

ON THE MAGNETIC RELAXATION DUE TO CARBON AND NITROGEN IN BCC IRON

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The relaxation strength and relaxation time constants have been measured from 210 to 410 K. It has been found that the relaxation strength is proportional to the reciprocal of absolute temperature and that it can be influenced by the annealing of relaxators during the loss-factor measurement. The relaxation time constants are in accordance with current literature data on carbon and nitrogen diffusion coefficients. A new modification of the disaccommodation measurements is described.

1. Introduction

At the present time the method of magnetic relaxation is used increasingly as an extremely sensitive technique for investigating point defects in ferromagnetic metals [1-3]. The relaxation is usually observed as the disaccommodation i.e., as an isothermal decrease of the initial susceptibility after demagnetization of the sample. The ac techniques are used [4] since they are more sensitive and much more easy to handle than the measurements exploring dc.

The disaccommodation due to carbon and nitrogen atoms in bcc iron originates, according to Néel [5, 6], from a direct non-mechanical interaction between interstitials and the magnetization vector. The interaction is caused by the disturbance of the electronic structure in the neighbourhood of interstitials [7]. Néel's theory has been confirmed by the experimental fact [8] that the magnetostrictive interaction proposed originally by Snoek [9] is negligible.

As Néel's theory implies, the relaxation strength of the orientation after-effect should be proportional to the concentration of relaxators in the sample and to the reciprocal of absolute temperature. The proportionality of the relaxation strength to the concentration of relaxators has been confirmed experimentally for carbon interstitials in bcc iron [10] but the temperature dependence of the relaxation strength as predicted by Néel's theory is not in accordance with the available experimental data [11, 12]. Tomono [11] observed,

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TABLE I

Diffusion coefficients $D = D_0 \exp(-E/RT)$ of carbon and nitrogen in bcc iron

Method	$10^7 D_0$ [m ² /sec]	E [J/mole]	Temp. range [K]	Year	Author	Ref.	Note		
C	mech.	20	84200		1950	Wert	[29]	compilation of different data	
		3.3 3.94	80800 80200	220-620 230-620	1965 1966	Mc. Lellan Lord	[13] [14]		
	mech. + mg.	19	83000	240-440	1967	Seeger	[28]		
	mg.	4.5	84800	260-370	1937	Richter	[34]	including other data	
			74000		1939	Snoek	[9]		
			95500	300-420	1952	Tomono	[11]		
		7.6	82000						
			± 200	240-450	1963	Hampe	[12]		
		19.8	83900 ± 5800	255-265	1968	Lachenmann	[31]		
	mg.	11.4 ± 8	82000 ± 1900	258-288	1971	Walz	[4]		
7.3 ± 3			81600 ± 900	240-275	1971	Kozłowski	[32]		
4.9 ± 1		81300 ± 200	210-410		this work				
N	mech.	3.0	76200		1950	Wert	[27]	compilation	
		4.88	76800	220-1740	1966	Lord	[14]		
	mg.	3.04	68200		1939	Snoek	[9]		
			76200 ± 2100	227-250	1960	Bosman	[33]		
		25.4	80100 ± 5800	241-252	1968	Lachenmann	[31]		
			5.1 ± 1	77000 ± 200	208-330		this work		

for bcc iron with carbon interstitials, a substantial decrease in the relaxation strength at temperatures above 370 K, whereas the relaxation strength measured by Hampe and Widmann [12] on Fe-3%Si with carbon interstitials is above 370 K higher than the theory

predicts. The aim of this investigation is to measure the relaxation strength due to carbon and nitrogen interstitials in bcc iron over a wide temperature range and to clear this discrepancy between theory and experiment by proper analysis of physical processes occurring during measurement.

The second point which is of particular interest is the temperature dependence of the relaxation time constants due to carbon and nitrogen interstitials in bcc iron. These time constants are simply related to the diffusion coefficient of carbon and nitrogen, respectively. The modern values of the carbon [13, 14] and nitrogen [14] diffusion coefficients (see Table I) have been found by summarizing the results of many authors and by using the least squares regression. In this research the magnetic methods did not play any great role. The results for temperatures below 500 K were obtained mostly by mechanical techniques. The values of the carbon and nitrogen diffusion coefficients evaluated from the previous magnetic measurements (see Table I) either differ from these recent values or they have been obtained in a narrow temperature range where the diffusion coefficient varied only very slightly.

