

# The influence of chemical composition and thermal treatments on the temperature-dependent internal friction of Fe–Ni alloys in the range of the phase transformation

S. Golovin<sup>a</sup>, I. Golovin<sup>b</sup>, Y. Rodionov<sup>c</sup> and V. Seleznev<sup>a</sup>

<sup>a</sup>Tula State Technical University, Tula 300600 (Russian Federation)

<sup>b</sup>Moscow State Aircraft Technology University, Moscow 103767 (Russian Federation)

<sup>c</sup>Moscow Research Institute of Physics of Metals, Moscow (Russian Federation)

## Abstract

Temperature-dependent curves of internal friction (TDIF) and modulus of elasticity (TDME) and the kinetics of the martensite transformation in Fe–Ni and Fe–24Ni–5Mo alloys with different carbon content have been investigated. The influences of chemical composition and preliminary heat treatment regimes on isothermal and athermal transformation and martensite decay due to heating were established.

## 1. Introduction

The influence of the initial  $\gamma$ -phase on the structural and kinetic peculiarities of the martensite transformation in Fe–Ni alloys are connected with the redistribution of atoms in submicrozones and the establishment of short-range order [1]. The influence of carbon content on the initial state of the  $\gamma$ -phase and on the character of the  $\gamma \rightarrow \alpha$  transformation has not been investigated so thoroughly. The increasing degree of short-range order of austenite leads to changes of the temperature-dependent internal friction (TDIF) and modulus of elasticity (TDME) spectra and martensite decay during heating [2, 3]. The aim of the present paper is to investigate the influence of chemical composition and heat treatment regimes on the IF and ME temperature spectra and the kinetics of the  $\gamma \rightleftharpoons \alpha$  transformation during quenching and heating.

## 2. Experimental details

The alloys investigated contained 29%Ni (0.01%C), 32%Ni (0.01%C) and 24Ni5Mo with differing carbon content (0.002–0.2%C). Water-quenching from 1100–1150 °C and annealing between 350 and 600 °C of ranging duration was performed. Measurements of TDIF, ADIF and TDME were made on samples with diameter 8 mm and length 180 mm at bending oscillations in the kHz range, between –180 and 600 °C and  $\epsilon = (0.2\text{--}100) \times 10^{-5}$ . The kinetics of the martensite

transformation on cooling ( $10 \text{ K min}^{-1}$ ) were measured by a magnetometric method (samples diameter 3 mm, length 30 mm).

## 3. Results

### 3.1. The influence of chemical composition

The TDIF and TDME curves of 29%Ni and 32%Ni are different in all structural states (Fig. 1). In the austenite of 29%Ni (quenched from 1150 °C) unstable TDIF is formed during heating with maxima at 60 and 160 °C and a wide peak at 320–400 °C. The  $\gamma \rightarrow \alpha$  transformation during cooling below the temperature  $M_1 = -5$  °C progresses smoothly. Isothermal growth of small martensite crystallites takes place. Only one peak at 360 °C appears for TDIF in the austenite of the 32%Ni alloy and an athermal (explosive) martensite transformation ( $M_A = -52$  °C) takes place during cooling and leads to noise-unstable TDIF formation and the appearing of large lenticular martensite crystals.

The TDIF curves of 24Ni5Mo alloys water-quenched from 1150 °C depend on carbon content (Fig. 1(a)). At low carbon content, up to  $C_c \approx 0.04\%$ , only a background of IF is present during heating; at higher carbon content an IF peak appears at 360 °C and increases. The increase of carbon content in the alloys leads to a decrease of the  $M_1$  point and an increase of the  $M_A$  point on cooling (Table 1). Simultaneously, an increase of the critical points of ADIF is observed, which attains a maximum at 0.04%C. In alloys with  $C > 0.04\%$  only

