ANALYSIS OF HEAT TREATMENT EFFECT ON MICROSTRUCTURAL FEATURES EVOLUTION IN A MICRO-ALLOYED MARTENSITIC STEEL

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Abstract
The microstructural evolution of a quenched and tempered medium-C micro-alloyed steel during tempering is analyzed. The steel was heat treated in order to develop fully martensitic microstructures after quenching with different prior austenite grain sizes (AGS). Main results are a very poor effect of AGS on packet size is found, as well as, high-angle boundary grains do not significantly grow after tempering; on the contrary, low-angle grain boundaries (cells) move, fully justifying the hardness evolution with tempering temperature.

Keywords: martensite, microstructure, packets, cells, tempering

1 Introduction
Martensitic transformations can occur in many metals and alloys provided the cooling rate is rapid enough to prevent diffusion-controlled transformations [1-4]. The transition from austenite to martensite in steels is the best-known and most important martensitic transformation because of the technological importance of hardened steel. The martensitic phase in steel can be simply described as a super-saturated solution of carbon in the ferritic phase, in which the carbon content leads to a tetragonal distortion of the lattice. Austenite to martensite phase change occurs when the sum of mechanical energy due to externally applied stress and the chemical driving force exceeds a critical value. In particular, in the temperature range above \( M_s \) and below \( M_a \), when the externally applied stress becomes over the yield limit of parent austenite, the transformation is dominated by strain-induced nucleation on new nucleation sites created by the plastic strain, occurring predominantly at shear-band intersections [5-8]. However, how the phase nucleates, is even today not completely understood because the high speed of formation makes the martensitic transformation a difficult process to be studied experimentally. In order to gain more insight into the kinetics of the martensitic transformation, the transformation progress upon continuous cooling has been investigated quantitatively by several authors by means of different experimental techniques (electrical resistivity, dilatometry, acoustic emission, quantitative metallography) [9-12].

The need of better understanding the kinetics of martensite evolution is also driven by the need in the development of new steels with very stringent requirements in terms of strength/toughness combinations, for which tempered martensite appears to be the most promising microstructure [13], together with weldability [14] and high temperature resistance [15]. In this paper the microstructure evolution during Quenching and Tempering (Q&T) of martensite in a medium-C steel is investigated.

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2 Experimental
A 0.30C-0.65Cr-0.65Mo-0.03Nb-0.07V steel was investigated. Various Q&T treatments were carried out on 16 mm thick specimens: austenitizing was performed in a muffle at temperature of 920 °C, 1000 °C and 1150 °C for 10 minutes holding time, in order to develop different austenite grain sizes (AGS). Austenitization was followed by quenching in stirred water with a cooling rate (CR) measured by a thermocouple inserted at mid-thickness. Tempering was carried out in the range 620 °C to 680 °C for 1 hour holding time. Microstructures were observed by means of Light Microscopy (LM). The austenite grain boundaries were revealed by etching in a saturated aqueous picric acid solution containing a few drops of a wetting agent (Teepol) and HCl. The austenite grain size was measured according to ASTM E112. The microstructural features taken into consideration in the present study were packets, defined as the crystal domains delimited by high-angle boundaries (>15 degrees) and cells, crystal domains bounded by low-angle grain boundaries (<15 degrees). Packet and cell size were determined by Orientation Imaging Microscopy (OIM) using Electron Back-Scattering Diffraction (EBSD) patterns, with a step size of 0.15 μm. Cleaning of data was performed according to a confidence index higher than 0.1. By means of this technique, the surface of a crystalline material is scanned and in each point the local orientation is determined in a fully automatic way. From these measurements some microstructural characteristics of the material were estimated, e.g. misorientations and types of grain boundaries, crystallographic orientations. Vickers hardness HV10 was measured; 10 hardness values were measured for each specimen and the average value was considered.

![Fig. 1 LM micrographs and EBSD map of as-quenched materials with different average values of AGS](image)

AGS=8.5 μm  AGS=25.0 μm
3 Results and discussion
3.1 AGS effects
Materials with a fully martensitic microstructure after quenching were considered. EBSD examinations showed a very limited effect of the prior AGS on the packet size (Figs. 1 from a to d).

In Fig. 2 results are compared to similar measurements on low-C bainitic steels where, on the contrary, the bainitic packet size tends to increase with increasing austenite grain size [16-18].

![Packet size versus austenite grain size](image)

**Fig. 2** Packet size versus austenite grain size

3.2 Tempering behaviour
The effect of tempering temperature on hardness reduction is reported in Fig. 3 showing a loss of 100 HV\textsubscript{10} with tempering temperature varying from 620°C to 680 °C. Fig. 4 shows that the effect of tempering on high-angle boundary grains (packets) is negligible. As a matter of fact, growth of such grains is not activated by temperatures typical of the tempering process (620-680°C). On the contrary, grains with low-angle grain boundaries (cells) move. The increase of cell size with tempering temperature is the mechanism which justifies the hardness variation with tempering temperature. In fact, the hardness of quenched and tempered steels can be related to the cell size according to the following Hall-Petch relation [19]:

![Hardness as a function of tempering temperature](image)

**Fig. 3** Hardness as a function of tempering temperature. Each HV\textsubscript{10} value is the average of 10 measurements. Error bars indicate one standard deviation.
Fig. 4 Packet and cells size as a function of tempering temperature.

$$HV = HV_0 + k_h d^{-0.5}$$  \(1\)

It is also expected it should affect the fatigue behavior [20].

In Fig. 5 the hardness of tempered martensite is plotted as a function of the cell size, in comparison to the hardness of tempered bainitic and mixed bainitic/martensitic microstructure. A \(k_y\) value of 10.0 HV/mm\(^{-0.5}\) is found, fitting both martensite and mixed bainitic/martensitic microstructures on a wide range of hardness values (HV\(_{10}\)= 220-390).

Fig. 5 Hardness as a function of cell size \(d_{cell}\).

4 Conclusions

The main conclusions for martensitic microstructures can be summarized as follows:

- A very weak effect of AGS on packet size was found.
- Low-angle grain boundaries (cells) move during tempering, fully justifying the hardness variation with tempering temperature.
References