2. Experimental

The samples used in the experiments were $6 \cdot 10^{-2}$ m long and $2 \cdot 10^{-3}$ m in diameter. They have been prepared from the zone-refined Johnson and Matthey spectrographically pure iron with the content of substitutional impurities below 15 ppm [15]. The samples have been annealed in dry hydrogen at 1023 K for three days and furnace cooled before carburization or nitrogenation. The carburization took place in a hydrogen-toluene atmosphere at 983 K for 3 hours and the process was terminated by water quenching. The nitrogenation has been performed in a hydrogen-ammonia mixture at 843 K for about 27 hours then the sample was furnace cooled.

The disaccommodation of the real part of the initial susceptibility after demagnetization of the specimen has been measured below room temperature by using the bridge method [16]. The bridge operated at a frequency of 1 kHz. The amplitude of the measuring field was about 0.6 A/m. The sensitivity of the bridge was better than $5 \cdot 10^{-4}$. The specimen was demagnetized in a 50 Hz magnetic field decreasing from $2.5 \cdot 10^3$ A/m to zero in several seconds. The temperature of the sample was stabilized with an accuracy ± 0.05 K.

The loss-factor $\tan \delta = \chi_2/\chi_1$ ($\chi = \chi_1 - i\chi_2$ being the complex susceptibility) has been measured above room temperature without demagnetization of the specimen. The bridge operated at a frequency of 1 kHz or 25 Hz and the temperature during measurement was maintained constant to within ± 0.1 K.

3. Analysis of the after-effect measurements

The magnetic after-effect observed on bcc iron containing carbon or nitrogen in solid solution originates from the interaction of Bloch walls with mobile interstitials besides the interaction with immobile defects as inclusions etc. The relaxation strength and relaxation time constant can be measured over a sufficiently wide range of temperatures only by

the combination of several measuring techniques. Below room temperature three modifications of the disaccommodation measurement have been used, whereas above room temperature the relaxation loss-factor has been measured.

The relaxation strength measured by using ac techniques is influenced by the skin effect. Its influence depends on the frequency of the measuring field, diameter and material constants of the cylindrical specimen [2] and on the concentration of relaxators [17]. The influence of the skin effect on the disaccommodation measurements has been eliminated by the method outlined in paper [17], whereas its influence on the loss-factor measurements has been eliminated by using the results of Ref. [18].

3.1. The disaccommodation measurements

After demagnetization of the specimen the mobility of the Bloch walls is reduced by the reorientation of interstitials into an energetically favorable position and consequently the initial susceptibility $\chi(t)$ decreases as a function of time. As Néel's theory [5, 6] implies, the isothermal increase of the reluctivity $r(t) = 1/\chi(t)$ observed by using ac magnetic field after demagnetization of the specimen is given by [19, 20]

$$r(t) = r(0) [1 + \eta G(t)] \quad (1)$$

where η is the relaxation strength dependent on the absolute temperature and on the concentration c of relaxators

$$\eta = A \frac{c}{T}. \quad (2)$$

The time function $G(t)$ for a simple relaxation process is given by

$$G(t) = 1 - \exp(-t/\tau) \quad (3)$$

with temperature dependent time constant

$$\tau = \tau_0 \exp(E/RT). \quad (4)$$

The relaxation time constant depends on the type of relaxators. For carbon and nitrogen interstitials in bcc iron they are related to the diffusion coefficients by the relation

$$\tau = \frac{a^2}{36D} \quad (5)$$

where a is the lattice constant.

A new modification of the disaccommodation measurements has been used for low temperatures besides the usual measurement of the isothermal and isochronal disaccommodation curves [1-3].

3.1.1. Measurements of the initial slope of isotherms

At low temperatures the relaxation time constant is very long ($\tau > 10^4$ sec) and only a small part of the isothermal increase of the reluctivity can be measured in a reasonable time after demagnetization of the specimen. In this case we have measured only the initial

slope of the isotherms in combination with direct measurement of the relaxation strength. Both measured quantities can be expressed from equations (1) and (3) as

$$\eta = \frac{r(\infty) - r(0)}{r(0)} = \frac{\Delta r}{r(0)} \quad (6)$$

and

$$\left. \frac{dr(t)}{dt} \right|_{t=0} = \frac{\Delta r}{\tau} \quad (7)$$

The relaxed value of the reluctivity $r(\infty)$ can be obtained at any desired temperature by measuring the reluctivity without demagnetizing the sample, whereas the unrelaxed value $r(0)$ can be measured directly after demagnetization only at low temperatures where the bridge can be balanced before perceptible relaxation takes place. By knowing both the relaxed and unrelaxed values of the reluctivity the relaxation strength has been determined from Eq. (6) and the relaxation time constant from Eq. (7).

