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Stabilization of Austenite Associated with Prior Formation of Martensite

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Abstract. The stabilization of retained austenite was studied via a two-step cooling procedure. Alloys with M_s temperatures below 0 °C were used. It was found that the martensitic transformation starting temperature, M_s , was lowered to M_s' during the second step of cooling. With increasing the amount of previously formed martensite, the difference in the transformation starting temperatures, $\Delta M_s (= M_s - M_s')$, increases. This stabilization effect can also be observed in an alloy having very low carbon content, and the magnetic properties of alloys do not have drastic effect on the nature of this stabilization phenomenon. The mechanism of the stabilization of retained austenite is proposed to be mainly due to the inhibition effect produced by the previously formed martensite. The aging processes are considered to be a necessary condition for the above mechanism to operate.

1. INTRODUCTION

It is known that, during an athermal martensitic transformation, if cooling is interrupted at a temperature of T_a ($M_f < T_a < M_s$) within the temperature range of martensitic transformation, and aging occurs at T_a or a higher temperature, the transformation stops, and it can recommence only upon additional cooling below the arrest temperature [1-7]. This stabilization of retained austenite has long been attributed to the immobilization of the interfaces between the martensite nucleus and the austenite matrix due to segregation of interstitial solute atoms to the dislocation arrays surrounding the martensite nucleus [2-4]. However, it has to be noted that most of the earlier studies [1-4] have been performed on alloys having M_s temperatures above room temperature, and with both the arrest and aging temperatures higher than 0 °C, where the diffusion of carbon atoms is significant. Studies performed at sub-zero temperatures [5,6] have shown that the stabilization of retained austenite can also develop rapidly in Fe-Ni-C alloys at aging temperatures as low as -90 °C. These results indicate clearly that the mechanism of the stabilization of retained austenite proposed by earlier researchers [2-4] is not complete since it can not be used to explain the stabilization phenomenon at low temperatures. In fact, it has been noticed in several studies that, for a given aging treatment, the greater the amount of previously formed martensite is, the larger will be the magnitude of stabilization of retained austenite [6,7].

In the present study, the stabilization effect of retained austenite has been studied using Fe-Ni-C alloys with M_s temperatures below 0 °C. The effects of both aging and the previously formed martensite on the stabilization of retained austenite are clarified. A thermodynamical analysis of the effects of the previously formed martensite on the stabilization of retained austenite is also performed. Part of the results have been published in Ref. [8]. In this report, some new data obtained from alloys with high Ni content are added.

2. EXPERIMENTAL

Two groups of Fe-Ni-C alloys with different magnetic properties, *i.e.*, paramagnetic matrix phase with low Ni-content and ferromagnetic matrix phase with high Ni-content [8,9], are used in the present study. The alloys, their heat treatment conditions, M_s temperatures and the related martensite morphology are listed in Table 1. The two-step cooling experimental procedure is as follows: the samples were first cooled to a temperature T_a below M_s and then heated to room temperature (RT), after being held at RT for a while, the samples were recooled to low temperatures (23 or 82 K) and then again heated to RT. The preparation of

the samples and the test procedure are described in details elsewhere [8,10]. The magnetic susceptibilities of the specimens were measured during the whole experimental procedure.

Table 1. Alloys, heat treatment conditions, M_s temperatures and martensite morphology

| Alloys (wt%) | Heat treatment condition | M_s (K) | Martensite morphology |
|------------------|--------------------------|-----------|-----------------------|
| Fe-9Ni-1.4C | 1150 °C / 1 h + WQ* | 248 | Lenticular |
| Fe-20Ni-0.73C | 1150 °C / 3 h + WQ | 226 | Lenticular |
| Fe-28Ni-0.2C | 1150 °C / 1 h + WQ | 237 | Lenticular |
| Fe-30Ni-0.3C | 1230 °C / 1 h + WQ | 236 | Lenticular |
| Fe-33.5Ni-0.009C | 1150 °C / 1 h + WQ | 151 | Lenticular |

* WQ = water quenched; 1150 °C / 1 h + WQ = samples were annealed at 1150 °C for 1 h, and then quenched into salt water.

