Internal friction in Invar Fe–35 % Ni alloy after combined SPD by hydroextrusion and drawing

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Received October 5, 2013

The temperature dependence of internal friction on frequency of ~3 Hz and ~60 Hz were studied in Invar Fe-35.0%Ni-0.49%Mn-0.03%C alloy after annealing at 1373 K and combined severe plastic deformation (SPD) by hydroextrusion and subsequent drawing with the total degree of accumulated deformation $\varepsilon_{\Sigma} = 4.69$. The reduction of the relaxation IF peak intensity is observed in deformed sample of the alloy at ~380 K (the activation energy 0.82-0.93 eV) due to decreasing of mechanical and magnetomechanical relaxation losses the contribution of which is renewed after ageing of the deformed alloy. The damping growth nearby temperatures 780-820 K after combined SPD and partial its reduction after ageing of the alloy were revealed. The estimated activation energy of the relaxation process is 1.82-1.97 eV.

Изучена температурная зависимость внутреннего трения (ВТ) на частоте ~3 Гц и ~ 60 Гц в инварном сплаве Fe-35.0%Ni-0.49%Mn-0.03%C после гомогенизации при 1373 К ниже и выше Т С и последующей комбинированной интенсивной пластической деформации (ИПД) методами гидроэкструзии и последующего волочения с суммарной степенью накопленной деформации $\varepsilon_{\Sigma} = 4.69$. В деформированном образце сплава при 380 К наблюдается снижение интенсивности углеродного релаксационного пика ВТ (энергия активации 0.82-0.93 эВ) за счет уменьшения механических и магнитомеханических релаксационных потерь, вклад которых восстанавливается после отжига. После комбинированной ИПД сплава обнаружен рост затухания колебаний вблизи температур 780-820 К и частичное их снижение после отжига. По оценке энергия активации релаксационного процесса составляет 1.82-1.97 эВ.

Внутрішнє тертя в інварному сплаві Fe-35% Ni після комбінованої ІПД методами гідроекструзії та волочіння. *Надутов В.М., Ващук Д.Л., Пилипенко А.М., Давиденко О.А., Бєлошенко В.О.*

Досліджено температурну залежність внутрішнього тертя на частоті ~3 Гц та ~ 60 Гц в інварному сплаві Fe-35.0%Ni-0.49%Mn-0.03%C після гомогенізації при 1373 К та наступної комбінованої інтенсивної пластичної деформації (ІПД) методами гідроекструзії і подальшого волочіння з сумарним ступенем накопиченої деформації $\varepsilon_{\Sigma}= 4.69$. У деформованому зразку сплаву при ~380 К спостерігається зниження інтенсивності релаксаційного піку ВТ (енергія активації 0.82–0.93 еВ) за рахунок зменшення механічних і магнітомеханічних релаксаційних втрат, внесок яких відновлюється після відпалу деформованого сплаву. Після комбінованої ІПД сплаву виявлено ріст загасання коливань поблизу температур 780-820 К сплаву і його часткове зниження після відпалу. За оцінкою енергія активації релаксаційного процесу становить 1.82-1.97 еВ.

1. Introduction

Special requirements are lodged to mechanical properties particularly to the mechanical quality factor of a product from Fe–Ni Invar alloy possessing low thermal expansion coefficient (TEC). For example, the high quality factor is required for precision ultrasonic equipment. By opposite, the high damping of elastic oscillations (high level of internal friction) in individual units of laser equipment and structural elements of telescopes is required. Taking into account magnetic subsystem as the main contribution to anomalous behavior of properties of the Fe–Ni alloys the study of different mechanisms of the internal friction (IF) in these materials is an important task.

The study results on IF in Invar Fe–Ni alloys after high temperature annealing are presented in [1–5]. In the studies of temperature dependence of the IF in Fe–Ni Invar alloy containing 0.01 %C and 0.25 %C on frequencies of 0.37 Hz and 0.78 Hz at the temperature below the Curie point (T_C = 533 K) [1–3] a carbon peak at 483 K and the abnormally large damping of oscillations with reducing temperature below the room one were interpreted as the Finkel'stein-Rozin relaxation peak and the magneto-diffusion losses respectively [2, 3]. The damping of elastic oscillations in binary Invar Fe–(30–36 %)Ni alloys or containing carbon ones after homogenizing heat treatment were studied at frequency 1.8 kHz [4, 5] that allows separate two relaxation contributions to the IF and thereby more accurately evaluate the activation energy of carbon diffusion in Fe–Ni austenite and the Young's modulus above and below the T_C. An anomalous growth of the IF background when the temperature drops to 200–300 K was revealed in [4]. The studies of the amplitude dependent IF in Fe–Ni Invar have shown that the damping of elastic oscillations can increase or decrease depending on Ni and C content [4].

