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INTERNAL FRICTION AND MAGNETIC AFTER-EFFECTS IN Fe-V-N ALLOYS

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Abstract.—Internal friction and magnetic permeability disaccommodation have been studied for a dozen or so samples of four Fe-V/0.34-1.0%N alloys nitrided in the ammonia atmosphere at 673 K. The obtained curves reveal three distinct bands of magnetic after-effect/A, B, C/ and a small peak of the Snoek relaxation. Band A corresponds to the fast-Weijlering relaxation and consists of three elementary processes with \( \tau \sim 10^{-15} \) s. Band C is identical with the relaxation discovered in 1973 by the K.H. Jack group while band B has not been observed in the literature till now.

1. Introduction.—Migrational relaxations in Fe-V alloys related to the presence of nitrogen in the solid solution have many times been studied since 1953 [1] by the internal friction /IF/ method. The most interesting results have been referred in [1-6]. In samples nitrided at 1223 K beside the Snoek relaxation a second high peak has been observed /the F-K relaxation/ [1-4]. Nitriding of Fe-V samples at 673 to 923 K leads to the appearance of a third relaxational process at yet higher temperatures [5,6].

In paper [7] relaxations in nitrided Fe-V alloys were investigated by means of magnetic permeability disaccommodation /MPD/ measurements. In the present paper are compared results obtained by both methods - IF and MPD.

2. Experimental and investigated samples.—Internal friction has been measured by means of an acoustic relaxometer [8]. Magnetic permeability disaccommodation has been measured on a Maxwell-Vien ac bridge. Isochronal MPD curves have been determined. In the numerical analysis of all the obtained curves the linear spectrum of relaxation times as well as the Wert-Marx law have been applied. In the case of several relaxations the MPD curve is described by the formula:

\[
\Delta(\frac{1}{\chi}) = \sum_i \frac{4}{\chi_i} \left[ \exp\left(-\frac{t}{\Theta_i}\right) - \exp\left(-\frac{t}{\Theta_i}\right) \right]
\]

where \( \Theta_i \) is the time-constant and \( \frac{4}{\chi_i} \) the intensity of the i-th relaxation; the latter value is proportional to the concentration /c_i/ of the i-th relaxator. Because the height of each relaxation peak /A_i/ is proportional to \( \frac{4}{\chi_i} \), the following formula is valid:
Some Fe-V alloys containing 0.34 to 1 % vanadium have been studied. After stress-relief annealing at 1223 K for 10 h and 1073 K for 1.5 h on isochronal MPD and IF curves no relaxational peaks have appeared /curve a in fig.2/. Samples have been nitrided at 673 K in the ammonia atmosphere and then aged for 30 min at higher and higher temperatures. By means of electron microscopy it has been shown that after nitriding the so-called tweed pattern appears resulting from superposition of regularly distributed pre-precipitation plates placed in \{100\} planes of alpha iron. These plates vanished after ageing at 1073 K and simultaneously the microhardness and the coercive force decreased.

3. Results.—Initial investigations have shown that the migrational relaxation spectrum in Fe-V alloys nitrided by the described method is very rich and consists, beside a trace of the Snoek relaxation, of three bands: A,B,C /figs. 1-3/.

\[ A_i = \text{const.} \quad \chi_i = \text{const.} \quad c_i \]  

\( \Delta (\chi^{-1}) \cdot 10^6 \) on temperature for the Fe-V/0.52 \% sample after nitriding at 673 K for 30 min; frequency \( f = 108 \) cps.

\( \Delta (\chi^{-1}) \cdot 10^6 \) on temperature for the Fe-V/0.52 \% sample: a/ after stress-relief annealing, b/ after nitriding at 673 K for 30 min; intensity of the magnetizing field 0.19 A/m; the maximum amplitude of the demagnetizing field 1100 A/m; \( f = 1050 \) cps, \( t_1 = 23 \) s, \( t_2 = 293 \) s.

Band A corresponds to the Fast-Heijering relaxation [1] while C to the relaxation discovered by the K.H. Jack group [5,9].

In the region of band A three discrete IF peaks or distinct inflexion points have usually been observed /figs. 1,3/ while the MPD
measurements have given one broad peak /fig.2/. In the region of B+C bands on IF curves a wide peak has been observed, visibly broadened form the B side while MPD has always split it into two separate relaxational processes.

![Figure 3](image_url)

*Fig.3.* The dependence of $Q^{-1}$ on temperature for the Fe-V/0.34% sample after nitriding at 673 K for 30 min; frequency $f = 87$ cps.

In fig.4 are shown as an instance the results of analysis of isochronal MPD curves obtained for the Fe-V/0.34% sample.

![Figure 4](image_url)

*Fig.4.* The result of analysis of the isochronal MPD curve obtained for the Fe-V/0.34% sample after nitriding for 30 min.

