Precipitation hardening of a FeMnC TWIP steel by vanadium carbides

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Abstract. A fine precipitation of spherical vanadium carbides is obtained in a Fe22Mn0.6C base steel during the final recrystallisation heat treatment. Precipitates formed in recrystallised grains have a cube-cube orientation relation with the matrix, confirmed by Moiré patterns observed in TEM. The theoretical size for loss of coherency is below the nm, much lower than the precipitates’ size. Deformation contrasts were observed around the precipitates and their residual coherency was measured. It was shown to decrease when the carbides’ size increases, to vanish above 30 nm. The net increase of the yield stress was estimated to be 140 MPa. Precipitation hardening by vanadium carbides do not alter the strain hardening rate by TWIP effect, as they do not seem to act as obstacles for the propagation of microtwins.

1. Introduction

The Fe22Mn0.6C TWIP steel has a low stacking fault energy and deforms by twinning at room temperature in addition to dislocation glide. Microtwins of secant variants form in each grain and define subgrain cells, which size decreases along with strain [1]. The microtwins stored in stacks [2] act as strong obstacles for dislocations and progressively decrease their mean free path. The resulting dynamical Hall&Petch effect leads to high strain hardening rate and an outstanding compromise between elongation (> 50\%) and mechanical resistance (> 1 GPa). On the other hand, the yield stress of this steel is rather low, below 400 MPa depending on the grain size, but can be increased by precipitation hardening. If the hardening effect of intragranular precipitates by blocking dislocations is well known, their interaction with mechanical twins has not been systematically studied. It is important that the presence of fine precipitates do not also block the expansion of the microtwins, in order to keep the high hardening rate. We characterize the precipitation of vanadium carbides by TEM, their interaction with twins and their resulting effect on the mechanical properties.

2. Studied material

The studied material is an austenitic Fe22Mn0.6C (wt\%) base grade with 0.21 at\% of vanadium furnished by ArcelorMittal, hot rolled at a temperature higher than 900\°C and air quenched at 20\°C/s. At this stage the steel is fully recrystallised and only 50 ppm of vanadium has precipitated, as predicted by models [3]. Sheets are then cold rolled down to a thickness of 1.2 mm and undergo a final recrystallisation and vanadium carbide precipitation optimized heat treatment [4]. Recrystallisation is...
complete and the final average grain size is 1.7 µm. TEM samples were prepared by mechanical (tripod) and ion (PIPS) polishing. The presence of fine intragranular spherical precipitates is confirmed by TEM observations. Their diameter varies from 2 to 18 nm with an average value 

\[ \langle d \rangle = 6.8 \text{ nm} \] 

and about 3 % of larger precipitates, up to 100 nm in size, are observed.

3. Lattice relation between austenite and vanadium carbides

3.1. Theoretical approach

The vanadium carbide phase has a stoichiometric VC composition and a B2 structure (NaCl type): f.c.c. vanadium structure with carbon atoms in each octahedral site. The lattice parameter is 

\[ a_{VC} = 0.4165 \text{ nm} \]

A cube-cube orientation relation in γ-austenite has been reported in the literature [5]: 

\[ (001)_γ // (001)_{VC}, [100]_γ // [100]_{VC} \]

The lattice parameter of Fe22Mn0.6C is 

\[ a_γ = 0.3605 \text{ nm} \] and the lattice mismatch with VC is high: 

\[ \delta = \Delta a/a_γ = 15.4 \% \]

If we consider a semi-coherent precipitate with a lattice mismatch \( \delta < \delta \) relaxed by interface dislocations, its total energy is:

\[ E_p = 4\pi R^2 e_s + E_{el}^{VC} (\delta^*) + E_{el}^γ (\delta^*) \]

where \( e_s = e_0 + e_{dis} \) is the energy per unit surface of the interface and \( E_{el}^{VC} \) and \( E_{el}^γ \) are the elastic energies stored respectively in the precipitate and the infinite matrix. \( e_0 \) is the specific surface energy of a coherent interface and \( e_{dis} \) is the energy of the interface dislocations, given by [6]:

\[ e_{dis} = \frac{\mu_γ b}{2\pi^2} \left[ 1 + \beta - (1 + \beta^2)^{1/2} \right] - \beta \ln \left[ 2\beta (1 + \beta^2)^{1/2} - 2\beta^2 \right] \]

with

\[ \beta = \frac{\pi (\delta - \delta^*)}{1 - \nu_γ} \]

\( \mu_γ = 60 \text{ GPa} \) and \( \nu_γ = 0.3 \) are respectively the shear modulus and Poisson ratio of the matrix, assumed equal to the ones of the precipitate in this expression and \( b = 0.255 \text{ nm} \) is the Burgers vector. In the case of a coherent precipitate, \( \delta = \delta \) and \( e_{dis} = 0 \). The elastic energy can be treated as in the case of an Eshelby spherical inclusion, with a radial displacement field and an eigenstrain applied to the precipitate equal to -\( \delta^* \). The general solution for the displacement field in spherical coordinates is:

\[ u_r = Ar + Br^{-2} \]

where A and B are constants specific to the precipitate and the matrix. After elastic relaxation of the eigenstrain, the strain induced by the precipitate is \( \delta^* \). By solving the problem with the boundary conditions: \( u_0(0) = 0 \) and \( u(R) = \delta - \delta^* \) for the precipitate and \( u(R) = \delta^* \) and \( u(\infty) = 0 \) for the matrix, the continuity of the radial stress at the interface leads to:

\[ \delta = \delta^* \left[ 1 + 4\mu_γ (3\lambda_{VC} + 2\mu_{VC})^{-1} \right]^{-1} = 0.75 \delta^* \]

where \( 3\lambda_{VC} + 2\mu_{VC} = 750 \text{ GPa} \), calculated from [6]. The elastic energies are thus:

\[ E_{VC} = 2\pi (3\lambda_{VC} + 2\mu_{VC}) (\delta^* - \delta^*)^2 \pi^3 \quad \text{and} \quad E_γ = 8\pi\mu_γ \delta^*^2 \pi^3 \]

