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S. Golovin, I. Golovin, V. Seleznev. Isothermal and Athermal Transformation of FeNiMo Alloys. Journal de Physique IV Proceedings, 1995, 05 (C8), pp.C8-305-C8-310. 10.1051/jp4:1995843 . jpa-00254092

HAL Id: jpa-00254092

<https://hal.science/jpa-00254092>

Submitted on 1 Jan 1995

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Isothermal and Athermal Phase Transformation of FeNiMo Alloys

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Abstract. Kinetic dependence of martensitic transformation and properties changes in Fe-24%Ni-5%Mo alloys with different carbon content have been obtained. Internal friction method was used for the precise investigation of structural changes after austenization, during martensitic transformation at sub zero temperature and during the postheat. The influence: (i) of frequency of oscillations, (ii) rate of cooling and (iii) amplitude of deformation on the time-temperature dependent internal friction during martensitic transformation have been shown. The curves of isothermal decay of austenite have been presented and we have discussed the type and kinetics of martensite transformation, and also the peculiarities of structural formation of martensite.

The influence of the composition on the character of phase transformation is connected with the redistribution of interstitials between the dislocations and austenite solid solution before the martensitic transformation at lower temperatures.

Introduction.

The existence of two types of martensitic transformation kinetics has been shown using 24Ni5Mo alloy [1,2]. Kinetics of martensitic transformation and martensite structure depend on the state of initial γ -phase and on the presence of the interstitials in solid solution [3,4]. Investigations of phase transitions and relaxation processes by the internal friction method in 24Ni5Mo with different carbon content provide the possibility of determining anelastic phenomena, connected with carbon distribution in alloyed austenite [4], and to find out mutual connection between γ -solid solution structure and martensite, formed during the subsequent γ - α transformation at lower temperatures. General kinetic laws of phase transformation in alloys with different carbon content in solid solution have been investigated in the present work.

Experimental details.

Alloys 24Ni5Mo with different carbon content (0.002...0.1 wt.%C) have been investigated. Water-quenching from 1150°C and annealing at 350...600 °C for variable times have been carried out. Measurements of temperature dependencies of internal friction (TDIF), amplitude dependencies of internal friction (ADIF) and modulus of elasticity (TDME) have been done on the samples $\varnothing = 8$ mm, $l = 180$ mm for bending oscillations in the kHz range, and on the samples $\varnothing = 0,8$ mm, $l = 60-100$ mm with the help of vacuum inverted torsion pendulum in the Hz range ($T = -180...600^\circ\text{C}$, $\gamma = (0.2-100) \cdot 10^{-5}$). Kinetics of martensitic transformation during cooling (10 K/min) was controlled by magnetometric induction method U (samples $\varnothing = 3$ mm, $l = 30$ mm).

Results

The structure of quenched solid solution depends on the content of interstitial atoms (IA). The peak discovered by Finkelshtain and Rosin (PFR) on temperature dependent internal friction (TDIF) curves, background of IF and slope of amplitude dependent IF ($\text{tg}\alpha$) are dynamic response on IA redistribution in

alloyed austenite [5]. PFR is due to the reorientation of couples of IA under the applied field of external stresses. The PFR appears on TDIF curves of 24Ni5Mo alloys with carbon content more than $C_{cr} > 0,04$ wt.% at 250 ($f \approx 2$ Hz) or 360°C (0,7 kHz). The height of PFR increases with the increase of carbon content in the alloy [4]. The experimental PFR is broader than standard relaxation peak with single relaxation time (Fig. 1,a). The computer simulation of PFR splitting into carbon and nitrogen peaks is presented in Fig.1,b.

Figure.1 TDIF of the quenched to austenite alloy 24Ni5Mo with 0.088%C at 1.85 Hz and 0.7 kHz (a) and theoretical description of maximum (b). C and N - partial dependencies of IF.

The analysis shows that the experimental data are the result of superposition of carbon (activation energy $H_C = 130$ kJ/mol) and nitrogen ($H_N = 116$ kJ/mol) peaks, the main contribution corresponding to carbon atoms. This is confirmed by the value of the activation energy (136 kJ/mol), which was determined with the help of frequency shift data. IF background and $tg\alpha_{ADIF}$ reflect the degree of dislocation pinning and crystalline lattice resistance to dislocation motion in applied stress, due to IA and their segregations, respectively. The values of $tg\alpha_{ADIF}$ decrease and achieve the saturation with the increase of carbon content up to C_{cr} :

C, wt% :	0.002	0.008	0.018	0.041	0.069	0.088
$tg \alpha_{ADIF}$:	0.42	0.37	0.32	0.26	0.25	0.25
$Q_{FR}^{-1} \cdot 10^{-4}$:	-	-	-	2.0	3.2	4.5
$M_s^{iso}, ^\circ C$:	+5	-10	-30	-50		
$M_s^{at}, ^\circ C$:	-140	-140	-137	-120	-118	-95

The data obtained reveal the existence of two different states of solid solution. At low carbon content in alloy ($< C_{cr}$) IA interact with imperfections of crystalline (mainly with dislocations), dislocations being not saturated by IA. At $C > C_{cr}$ the carbon concentration at dislocations reaches equilibrium at room temperature.

