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ATHERMAL, STRESS AND STRAIN INDUCED TRANSFORMATIONS IN MULTIPHASE STAINLESS STEELS

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ABSTRACT.- An investigation has been made of athermal, stress and strain induced transformations in multiphase (duplex, triplex and quadruplex) stainless steels with a chemical composition of the type 301, 302 and 304. Special thermomechanical treatments consisting in various sequences of athermal transformations, deformation induced transformations, deformation, tempering and recrystallization were developed. This resulted in multiphase microstructures consisting of composite networks of austenite, ferrite, athermal martensite and stress/strain induced martensite (MFAFM) with high strength and high ductility. The effect of dispersed particles of ferrite, athermal martensite, γ/a interfaces, γ/twin and grain boundaries, amount of deformation and deformation temperature on the nucleation, growth, morphology, substructure, strength and ductility of the transformation products are discussed.

DESIGN OF MULTIPHASE STAINLESS STEELS/EXPERIMENTAL METHODS

During the present investigation four types of microstructures were designed and studied: (I) duplex: austenite (γ) and deformation induced martensite (MD); (II) triplex: austenite, athermal martensite (MA) and deformation induced martensite; (III) triplex: austenite, ferrite (α or δ) and deformation induced martensite and (IV) quadruplex: austenite, ferrite, athermal martensite and deformation induced martensite. The first and second type of microstructures were developed through the application of type I of the thermomechanical treatments and the third and fourth type of microstructures were developed through the application of thermomechanical treatments type II and type III. Thermomechanical treatments type I were designed to obtain a dispersion of deformation (stress or strain) induced martensite and/or athermal martensite within an austenitic (containing between 6 and 12 wt % nickel), stainless (containing between 16 and 20% chromium) matrix. This microstructure was then submitted to thermomechanical treatments type II: deformation at different temperatures to 50% strain and then tempering prior to recrystallization within the α+γ or δ+γ phases field; followed by water quenching, liquid nitrogen requenching and deformation to a strain sufficient to obtain a considerable volume fraction (20.3) of deformation induced martensite. In order to achieve further refinement these microstructures were submitted to thermomechanical treatments type III, which consisted in the reapplication of one or various cycles of thermomechanical treatments type II. This resulted in a very fine micromultiphase structure, with a typical average grain size between 1 and 5 μm.

RESULTS AND DISCUSSION

Microstructures Type I and II/Thermomechanical Treatments Type I

Abundant formation of deformation induced (stress and/or strain) martensite was observed in all steels at all temperatures within the range 77K to 273K. This resulted in a considerable increase in the diamond pyramid hardness (DPH), yield strength (YS) and ultimate tensile strength (UTS), but was accompanied by a significant decrease in the elongation (Figs. 1 and 2). For the same amount of deformation, the amount of deformation induced martensite and the increase in DPH, YS and UTS were observed to increase with decreasing deformation temperatures for all steels investigated. As shown in figures 1 and 2 a much higher volume fraction of martensite and a much higher strength were obtained for a given amount of deformation at 77K than at 298K. This is expected since the temperature range 77-298K falls within the temperature...
Fig. 1 Effect of the strain and temperature on martensite formation and mechanical properties of: (a) & (b) 302, (c) & (d) 304 and (e) & (f) 304 B stainless steels.

range \( H_a - H_d \) for the 302 and 304 steels and within the latter the total driving force for the formation of martensite \( \Delta F_T \) increases with decreasing deformation temperatures. This results in a higher amount of martensite being formed for the same amount of strain at lower temperatures and a lower stress and/or strain being needed to form the same amount of martensite at lower temperatures.