3.1.2. Measurements of the isotherms

In the temperature range where the time constant of relaxation is of the order 10^3 sec a substantial part of the isotherms can be observed experimentally in a reasonable time after demagnetization of the sample [1-3]. In this case the theoretical curve (1) has been fitted to the measured isotherms by using the method of least squares. The computer fitting yielded both desired parameters, the relaxation strength and the relaxation time constant.

3.1.3. Determination of isochrones

The isochronal relaxation curves may be constructed from the measured isotherms by a method described by Kronmüller et al. [1-3]. The isochronal change of the reluctivity within a fixed time interval $t \in (t_1, t_2)$

$$r(t_2) - r(t_1) = \Delta r [\exp(-t_1/\tau) - \exp(-t_2/\tau)] \quad (8)$$

is determined as a function of temperature. This isochronal change is temperature dependent primarily because of the strong temperature dependence of the relaxation time constant. The given isochronal curve has a maximum at temperature T_m because at lower temperatures the relaxation proceeds very slowly and at higher temperatures the relaxation has almost completely decayed before the time t_1 .

The relaxation strength and relaxation time constant can be determined from the given isochronal curve only at the temperature of its maximum T_m and their temperature dependence must be measured by the variation of t_1 and t_2 , i. e., by constructing another isochronal curve. The relaxation time constant is determined from equation [1-3]

$$\tau = \frac{t_2 - t_1}{\ln(t_2/t_1)}, \quad (9)$$

and afterwards the relaxation strength is calculated from the height of the maximum by using Eq. (8) in combination with the measured value of the relaxed reluctivity. The height and the temperature of maximum of a given isochronal curve have been found by computer analysis. The time constants determined by this method are about 10^2 sec.

3.2. The loss-factor measurements

With ac techniques the relaxation loss-factor $\tan \delta$ is also usually measured by varying the temperature during measurement (or the angular frequency of the measuring magnetic field ω). In this case the interstitial atoms follow the periodic motion of Bloch walls with a phase shift which implies the observed dissipation of energy. The relaxation strength and the time constant of relaxation have been evaluated from the height of the maximum of the loss-factor $\tan \delta_m$. This maximum is situated at a temperature where the relaxation time constant satisfies the equation

$$\omega\tau = \frac{1}{\sqrt{1+\eta}} \quad (10)$$

and its height is given by

$$\tan \delta_m = \frac{\eta}{2\sqrt{1+\eta}} \quad (11)$$

as has been derived [21, 20] from Néel's theory.

4. Experimental results

In Fig. 1., the measured loss-factors are presented as a function of temperature. The measurements have been obtained on carburized and nitrogenized samples by using frequencies 25 Hz and 1 kHz. The peaks are superimposed on the background which has been found to be rather high for 1 kHz in contradiction to the measurements at 25 Hz.

For the carburized sample, at first the disaccommodation and the relaxation loss-factor at 25 Hz have been measured. Afterwards the loss-factor has been measured at 1 kHz by heating the sample above 420 K. Repeating the disaccommodation measurement reveals a lowering of the observed relaxation strength and therefore the tangent of the loss angle has been remeasured once more at 25 Hz. This result which has already been reproducible is reproduced in Fig. 1 and Fig. 2 as the second measurement. In Fig. 2 the measured relaxation strength is shown as a function of the reciprocal of absolute temperature for both carburized and nitrogenized samples. The relaxation strength due to carbon interstitials has been found to be proportional to the reciprocal of absolute temperature giving $\eta_c = (37.5 \pm 0.2)/T$ in the first and $\eta_c = (33.1 \pm 0.2)/T$ in the second measurement. It may be seen from Fig. 2 that the point at 410 K which has been obtained from the measurement of the loss-factor at 1 kHz lies on the line corresponding to the second measurement.