The standard Invar $Fe_{36}Ni$ alloy has low mechanical properties [12]. Severe plastic deformation (SPD) is an effective hardening method and it is frequently used for metal products manufacturing. The structure and properties of the f.c.c.-Fe–Ni alloys was studied after the plastic deformation by tension [6], rolling [7], equal-channel angular pressing [8, 9], hydroextrusion [10]. Effect of structure grinding by combined treatment including hydroextrusion (HE) and subsequent drawing on thermal expansion, hardness and magnetic properties of Invar Fe–35.0 %Ni alloy was studied in [11]. At the same time the influence of such treatments particularly their combination, resulting in formation of the particular structural states in Invar [10, 12], on the dissipation of the elastic energy and elastic properties of the Invar are not described in the literature. Although these data accessing for understanding the factors resulting in the Invar anomaly and the creation prospects at such approach the stronger structural states is an important task.

In this work, the SPD was carried out by combination of HE and drawing. The damping of elastic energy in the Invar alloys after their hardening by means of denoted methods has not been studied yet. Such studies are important taking into account the wide application of the HE and drawing techniques for manufacture of hardened metal bar and wire items.

2. Experimental

Our study was carried out on industrial Invar Fe–Ni alloy containing 35.0 %Ni, 0.49 %Mn, 0.032 %C, 0.07 %Cu, 0.03 %C (hereinafter Fe–35.0 %Ni–0.49 %Mn–0.03 %C). A sample of the alloy of 2 mm in diameter for measurements of the temperature dependence of internal friction (IF) was machined and then heated at 1373 K in vacuum of 10^{-5} Torr for 0.5 h and subsequently quenched in oil. Cylindrical billets of 21 mm in diameter were annealed at 1373 K (0.5 h) and then quenched in oil before combined SPD, which was performed consistently by the HE and drawing with a total degree of $\varepsilon_\Sigma = 4.69$ (the degree of plastic deformation was determined using expression $\varepsilon = 2\ \ln(d_0/d)$, where d_0 and d are diameters of a sample before and after SPD). The strain degree before drawing resulting from multiple passes through the extruder was achieved $\varepsilon_\Sigma = 3.47$. The restricted degree of plastic deformation caused by this value because of the formation of the substructure boundaries accompanying with strengthening and decreasing TEC of the Invar is practically completed at $\varepsilon_\Sigma = 2.2$–3.47 [12].

The temperature dependence of IF Q^{-1} and the shear modulus G were measured within the temperature range 300 K–973 K in vacuum of 10^{-2} Torr by means of automated system of the relaxation spectroscopy based on reverse torsion pendulum in the mode of natural damped oscillations on frequencies of ~3 Hz and ~60 Hz. The samples were of 2 mm in diameter and 90 mm in

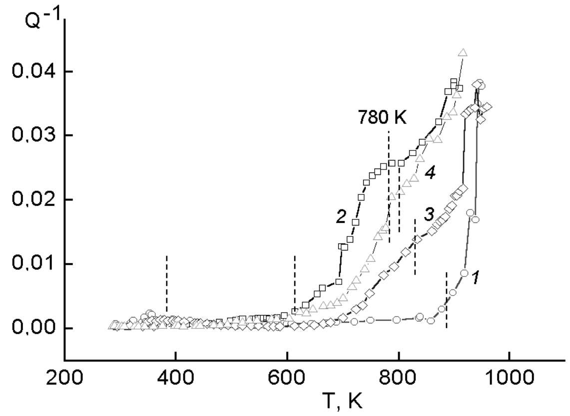

Fig. 1. Temperature dependences of IF in the Invar Fe–35.0% Ni–0.49 % Mn–0.03 % C alloy: after homogenization, $f \approx 3$ Hz (*1*); after combined SPD ($\varepsilon_\Sigma = 4.69$), $f \approx 3$ Hz (*2*) and $f \approx 60$ Hz (*4*); after reheating of the deformed sample, $f \approx 3$ Hz (*3*). Vertical lines indicate the most characteristic changes on the curves.