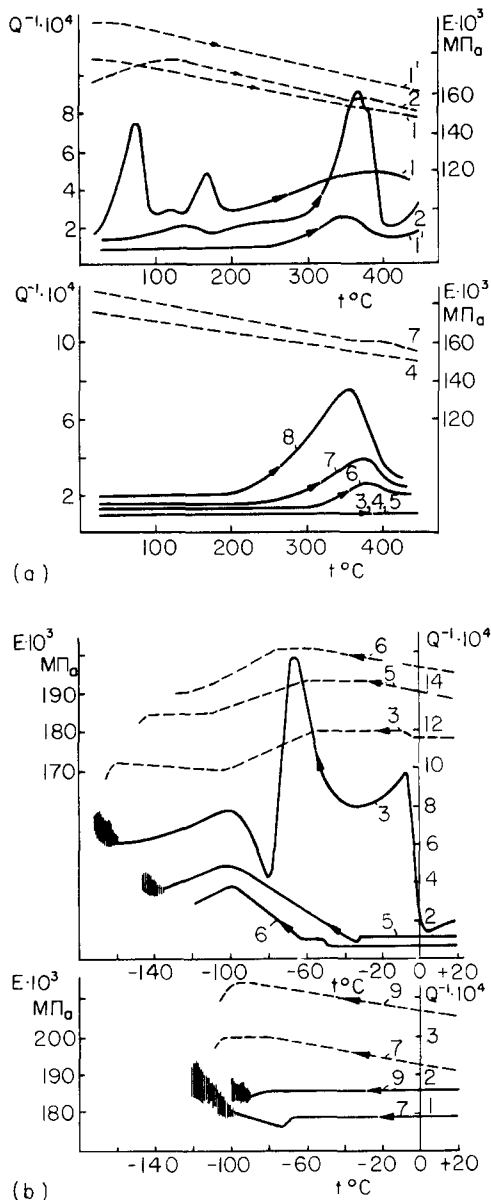


Fig. 1. TDIF (lines) and TDME (dashed lines) quenched from 1150 °C in water for alloys (a) during heating; (b) during cooling. Alloys are as follows: 1, 29Ni; 2, 32Ni; 3, 24Ni5Mo, 0.002% C; 4, 24Ni5Mo, 0.008% C; 5, 24Ni5Mo, 0.018% C; 6, 24Ni5Mo, 0.041% C; 7, 24Ni5Mo, 0.069% C; 8, 24Ni5Mo, 0.088% C; 9, 24Ni5Mo, 0.2% C; 1', 29Ni, quenched and annealed at 450 °C for 12 h.

the athermal martensite transformation takes place (Fig. 1(b)).

### 3.2. Influence of preliminary heat treatment

Increasing the annealing time at 450 °C for 29%Ni and 32%Ni alloys leads to the disappearance of the low-temperature peaks and the formation of a 360 °C peak (Fig. 1(a)). As a result of the heat treatment, the athermal (explosive) martensite transformation appears in the 29%Ni alloy with isothermal transformation

and becomes stronger in the alloy 32%Ni (Table 1). Long-term annealing of 24Ni5Mo at 450 °C leads to the disappearance of the IF anomaly on heating and to the suppression of the  $M_A$  temperature. The maximum of TDIF after annealing of alloys with  $C > 0.069\%$  remains and an athermal  $\gamma \rightarrow \alpha$  transformation takes place.

### 3.3. Influence of cooling regime on alloy structure formation

The TDIF curves of the investigated alloys on heating after the martensite transformation depend on their chemical composition, heat treatment and transformation depth. After the  $\gamma \rightarrow \alpha$  isothermal transformation during heating, the low-temperature IF peak shifts to higher temperatures. However, for alloys with an athermal  $\gamma \rightarrow \alpha$  transformation, asymmetrical peaks form in the range the of athermal martensite transformation (Fig. 2(a)). Two TDIF peaks,  $Q_M^{-1}$  at 220 °C and  $Q_{FR}^{-1}$  at 300–360 °C, appear at high temperature. The  $Q_M^{-1}$  peak increases and the  $Q_{FR}^{-1}$  peak decreases with decreasing temperature of the  $\gamma \rightarrow \alpha$  martensite transformation.