Assuming that the nucleation of martensite is heterogeneous and without specifying any shape or size for the embryos, the thermodynamic driving force for the formation (nucleation and growth to the measured completion) of martensite can be expressed by the following equation:

\[
\Delta F_T = \Delta F_V + \Delta F_S + \Delta F_E + \Delta F_D
\]

where \( \Delta F_T \) is the total free energy change measured per unit volume due to the formation of martensite (athermal, stress assisted and/or strain induced); \( \Delta F_V \) is the free energy change associated with the bulk material, largely determined by electronic configurations; \( \Delta F_S \) is the surface energy associated with the atomic misfit between the parent (austenite) and product (martensite) phase; \( \Delta F_E \) is the stress which has to be overcome to shear the austenite into martensite, which includes the free energy change associated with the growth of one martensite crystal (block, lath or plate) in the strain field created by previously formed martensite crystals and \( \Delta F_D \) is the free energy of the microstructural defect which is destroyed due to the formation of martensite. The terms (\( \Delta F_S \), \( \Delta F_E \) and \( \Delta F_D \)) are always positive and therefore oppose the transformation at all temperatures. The terms \( \Delta F_V \) and \( \Delta F_D \) may become negative and have to overcome the terms \( \Delta F_V + \Delta F_E + \Delta F_D \) in order that the martensite crystals can complete their transformation.

For each two deformation temperatures the experimentally measured differences in the volume fractions of martensite and in the correspondent strength (DPH, YS, UTS) were observed to be a function of the amount of deformation, a maximum being obtained in the deformation range of 20% to 40% strain. It was observed that in the 304B steel the amount of martensite formed by deformation in the temperature range 77-298K increased continuously and very rapidly with increasing strain through the entire strain range. However in the case of the 302 and 304 steels the amount of martensite formed exhibited plateaux between 15% and 35% strain, as shown in figure 1.
Fig. 2 Nucleation, morphology and substructure of martensite upon TMT Type I: (a) $M_D$: lenticular twinned: 302 $\varepsilon=2\%$, 77K; (b) $M_D$: very fine lenticular nucleates at $M_A$: zig-zag dislocated and $T_1$: 304B, $\varepsilon=2\%$, 77K; (c) $M_d$: zig-zag dislocated: 304B, $\varepsilon=8\%$, 77K; (d) $M_d$: very fine lenticular nucleates at $M_d$: dislocated: 304B, $\varepsilon=16\%$, 77K; (e) $M_D$ nucleates at $T_1$: 304, $\varepsilon=10\%$, 298K and (f) $M_1$: lenticular twinned and faulted: 304, $\varepsilon=15\%$, 298K; internal twins in plate $P_1$ continue in the austenite as plates $P_2$ & $P_3$.

Fig. 3 The role of $M_A$ and $M_D$ on tempering and on the nucleation of $\alpha/\delta$ and $\gamma$ during recrystallization and of $\alpha/\delta$ and $\gamma$ on the formation of $M_1$ and $M_D$ upon TMT Type II: (a) 304B, 673K; (b) 304, 773K and (c) 301, 1073K followed by 10% strain at 298K.
This is attributed to a transition from stress assisted to strain induced martensite in the 302 and 304 steels between 15% and 35% strain. However in the 304B steel martensite is considered to be strain induced through the entire strain range. Those plateaux coincide with the last stage of formation of the stress assisted martensite and can be explained by the partial stabilization of the austenite relatively to the formation of stress assisted martensite, due to previous transformation. This is due to the transformation itself and the fact that the specific volume of martensite is 4.3% larger than that of the parent austenite, which cause a distortion that must be accommodated by the generation of dislocations, twins or stacking faults within the austenite. The reverse of the austenite stabilization (i.e. instabilization relatively to the formation of strain-induced martensite) would be expected if the martensite was strain-induced, due to the autocatalytic effect associated with the formation of nucleation sites by plastic deformation which would sharply increase the slope of the curve, as observed in the 304B steel. It is also important to explain why this stabilization of the austenite, observed only in the last stages of formation of stress assisted martensite (in the 302 and 304 steels) does not occur for strain levels below 15%. This is attributed to the fact that the mechanical work (8% to 15%) produced during deformation is not being stored in the austenite through the formation of dislocations, twins or stacking faults, but is instead used as the strain energy need to propagate the martensite interface. This is in agreement and explains why no significant number of dislocations, twins or stacking faults were observed in the austenite, even adjacent to the martensite plates.