Coherency is energetically favorable for small radii and semi-coherency for large radii. By an iterative calculation on \( \delta^* \) and R, the critical radius for the loss of perfect coherency is \( R_{crit} = 0.275 \text{ nm} \), which is very small due to the high initial lattice mismatch, and \( \delta^* = 2.9 \% \). No perfectly coherent precipitate should be observed and semi-coherent precipitates should have a small residual coherency.

3.2. TEM observations

Spherical precipitates are observed, as shown in Figure 1. In two wave conditions and certain values of the Bragg vector s, Moiré patterns perpendicular to the diffraction vectors g are observed in small precipitates (Figure 1.a) with a wave length of 1.6 nm for \( g = <111> \). Interferences between beams diffracted by parallel planes with different intertercular distances \( d_{VC} \) and \( d_r \) leads to fringes distant by

\[ \Delta s_{moire} = d_{VC} d_r (d_{VC} - d_r)^{-1} = 1.56 \text{ nm} \] in the case of unstrained \{111\} planes in the two phases. This is consistent with the cube-cube orientation relation and small deformations due to residual coherency.
In other conditions, deformation contrasts are observed around the precipitates (Figure 1.b), which confirms their semi-coherent character. The coffee bean shape of these contrasts is characteristic of a radial displacement field $u$ around the precipitates: in the plane normal to $g$, $g \cdot u = 0$ and the contrast vanishes. The displacement scales like $r^{-2}$ and the width of the contrast increases with the deformation due to residual coherency. By measuring the width at which the intensity around the precipitate is 80% of the continuous intensity of the micrograph (see Figure 2.a) and using the method and tables proposed by [8], we measured the residual coherency of several precipitates. As shown in Figure 2.b, the residual coherency of small precipitates decreases when their size increases. A linear regression predicts that precipitates lose their semi-coherent character above 30 nm in diameter.

Larger precipitates show neither Moiré pattern nor deformation contrast. As precipitation and the final recrystallisation occur during the same heat treatment, these large precipitates formed in not recrystalised grains and are randomly oriented with the matrix and incoherent after the recrystallisation is complete. Small precipitates nucleated in recrystallised grains have the cube-cube orientation and a semi-coherent character below 30 nm in size.

### 4. Mechanical properties

Figure 3.a shows the true stress-strain curves of the present steel and of the reference Fe22Mn0.6C carbide free steel with a similar grain size (2.5 µm). Knowing that the contribution of the grain size $D$ to the yield stress is $0.4 \mu(b/D)^{1/2}$ in FeMnC steels, the estimated effect of VC is an increase of the yield stress by 140 MPa. The theoretical hardening by incoherent precipitates is given by [9]:

$$\Delta \sigma_{th} = M \cdot 0.81 \left( \frac{6 f_y}{\pi} \right)^{1/2} \frac{\mu v b}{<d>},$$
where \( M \sim 3 \) is the Taylor factor, and \( f_v \) is the volumetric fraction of precipitates. If all the vanadium had precipitated, \( f_v = 0.32\% \) and the resulting hardening would be \( \Delta \sigma_h = 430 \text{ MPa} \). This shows that an important quantity of vanadium is still in solid solution and/or that the hardening efficiency of the large precipitates is poor, while they consummate a high amount of vanadium.

Figure 3a also shows that the strain hardening rate is not affected by the presence of carbides: the entire curve is simply shifted to higher stresses by the amount of the initial hardening. The presence of precipitates does not alter the TWIP effect. Carbides do not seem to block microtwins, as shown on Figure 3: a microtwin continues on both sides of a precipitate. Although unknown, the crossing mechanism is likely to be of Orowan type because the friction stress of carbides is very high and their residual coherency is low. But, as the microtwin is 13 dense plane thick, it leaves 13 Shockley loops at the interface, which induces high stresses and should promote the emission of secondary dislocations.

**Figure 3.** Compared true stress-strain curves of the precipitation hardened steel and the reference steel.

**Figure 5.** TEM micrograph of a carbide on the path of a microtwin seen edge-on.

### 5. Conclusion

A fine precipitation of vanadium carbides increases the yield stress of a Fe22Mn0.6C by 140 MPa, without decreasing the strain hardening rate. The hardening is performed by small precipitates formed in recrystallised grains. They are semi-coherent below 30 nm in size and have a cube-cube orientation relation with the matrix. Larger precipitates formed in non recrystallised grains do not participate much to hardening. The presence of carbides does not seem to block the propagation of microtwins and do not alter the hardening efficiency by TWIP effect.

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