### 3.4. Kinetics of martensite decay

The annealing of quenched martensite 24Ni5Mo samples (0.069% C; curves 1 and 4 in Fig. 2(a) at the temperatures of the  $Q_M^{-1}$  and  $Q_{FR}^{-1}$  maximum leads to a decrease in  $Q_M^{-1}$  [1] but hardly alters the height of the IF peaks [4]. The relative changes of the subsequent parameters of solid solution decay,  $q_M$  and  $q_{FR}$  ( $q = Q_0^{-1} - Q_t^{-1} / Q_0^{-1}$ , %) are shown in Fig. 2(b).

## 4. Discussion

The TDIF spectrum of austenitic Fe–Ni alloys quenched from 1150 °C reflects a heterogeneous distribution of carbon atoms near the substitutional elements and dislocations (IF peaks at 60–120 °C [2]). Short-range order in these alloys leads to thermal stabilization of the austenite phase and the formation at 360 °C, of the Finkelshtain–Rozin peak, corresponding to the migration of carbon atoms in the alloyed austenite. This state is achieved as a result of increasing Ni content in Fe–Ni systems and C content above 0.04% in Fe–Ni–Mo alloys (Fig. 1(a)). Interstitials raise the rate of ordering, and the influence of temperature, type and morphology of martensite transformation. As a rule, the presence of the FR peak reflects the athermal type of martensite transformation following cooling.

In alloys with an isothermal transformation (29Ni, 24Ni5Mo with low carbon content), long-term (1–12 h) annealing at 300–600 °C leads to the formation of short-range order in the substitutional solid solution

TABLE 1. The influence of chemical composition and heat treatment regimes on isothermal ( $M_I$ ) and athermal ( $M_A$ ) martensite transformation, and the temperatures of maximum modulus of elasticity and internal friction ( $T_G$  and  $T_I$ ), during isothermal transformation

Composition	Heat-treatment regime	$M_I$ (°C)	$M_A$ (°C)	$T_G$ (°C)	$T_I$ (°C)
29%Ni	Quench from 1150 °C in water	-5		-3	-12
32%Ni	Quench from 1150 °C in water		-52		
	Anneal at 300 °C for 1 h		-75		
24Ni5Mo					
0.002%C	Quench from 1150 °C in water	+5	-140	-12	-8
	Anneal at 450 °C for 12 h	-65	-155	-58	-68
0.018%C	Quench from 1150 °C in water	-30	-137	-60	-105
	Anneal at 450 °C for 12 h	-73	-132		
0.041%C	Quench from 1150 °C in water	-50	-120	-60	-100
	Anneal at 450 °C for 12 h	-100	-110		
0.069%C	Quench from 1150 °C in water		-118		
0.088%C	Quench from 1150 °C in water		-115		
	Anneal at 450 °C for 1 h		-128		
	Anneal at 500 °C for 1 h		-156		
	Anneal at 550 °C for 1 h		-150		
	Anneal at 600 °C for 1 h		-125		
0.2%C	Quench from 1150 °C in water		-95		
	Anneal at 450 °C for 1 h		-100		
	Anneal at 500 °C for 1 h		-100		

