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Internal friction and Young's modulus in Invar Fe–Ni–C alloys

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The temperature dependence of internal friction (IF) and the amplitude-dependence of IF (ADIF) in kHz frequency range were studied in the f.c.c. Invar Fe–Ni and Fe–Ni–C alloys after homogenization at 1373 K and ageing at 973 K. The relaxation carbon peak at 520–550 K has been revealed on the temperature dependence of IF for the Fe–Ni–C alloys. The effect of nickel and carbon content as well as of heating on the peak intensity, the value of ADIF and the Young's modulus was studied.

Изучена температурная зависимость внутреннего трения (ВТ) и амплитудная зависимость внутреннего трения (АЗВТ) в кГц диапазоне частот в ГЦК сплавах Fe–Ni и Fe–Ni–C после гомогенизации при 1373 K и отжига при 973 K. На температурной зависимости ВТ сплавов Fe–Ni–C обнаружен релаксационный углеродный пик при 520–550 K. Исследовано влияние содержания никеля и углерода, а также нагрева на интенсивность максимума ВТ, величину АЗВТ и модуль Юнга.

The Invar Fe–Ni alloys are used as materials for precision elements of instruments and devices. To ensure high accuracy of these elements in operation, they should exhibit high mechanical properties and quality factor, i.e. a low level of internal friction (IF). One of effective ways to improve mechanical properties of Invar is alloying with carbon. The IF technique is widely used to study the behavior of interstitials in metals, particularly of carbon. The IF nature in Invar alloys was not studied enough [1–4] and there is no clear idea in the problem of the quality factor improvement. The IF in Fe — 36 % Ni — 0.25 % C Invar alloy in Hz frequency range was studied by I.B.Kekalo [2, 4] where the magneto-diffusion relaxation peak of IF at a temperature below the Curie point was first revealed there. This peak coincides with the commonly observed carbon peak, so-called Finkelshtein-Rozin one, and is not separated from magnetic contribution.

Further studies of the magneto-elastic relaxation effect in Invar-type Fe–Ni–C alloys of different compositions within a higher frequency range (kHz) are pretty important, because they will allow the observation of carbon peak in paramagnetic state at temperatures above the Curie point. Moreover, in order to find new Invar compositions with the required physical and mechanical properties the regularities in changing of elastic and non-elastic properties variation of carbon-containing Invar alloys under heat treatment should be studied. To date, the influence of carbon on Invar properties of Fe–Ni–C alloys is not cleary so far. The new studies in this direction will provide understanding of the mechanism of anomalous behavior of Invar alloys.

In this work, the amplitude and temperature dependences of IF and the Young's modulus in the Fe — 30 % Ni — 1.31 % C, Fe — 36.1 % Ni — 0.55 % C and Fe — 35.9 % Ni — 0.61 % C alloys annealed at 1373 K in vacuum and subsequently quenching at 973 K during the measurements were studied. The carbon-free alloys with the close Ni concentration (Fe — 29.2 % Ni and Fe — 36 % Ni) were studied in order to compare with the carbon-containing ones. The logarithmic decrement of natural vibrations (δ) was determined in 1–

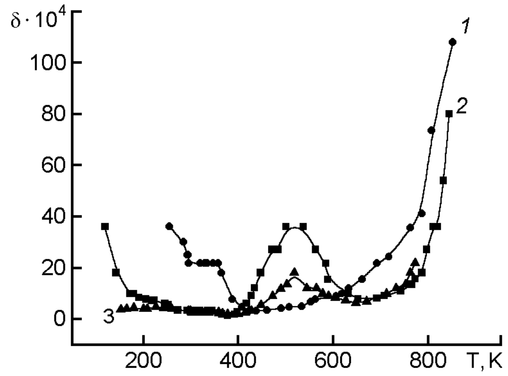

Fig. 1. Temperature dependence of internal friction in Fe–Ni and Fe–Ni–C alloys quenched from 1373 K: Fe — 36 % Ni (*1*), Fe — 36.1 % Ni — 0.55 % C (*2*), and after the following ageing of the Fe — 36.1 % Ni — 0.55 % C alloy at 973 K during the first measurements (*3*).

