Mechanical properties of low carbon steel hardened by the Fe$_2$SiTi phase at high volume fraction

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Abstract. The addition of Si and Ti to low carbon steel allows to obtain a nanometric precipitation of an ordered intermetallic phase, Fe$_2$SiTi, with a volume fraction of the order of 6%; which is much higher than usual for microalloyed steels and more comparable to the situation encountered in aluminum alloys. The resulting precipitation hardening leads to extremely high mechanical properties, depending on the optimization of metallurgical route, which makes these materials very promising for the development of innovative, weight saving automotive solutions. In this contribution the precipitation sequence and kinetics are studied in details by a combination of experimental techniques including small angle neutron scattering and scanning or transmission electron microscopy. Some properties (yield stress, strain hardening, strain to fracture) are discussed in view of the measured precipitate characteristics, notably their size and volume fraction. The fracture mechanism, and particularly the occurrence of brittle fracture, is discussed in view of the state of precipitation.

1. Introduction

The Iron-Silicon-Titanium (Fe-Si-Ti) system is known since the 1930’s to show precipitation hardening capability. It has been further assessed by several authors (e.g. [1]), that the Fe$_2$SiTi phase provided significant precipitation hardening to steels. However, it was also found ([1], [2]) that such precipitation-hardened alloys are rather brittle, limiting the possible applications of this system. Obtaining a useful compromise of properties is only possible if the development of microstructure and related mechanical properties is rationalized. The purpose of this work is therefore to carry out a systematic quantitative investigation of precipitation kinetics and mechanical properties along various heat treatments and to investigate the microstructure / property relationships, on a ternary Fe-Si-Ti alloy.

2. Methodology

The iron-based alloy studied here contains 2.5wt% Silicon and 1wt% Titanium. It was hot rolled between 1250°C and 950°C with long interpass times (30 s), in order to make recrystallization easier. It was then cold rolled up to 75% thickness reduction in the aim to have smaller ferritic grains. The cold-rolled samples, 0.9mm in thickness, were then subjected to heat treatments starting with a quench from 900°C (temperature of solution treatment) followed by ageing treatments between 450°C and 650°C for different times. The matrix remains fully ferritic all along the heat treatment, including the 900°C solution treatment, with a grain size of about 25µm.

The methods used for precipitation characterization were: Scanning Electron Microscopy with a Field Emission Gun (SEM-FEG), Transmission Electron Microscopy (TEM) and Small Angle Neutron Scattering (SANS), carried out at ILL on beamline D11 (Grenoble, France). SANS measurements were carried out under a high magnetic field. Both the nuclear and magnetic contributions could be separated and analyzed to give precipitate size (using a standard Guinier
analysis) and volume fraction values (using the Fe$_2$SiTi composition for nuclear contrast and the magnetic hole assumption for magnetic contrast). The nuclear and magnetic contributions were shown to provide consistent information. Here only the results from the magnetic contribution will be presented (for more details about the method see [3]). For the study of mechanical properties microhardness (Vickers) and tensile tests were performed.

3. Results

3.1. Precipitation characterization

Microscopic observations (Figure 1a, b, c and d) confirm that a nano-scale precipitation occurs during ageing. Depending on the ageing temperature, the precipitates can reach diameters ranging from 3nm to 100nm. The images obtained by SEM-FEG show a large number of precipitates spread homogeneously within the grains (Figure 1a). Electron diffraction patterns confirmed that the precipitate crystal structure is consistent with the metastable phase Fe$_2$SiTi (fcc, a = 0.5709 nm) (Figure 1c). More detailed observations made on TEM thin foils confirm the observations made with the SEM-FEG (Figure 1b and d): radius of about 10nm and density of 1e$^{17}$ precipitates per m$^3$.

Quantification of the precipitate average size was achieved on dark-field TEM images for selected heat treatments (see Figure 2a). More systematic and global information were obtained from the SANS measurements. For heat treatments in the temperature range 500°C-580°C, Figure 2a shows the average precipitate size obtained by SANS and TEM, while the evolution of precipitate volume fraction derived from SANS is given in Figure 2b.