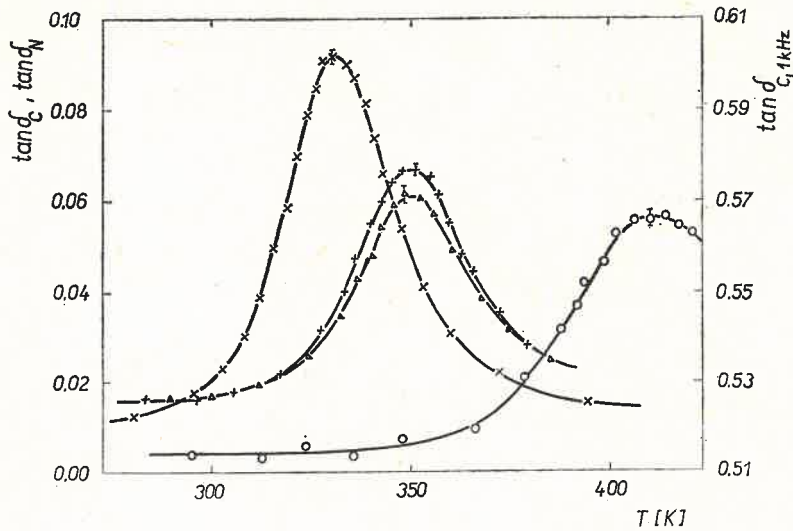


Fig. 1. The measurement of the loss-factor $\tan \delta$ as a function of temperature. \times — nitrogenized sample, $f = 25$ Hz; $+$ — carburized sample, $f = 25$ Hz, first measurement; Δ — carburized sample, $f = 25$ Hz, second measurement; \circ — carburized sample, $f = 1$ kHz

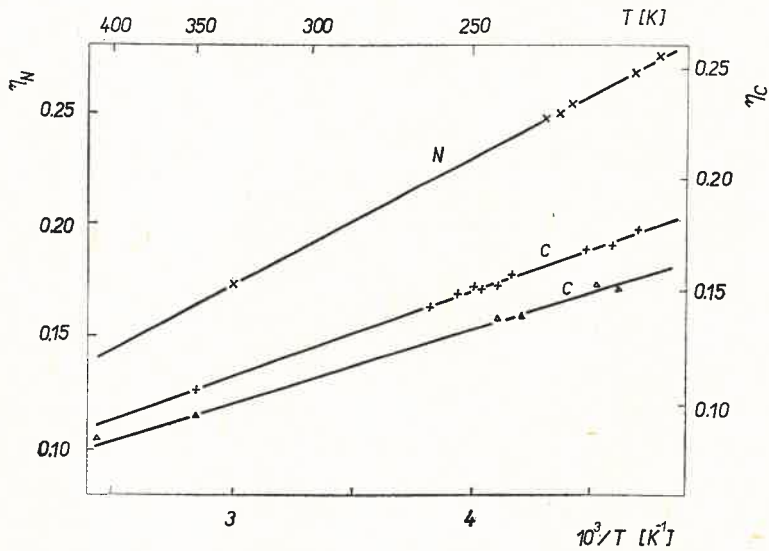


Fig. 2. The temperature dependence of the relaxation strength \times — nitrogenized sample, $\eta_N = 57.5/T$; $+$ — carburized sample, first measurement, $\eta_C = 37.5/T$; Δ — carburized sample, second measurement, $\eta_C = 33.1/T$

The difference between the first and second measurement indicated a lowering of the relaxation strength during the heating of the specimen. Therefore, segregation of carbon atoms from solid solution has been measured on a comparable sample. This sample has been annealed at annealing temperature T_a for 600 sec and the content of carbon in solid

