Contents lists available at ScienceDirect

Materials Science & Engineering A

Evaluation of dislocation density and interstitial carbon content in quenched and tempered steel by internal friction

J.J. Hoyos [a,*], A.A. Ghilarducci [a], D. Mari [b]

[a] Centro Atómico Bariloche, Comisión Nacional de Energía Atómica, Instituto Balseiro-Universidad Nacional de Cuyo, Consejo Nacional de Investigaciones Científicas y Tecnológicas, Av. Bustillo 9500, CH 8400 Bariloche RN, Argentina
[b] Institute of Condensed Matter Physics, Ecole Polytechnique Fédérale de Lausanne, CH 1015 Lausanne, Switzerland

ARTICLE INFO

ABSTRACT

In this work, mechanical spectroscopy (internal friction) is used for a qualitative evaluation of interstitial carbon content in martensite and of the dislocation density in bulk samples of quenched and tempered steel. On one hand, the decrease of the amplitude of a local maximum at 380 K is correlated with the reduction of interstitial carbon content in the martensite matrix, which is due to the carbide precipitation during the first stage of tempering. On the other hand, the amplitude change of an internal friction peak that appears at 500 K is correlated to the variation of the dislocation density. Both amplitude variations follow a similar trend during quenching and tempering, indicating a correlation between the interstitial carbon content and dislocation density in martensite. This correlation is in agreement with the results obtained by X-ray diffraction, thermoelectric power, and hardness. Additionally, it is possible to observe the Snoek effect in samples that contain ferrite and martensite with low tetragonality due to intercritical austenization or tempering.

1. Introduction

In carbon steels, quenching and tempering allow to obtain a wide range of mechanical properties due to the allotropic transformation of iron, and the hardening caused by interstitial carbon [1]. Mechanical spectroscopy can help in improving the microstructural characterization, and understanding of the hardening mechanism of these steels. This technique measures the mechanical loss (internal friction, Q^{-1}) when an oscillating periodic stress is applied to the material, and it is sensitive to microstructural changes such as interstitial atoms diffusion and the dislocation movement [1].

In the last years, some works about internal friction of quenched and tempered steels have been reported for low frequencies (1–3 Hz), correlating several relaxation mechanisms with the microstructure [2–6]. On one hand, a local maximum (a non-thermally activated peak) appears around 380 K in quenched and low temperature tempered steels. This local maximum has been related to the decrease of carbon content in martensitic matrix due to transition carbide precipitation during the first stage of tempering [2–5]. On other hand, a thermally activated peak appears around 480 K in quenched and tempered steels. The peak amplitude is proportional to the density of dislocation segments, Λ, and the active dislocation segment length, L, according to ΛL^2 [5,6].

In this work, the use of mechanical spectroscopy (internal friction) as a technique for microstructural characterization is promoted. Using several austenitizing conditions, qualitative correlations are established between the amplitudes of internal friction peaks occurring at about 380 K and 500 K, and the interstitial carbon content in martensite, or dislocation density in bulk samples, respectively. From this, a correlation between the interstitial carbon content and dislocation density in martensite is established, which is in agreement with the model of Morito et al. [7]. On the other hand, the Snoek peak appears in samples with lower martensite tetragonality, which could be associated to ductile deformation.

2. Experimental

The chemical composition was measured in an optical emission spectrometer, with an error of 1×10^{-2} wt% (Table 1). The samples of 40 mm × 4 mm × 1 mm were encapsulated under argon to avoid decarburization during quenching. In both, quenching and tempering, the cooling was made in water. The Table 2 shows the heat treatments that were carried out.

The microstructure was observed by Scanning Electronic

---

* Corresponding author.

E-mail addresses: quinteros@cab.cnea.gov.ar (J.J. Hoyos),
friccion@cab.cnea.gov.ar (A.A. Ghilarducci), daniele.mari@epfl.ch (D. Mari).

Table 1
Chemical composition of carbon steel.

Element	C	Si	Mn	S	P	Cr	Ni	Mo	Cu	Sn	Ti	Sb	Fe
(wt%, 10^{-2})	70.8	30.2	76.2	1.8	1.6	4.9	2.9	1.4	3.2	0.4	0.2	0.9	–

Table 2
Description of heat treatments.

Sample	Austenitizing temperature (K)	Austenitizing time (min)	Tempering temperature (K)	Tempering time (min)
A1093K5	1093	5	–	–
A1093K10	1093	10	–	–
A1173K5	1173	5	–	–
A1173K10	1173	10	–	–
A1173K5R380K	1173	5	380	10
A1173K5R480K	1173	5	480	10

Microscopy (SEM), etching the polished surfaces with 2% nital solution (2 ml $HNO_3 + 98$ ml of ethanol). Hardness was measured in a Vickers HV Mitutoyo durometer, with a charge of 1 kg, and five tests on each sample, reporting the mean value. X-Ray Diffraction (XRD) measurements were performed at room temperature using CuKα radiation ($\lambda = 1.542*10^{-10}$ m), and scanning from 20° to 99°, with a step of 0.01°. The thermoelectric power (TEP) was measured at a temperature of 20 °C ± 10 °C, with a gradient (ΔT) of 20 °C between the extremes of the sample, which is held by copper blocks. This gradient induces a voltage difference ΔV due to Seebeck effect. The value obtained, ΔS, is the difference between the absolute value of the sample S^* and the relative value to the TEP of copper blocks, S_o (Eq. (1)) [8].