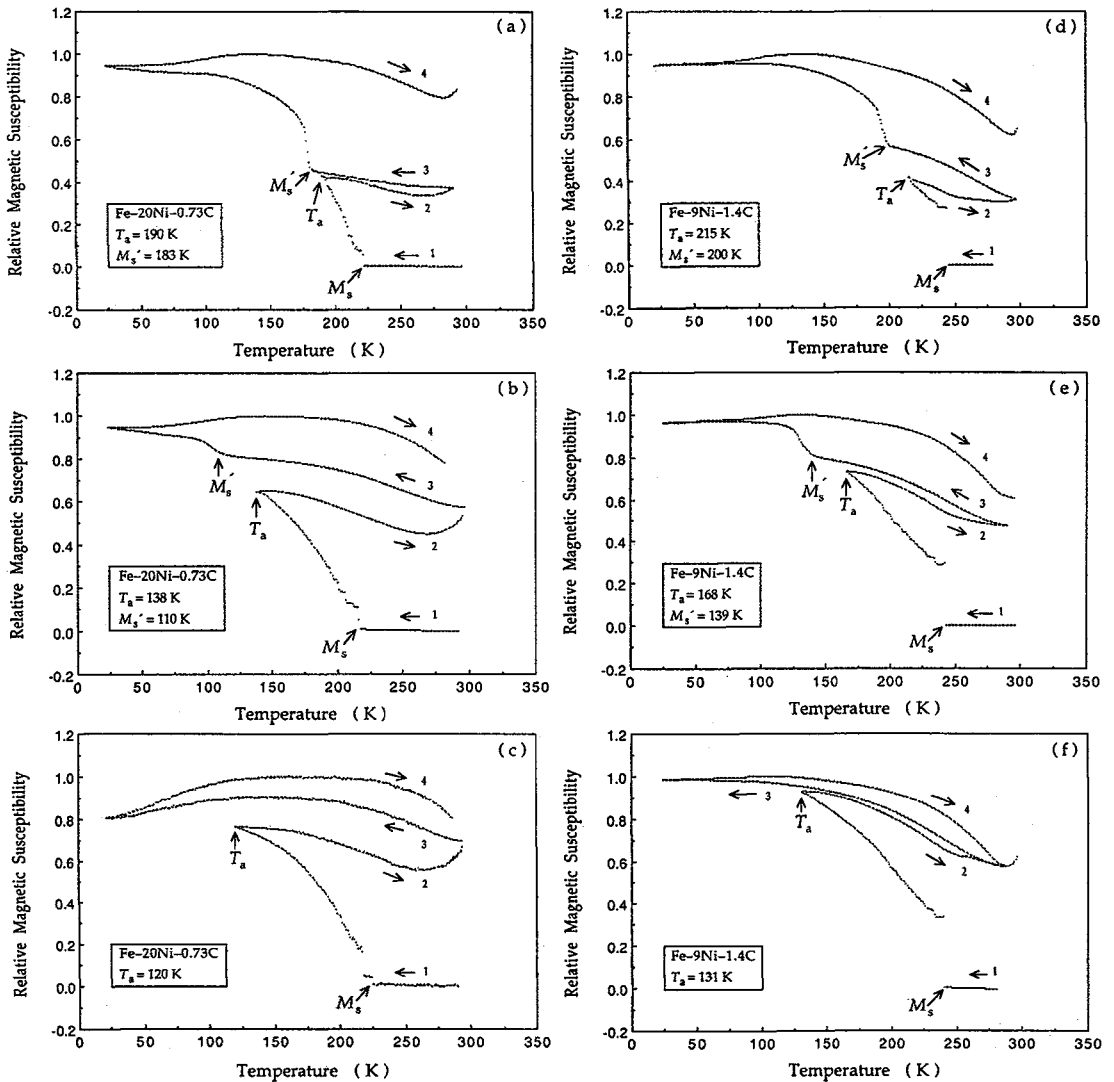


Figure 1: Relative magnetic susceptibility of alloys having lower Ni-content during two-step cooling experiment.

3. RESULTS AND DISCUSSIONS

Several magnetic susceptibility results of the alloys measured during the two-step cooling procedures with changing T_a temperatures are shown in Figs 1 and 2. It can be seen that, for both groups of alloys with different magnetic properties, martensitic transformation started at M_s temperature during the first step of cooling, and the M_s temperature was lowered to a much lower temperature of M_s' during the second step of cooling. It can also be noted that with decreasing T_a (increasing the amount of martensite), both the M_s' temperature and the intensity of the transformation occurring at M_s' temperature decrease for all the alloys. These observations suggest that the magnetic properties of the alloys have no drastic effects on the stabilization of retained austenite. If T_a is low enough, the martensitic transformation can be completely inhibited during the second step of cooling as shown in Figs 1c and 1f. The fact that the stabilization phenomenon takes place even in an alloy containing a very small amount of carbon, e.g., Fe-33.5Ni-0.009C alloy as shown in Fig. 2, indicates that, in addition to the carbon diffusion, the previously formed martensite may also contribute to the stabilization of retained austenite.