The initial state of γ -solid solution determines the kinetics of the following martensitic transformation on cooling (Fig.2). TDIF curves below subzero temperatures have been measured at cooling rate $V_c = 10$ K/min, frequency $f = 0.7$ kHz (Fig.2,a) and at $V_c = 1.7$ K/min, $f = 1.85$ Hz (Fig.2,b). The smooth change of IF is typical for the first set of samples with carbon content bellow 0.04%C. Isothermal appearance of small martensite crystallines takes place. That is the result of isothermal $\gamma \rightarrow \alpha$ transformation which is followed by the "burst" athermal transformation.

Fe-24%Ni-5%Mo

Figure 2. Kinetic curves of cooling the alloy 24Ni5Mo with 0.002 (1) and 0.088 %C (2), quenched to austenite: a - 0.7 kHz, b - 1.85 Hz; 1, 2 - Q^{-1} ; 1', 2' - U . Figures near curves on Fig.2,b show carbon content, %.

The isothermal transformation γ -solid solution is in agreement with location of the temperature ranges of iso- and athermal martensitic transformations. In order to obtain quantitative estimations of the degree of austenite phase transformation, isothermal low frequency IF measurements of quenched austenite have been carried out in the temperature range from -10 to -150 $^{\circ}\text{C}$. The degree of interstitial solid solution decay has been determined with the help of initial (Q_0^{-1}) and transient (Q_{tr}^{-1}) values of IF by $W = Q_0^{-1} - Q_{tr}^{-1} / Q_0^{-1}$ equation. The T-T-T diagrams (isothermal) have been obtained on this base (Fig 3). Under the above mentioned cooling conditions, the martensitic transformation in alloy with 0.002 %C is isothermal (Fig.3,a). With the increase of carbon content to 0.041 %C the isothermal type of martensitic transformation changes to athermal on cooling (Fig.3,b). The type of martensitic transformation changes, depending on cooling rate and more over the co-existence of different types of martensite transformation is possible.

Fe-24%Ni-5%Mo

Figure 3. Curves of isothermal decay of alloy 24Ni5Mo with 0.002 (a) and 0.041 %C (b) as the result of IF measurement. Figures near curves show the amount of martensite formed, %.

Unstable scattering behavior of IF typical for athermal martensitic transformation occurs in alloys with carbon content greater than 0.04 %C. There is appearance of large-size lens-type martensite crystals. Athermal martensitic transformation during IF measurements (kHz equipment) has been accompanied by noise effects. The results of the influence of parameters of measurements (cooling rate, frequency of oscillations) on isothermal martensitic transformation in alloys with carbon content 0.002 %C are presented in Fig. 4. The two staged character γ -solid solution decay (IF peak at -70°C and plateau around -100°C are observed on TDIF curves) is better observed during slow cooling 1.7 K/min ($f = 1.85 \text{ Hz}$, $\gamma = 2.8 \cdot 10^{-5}$ Fig.4,a) and with the decrease of frequency of measurements from 1.85 to 1.2 Hz ($V_c = 1.7 \text{ K/min}$, $\gamma = 2.8 \cdot 10^{-5}$ - Fig.4,b). In general the IF is proportional to the cooling rate and is inverted proportional to the frequency of oscillations. The systematic influence of amplitude deformation influence on IF level was not established.

Fe-24 % Ni-5 % Mo-0.002 % C

Figure 4. The influence of cooling rate (a) and frequency of oscillation (b) on the TDIF during cooling of the quenched to austenite alloy 24Ni5Mo with 0.002 %C.