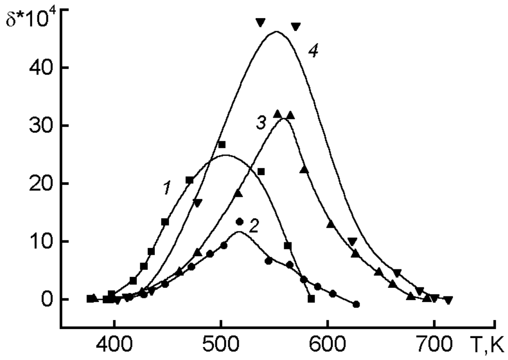

Fig. 2. Internal friction peak in the Fe–Ni–C alloys quenched from 1373 K: Fe — 36.1 % Ni — 0.55 % C, f_{max} = 1503 Hz (*1*), Fe — 36.1 % Ni — 0.55 % C, f_{max} = 1524 Hz, 1373 K + 973 K during measurement (*2*), Fe — 35.9 % Ni — 0.61 % C, f_{max} = 1937 Hz (*3*), Fe — 30 % Ni — 1.31 % C, f_{max} = 1950 Hz (*4*). These peaks were obtained from experimental curves by subtraction of background.

2 kHz frequency range for cylindrical samples of $d = 5$ mm diameter and $l = 100$ mm length. The δ values of the order of 10^{-4} were measured to within 1 %.

The Young's modulus (E) was determined using formula [1]:

$$E = 1.606 \cdot 10^{-10}\left(\frac{l}{d}\right)^4 \frac{m}{l} f^2 \ (GPa), \tag{1}$$

where l, d are the sample length and diameter, cm; m is the sample mass, g and f_0 is the sample natural frequency, Hz. The natural frequency was measured by means of frequency meter within the accuracy of ±0.1 Hz. However, the temperature variation was approximately ±2°C, therefore, the accuracy of frequency measurements was lower but not worse than ±1 Hz. The absolute error in the Young's modulus estimation was determined by the accuracy of the sample size measurements. The mean square error in determination of absolute value of E was ±0.25 GPa. The IF was measured in the absence of magnetic field. The sample vibration amplitudes at the measurements of IF temperature dependence was $2 \cdot 10^{-6}$.

The temperature dependences of IF for the studied alloys are presented in Fig. 1 and Fig. 2. The following peculiarities were revealed in IF curves. In the carbon-containing alloys, a maximum in the 520–550 K temperature range is observed. The peak height grows with the increasing carbon concentration (Fig. 2) and decreases after heating of the Fe — 36.1 % Ni — 0.55 % C alloy in the 773–873 K range (Fig. 1, curve 3). The reduction of damping after heating is accompanied with the Young's modulus increase. This difference of Young's modulus of the Fe — 36.1 % Ni — 0.55 % C alloy is of about 0.8 %, that of the Fe — 35.9 % Ni — 0.61 % C, 0.85 % and Fe — 30 % Ni — 1.31% C, 2.35 % as compared to that for quenched samples. The position of IF peak shifts towards lower temperatures with the decreasing frequency. The process activation energy, H, was calculated using the Marx-Wert formula [1]:

$$H = RT_{\max}\ln\frac{kT_{\max}}{hf_{\max}}, \tag{2}$$

where T_{max} and f_{max} are the IF peak temperature and frequency, respectively. The calculations have shown that the activation energy lies in the 1–1.1 eV range which is close to the activation energy of carbon diffusion in a f.c.c. Fe–Ni alloy [2]. The comparison of behavior of the δ(T) curve with the temperature dependence of the Young's modulus shows that the IF peak position either coincides with anomalous behavior of modulus E in the Fe — 35.9 % Ni — 0.61 % C alloy (Fig. 3) due to transition from ferromagnetic to paramagnetic state or this peak lies in the linear range of $E(T)$ for the Fe — 30.0 % Ni — 1.31 % C alloy

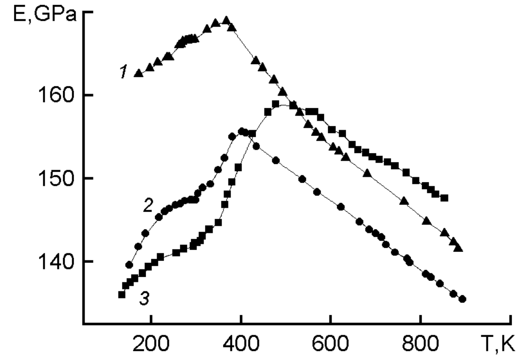

Fig. 3. Temperature dependence of the Young's modulus of Fe–Ni and Fe–Ni–C alloys quenched from 1373 K: Fe — 29.2 % Ni (*1*), Fe — 30 % Ni — 1.31 % C (*2*), Fe — 35.9 % Ni — 0.61 % C (*3*).