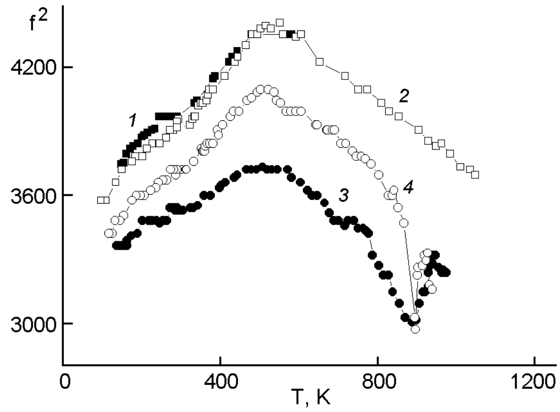

Fig. 2. Temperature dependences (taken at frequency of ~60 Hz) of the square frequency of elastic torsion oscillations f^2 that is proportional to the shear modulus G of the Invar Fe–35.0% Ni–0.49 % Mn–0.03 % C alloy: after annealing at 1373 K in vacuum and subsequent quenching in oil (*1* — first measurement, *2* — second measurement); after combined SPD $\varepsilon_\Sigma = 4.69$ (*3*, *4*), (*3*) is the first measurement of the deformed specimen, (*4*) is the reheating at measurement of the same sample.

length (the distance between clamps of a sample). The rate of the temperature change during heating was 2 K·min^{-1}. The relative cyclic deformation on the surface of a sample during measuring of the IF did not exceed $3 \cdot 10^{-5}$. Shear modulus (G) was determined as a value proportional to the square of frequency (f^2) of the torsion oscillations.

3. Results and discussion

Temperature dependence of the IF $Q^{-1}(T)$ in Fe–35.0 % Ni–0.49 % Mn–0.03 % C alloy is shown in Fig. 1. The damping of elastic oscillations in the alloy after annealing at 1373 K in vacuum and subsequent quenching in oil is almost unchanged with the elevating temperature up to $0.5T_s$ (T_s = 1728 K is the melting point of the Invar [13]) except its weak growth within the temperature range of 350–400 K. The dramatic rise of the $Q^{-1}(T)$ curve is started above $0.5T_s$ in the range of 870–940 K that is above the Curie point T_C = 495 K of the alloy [11] (Fig. 1, curve *1*). Similar growth of dumping of the elastic oscillations at heating that close to exponential one was observed in the Invar alloys containing 0.01 % C and 0.26 % C on the frequencies of 0.37 Hz and 0.78 Hz [2, 3] and containing 0.55 % C on higher frequency of 1503 Hz [4] (Fig. 2, curve *2*).

Dramatic increase of a background on the IF temperature dependence in the both carbon-containing and carbon-free Invar ally at ~3 Hz (Fig. 1, curve *1*), ~60 Hz (Fig. 1, curve *4*) and 1503 Hz (Fig. 3, curve *2* [4]) occurs in approximately the same temperature range and does not depend on frequency, which indicates the nonrelaxation nature of high damping of the elastic oscillations. There is no unified point of view concerning the microscopic mechanism of the high-temperature IF background in austenite [14, 15]. One of the reasons for the increasing IF background above 0.5 T_s can be intensive dislocation movement (climb) due to the activation of diffusion processes in the crystal lattice under high temperatures.

The combined SPD of the alloy which forms substructures with the high level of elastic microstresses [12] leads to the IF dramatic increase (f ~ 3 Hz) under the temperatures above 620 K and appearance of distinctive satellite component nearby 780 K (Fig. 1, curve *2*). At frequency of 60 Hz the IF maximum was exhibited under 800 K (Fig. 1, curve *4*). The possible reason for observed dumping of the elastic oscillations in the deformed Invar can be interaction of carbon atoms with the dislocations and their climb under the alternating fields of elastic stresses. The pronounced IF peak caused by this mechanism was observed in the austenitic steels nearby ~600 K, the intensity of which was increased with the rate of drafting and the activation energy estimated upon the frequency shift of the IF peak was 1.48 eV [16]. Smeared dislocation maximum on the IF curve of the technical purity f.c.c. nickel after plastic deformation and aging was also revealed nearby the temperature of 550 K [17].