Figure 1: observations of Fe$_2$SiTi precipitates with a) SEM-FEG for sample aged at 550°C for 3h; b) TEM bright field for sample aged at 550°C during 3h; c) TEM diffraction pattern in [110] axis (sample aged at 550°C for 6h) and d) TEM dark field for sample aged at 550°C during 15h
Figure 2a shows the evolution of the mean radius of the precipitates in Fe-Si-Ti system, obtained by SANS measurements. Two remarkable aspects of the radius evolution can be noticed. First, a reduction of the mean radius is observed after 3 hours ageing. Secondly, the TEM measurements on dark field images, give radius values that are in good agreement with SANS measurements for samples aged under 3 hours (1h25 and 2h). Values for samples aged 3 hours and more are not consistent anymore with the ones measured with TEM using picture Figure 1d (3nm vs. 10nm). These two elements could be explained by the formation of a new precipitate population at this stage. In fact, SANS method enables to measure the radius and volume fraction of the phase which is present predominantly. Hence, if we imagine the nucleation of a new phase at about 3 hours ageing with a smaller size, the apparent mean radius would decrease suddenly. Thus, if the new phase nucleates rapidly, we could imagine that in SANS observations these are predominant. This would also explain why the TEM measurements are not in agreement, since with TEM we can only see the bigger particles (3nm is probably too small for TEM resolution here). These observations are to be brought in parallel with the measures of microhardness performed on the same samples (see section 3.2). This possible nucleation of a new phase was already mentioned in former studies ([1]), though it needs to be confirmed by additional observations: atom probe or by high temperature Differential Scanning Calorimetry (DSC) could be used.

3.2. Mechanical properties
Microhardness tests showed a wide range of hardening values depending on the annealing temperature (Figure 3a). The first hardness peak observed at 550°C appears after 2 hours, whereas, at 500°C, hardness still increases after 24 hours, in agreement with the slower precipitation kinetics at lower temperature measured with SANS. Peak hardness is observed to be associated with a precipitate radius of about 3 nm at 550°C.

In order to find a relation between precipitate size, volume fraction and the ductility, tensile tests were performed (Figure 3b). These show the 3.5 nm large precipitates at peak hardness for 550°C give the best compromise between yield stress and ductility. The sample over-aged for 3h has a deformation of 15% but the yield stress decreases suddenly, probably due to a change in the deformation mechanism, namely from shearing to Orowan by passing of the precipitates.
Figure 3: a) Hardness evolution of the Fe-Si-Ti as a function of time at various temperatures and b) tensile test curves showing the evolution of true stress with true strain for different annealing conditions

For samples with very small precipitates (2-3nm) as for example at 500°C on Figure 2a, the hardness reaches very high values (see Figure 3a). However, tensile tests performed on these samples reveal a very brittle behavior (cleavage fracture observed) with a uniform elongation of only 2-3% (Figure 3b). Thus the presence of very small precipitates with high volume fraction does not provide a satisfying strength-ductility compromise. A better solution would be to increase the radius at the cost of a decrease of precipitate density like for example at 550°C. Hardness peak is reached more rapidly, at lower value, but the uniform elongation can reach up to 15%.

4. Discussion

We have seen that, contrary to what has been said in earlier works ([1] and [2]), Fe-Si-Ti alloys can combine remarkable hardening characteristics and interesting deformation capabilities, depending on the heat treatment temperature. This is the result of the choice of an appropriate compromise between mechanical properties and precipitate size and density. Our measurements provide evidence that age hardening at low temperature with a high density of extremely small precipitates leads to embrittlement. At precipitate sizes equal to or above 3nm (corresponding to the ones at peak strength), part of the precipitates become non-shearable, which gives rise to a less pronounced localization of plastic flow and probably explains the ductility improvement.

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