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Kinetics of Austenite to Martensite Transformations in Iron-Nickel-Carbony Alloys During their Cooling or Tensile Testing

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Abstract. Two austenitic Fe-Ni-C alloys, one having 23.9 wt.% Ni, 0.39 wt.% C, and a martensite start point of 228 K, the other with 21.8 wt.% Ni, 0.48 wt.% C, and an M_s of 233 K, were tested to determine the kinetics of their austenite to martensite transformation both during cooling, at a rate of $5 \text{ K} \cdot \text{min}^{-1}$, and during tensile testing at strain rates from 8.3×10^{-5} to $8.3 \times 10^{-3} \text{ s}^{-1}$ and at temperatures from 233 to 323 K. Both alloys displayed variations in the critical plastic strain ϵ_c for the onset of serrated yielding. Metallography confirmed that serrated yielding can occur in austenite before any strain-induced martensite is formed. Although strain-induced martensite formed at all of the examined strain rates and temperatures, inflections on the true stress versus true strain, or σ - ϵ diagrams, were observed only at low test temperatures. The effect of transformation-induced plasticity, or TRIP, assessed by the maxima of uniform plastic strain ϵ_u ($\epsilon_u = 0.8$), was detected only in tests run at 253, 273 and 293 K. Kinetic diagrams for the formation of strain-induced martensite, in dependence on ϵ , were compiled for all the test temperatures and strain rates, and were compared with kinetic diagrams of heat release gained by differential scanning calorimetry for the austenite to martensite transformation. Diagrams of the amounts of heat released, $\Delta Q^{A \rightarrow M}$, in the martensite formation process, against the plastic strain magnitude in the presence of similar proportions of martensite, were found to yield linear relationship.

1. INTRODUCTION

Martensite transformations are often investigated on Fe-Ni-C alloys, where the process kinetics can be studied by relatively simple experimental procedures (1, 2, 3). Martensite arises either by quenching to below the martensite start temperature M_s , or else by strain ϵ at a temperature above M_s but below the thermodynamic equilibrium temperature T_0 . The work reported here examined the kinetics of martensite transformations induced by both of these alternative means, and shows how the findings can be compared.

2. EXPERIMENTAL WORK

This research was performed on two alloys, designated A and B respectively, with the chemical compositions and M_s temperatures listed in Table 1.

Alloy	%C	%Ni	%Si	%P	%S	%Mn	M_s [K]
A	0,39	23,87	0,44	0,0070	0,020	0,42	228
B	0,48	21,80	0,35	0,0060	0,019	0,43	233

Table 1: Compositions and M_s points of the examined alloys

The preparation and processing of the specimens have been described previously [4, 5]. Their two stage austenitizing treatment produced a homogeneous austenitic structure and a mean grain size of 0.080 mm.

Alloy A was utilised to ascertain the kinetics of martensite transformations induced by quenching and by strain, and to compare them. Quenching was performed in a Setaram DSC 111 calorimeter at a cooling rate of $5\text{K}\cdot\text{min}^{-1}$ from room temperature down to 153 K. The calorimetry records then yielded heat diagrams showing the amounts of energy released during the transformation process, and integration revealed the amounts of energy released in the formation of a given quantity of martensite in quenching down to 153 K. The quantity of martensite formed by this quench was established by metallography, by a point-to-point method.

If we assume that the amount of heat liberated in the formation of each per cent of martensite remains constant throughout the transformation process, then the total amount of heat ΔQ released between 153 K and M_s by the formation of some per cent of α'_T martensite can be established as :

$$\Delta Q_{(\% \alpha')_T} = \frac{\Delta Q_{153\text{K}}}{\% \alpha'_{153\text{K}}} \cdot \% \alpha'_T$$

This equation then allows us to compile kinetic diagrams for martensite transformations in terms of the amounts of energy ΔQ versus the percentages of α'_T martensite.

The kinetics of strain-induced martensite transformations were examined by tensile tests at a strain rate of $8.33 \cdot 10^{-4} \text{s}^{-1}$ and test temperatures ranging from -40 to $+50^\circ\text{C}$ (233 to 323 K). To permit the construction of kinetic diagrams, we conducted both conventional tensile tests to rupture, and interrupted tests in which the specimens were subjected to only a certain amount of plastic strain and then unloaded. The proportions of martensite were then determined in all the specimens, always in the regions which had undergone only uniform strain without any necking.

Alloy B was used to investigate how the strain rate $\dot{\epsilon}$ and the test temperature affect the martensite transformation. Tensile testing was carried out at strain rates of $\dot{\epsilon}_1 = 8.33 \cdot 10^{-3} \text{s}^{-1}$, $\dot{\epsilon}_2 = 8.33 \cdot 10^{-4} \text{s}^{-1}$ and $\dot{\epsilon}_3 = 8.33 \cdot 10^{-5} \text{s}^{-1}$ and test temperatures of $T_1 = 20^\circ\text{C}$ (293 K), $T_2 = 5^\circ\text{C}$ (278 K) et $T_3 = -20^\circ\text{C}$ (253 K).

3. RESULTS

Fig.1 presents heat release diagrams showing the enthalpy of a martensite transformation, as obtained by calorimetry. The transformation will be seen to have a "burst" character. The M_s temperatures were always ascertained at the points where the bursts arose. In the first case, M_s was 216 K (-57°C), in the second case, it was 213 K (-60°C).

Fig. 1 : Heat release diagrams for the enthalpy of alloy A in specimens 1 and 2.

Fig. 2 indicates the kinetics of martensite transformations. It will be noted that a burst causes very rapid onset of the transformation (some 20 to 30% of martensite arise during cooling by roughly 10 K beneath the M_S point), and that later this process slows down.