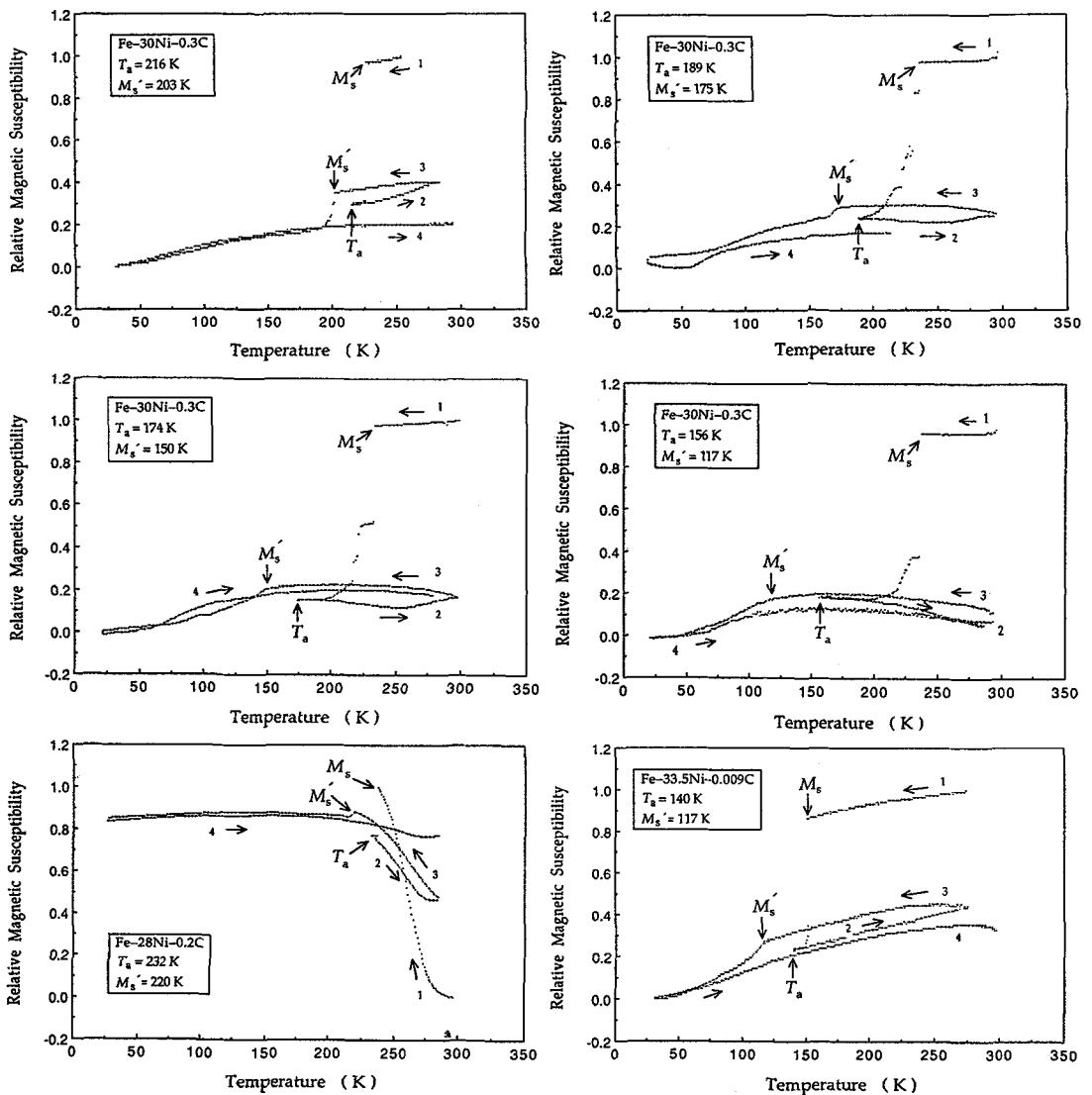


Figure 2: Relative magnetic susceptibility of alloys having higher Ni-content during two-step cooling experiment.