Nature of an intensive growth of the high temperature damping of the elastic oscillations can be associated also with the recrystallization processes under the heating. To clarify the origin of high-temperature rise of IF the measurement was repeated at frequency of 3 Hz for the sample after heating in the previous experiment. Between the first and second heating the sample was not taken off the instrument. The dislocation maximum on the temperature dependence of IF in the deformed nickel was disappeared after annealing [17]. Reheating of the Fe–35.0 %Ni–0.49 % Mn–0.03 %C alloy reduces the intensity and shifts of the position of satellite peak on the $Q^{-1}(T)$ curve (f_{max} = 3.1 Hz) to the higher temperature, 820 K (Fig. 1, curve *3*).

Assuming the relaxation mechanism of damping of the oscillations in the deformed sample at 780 K–820 K (Fig. 1, curves *2*, *3*, *4*) we estimated the activation energy using the Marx-Werth formula [14]:

$$U_1 \equiv RT_{\max}\ln\frac{k_B T_{\max}}{hf_{\max}}, \qquad (1)$$

where R is the universal gas constant; k_B is the Boltzmann constant; T_{max} is the IF peak temperature; h is the Planck's constant; f_{max} is oscillation frequency corresponding to the IF maximum, Hz. The values of the activation energy for different frequencies are as follows: a) U_1 = 1.97 eV for f_{max} = 3.44 Hz and T_{max} = 780 K, b) U_1 = 1.82 eV for f_{max} = 57.2 Hz and 800 K and they are approximate values that suggesting the relaxation nature of the oscillation damping in the alloy at the considered temperatures. The dislocation satellite peak on the $Q^{-1}(T)$ curve at T_{max} = 820 K on frequency f_{max} = 3.1 Hz at reheating of the deformed specimen is observed (Fig. 1) that corresponds to the activation energy U_1 = 2.08 eV indicating changes in the structure after the first measurement. It could be noted that obtained values of the activation energy is higher than 1.40–1.56 eV obtained by frequency shifted dislocation peak for deformed austenitic steels [16]. The difference may be caused by peculiarities of dislocation

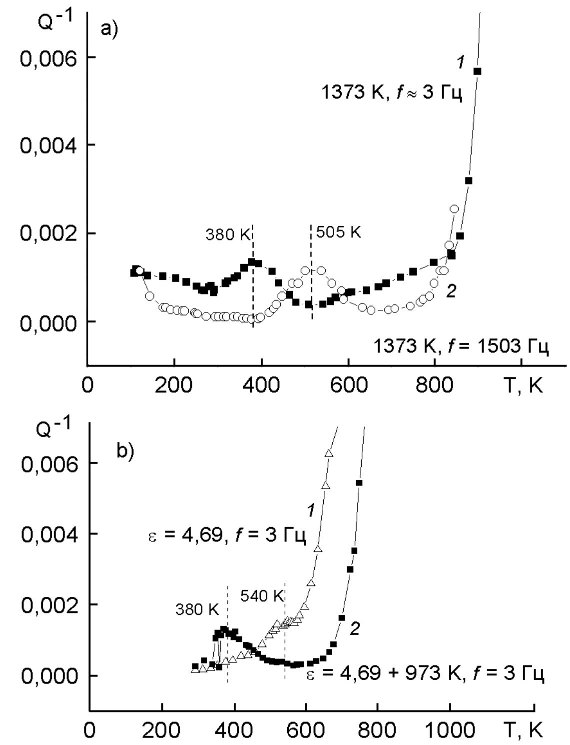

Fig. 3. The temperature dependence of IF in: a) the 1373 K homogenized Fe-35.0%Ni-0.49%Mn-0.03%C (f ≈ 3Hz, curve *1*) and the Fe-36.1%Ni-0.55%C alloys (f ≈1504 Hz, curve *2*, [4]); b) the Fe-35.0%Ni-0.49%Mn-0.03%C alloy after combined SPD with ε_Σ = 4.69 (curve *1*) and after the heating of the same sample to 973 K during the first IF measurement (curve *2*).

structure of the investigated Invar alloy formed under combined SPD [12] and by possible subsequent recrystallization during the following heating.