In the F-M region three elementary processes /I,II,III/ have been obtained with activation energies $Q_I = 0.87$ eV, $Q_{II} = 0.94$ eV, $Q_{III} = 0.99$ eV, for $\Theta$, the value $4.5 \cdot 10^{-15}$ s [9] has been assumed. For experimental peaks B and C two elementary processes have been obtained for each of them. However, it should be noticed that in the case of these peaks a continuous spectrum should rather be applied; it results from proposed in the literature mechanisms giving rise to these relaxations /section 4/.

Isochronal ageing of nitrided Fe-V samples in the range 673 - 1173 K resulted in the decay of the Snoek relaxation /process O/ and A,B and C peaks. In fig.5 are presented contributions of peak O.
and peaks $A/_{A_I} + A/_{II} + A/_{III}$, $B/_{A_{IV}} + A/_{V}$ and $C/_{A_{VI}} + A/_{VII}$ to the total KPD curve in dependence on the ageing temperature. Because the peak heights $A_i$ are proportional to the concentration $c_i$ of the $i$-th relaxator /formula (2)/ it can be assumed that $A_i/\Sigma A_i$ state for the atomic fractions of nitrogen taking part in each relaxation.

Fig. 5. The dependence:

Curve $a$ of $A_i + A_{II} + A_{III}$

$A_1^2 + A_2^2$  $A_1^2 + A_2^2$

$A_3^2 + A_4^2$ versus ageing temperature; sample Fe-V/0.34 %/ after nitriding for 30 min.

4. Discussion.- In figs. 1 and 3 on the temperature axis are marked peak temperatures calculated for the Snoek relaxation, processes I, II, III and for bands B and C on the basis of numerically obtained data for MPD curves. It can be seen that IF and MPD measurements give the same results.

Band $A$. Three elementary processes in band $A$, with pre-exponential factors $\sim 10^{-15}$ s, have also been obtained by Welch and Carpenter [4]. They consider that in the case of this relaxation nitrogen atoms order directionally in the vicinity of an immobile V-N pair called by them the VN defect. This defect consists of a substitutional V atom in the lattice point, and of an immobile N atom occupying a tetrahedral site situated as near as possible to the vanadium atom.

Fig. 6 presents the dependence of heights of individual elementary processes, forming peak $A_1$, on the square height of the Snoek peak $(A_o^2)$. It can be seen that there is a linear correlation between $A_i$, $A_{II}$, $A_{III}$ and $A_o^2$ while there is no such dependence for $A_1 = f/A_o^2 / i= 1,2,3$.

It seems possible that the above fact may be explained in the following way. If we assume that the concentration of VN defects is proportional to the number of N atoms responsible for the Snoek relaxation and the concentration of relaxators - single N atoms, related to each peak forming the F-M relaxation /I,II,III/, is proportio-
nal to the concentration of VN defects as well as to the concentration of N atoms, giving rise to the Snoek relaxation, then the following relation should be valid:

\[ A_i = \text{const}. A_0^2 \quad i = I, II, III \]

\[ \text{Fig. 6. The dependence of heights of elementary processes } I, II, III \text{ on the square height of the Snoek peak; the sample Fe-V/0.34 %/} \]

The result of this argumentation seems to be confirmed by the experimental results presented in fig. 6.

**Band C.** Experimental peak C is related to the directional ordering of N atoms in the vicinity of V-N plates situated in \{100\} planes of the matrix \[5,6\]. According to D.H. Jack \[10\] edges of these plates are of interstitial type dislocation loops nature so in their environment a part of free N atoms collects. The binding energy of N atom in the strain field of the plate quasi-dislocations increases its activation energy of migration in consequence of what the peak related to the directional ordering is situated considerably higher than the temperatures of the Snoek and the F-M peaks.

**Band B.** Nitriding of Fe-V samples in conditions given above leads also to the appearance of structural elements intermediate between VN defects and coherent with the matrix V-N plates. The mentioned plate structural elements, containing Fe, V, N atoms, are sometimes called G-P zones of a new type /Fe-V-N zones/ \[5,6\].

The authors of \[6\] have mentioned indeed about additional internal friction related to elements of such a type, however, this IF has not been observed in the form of a separate peak. After-effect bands B and C have already been called \[11\] the plate or J-J relaxations.

The obtained results suggest the following model of structural variations proceeding in Fe-V-N alloys. During nitriding at 673 K in the ammonia atmosphere are formed simultaneously VN defects,
G-P zones of a new type and coherent V-N plates. The isochronal ageing in the temperature range 673 - 1173 K causes migration and clustering of N atoms and VN defects and formation of additional G-P zones of a new type. In consequence of this the intensities of the Snoek and the F-M relaxations decrease and the contribution of peak B increases /fig.5/. As a result of replacement of Fe atoms in zones by V atoms and of ordering of the stoichiometric composition of pre-precipitates new V-N plates appear which with increasing ageing temperature enlarge their dimension and at 1073 K transform into precipitates of incoherent VN nitride. At this temperature the tweed pattern vanishes as well as the migrational relaxations.

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