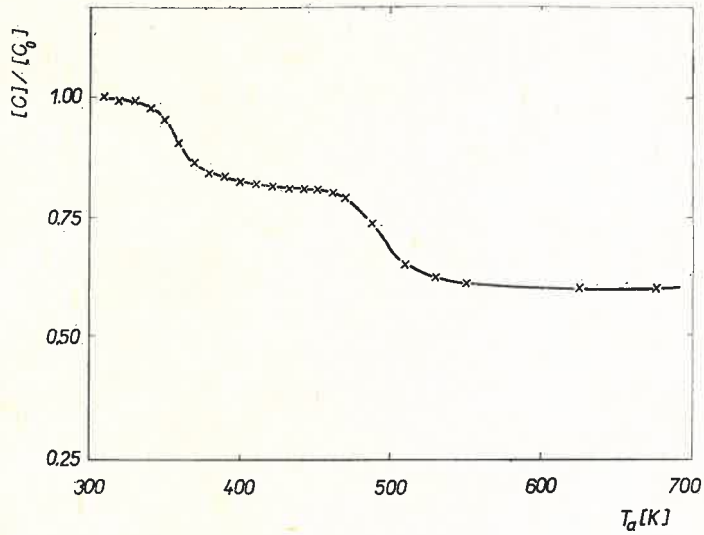


Fig. 3. Fraction of carbon in solid solution as a function of annealing temperature measured by isochronal anneal ($\Delta t_a = 600$ sec). The influence of the skin effect on the measurement has been eliminated by using the results of Ref. [2]

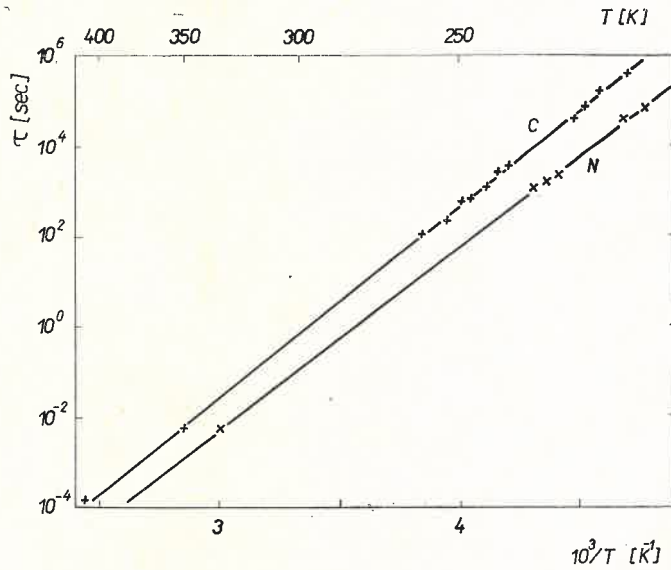


Fig. 4. The temperature dependence of the relaxation time constants. \times — nitrogenized sample; $+$ — carburized sample

solution has been determined by the disaccommodation measurement [10]. The normalized isochronal annealing curve is shown in Fig. 3 as a function of annealing temperature. It may be seen that about 17% of the interstitial carbon atoms anneal out from the solid solution during the first stage of segregation at about 360 K as well as the second stage

at about 490 K. This measurement clearly shows that the interstitial carbon content can change during the loss-factor measurement by the segregation of carbon atoms from the solid solution.

For the nitrogenized sample, first the relaxation loss-factor at 25 Hz and afterwards the disaccommodation has been measured. As it may be seen from Fig. 2, the relaxation strength has also been found to be proportional to the reciprocal of absolute temperature in accordance with equation (2) giving $\eta_N = (57.5 \pm 0.2)/T$.

The relaxation time constants determined by the use of the methods mentioned above are shown in Fig. 4 as a function of the reciprocal of absolute temperature for both carburized and nitrogenized samples. The time constant of relaxation due to carbon interstitials in bcc iron has been found to comply with Arrhenius Eq. (4) with the activation energy $E_C = 81300$ J/mole and with the preexponential factor $\tau_{0C} = 4.6 \cdot 10^{-15}$ sec. The time constant of relaxation due to nitrogen interstitials in bcc iron can be described by the Arrhenius equation with the activation energy $E_N = 77000$ J/mole and with the preexponential factor $\tau_{0N} = 4.5 \cdot 10^{-15}$ sec. The corresponding carbon and nitrogen diffusion coefficients calculated using Eq. (5) are given in Table I together with other published data.

5. Discussion

The relaxation strength and the relaxation time constants have been measured as a function of temperature on carburized and nitrogenized samples of bcc iron in as wide as possible temperature range by the combination of several measuring techniques. Particularly by using the measurements of the initial slope of isotherms, the temperature range of the magnetic after-effect measurements has been extended to lower temperatures.