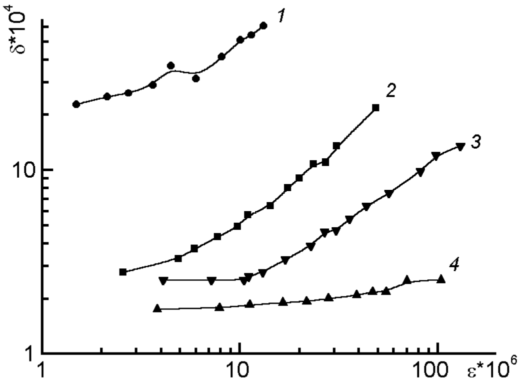

Fig. 4. Amplitude dependence of internal friction in Fe–Ni and Fe–Ni–C alloys quenched from 1373 K: Fe — 36 % Ni (*1*), Fe — 36.1 % Ni — 0.55 % C (*2*), Fe — 30 % Ni — 1.31 % C (*3*), Fe — 29.2 % Ni (*4*).

at the temperature above the Curie point (Fig. 3). We assume that IF maximum at the temperatures 520 to 550 K is a result of directed ordering of C atoms in the lattice of Invar under the repeated variable stresses. Such a peak was observed in austenitic carbon-containing steel and explained by Finkelshtein and Rozin [3]. The height of this peak is proportional to carbon concentration in the solid solution.

The anomalous behavior of IF $\delta(T)$ was revealed at temperatures lower than 250 K (Fig. 1). The increase of damping is much more pronounced for quenched Invar Fe — 36 % Ni and Fe — 36.1 % Ni — 0.55 % C alloys (Fig. 1, curves 1, 2). The increase of IF in the Fe — 29.2 % Ni and Fe — 30 % Ni — 1.31 % C at low temperatures is weaker. It is worth to note that addition of carbon into the alloy with approximately 36 % Ni shifts this anomaly to lower temperatures (Fig. 1, curve 2) and after heating to 973 K, the damping increase disappears (Fig. 1, curve 3). A similar growth in IF with the decreasing temperature was observed previously in an Invar alloy, but its nature was not discussed [4].

The behavior of the $\delta(\varepsilon)$ and $E(\varepsilon)$ curves for the Fe — 30 % Ni — 1.31 % C alloy after the low-temperature measurements follows the dependences for the quenched sample. In the carbon-free alloy (Fe — 29.2 % Ni), this behavior is considerably different, i.e. the damping increases and the Young's modulus decreases. These changes result from martensitic transformation [5]. In martensitic phase, the level of elastic energy losses is higher due to a high level of elastic stresses [6]. In the carbon-containing alloy, Fe — 30 % Ni — 1.31 % C, this transformation does not occur and the parameters of IF and the Young's modulus remain unchanged.

A reduction of the Young's modulus with the temperature decreasing in ferromagnetic range was observed in the Fe — 30 % Ni — 1.31 % C alloy which is typical for Invar, i.e. the thermal coefficient of E is positive [7]. The ferromagnetic-paramagnetic transition has a smeared character that is attributed to Invar alloys [8, 9]. As shown in Fig. 3, the alloying of alloy with carbon Fe — 30 % Ni — 1.31 % decreases the Young's modulus and therewith shifts the Curie point to higher temperatures. The result is consistent with study of elastic moduli in Invar Fe–Ni–C alloys [10] and with the data on magnetic properties [7, 8].

The obtained results on the amplitude dependence of IF (ADIF) in the Fe — 36.1% Ni — 0.55% C alloy (Fig. 4) are in good correlation with the results on thermal dependence of δ and E. In particular, a carbon additive decreases the ADIF level (Fig. 4, curve 2). As established, the heating during the $\delta(T)$ measurement in 300–973 K interval reduces also the level of the amplitude dependence losses.

It is known that the vibration damping in the kHz frequency range is caused by motion of dislocations. In ferromagnetic materials, the magneto-elastic damping resulting from reversible and irreversible displacement of domain walls under elastic stresses (magneto-mechanical hysteresis associating the stress σ with the magnetostriction component of strain, ε_λ) becomes superimposed on dislocation damping. These magnetico-mechanical losses are determined by the hysteresis loop area, similar to determination of the magnetic losses from magnetic hysteresis [1]. The damping at smaller vibration magnitude (10^{-6}–10^{-4}) is defined by the following relationship [1]:

$$\delta_h = \frac{4}{3} bE\sigma = \frac{4}{3} bE^2\varepsilon, \tag{3}$$

where b is the magneto-mechanical constant corresponding to the Rayleigh factor for magnetic hysteresis, and $\varepsilon = \varepsilon_{elastic} + \varepsilon_\lambda$.