Fig. 2 : Kinetics of quench-induced martensite transformation in alloy A, specimens 1 and 2.

The M_S temperature ascertained by calorimetry is lower than that established by metallography. It has been thought to be due to the stabilization of austenite by its slow cooling [6, 7] in the calorimeter, as against its more rapid cooling in metallographic tests.

The kinetics of strain-induced martensite transformations were examined at four temperatures : 233, 253, 293 and 323 K, or -40, -20, +20 and +50°C. The findings are summarized in Fig. 3.

Fig. 3 : Kinetics of strain-induced martensite transformations at test temperatures of : a) 323 K, b) 293 K, c) 253 K, and d) 233 K.

The shape of the curves in these diagrams is much the same regardless of the test temperature ; what alters is the instant when the martensite transformation begins. At a temperature very close to M_s , at 233 K or -40°C , the transformation begins immediately upon the onset of plastic strain. The higher the test temperature, the more plastic strain is needed before the transformation starts, and the less martensite is produced.

Fig. 4 affords a comparison of the kinetics of quench-induced and of strain-induced martensite transformations. In this diagram, the amount of heat released during the transformation (on the y axis) is plotted against the strain required for the formation of an equal amount of martensite (on the x axis).

Apart from the onset of martensite transformation, which varies with the test temperature, there is a linear relationship between the quantity of heat released in quench-induced transformations and the amount of strain needed for strain-induced martensite to arise at any given test temperature. If the test temperature is taken into account, this relationship can be described by

$$\Delta Q = -2609 + 1693.\epsilon + 667505.\frac{1}{T}[\text{J.mol}^{-1}]$$

Tensile testing at some of the applied temperatures produced evidence of a TRIP or transformation-induced plasticity effect. Fig. 5 shows that at temperatures close to M_s , in our case 233 K, we gain a large amount of martensite (about 96%), but that this transformation does not enhance plasticity. At temperatures very far from M_s , such as 323 K, we obtain only a little amount of martensite (roughly 18%), but plasticity is not improved either. Only at test temperatures between these two extremes, i.e. at 253, 273 and 293 K, did we obtain some 80 to 90% of martensite while plasticity rose approximately from 0,4 to 0,8.

Fig. 4 : A comparison of the kinetics of quench-induced and of strain-induced martensite transformations.

The TRIP effect became apparent only in the temperature interval between $M_s + 25$ and $M_s + 65$, and only at a strain rate of $8.33 \times 10^{-4} \text{ s}^{-1}$.

Inflection point ϵ_i on the rational $\sigma_i - \epsilon_i$ curve is sometimes claimed to be related to the onset of a strain-induced martensite transformation [8]. In our tests at 323 K no inflection was observed, and the ruptured specimen was found to contain 17% of martensite. Lower test temperatures always resulted in an inflection, but that did not mark the start of martensite transformation, because at this point the transformation was already in progress : at 293 K, $\epsilon_i = 33\% \alpha_D$; at $T = 253$ K, $\epsilon_i = 19\% \alpha_D$; and at $T = 233$ K, $\epsilon_i = 68\% \alpha_D$. The inflection on the $\sigma_i - \epsilon_i$ diagram merely indicates that the material contains

enough martensite for its mechanical properties to alter : it does not signal the onset of martensite transformation.

Fig. 5 : Strain and the amount of martensite formed as functions of the test temperature.

The tensile test records also revealed some serrated yielding ; its initial stages, at ϵ_c , were observed both in the purely austenitic, as yet untransformed structures at 323 and 293 K, and in structures already undergoing martensite transformations : at 253 K, $\epsilon_c=19\%\alpha_D$ and at 233 K, $\epsilon_c=88\%\alpha_D$.

Fig. 6 demonstrates the test temperature dependence of ϵ_c in alloy A at one strain rate, $8.33 \times 10^{-4} \text{ s}^{-1}$, and Fig. 7 shows the same dependence in alloy B at three different strain rates. The evidence is that ϵ_c declines as the temperature increases and as the strain rate diminishes.

Fig. 6 : Temperature dependence of the critical strain that starts serrated yielding in alloy A.

Fig. 7 : Temperature and strain rate dependences of the critical strain for the start of serrated yielding in alloy B.

4. DISCUSSION AND CONCLUSION

In this work, we determined the relationship between the kinetics of quench-induced and of strain-induced martensite transformations. An equation presented here describes the relationship between the enthalpy liberated in the formation of a certain amount of quench-induced martensite, and the strain needed to produce the same amount of strain-induced martensite at the same test temperature (above the M_S point).

This relation does not apply to the initial phase of the martensite transformation : it becomes valid only from strains of about 0.2 upwards. Below this threshold of 0.2, the computed ΔQ values are higher than the real level.

In alloy A, a TRIP effect was discerned in the temperature interval between $M_S + 25$ and $M_S + 65$, but it did not become apparent at test temperatures close to M_S ; strain easily produced large proportions of martensite, about 96%, but the final amount of strain was roughly equal to that observed at test temperatures far from the M_S point, at which strain temperatures rendered only little martensite (about 18%). The martensite transformation enhances plasticity only at temperatures which lie between these two extremes, improving it to as much as 0.8.

This investigation failed to define the onset of strain-induced martensite transformation from charts recorded during tensile testing or from rational diagrams. Inflection points on the rational $\sigma_t - \epsilon_t$ diagrams appeared only at a stage when the specimen already contained 19% or more of martensite ; at 323 K no inflection was discovered, yet a ruptured specimen was found to hold 17% of martensite.

The onset of serrated yielding, at ϵ_c , does not signal the start of martensite transformation : serrated yielding was detected in both austenitic and martensitic structures.

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