The experimental results have been evaluated by using Néel's theory of pure orientation after-effect although we deal with the combined after-effect due to carbon or nitrogen interstitials in bcc iron. This after-effect [22] is a combination of the pure orientation after-effect which originates from the reorientation of anisotropic defects and the pure diffusion after-effect [23-25] which originates from the long-range diffusion of isotropic defects inside Bloch walls. The theory of the combined after-effect implies [22] that the description of the combined after-effect by Néel's theory is still valid at times $t \approx 10 \tau$ because the other terms coming into the interaction energy have time constants about five orders of magnitude longer.

The foregoing equations which have been used for the evaluation of the disaccommodation measurements are valid for ac measurements only if the condition $\omega\tau \gg 1$ is satisfied [19, 20]. Our disaccommodation measurements have been performed in an ac magnetic field with $\omega\tau > 5 \cdot 10^5$ well satisfying the condition mentioned above.

On the other hand, our loss-factor and disaccommodation measurements have been influenced by the skin effect; the characteristic constant r_0/r_s being about 2.3 for 1 kHz. Its influence on the disaccommodation, which can be exploited for the determination of the radial distribution of relaxators in the sample [26], has been eliminated by using the results of paper [17]. Its influence on the loss-factor measurements has been found to be very small at 25 Hz whereas at 1 kHz it was notable. At 25 Hz the skin effect gives rise

solely to a small background. The shape of curves presented in Fig. 1 is for 25 Hz in good agreement with theory [21, 20] if we use the relaxation strength and the temperature dependent time constants as determined above. At 1 kHz the background is high and the shape of the peak is modified. The measured loss-factor is no longer a simple sum of the relaxation and skin effect loss-factors as it was approximately valid for 25 Hz [18] and the correct value of the relaxation strength can be obtained only after appropriate elimination of the influence of the skin effect on measurement.

The carbon and nitrogen diffusion coefficients in bcc iron determined from the measurements of the relaxation time constants (see Table I) can be compared with other published data. The diffusion coefficient of nitrogen found here by the magnetic method is in good agreement with the value of Lord and Beshers [14] based on the compilation of nitrogen diffusion data of many authors. The new values are only a little different from the original value of Wert [27]. The diffusion coefficient of carbon found in this paper is in agreement with the value of Lord and Beshers [14] and in good agreement with the results of Mc. Lellan et al. [13] whereas the results of Seeger [28] seem to be somewhat higher. Our result is therefore a new confirmation obtained by the magnetic method that the diffusion coefficient of carbon in bcc iron differs appreciably from the original value of Wert [29] which is associated with the anomaly of the carbon diffusion coefficient at high temperatures [30]. In previous magnetic measurements [4, 31-33], the measured relaxation time constant varied only by about two decades whereas in the present work it varied by about ten decades. Therefore, the results given for the carbon and nitrogen diffusion coefficients are more reliable. Moreover, the diffusion coefficients determined above by using the measurement of the initial slope of isotherms are extremely low (lower than 10^{-25} m²/sec). Such low diffusivities of carbon or nitrogen in bcc iron have not been measured by any other technique up to now, as far as we know.

Our experimental results can be described by a single temperature dependent relaxation time constant corresponding to a well defined value of the diffusion coefficient. On the other hand, it was considered in the literature that the description of the magnetic after-effect measurements requires the introduction of an entire spectrum of relaxation time constants [34, 35]. More recently, the results of magnetic after-effect measurements have been described, like mechanical after-effects, by means of one or a sum of a few discrete relaxation processes [4, 31, 36-38]. We believe that the continuous spectrum of relaxation time constants is associated with the high content of different substitutional impurities in the sample or with the description of the experimental data by using the phenomenological theory [2] instead of the Néel's theory.

The measured relaxation strength has been found to be proportional to the reciprocal of absolute temperature in accordance with Eq. (2) even though some of the constants of Néel's theory coming into the proportionality factor A could be slightly temperature dependent. This proportionality we have found also on other samples of bcc iron with substantially different concentration of relaxators (0.5 — 40 wt-ppm of carbon). But these measurements have been performed below room temperature only by using disaccommodation techniques. In previous experiments [11, 12] this proportionality has not been found. In paper [12] the experimental results have been obtained for Fe-3% Si samples

and the departure of experimental results from Eq. (2) has been ascribed to the influence of substitutional Si-atoms. The measurements of Tomono [11] and our results on a carburized sample revealed the decrease of the relaxation strength at temperatures above 370 K. This decrease is irreversible as indicated by repeated measurements of the disaccommodation and of the 25 Hz loss-factor. It cannot therefore be interpreted as the temperature dependence of the proportionality factor A in Eq. (2) or as to be caused by the influence of the skin effect on the measurements. Besides of the irreversibility of this decrease, the skin effect raises the height of the observed loss-factor peak [18] and if its influence is not properly eliminated the obtained relaxation strength would be higher than the correct value.

