperatures there are certainly many vacancies in the samples (e.g. 10^{-4} pct at 1223 K), but during quenching and ageing most of them annihilate.

In samples with higher dislocations densities, nitrogen precipitates at a greater rate, too, as well during homogeneous (Fig. 8), as heterogeneous precipitation (Fig. 3). And so e.g. immediately after deformation by elongation ($\epsilon = 4$ pct) the precipitation at room temperature finishes, practically, as early as after 1.5 h (Fig. 3).

As concerns the time law of precipitation at free dislocations the value of the exponent n is of greatest interest. And so, the isochronal curve in Fig. 3 could be described by means of formula (4) with $n = 0.52$ from $t = 8$ min.

As mentioned in chapter 3, it was shown in [23] that in ageing processes in $\alpha\text{Fe}-\text{C}$, in the range from 263 K to 300 K, two stages could be identified. The first is related to the formation of Snoek atmospheres, the second — to the formation of Cottrell-Bilby atmospheres. For the first $n = \frac{2}{3}$ was obtained, for the second — $n = \frac{1}{2}$. The exponent, determined in this paper for $\alpha\text{Fe}-\text{N}-\text{C}$ — $n = 0.52$, is practically equal to the second value. This means that the heterogenous precipitation, observed in this work, may be connected with the formation of the Cottrell-Bilby atmospheres.

In the case of strained samples ($\epsilon = 30$ pct by rolling), annealed at 623 K/1h and then quenched in liquid nitrogen, the isothermal curve could be described by formula (6) and (4) with $n = 1$ (Fig. 6 and 7). Thus, it can be assumed that N precipitates homogeneously in these samples.

It is also worth-while to pay attention to the fact that the influence of point defects created during plastic deformation, mainly vacancies, on the kinetics of precipitation is no longer as great as previously. In metals, as is known from the literature, vacancies annihilate at the IV stadium of regeneration, which for iron lies between 373 and 553 K. Hence in the investigated samples, the concentration of vacancies at 623 K is given by the thermodynamic equilibrium and therefore is small ($c_v = 5 \times 10^{-13}$ pct). The last conclusion is corroborated by the following results. The values of T_m obtained for two kinds of samples (Table I), the quenched one and the one slowly cooled with the furnace, which seems to be in equilibrium, are very near: 424 and 427 K, respectively, whereas in samples strained by 30 pct the temperature shift amounts to about 50 K (Fig. 8).

The results of this work show also that in samples plastically deformed by 30 pct, annealed at 623 K/1h and then quenched, no nucleation stage could be observed in the isothermal curves, measured between 313 K and 383 K. If such a stage exists, even at 313 K it should be shorter than a few minutes.

Hence, it seems that changes in the kinetics of homogeneous precipitation in samples with higher dislocation densities are probably connected with a shortening of the nucleation stage. Perhaps, in these samples the stress fields of dislocations saturated with nitrogen atoms make the formation of nuclei of precipitates easier, in some regions of the perfect matrix. Diffusion, directed to these regions, causes the precipitates to grow steadily.

The activation energy of homogeneous precipitation in $\alpha\text{Fe}-\text{N}$ samples with higher dislocation densities is much smaller than the activation energy of nitrogen migration: $E = 0.43$ eV, whereas according to [34] $Q = 0.77$ eV. The same result was obtained in [16] for undeformed $\alpha\text{Fe}-\text{N}$ samples.

It is worth-while to emphasize that also in the case of heterogenous precipitation in $\alpha\text{Fe}-\text{C}$, E was observed to be smaller than Q [23].

6. Conclusions

1. On the basis of isochronal ageing curve measurements, it was shown that the annealing temperature of α Fe—N samples, which were subsequently quenched in liquid nitrogen, influences strongly the precipitation of N: the higher the annealing temperature, the greater the precipitation rate.

2. The isothermal ageing curve determined for an α Fe—N sample plastically deformed by elongation ($\varepsilon = 4$ pct), could be described by the $t^{\frac{1}{2}}$ law.

3. The isothermal ageing curves of α Fe—N samples, plastically strained by 30 pct and then annealed at 623 K/1h and quenched, comply with a first order kinetic equation. For these samples, even at 313 K no nucleation stage could be observed.

4. The activation energy of homogeneous precipitation E , calculated for α Fe—N samples strained by 30 pct, is distinctly smaller than the activation energy of migration Q of nitrogen: $E = 0.43$ eV, $Q = 0.77$ eV.

5. It was shown, that for aged α Fe—N samples the electrical resistance is proportional to the degree of plastic deformation.

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