Like to behavior of the Rayleigh constant, the hysteresis damping depends on lattice defects, inclusions of the second phase, the internal stresses, etc. The high level of amplitude-dependent damping within the range of amplitude strength ε 10^{-6}–10^{-4} (Fig. 4, curves 1 and 2) points to the fact that the magneto-mechanical losses provide the main contribution into IF in these alloys at temperatures below the Curie point. As it was mentioned above, the heating of the Fe — 36.1 % Ni — 0.55 % C alloy to 973 K during the measurements results in reduced height of carbon maximum (Fig. 1 and Fig. 2) and simultaneously suppresses the amplitude. We assume that the observed changes in the IF parameters are caused by redistribution of carbon atoms and graphitization [9] resulting in pinning of domain walls and reduction of mobility thereof.

A considerably different picture is observed for the Fe — 30 % Ni — 1.31 % C Fe — 29,2 % Ni alloys. The ADIF in the Fe — 29.2 % Ni and Fe — 30 % Ni — 1.31 % C alloys is much lower than in the Fe — 36 % Ni and Fe — 36.1 % Ni — 0.55 % C compositions (Fig. 4). We suppose that this is attributed to the decrease of magnetic contribution into the total elastic energy losses with the Ni concentration is reduced. The fact has arrested our attention is that, in contrast to the case of standard Invar where ADIF decreasing is observed (Fig. 4, curves 1 and 2), alloying of the alloy containing ~30 % Ni with carbon results in 2–5 times ADIF increasing (Fig. 4, curves 3 and 4). As it has been shown in [7, 9, 11], carbon addition results in magnetic ordering and formation of inhomogeneous magnetic structure in the Fe–Ni–C alloys containing approximately 30 % Ni. This means that carbon in the solid solution enhances magnetic contribution into damping of elastic vibrations. However, some fraction of carbon reduces the mobility of the domain walls leading to the lower than in the Fe — 36.1 % Ni — 0.55 % C alloy elastic losses (Fig. 4).

Thus, an internal friction relaxation peak at 520 to 550 K and decreasing its intensity after ageing at 973 K have been revealed in the f.c.c. Fe — 30 % Ni — 1.31 % C and Fe — 36.1 % Ni — 0.55 % C alloys. This peak height is proportional to the carbon concentration in the solid solution. The activation energy H is estimated to be 1–1.1 eV that corresponds to the activation energy of carbon diffusion in a Fe–Ni austenite. The Young's modulus of the Fe — Ni and Fe — Ni — C alloys within the 100–900 K temperature range has been calculated. It has been shown that carbon decreases E approximately by 5 % and this change depends on Ni concentration. The temperature dependence of E is not monotonous and shows anomalous behavior near the temperature of magnetic transition. The increase of carbon content increases the Curie temperatures of Invar Fe–Ni–C alloys. The ADIF losses in the Fe — 36 % Ni and Fe — 36.1 % Ni — 0.55 % C alloys grow at small strains $\varepsilon = 10^{-6}$, therewith, addition of C reduces these losses approximately by one decimal order. The ADIF level is much lower in the alloys containing lower Ni concentration (Fe — 29.2 % Ni, Fe — 30 % Ni — 1.31 % C), contrary alloying with carbon results in increased ADIF losses caused by magnetic contribution. However, they remain by more than one order less as compared to the alloys with 36 % Ni.

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Внутрішнє тертя та модуль Юнга в інварних сплавах Fe–Ni–C

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Досліджено температурну залежність внутрішнього тертя (ВТ) та амплітудну залежність ВТ (АЗВТ) у кГц діапазоні частот у ГЦК сплавах Fe–Ni та Fe–Ni–C після гомогенізації при 1373 К та відпалу при 973 К. На температурній залежності ВТ сплавів Fe–Ni–C виявлено релаксаційний вуглецевий пік при 520–550 К. Досліджено вплив вмісту нікелю і вуглецю, а також нагріву на інтенсивність максимуму ВТ, величину АЗВТ та модуль Юнга.