The measurements presented in Fig. 3 clearly indicate that the observed decrease of the relaxation strength is to be ascribed to the segregation of carbon atoms from the solid solution during the first loss-factor measurement, i. e., to the lowering of the concentration of relaxators c in Eq. (2). The observed decrease of the relaxation strength between the first and second measurement in Fig. 2 (about 12%) is in good accordance with the measured decrease of the concentration of interstitial carbon atoms during the first stage of segregation in Fig. 3 (about 17%). The segregation of carbon atoms from the solid solution in bcc iron above 360 K has also been observed in literature by using the disaccommodation technique [39-41] or by resistivity measurements [42].

The sample containing nitrogen interstitials has not been quenched and the relaxation loss-factor has been measured while decreasing the temperature before the disaccommodation measurement. As a consequence of this treatment, the results presented in Fig. 2 are unaffected by the segregation of nitrogen atoms from the solid solution during the experiment.

In the previous paper [10], the proportionality between the relaxation strength and the concentration of interstitial carbon in bcc iron has been confirmed experimentally at about 250 K. The following values for the proportionality factors A in Eq. (2) can be obtained from the calibration given in [10]

$$\eta_C = 3.03 \frac{c_C}{T}$$

and

$$\eta_N = 1.39 \frac{c_N}{T}$$

where c_C and c_N denotes the concentration (in at-ppm) of the interstitial carbon and nitrogen, respectively. Now, the content of the interstitial carbon and nitrogen in our samples can be deduced. The nitrogenized sample contained about 41 at-ppm of nitrogen in solid solution and the carburized sample about 12.5 at-ppm of carbon in the first and 11 at-ppm in the second measurement.

For quantitative determination of the concentration of relaxators in the sample the relaxation strength η is measured. It can be determined from the measurements of the

disaccommodation or from the maximum of the loss-factor peak. Comparison of both methods implies that the loss-factor measurements are unadvisable and the disaccommodation measurements which are performed at lower temperatures should be preferred. In contradiction to the loss-factor measurements, relaxators of any kind make only a few atomic jumps during the disaccommodation measurements and cannot therefore anneal out in the course of the experiment.

6. Conclusions

The magnetic relaxation has been measured on the carburized and nitrogenized samples of bcc iron over a broad temperature range 210 K to 420 K by use of several measuring techniques. Particularly, a new modification of the disaccommodation measurement allows the determination of such low carbon and nitrogen diffusion coefficients which have not been measured by any other technique up to now. From the measured temperature dependence of the relaxation time constants, the following values for the carbon and nitrogen diffusion coefficients in bcc iron have been obtained

$$D_C = 4.9 \cdot 10^{-7} \exp(-81300/RT) \text{ m}^2/\text{sec}$$

and

$$D_N = 5.1 \cdot 10^{-7} \exp(-77000/RT) \text{ m}^2/\text{sec}$$

where activation energies are given in J/mole. This result is a magnetic confirmation of the values of carbon and nitrogen diffusivities [13, 14] based primarily on the mechanical techniques.

The measured relaxation strength has been found to be proportional to the reciprocal of absolute temperature in accordance with Néel's theory. The previous discrepancy between theory and experimental results in [11] has been cleared up by the annealing of relaxators during the measurement. The segregation of carbon atoms from the solid solution was observed also in our experiments. On the basis of experimental data published in [10] the following relation between the relaxation strength, concentration of carbon and nitrogen interstitials in bcc iron (in at-ppm) and absolute temperature have been derived

$$\eta_C = 3.03 \frac{c_C}{T}$$

and

$$\eta_N = 1.39 \frac{c_N}{T}$$

The phenomena which can influence the magnetic after-effect measurements have been discussed. It follows that the disaccommodation measurements are fully recommendable in contradiction to the measurements of the loss-factor.

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