$$\Delta S = S^* - S_o = \frac{\Delta V}{\Delta T} \tag{1}$$

Internal friction was measured from the free decay of the oscillations, with resonance frequency of 2 Hz, amplitude of deformation of 2×10^{-4} and heating rate 0.8 K/min, in a temperature range from 100 K to 800 K. The deconvolution of peaks is made with a background exponential, and Debye functions (Eq. (2)), φ, considering a peak broadening factor, α, where Δ is the relaxation amplitude, ω is the angular frequency, and τ is the relaxation time. The broadening factor is equal to 1 when the peak is symmetrical [5].

$$\varphi = \Delta \frac{(\omega\tau)^\alpha}{1 + (\omega^2\tau^2)^\alpha}, \ 0 < \alpha \le 1 \tag{2}$$

3. Results and discussion

Fig. 1 shows the microstructure of the sample A1173K5, a martensitic matrix (α'), with regions of ferrite (α) and undissolved carbides (c). The same phases are present in all quenched samples. The ferrite appears due to incomplete austenization. Thus, the size and the amount of ferrite areas decrease when the austenitizing temperature is higher. On the other hand, in the samples austenitized at 1173 K, the tempering at 380 K and 480 K does not generate significant changes in the microstructure [1].

Fig. 2 shows the XRD diffractograms of all samples. In quenched samples, the austenite peaks are observed only when the austenitizing temperature is 1173 K, but with a low intensity. The ferrite and martensite peaks appear in all samples, overlapping. Therefore, it is not possible distinguish between the peaks of these phases. However, the overlapped peaks are broader when the
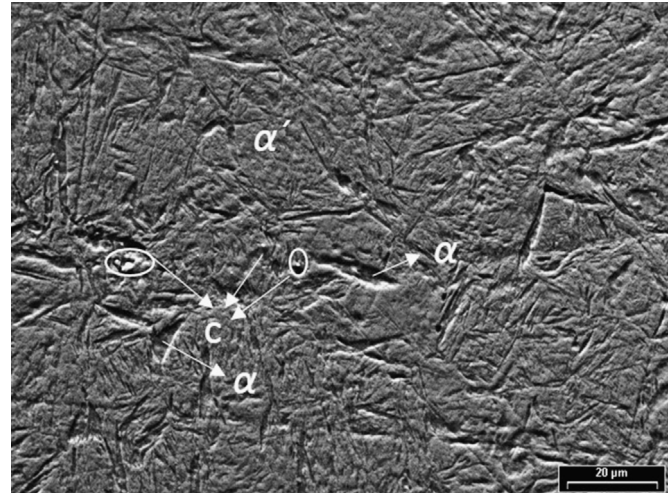

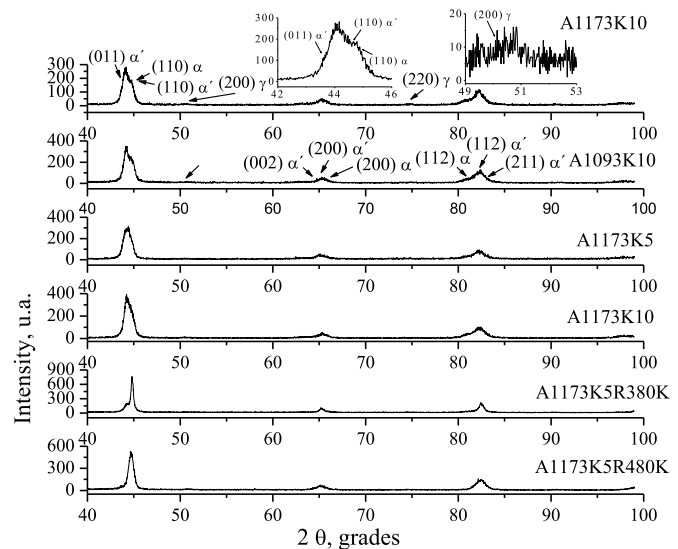**Fig. 1.** Microstructure of the sample A1173K5.

Fig. 2. X-ray diffractograms of quenched and tempered samples.

temperature and time of austenitizing are higher due to the increase of the interstitial carbon content in martensite (martensite tetragonality), which produces a higher separation between the diffraction lines of both phases. During tempering of samples austenitized at 1173 K for 5 min, the width of overlapped peaks decrease in comparison with quenched sample. This is attributed to the decrease of interstitial carbon content of the martensite during tempering, and therefore of martensite tetragonality.

The variation of the interstitial carbon content in martensite is also evaluated by TEP (Fig. 3). This technique shows that interstitial carbon content increases when the temperature and time of austenitizing get larger. Tempering clearly produces a major drop in interstitial carbon content as shown by the increase of TEP. It is known, that TEP at room temperature is inversely proportional to the carbon concentration in ferrite and martensite [5]. The contributions due to other defects such as dislocations and carbides