Taking into account the relationship of frequency and shear modulus $f^2_{max} \sim G$ the changes of the elastic modulus under heating of the Invar alloy in quenched and deformed states was analyzed that allows conclude as follows. The temperature dependences of the shear modulus of the quenched and deformed Invar alloy are qualitatively similar (Fig. 2). However, the shear modulus of the quenched sample after its heating to 500 K during the first measurement was not changed (curves *1* and *2*) and of the deformed alloy was significantly lower (Fig. 2, curve *3*) increasing slightly after the heating to 973 K during the first measurement (Fig. 2, curve *4*).

Such a behavior of shear modulus usually occurs along recrystallization processes in severe deformed material under its heating, particularly in well studied copper [18]. Therefore, there is the reason to assume that growth of dumping in the range of 780–820 K at heating of the deformed Invar alloy to 973 during the measurement caused by partial recrystallization along with the relaxation contribution (Fig. 1). One can assume that this process is an important circumstance explaining discrepancy of the activation energies estimated for studied Invar alloy (U_1 = 1.82–1.97 eV) and austenitic steels (1.40–1.56 eV) [16].

Thus, intensification of the elastic oscillations dumping in the deformed Invar Fe–35.0 %Ni–0.49%Mn–0.03 %C alloy above T_C can be caused by superposition of two mechanisms that are the migration of dislocation atmospheres under the alternating field of elastic stresses and the dislocation restructuring. Therefore obtained values of U_1 = 1.82–1.97 eV can be considered as the estimated and approximate ones.

The damping of the elastic oscillations at frequency of 3 Hz in Fe–35.0%Ni–0.49 %Mn–0.03 %C alloy upon cooling below the Curie point is much lower than at the higher temperatures. However the low intensive IF peak in the temperature range of 350–400 K is observed (Fig. 3). According to X-ray diffraction data the alloy at cooling is in the austenitic state and hence the observed growth of Q^{-1} is not associated with the martensitic transformation but rather due to relaxation losses. Indeed, increase of the dumping in the Invar Fe–Ni carbon-containing alloys during cooling below the Curie point associated with the Finkelstein-Rosin relaxation was observed on the frequencies of 0.37 Hz and 0.78 Hz at 473 K [2, 3] and frequency of 1503 Hz at 505 K [4]. The IF curve *2* in Fig. 3a for kilohertz frequency range was taken from [4]. The pronounced dumping of relaxation nature at 550–566 K and on the frequencies of 1887–1943 Hz was observed in Fe–Ni–C alloys with Ni concentration (29–30 %) lower than in the standard Invar (35–36 %) as well as in the alloys with the same low Ni content but doped additionally with Co or Mn [19].

Assuming the relaxation mechanism of damping in the investigated Invar Fe–35.0 %Ni–0.49 %Mn–0.03 %C alloy at T_{max1} = 380 K on the frequency of 3.31 Hz (Fig. 3a, curve *1*) the activation energy of the process was calculated by the Marx-Werth formula (1) and value U_1 = 0.93 eV received. Since the peak on the $Q^{-1}(T)$ curve for Fe–36.1 %Ni–0.55 %C alloy with the increasing frequency to f_{max2} = 1503 Hz [4] is shifted to the temperature T_{max2} = 505 K (Fig. 3a, curve *2*), the activation energy was estimated also by the frequency dependence [2]:

$$U_2 = R\frac{T_{\max1}T_{\max2}}{T_{\max2} - T_{\max1}}\ln\frac{f_{\max2}}{f_{\max1}}, \qquad (2)$$

which is equal to U_2 = 0.82 eV.