Values of M_s , T_a , M_s' and the corresponding volume fraction of martensite, $f(T_a)$, are given in detail in [10]. By plotting ΔM_s as a function of $f(T_a)$ in Fig. 3 for two alloys with low Ni-content, it can be seen that ΔM_s increases with increasing $f(T_a)$. By plotting ΔM_s as a function of $M_s - T_a$, which can be assumed to be proportional to the volume fraction of martensite, for Fe-30Ni-0.3C alloy in Fig. 4, it can be seen that ΔM_s increases with increasing $M_s - T_a$, i.e., it increases with increasing $f(T_a)$. These results indicate that, for both paramagnetic and ferromagnetic austenite phases, the stabilization phenomenon is strongly related to the amount of martensite formed previously in specimens. Under the same aging conditions, the larger the amount of martensite formed during the first cooling, the more difficult will be the transformation of retained austenite into martensite during the second step of cooling.

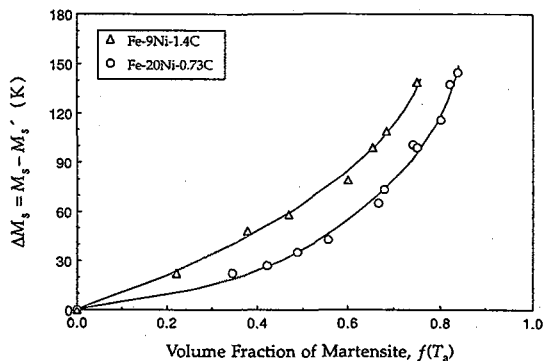


Figure 3: ΔM_s as a function of the volume fraction of martensite formed during the first step of cooling for the alloys indicated.

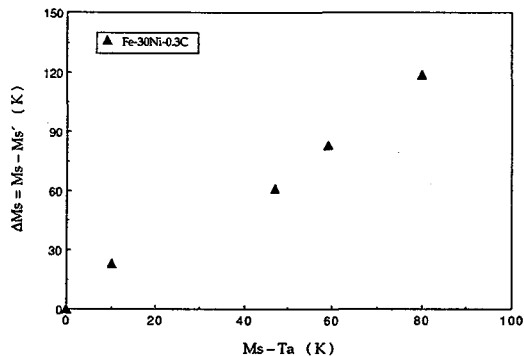


Figure 4: ΔM_s as a function of undercooling below M_s during the first step of cooling, $M_s - T_a$, for an Fe-30Ni-0.3C alloy.

It is known that, when a crystallite of martensite phase is produced, accommodation of the volume expansion and transformation shear strains leads to a spontaneous deformation of the austenite surrounding the growing martensite embryo, which will produce dislocation arrays in the austenite/martensite boundaries [11] and an internal stress field in the retained austenite phase [12,13,14]. When the athermal martensitic transformation is stopped at an arrest temperature T_a , and aging occurs, segregation of carbon atoms to martensite/austenite phase boundaries and relaxation of stresses at these boundaries will not only reduce the amount of available nucleation sites but also anchor the martensite/austenite phase boundaries.

X-ray diffraction results [13,14], have also shown that the austenite lattice parameter decreases linearly with decreasing temperature before the martensitic transformation starts, while a significant drop at the start of martensitic transformation occurs and it decreases continuously with lowering the temperature (increasing the amount of martensite). The austenite lattice parameter and the austenite peak width was not recovered to the original values when the sample was heated back to room temperature. In addition, the diffraction peaks of the austenite phase broadened. The decrease in austenite lattice parameter during cooling at temperatures above M_s temperature is due to the thermal contraction of the lattice. The broadening of the austenite diffraction peaks due to martensitic transformation was explained by a non-uniform internal stress field in retained austenite due to the existence of the martensite phase, while the reduction of the austenite lattice parameter was attributed to the result of a compressive stress field acting on the retained austenite by its surrounding martensites. The results indicate that the internal stress field in retained austenite induced by the martensitic transformation can not relax completely at room temperature. Although it is generally accepted that the retained austenite is stressed during the martensitic transformation, a complete pattern of the internal stress field is still not yet obtained.

Since the internal stress in retained austenite increases with increasing the amount of martensite and the internal stress field exists in retained austenite (can not relax completely) even after the sample was aged at RT [13,14], it is then a logical approach to assume that the further martensitic transformation process during the second cooling was inhibited by the internal stress. Thus, further cooling below T_a temperature is needed in order to obtain enough driving force for the transformation to take place.

Thus, we propose that the direct reason for the stabilization of retained austenite during two-step cooling experiments is due to the internal resisting stress induced by the previously formed martensite. Segregation of carbon atoms to martensite/austenite phase boundaries and stress relaxation at these boundaries can reduce the amount of available potential nucleation sites and anchor the previously formed martensite/austenite phase boundaries, and thus they are the necessary conditions for the stabilization phenomenon to occur.