The character of IF change due to heating of quenched austenite samples of 25Ni5Mo alloys having different carbon content has been investigated on the same samples after their cooling from 20 to -180°C in pendulum, followed by heating up to 450°C (Fig.5). IF peak below zero temperature appears in all the samples, independently on the type of $\gamma - \alpha$ transformation during preliminary cooling. The next IF peak ($\approx 160^\circ\text{C}$ at 1.85 Hz) appears and increases with the increase of IA content in alloys of the athermal type of martensitic transformation ($C > 0.04 \text{ \%C}$). This peak is usually observed for Fe-Ni-twinned martensite structure [6]. It shifts to $220-260^\circ\text{C}$ for $f = 0.7 \text{ kHz}$ (Fig.6). The height of this IF-peak is $(10-12) \cdot 10^{-4}$ for alloys $< 0.041 \text{ \%C}$ and the peak rapidly grows with the increase of carbon content. In the temperature range $320-360^\circ\text{C}$ a FR - peak appears in retained austenite.

Discussion.

The structure of quenched austenite for the investigated alloys is disordered for interstitial I and substitution S atoms. In alloys with low carbon content IA are mainly distributed in the imperfections of crystalline lattice. Unstable IF curves due to heating reproduce alloy's heterogeneity and the appearance of ordered zones of I and S atoms. At $C = C_{cr}$ the dislocations become unmovable due to their pinning, and IA dissolve in FCC solid solution. The type of the subsequent martensitic transformation is connected with these two initial states of austenite.

Fe-24%Ni-5%Mo

Figure 5. TDIF during heating after cooling of the alloy 24Ni5Mo with 0.002 (a), 0.041 (b) and 0.088 %C (c). Frequency of oscillation is 1.85 Hz.

The effect of IF peak appearance due to heating at -100...-60°C (from -190°C) is present for alloys with all types of direct martensitic transformations (Fig.5). Its height decreases and temperature location increases as the carbon content increases. This peak might be connected with stress relaxation and dislocation pinning during heating as well as with low temperature isothermal martensitic transformation due to reversed α - γ transformation under conditions when diffusion processes are absent because of low temperatures.

The distribution of atoms in martensite is inherited from initial austenite state. Carbon atoms in γ solid solution are located around twin boundaries in martensite. Their mutual motion under the applied stresses leads to IF peak at 160°C ($f = 1.85$ Hz) or at 240°C (0.7 kHz) - Fig.5. If the direct martensitic transformation is not finished (Fig.6, curve 2) two IF peaks are present: martensite and PFR in retained austenite.

The γ - α transformation is isothermal and might be finished by athermal "burst" transformation in alloys with easy movable dislocations ($C < C_{cr}$). The M_s^{iso} temperature decreases and M_s^{at} temperature increases with the increase of carbon content in the alloy. Inspection of IF curves reflects the presence of two stages of isothermal decay of alloyed austenite (Fig.2). There are two temperature intervals of TDIF with maximum of martensite transformation rate (-60-70°C and -100-110°C) and two corresponding plateaus TDME (f^2) during cooling for alloy with 0.002 %C. Assuming that before γ - α transformation the initial austenite is heterogeneous, the direct martensitic transformation could take place in enriched and impoverished by alloying elements zones [3]. Initial heterogenities could be broken due to dislocation motion during γ - α transformation. Simple analysis shows that the transient part of IF (Q_{tr}^{-1}) could be explained in terms of well known equation:

$$Q_{tr}^{-1} = (dV/dT) \cdot (T/\omega).$$

The sequence of different types of martensite transformation could be explained from the point of view of temperature ranges changing of iso- and athermal martensitic transformation due to the increase of IA content in the alloy (Fig.3). Dislocation pinning by IA as well as IA solution in alloyed austenite suppress isothermal type of martensitic transformation, shift M_s^{at} to higher and M_s^{iso} to lower temperatures. Martensitic transformation starts from athermal one in alloys with carbon content 0.069 %C or greater. In this case the possibility of isothermal martensite transformation at lower temperature is determined by the temperature of the end of athermal martensitic transformation. The change of cooling rate for 24Ni5Mo alloys with both types of martensitic transformation gives the opportunity to have any type of martensitic transformation.

Figure 6. TDIF of the quenched to austenite alloy 24Ni5Mo with 0.088 %C (1) during heating after cooling from temperature -150° (2) and from liquid nitrogen (3); and after aging at room temperature during 72 hours (4).

Conclusion.

The analysis of anelastic phenomena in 24Ni5Mo alloy with different carbon content has shown that homogeneous distribution of IA is being formed just in freshly quenched to austenite samples. This predetermines the possibility of different types of γ - α transformations. Saturation of defects of crystal lattice of γ -solid solution provides the transition of isothermal type to athermal type of martensitic transformation, due to the displacement of the athermal transformation start. Kinetics and the type of solid solution decay depend on the cooling rate and can have different mechanisms of decay. The initial state of solid solution connected with IA distribution is maintained during quenching to martensite. With the increasing of $C > 0.04\%$ the structure of twinned types begins to develop.

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