The values of the activation energies founded by the Marx-Werth formula (1) and the frequency dependence (2) for Fe–35.0 %Ni–0.49 %Mn–0.03 %C alloy are approximate in magnitude but slightly below the values for the Invar alloys obtained at lower frequencies 0.37 and 0.78 Hz (1.5 eV [2, 3]) and at the higher ones 1887–1943 Hz (1.0–1.1 eV) [4, 19] as well as calculated in [20] values of activation energy of carbon diffusion in Ni–Fe austenite 1.24 eV. The observed difference may be due to additional peculiarities of the magnetic contributions below T_C in damping of the elastic oscillations in the alloys with different content of nickel and carbon. It is known that the magnetic order in Fe–Ni–C austenite is highly sensitive to slight changes in the concentration of Ni and C [21]. Therefore, the magnetoelastic hysteresis* and magnetomechanical relaxation loses** can be additional contributions to the damping in the investigated Invar alloy below T_C nearby 380 K. The both types of losses can effect on an uncertainty in evaluation of the Finkelstein-Rozin peak position on the temperature scale and respectively of the activation energy. Taking into account the IF data obtained for the Invar alloy without and in magnetic field 250 Oe excluding magnetoelastic and magnetomechanical relaxation loses [2] we estimated the IF peak position uncertainty (10–20 K) that increases the activation energy by approximately 0.05 eV. One can conclude that the obtained activation energies U_1 = 0.93 eV and U_2 = 0.82 eV have underestimated and should be higher by this value.

*Magnetoelastic hysteresis is the irreversible shift of magnetic domain boundaries under influence of the external periodic elastic stresses, which are removed by application of constant or alternating magnetic fields [2].

**Magnetomechanical relaxation is appeared due to the phase delay of movement of the domain boundaries with respect to the external mechanical stresses when their displacement becomes dependent on the degree of ordering of carbon atoms in the interior of these boundaries as well as the secondary their directional ordering in volume remagnetizing as a result of displacement of domain boundaries under the influence of external stresses [21].

Obviously, the considered contributions into IF are sensitive to the structural state of the alloy and can be weakened after plastic deformation or reinforced by the subsequent heat treatment. The temperature dependence of IF with the deformation of $\varepsilon_\Sigma = 4.69$ suppressed the damping in the temperature range of 380–480 K (Fig. 3b, curve *1*) that caused by blocking effect of the high density defects and the high level of microstresses [12] both on mobility of carbon atoms in the field of elastic stresses and the mobility of the magnetic domain boundaries. Heating of the deformed sample to 973 K during the first measurement, which unlocked carbon atoms due to restructuring the subgrain structure and reducing the elastic microstresses [12], recovered the low temperature IF peak at 380 K at the repeated measurement (Fig. 3b, curve *2*). Thus, the high level of dumping of elastic oscillations in Fe–35.0 % Ni–0.49 % Mn–0.03 % C alloy at the temperatures below the Curie point is provided by the mechanical Finkelstein-Rozin losses at superposition of magnetomechanical ones whose contribution decreases after combined SPD by HE and the following drawing ($\varepsilon_\Sigma = 4.69$) and it is recovered again after annealing of the alloy.

4. Conclusions

Combined SPD of the Invar carbon-containing Fe–35.0% Ni–0.49 % Mn–0.03 % C alloy by hydrostatic methods and subsequent drawing with the degree of accumulated strain $\varepsilon_\Sigma = 4.69$ differently affects the elastic energy losses at the temperatures below and above the Curie point (495 K).

Growth of damping of the elastic oscillations was revealed below T_C of the Invar alloy (IF peak at 380 K) after annealing caused by the mechanical Finkelstein-Rozin relaxation losses with the supperposition of losses of the magnetic nature. After the combined SPD ($\varepsilon_\Sigma = 4.69$, hydroextrusion and subsequent drawing) these losses decreases as a result of interaction of carbon atoms with the crystal structure defects which reduce also the magnetic contribution. These losses are renewed again after annealing of the deformed alloy. The estimated activation energy of carbon microdiffusion in the alloy is 0.82–0.93 eV although these values are underestimated by approximately 0.05 eV through magnetic losses affecting an uncertainty in the relaxation peak position on the temperature scale.

Above T_C the dramatic increase of the IF background in the quenched Invar alloy is added after combined SPD by satellite component at 780–820 K caused by movement of dislocation atmospheres under alternating fields of the elastic stresses with superposition of the recrystallisation process. Neglecting the last one the estimated activation energy of the relaxation process is 1.82–1.97 eV.

Acknowledgements. This work was carried out with partial financial support of the competitive project 92/13-N of Target Complex Program of Fundamental Research of National Academy of Sciences of Ukraine "Fundamental problems of nanostructured systems, nanomaterials, nanotechnologies".

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