MECHANICAL LOSS SPECTRUM OF THE Fe-20%Ni-C ALLOYS IN THE TEMPERATURE INTERVAL 300 K–700 K

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Abstract. The present paper studies the mechanical loss spectrum of Fe-20% (mass) Ni-C in the temperature range 100–300 K by means of the X-ray and internal friction method. At temperatures of the order of 250 K relaxation maximum is observed with relaxation parameters – activation energy $H = 10000$ [cal/mol] and relaxation constant $\tau_0^{250} = 6.15 \times 10^{-11}$ [s]. The maximum has behaviour similar to that of the Bordoni maximum in alloys with face-centered cubic lattice, related to the fundamental properties of the dislocations. The relationship established between the values of the microstructural parameters of martensite and the hardening temperatures showed that there is a certain critical size of the dislocation segments, determined by the carbon concentration, below which the relaxation mechanisms defined by Seeger and Pare are not realized.

Keywords: X-ray, internal friction, mechanical loss spectrum, relaxation maximum, relaxation parameters, dislocations, microstructural parameters of martensite.

1. INTRODUCTION

In the Fe-20% (mass) Ni-C alloy, the austenite-martensitic phase transformation proceeds in a diffusion-free path at a low rate [1–3]. The properties of this alloy and the mechanism of $\gamma \Rightarrow \alpha$ martensitic phase transformation are based on the microstructure of the solid solution, determined by its dislocation structure of point defects and the atomic configuration of matrix iron atoms and those of nickel and carbon.

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The aim of the present article is to study the behaviour of the highly stressed and rich in structural defects matrix as a consequence of the integral method of internal friction.

2. MATERIAL AND METHODOLOGY

The alloy, Fe-20% (mass) Ni-135 ppm carbon, is made in a vacuum furnace from materials of high purity. The dynamic relaxation spectrum (internal friction) of the test bodies is recorded with apparatus and by a method analogous to that described in [4]. The studies are performed under cooling and subsequent heating at a rate of 2 °C/min in the temperature range 300–700 K, where the course of α -phase transformation is excluded and the dynamic relaxation spectrum includes only effects from the structural states of the solid solution.

The samples are hardened from the austenitic region in quartz ampoules with argon, in water, at temperatures of 680, 740, 800 and 900 °C.

At three operating frequencies the kinetic parameters of relaxation are determined by using the known dependence of the arenius type [5]:

$$\tau = \tau_0 \exp(H/RT), \quad (1)$$

where τ is the relaxation time [s]; T – the absolute temperature K; H – the activation energy [cal/mol]; τ_0 – the relaxation constant [s]; K – the fundamental gas constants [cal/mol.K].

The analysis of the width of the relaxation maximum is performed according to the methodology described in detail in [5]. It is based on the fact that if the width of the relaxation maximum is greater than that corresponding to one time relaxation process, i.e. there is a distribution of relaxation times with a distribution parameter β .

3. EXPERIMENTAL RESULTS

A typical temperature dependence of the internal friction after hardening of the alloy is shown in Fig. 1, where there are two relaxation maxima – the first at temperatures of the order of 375 K and the second at 485 K.

While the 485 K maximum is the well-known in the literature, Snoek–Koester (S–K) maximum in martensite [5, 6], the relaxation maximum observed from its low-temperature part is not unambiguously defined in the world literature.

Fig. 1. Temperature dependence of internal friction at four hardening temperatures

Our research has shown that the main features of the two maxima can be briefly defined as follows:

- Two maxima exist in the presence of atoms of incorporation into the alloy, in this case – carbon atoms;

Fig. 2. Determination of the kinetic parameters of the relaxation maxima

- The two maxima appear after hardening, i.e. embedding atoms must be in the martensite lattice;
- Heating from temperatures above 700 K leads to the disappearance of both maxima;
- The kinetic parameters of relaxation for both maxima are: $H_{375} = 15640$ [cal/mol]; $\tau_0^{375} = 1.45 \times 10^{-10}$ [s]; $H_{485} = 34117$ [cal/mol]; $\tau_0^{485} = 1.65 \times 10^{-16}$ [s]; $\beta^{375} = 1.75$ and $\beta^{485} = 3.85$, Fig. 2(a, b).
- The distribution parameters β and the heights of the relaxation maxima do not depend on the hardening temperature, as the concentration of carbon in the alloy is lower than the maximum solubility of carbon at the considered hardening temperatures.

4. DISCUSSION OF THE EXPERIMENTAL RESULTS

The first studies of Ke in an alloy similar to the one studied by us show the presence of two relaxation maxima in the temperature range 433 K and 523 K [7]. The authors also established the presence of a 373 K maximum, hidden by a 433 K maximum, but due to the complicated division of the maxima it is not given a specific interpretation. A similar 383 K maximum was found by other authors in similar alloys after cold plastic deformation [8]. It has been suggested that this maximum is caused by the interaction of carbon with point structural defects and high internal stresses, analogous to the interaction of the intercritical atoms with the dislocations at the Snoek–Koester maximum.

The presence of 433 K maximum of martensite, established by some authors [9] in relatively carbon-rich iron-nickel alloys, is associated by Ke with the release of low-temperature ε -carbide in martensite. Klems and co-workers [9], as well as the studies we conducted, show that they challenge this assumption, proving that the release of low-temperature ε -carbide is not associated with the appearance of a relaxation maximum. The 433 K maximum of Klems has features close to those obtained by us for the 375 K maximum of nickel martensite. The common properties are:

- The two maxima are caused by the presence of carbon;
- They appear in deformation or hardening;
- The height of the maximum is less sensitive to the carbon content in the alloy, compared to the S–K maximum;
- The two maxima have a width corresponding to a relaxation time.