The nickel content and the nickel equivalent of the 302 and 304 type steels is approximately the same and higher than those of the 304B steel. This results in a lower Ms temperature for the 302 and 304 steels. Therefore it is also expected that Ms (the temperature below which "yielding" of the austenite is initiated by martensite transformation, and above which yielding of the austenite is initiated by perfect or dissociated slip, i.e. M_S < M_S < M_F) in the 302 and 304 steels will be lower than that of the 304B steel. It is therefore possible for the formation of martensite in the 302 and 304 steels to be stress assisted for strain levels below 15% and strain induced for strain levels above 35%, where significant number of nucleation sites have been formed by plastic deformation.

In the 302 and 304 steels the morphology of the deformation induced martensite was observed to be considerably influenced by the deformation temperature. The number of variants of deformation induced martensite was observed to increase with decreasing deformation temperatures. At lower temperatures a greater number of shorter and thinner plates and plate variants were observed (Fig. 2). Upon deformation at 298K only one or two variants of martensite were observed within each austenite grain. However, upon deformation at 77K an average four variants of deformation induced martensite were observed. These observations are in agreement with previous results obtained by Marques for zirconium and titanium based deformation induced martensite (2,3) and are consistent with a stress assisted heterogeneous martensite formation. The stress assisted modes will be those whose transformation shear distortion is minimized by the applied stress. Thus the applied stress will only assist the formation of very few plate variants, the other being opposed. It is expected that the formation of these plates will produce strains parallel and normal to the habit plane. This is in agreement and explains the morphology of these martensites and in particular the orientation relationships between the successive variants of martensite (Figs. 2 and 3). The larger density of plates and plate variants observed at lower temperatures can be explained since at lower temperatures AFV becomes larger, AF becomes smaller, the critical radius decreases and the number of critical embryos increases. The smaller size of the plates observed after growth is completed, at lower temperatures, can be explained by the higher strength of the austenite and therefore a larger AFV.

Although the exact nature of the martensite embryos is not yet fully established it is expected (2) that the stress assisted nucleation of martensite depends on the same nucleation sites or embryos which are responsible for the usual athermal transformations. This would explain the effect of the deformation temperature on the morphology of the stress assisted martensites observed in the 302 and 304 stainless steels. However, the strain induced nucleation of martensite depends on the creation of new sites and embryos by plastic deformation, with an expected autocatalytic nucleation effect. It is generally believed that local stress concentrations act as
nucleation sites. Intersections of two active slip systems of the type (111)<112>FCC (4); intersection of two ε martensite plates (5); intersection of an ε plate with an austenite twin and/or grain boundary (7,8) and strain induced stacking faults (5,9,10) have been suggested as possible embryos. However in each of the above cases the region of intersection is defined as a lath or rod shaped volume which controls the morphology of the strain induced martensite, but which is not significantly influenced by the deformation temperature. This further supports the view that the present deformation induced martensites, observed in the 302 and 304 steels are stress assisted. During this investigation it was observed that the deformation induced martensite nucleated heterogeneously at the following favorable sites: (a) annealing twins within the austenite (Fig. 2); (b) athermal martensite plate boundaries (Fig. 2 and 3); (c) austenite grain boundaries (Fig. 2); and (d) interfaces between the austenite and ferrite phases (Fig. 3).

Upon quenching/requenching to 77K a significant amount of athermal martensite was observed in the 3040 steel, but not in the 302 or 304 steels. This is explained by the lower nickel content of the 304B steel and consequently higher Ms temperature. This athermal martensite had a lenticular zig-zag morphology (1) with a large number of plate variants which made it easily distinguished from the deformation induced martensite (Figs. 2 and 3).

The substructure of the deformation induced martensites was observed to be highly faulted and mainly internally twinned (Fig. 2). This heavy internal faulting is probably associated with stresses due to the complex interactions between the lattice invariant deformation occurring during the martensite formation and the applied stress/strain. However the substructure of the lenticular zig-zag athermal martensite was observed to be dislocated. The factors that control the morphology, substructure and the morphological and substructural transitions are discussed elsewhere (1). The athermal martensite in the 304B steel was observed to provide a considerable volume fraction of nucleation sites for the formation of deformation induced martensite and to considerably increase the volume fraction of Ms upon subsequent deformation at all deformation temperatures (Figs. 2 and 3). The role of the athermal martensite on subsequent nucleation of the deformation induced martensite is shown in figure 1: for a 304B steel 100% of Ms was obtained with 20% strain at 77K and 45% strain at 298K. However in the 302 steel 100% Ms was obtained with 40% strain at 77K and 50% strain at 298K only produced 65% Ms. In the 304 steel 50% strain only produced 85% Ms at 77K and 55% Ms at 298K.