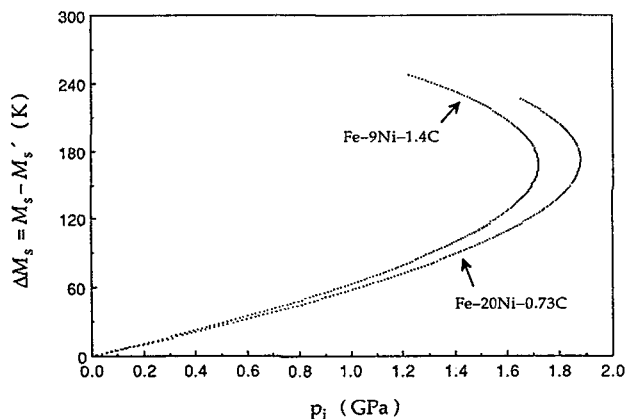


Figure 5: Calculated relationship between the hydrostatic pressure, p_i , and ΔM_s for the alloys indicated.

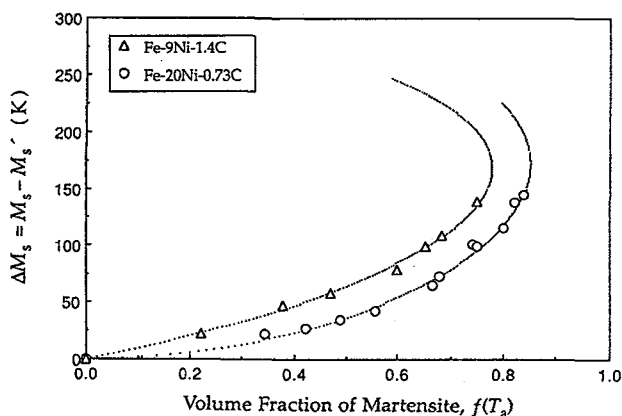


Figure 6: Calculated relationship (dashed lines) between volume fraction of previously formed martensite, $f(T_a)$, and ΔM_s for the alloys indicated. Δ and \circ are experimental results.

By assuming that the total internal stress resisting the subsequent martensitic transformation in retained austenite phase can be simplified as a hydrostatic compressive stress, p_i , the results can be analysed thermodynamically. According to Patel and Cohen [15], the change in M_s temperature due to the presence of the hydrostatic pressure (simplified internal resisting stress), p_i , can be calculated from the following equation:

$$\Delta G_c^{\gamma \rightarrow \alpha'} \Big|_{T=M_s'} - \Delta G_c^{\gamma \rightarrow \alpha'} \Big|_{T=M_s} + p_i \epsilon_0 V_m = 0 \quad (1)$$

Here the Gibbs chemical free energy change accompanying the transformation is calculated according to Kaufman and Hillert [16]. By using approximate values of $\epsilon_0 = 0.0298$ and $V_m = 7.11 \text{ cm}^3/\text{mol}$ [17], the relationship between p_i and ΔM_s can be calculated by using Eq. (1) as shown in Fig. 5 [8,10].

It needs to be kept in mind that the relationship between the internal resisting stress in retained austenite and the amount of previously formed martensite is not exactly known at present. However, if the

relationship between the internal resisting stress, p_i , and the amount of previously formed martensite, f , obeys a quadratic relationship [8, 10]:

$$p_i = af + bf^2 \quad (2)$$

the calculated relationship between ΔM_s and $f(T_a)$ fits well with the experimental data as shown in Fig. 6.

4. CONCLUSIONS

Based on the results obtained in the present work the following conclusions can be drawn:

- 1) The magnetic properties of alloys do not have drastic effects on the nature of the stabilization of retained austenite. The stabilization phenomenon can also be observed in an alloy having very low carbon content, *i.e.*, Fe-33.5Ni-0.009C.
- 2) Under the same aging conditions, the change in M_s temperature, ΔM_s , increases with increasing the amount of previously formed martensite, $f(T_a)$. The transformation of retained austenite to martensite can be completely inhibited if the arrest temperature, T_a , is low enough.
- 3) It is proposed that the direct reason for the stabilization of retained austenite during two-step cooling experiment is due to the internal resisting stress induced by previously formed martensite. The segregation of carbon atoms to lattice defects and martensite/austenite phase boundaries and the stress relaxation at these boundaries are the necessary conditions for the stabilization phenomenon to occur.
- 4) If the internal resisting stress in retained austenite can be simplified to be a hydrostatic compressive stress, p_i , and if p_i and $f(T_a)$ obey a quadratic relationship: $p_i = af + bf^2$, the calculated relationship between ΔM_s and $f(T_a)$ fits well with the experimental results.

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