The differences between them are mainly defined by two points:

- A maximum of 433 K occurs only in alloys with high carbon content compared to alloys in which a maximum of 375 K appears;

- Kinetic parameters of relaxation at 433 K maximum are characterized by higher activation energy and lower relaxation constant $H_{375} = 15640$ [cal/mol], $H_{433} = 2300$ [cal/mol], $\tau_0^{375} = 10^{-10}$ [s], $\tau_0^{433} = 10^{-13}$ [s].

In such a case, it can be assumed that the 433 K maximum in Fe-20% Ni alloys established in the literature is of a similar nature to that of the 375 K maximum obtained by us. The obtained value of the activation energy speaks of a mechanism that is related to the diffusion mobility of carbon in the martensite lattice. The high content of nickel atoms makes it impossible for the pure relaxation mechanism of jumps of carbon atoms in the crystal pores of the iron lattice under the action of the applied micro stresses. Conditions are created for more complex mechanisms of relaxation. The lower value of the activation energy is thermodynamically justified by the influence of the environment of the nickel atoms. Like Si and Co, they increase their diffusion mobility. On the other hand, the value of the relaxation constant indicates an existing collective relaxation atomic process. It can be assumed that the mechanism of relaxation rests on the so-called $i-s$ pairs, i.e. atomic complex – intercritical atom-substitution atom.

In Face-Centered Cubic (FCC) metals, this mechanism is sufficiently studied and can be relatively easily modeled. In Body Centered Cubic (BCC) lattices, $i-s$ pair cannot switch to an equivalent configuration in the position of a close neighbor of the embedding atom during the relaxation jump. The theory of relaxation of $i-s$ pair [5] proposes a simplified situation in which the pair is located only in a tetragonal configuration – nn-pair and orthorhombic with an axis of symmetry $[110]_\gamma$, so-called – nnn-pair, Fig. 3.

Fig. 3. Possible configurations of the atoms in the Fe-Ni-C system:
 (a) arrangement of atoms in $i-s$ configuration (nn-pair);
 (b) arrangement of atoms in $s-i-s$ configuration (nnn-pair)

From the selection rule it is established that in this case three relaxation maxima are possible [5]. The presence of nickel atoms in the alloy with a diameter smaller than the diameter of the iron atoms causes local stresses in the crystal lattice that interact with the stresses created by the embedding atoms and the externally applied stress. This causes local jumps in the carbon atoms, which are the more mobile element $i-s$ pair.

In a simplified form, the relaxation mechanism of $i-s$ pair is shown in Fig. 3(a). Taking into account the atomic weight of nickel and iron, as well as the coordination number of the BCC lattice, it can be calculated 1.71 nickel atoms per elementary cell, i.e. the model involving one nickel and one carbon atom in the atomic relaxation complex is very probable. However, it is not excluded that at this high concentration of carbon atoms, at which there is a 433 K relaxation maximum, there is a mechanism in which two nickel atoms participate.

Similar relaxation processes are known in the literature for other Nb-Zr-N complex alloys [10, 11]. For these alloys, the possible configuration is shown in Fig. 3(b). These are respectively orthorhombic defects with an axis of symmetry $[100]_\gamma$. The two mechanisms exist in similar alloys in certain quantitative ratios and distribution of relaxation times established by the mass balance equation [5].

Assuming the mechanism for the existence of $i-s$ and $s-i-s$ relaxation complexes, it is clear why the microstructural parameters of martensite, which change in a certain way, do not affect the height of the relaxation maximum when quenched by different temperatures. The disappearance of the maximum on heating is probably related to the course of annealing processes in the martensite, which lead to the separation of the cementite in the solid solution, thus releasing the martensite solid solution from the carbon in its lattice.

The presence of a 485 K maximum in martensite according to kinetic data corresponds to the so-called 200 °C maximum of S-K in deformed α -Fe. This maximum has been studied in detail in the literature and can be briefly formulated as a manifestation in the relaxation relationship of the interaction of the intercritical atoms with the dislocations in the martensite.

Our studies have shown the relationship between the microstructural parameters of martensite and the hardening temperatures [4]. However, these results show certain discrepancies between the proposed mechanisms of the S-K maximum and our experimental results, namely: changes in the dislocation structure of martensite determined by the value of the hardening temperature do not affect the width and height of the S-K maximum. The heating of the alloys to high temperatures and their slow cooling from high temperatures

practically leads to the disappearance of the maximum, which is associated with the release of carbon from the lattice of martensite and the formation of cementite.

5. CONCLUSION

The studies of the temperature dependence of the internal friction in the temperature range 300 K–700 K of Fe-20% Ni alloy can be summarized as follows:

The established relaxation maximum at 375 K with relaxation parameters $H_{375} = 15640$ [cal/mol], $\tau_0^{375} = 1.45 \times 10^{-10}$ [s] showed that the relaxation mechanisms indicated by Ke [7] and Klems [9] are unlikely, and it is logical to assume that the appearance of the maximum is caused by the presence of $i-s$ complexes in the structure of martensite.

The presence of S–K maximum of martensite at temperatures of 485 K with relaxation parameters $H_{485} = 34117$ [cal/mol]; $\tau_0^{485} = 1.65 \times 10^{-16}$ [s] is well known in the literature, as well as its mechanism. However, it was found that the change in the microstructural parameters of martensite with a change in the hardening temperature does not affect the width and height of the maximum, which is normally in contradiction with the accepted mechanism of relaxation of the S–K maximum.

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