Upon thermomechanical treatments type I and for a given pair: amount of deformation-deformation temperature, the amount of martensite induced by deformation was observed to be controlled mainly by the nickel content of the austenite phase of the particular steel and the amount and type of athermal martensite previously formed during quenching/requenching to 77K.

In order to increase the strength (DPH, YS, UTS) of these stainless steels with a small decrease in their formability and fracture toughness a second type of thermomechanical treatments (Table I) was applied to the microstructures obtained at the end of TMT type I. During the deformation of TMT type II the dispersed crystals of athermal martensite and deformation induced martensite were observed to play four important roles: (a) Ms provided considerable volume fraction of sites for the nucleation of Ms, in addition the dispersed crystals of both Ms and Ms were observed to: (b) provide sites for the nucleation of a large number of dislocations; (c) provide sites for the precipitation of carbide particles during tempering; (d) provide sites for the nucleation of recrystallized grains of ferrite and austenite; and (e) impede the motion of reaction fronts, which considerably limited the grain growth during recrystallization. As shown in Table I and figure 3 for a 302/304 steel annealing at 674K resulted in a multiphase structure containing ferrite, austenite and 80% retained martensite. Annealing at higher temperatures (973K and 1073K) resulted in very fine (4-5 μm: 973K and 7-10 μm: 1073K) fully recrystallized austenite-ferrite microstructures. Similar microstructures were obtained in the 304B steel, however both the recrystallization temperatures and the grain sizes achieved were shifted to lower values, which is explained by the role played by the athermal martensite. These recrystallized microstructures were water quenched and requenched
into liquid nitrogen and further deformed to various strain levels by rolling in the temperature range 77K to 298K. This again resulted in considerable amounts of transformation of the austenite into martensite at all temperatures. The achievement of such fine grain size with only one cycle of TMT is attributed and explained by the role played by the disposed crystals of MA and MD.

**TABLE 1** Thermo-Mechanical Treatments Type II (1 Cycle: 1-6) and Type III (2 Cycles: 1-8). Results for a 302 steel.

<table>
<thead>
<tr>
<th>Temperature</th>
<th>Phase Nature After WQ</th>
<th>Phase Nature After 10% Deformation</th>
</tr>
</thead>
<tbody>
<tr>
<td>673</td>
<td>$\alpha + \gamma + M_R(80%)$</td>
<td></td>
</tr>
<tr>
<td>773</td>
<td>$\alpha + \gamma + M_R(30%)$</td>
<td></td>
</tr>
<tr>
<td>973</td>
<td>$\alpha + \gamma + M_R(20%)$</td>
<td></td>
</tr>
<tr>
<td>1073</td>
<td>Recryst. Grains (4-5/2-3μm)</td>
<td>Recryst. Grains</td>
</tr>
<tr>
<td>1173</td>
<td>Recryst. Grains (7-10/5-6μm)</td>
<td>Recryst. Grains</td>
</tr>
</tbody>
</table>

Micromultiphase Structures Type III and IV/Thermomechancial Treatments Type III

In order to refine the microstructures even further two and three cycles of TMT were carried out (Table 1, Fig. 3). This resulted in refined recrystallized micromultiphase structures containing ferrite and austenite with a very high density of annealing twins ($T_a$). Typical grain sizes for 302 and 304 steels after two cycles of TMT and recrystallization at 1073K were between 2 and 3 μm. After three cycles of TMT the 304B steel exhibited grain sizes of approximately 1.5 μm. These micromultiphase structures were water quenched and requenched to liquid nitrogen and further deformed to various strain levels within the temperature range 77K to 298K. This resulted in the formation of very fine crystals of $M_D$ in the 302 and 304 steels and in a mixture of very fine $M_A$ and $M_D$ crystals in the 304B steel. These microstructures have UTS values above 200 Kpsi and tensile elongations above 80%.

REFERENCES
5. VENABLES, T.A., Phil. Mag. 7 (1962) 35.