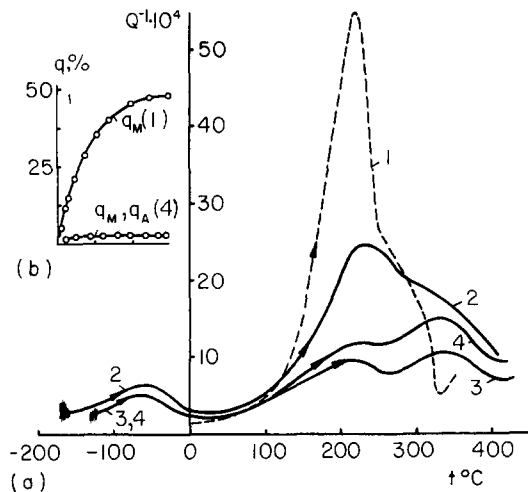


Fig. 2. (a) TDIF during heating of alloy 24Ni5Mo, 0.069%C and (b) fraction of martensite ( $q_M$ ) and austenite ( $q_A$ ) decay during annealing of quenched steel: 1, quenched from 1150 °C in water and liquid nitrogen; 2, quenched from 1150 °C in water plus annealing at 450 °C for 1 h and cooling ( $\gamma \rightarrow \alpha$  transformation); 3, quenched from 1150 °C in water plus annealing at 550 °C for 1 h and cooling ( $\gamma \rightarrow \alpha$  transformation); 4, quenched from 1150 °C in water plus annealing at 650 °C for 1 h and cooling ( $\gamma \rightarrow \alpha$  transformation).

and suppresses heterogeneity in the carbon distribution (peaks at 20–160 °C) due to dislocation pinning. This leads to retardation of the isothermal transformation, but does not influence the final result. In this case the FR peak is not revealed in TDIF. The decrease of the  $M_I$  point is a result of the decreasing free energy of

the austenite, not only because of ordering but also because of the dislocation pinning by atom segregation. The parameters of the amplitude-dependent internal friction (ADIF) (background,  $\epsilon_{cr}$ ,  $\text{tg } \alpha$  ADIF) after 1 h annealing at 100–600 °C achieve their extreme values at 450 °C ( $\epsilon_{cr} \approx 15 \times 10^{-5}$ ). Annealing at 450 °C for 12 h changes the kinetics of the martensite transformation to athermal [2].

In alloys with an athermal  $\gamma \rightarrow \alpha$  transformation (32Ni, 24Ni%Mo with  $C \geq 0.04\%$ ), annealing does not change the character of TDIF that has an FR peak. There were no regularities of influence of  $Q_{FR}^{-1}$  and  $M_A$  (Table 1, 0.088%C) in annealed alloys. The disappearance of Ni and Mo segregations and dislocation depinning are possibly due to the increasing temperature and the annealing time. Simultaneously, the precipitation of dispersed particles of  $\text{Mo}_2\text{C}$  takes place at grain boundaries and dislocations.

In spite of the complex nature of the IF maxima  $Q_M^{-1}$  [2–4] and  $Q_{FR}^{-1}$ , the change of their heights corresponds to the kinetics of martensite decay and permanent austenite respectively. The stage of permanent austenite decay during heating of high-Ni steel is not revealed.

## 5. Conclusions

1. The nature of the influence of carbon atoms on the kinetics of martensite transformation in Fe–Ni and Fe–Ni–Mo alloys has been investigated with the help of the internal friction method.

2. It was shown that an increased content of carbon interstitials suppresses the relaxation processes due to  $\gamma \rightarrow \alpha$  transformation and changes the transformation type from isothermal to athermal.

3. The critical impurity concentration that leads to the change of transformation type was established as 0.04%C.

4. Short-range ordering in Fe and Ni distribution takes place on heating up 400–500 °C, as carbon complexes are formed around Fe atoms. These processes lead to dislocation pinning and change the structural and kinetic form of the martensite.

## References

- 1 V.N. Zambrgitskij, O.P. Maximova, P.L. Gruzin, U.L. Rodionov and V.N. Seleznev, *Phys. Met. Met. Sci.*, **3** (1988) 562 (in Russian).
- 2 S.A. Golovin and K.N. Belkin, *Phys. Met. Met. Sci.*, **5** (1965) 763 (in Russian).
- 3 V.G. Gavriluck, V.A. Duzz and Ju. N. Jugodzinski, *Phys. Met. Met. Sci.*, **4** (1996) 197 (in Russian).
- 4 G. Klems, R. Miner, F.A. Hultgren and R. Gibala, *Met. Trans.*, **6** (1976) 839.