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Anelastic Effects on Martensitic Carbon Steels During First Stage of Tempering

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Abstract. The evolution of martensitic carbon steel during low temperature tempering was studied using internal friction. The steel containing 0.71wt.% carbon was heated at 1093K for 5min and then rapidly cooled into water (quenched), and tempered for 10 min at 340, 380, 420 and 460K. Additionally, other samples were tempered at 380K for 1 and 20 hours. Internal friction was measured by using a forced vibration pendulum, in a temperature range from 300 to 600K, with deformation amplitude 3 x 10^{-6} and temperature rate of 0.8 K/min. The internal friction spectrum is decomposed into three Debye peaks: P1 at 380K, P2 at 420K and P3 at 480K, for 3 Hz. P1 is attributed to the epsilon carbide precipitation. P2 and P3 are associated to the dislocation relaxation process. P2 appears when dislocations are pinning with epsilon carbide and P3 appears when dislocations are pinning with cementite carbide.

Introduction

The static and dynamic interactions of dislocations with carbon atoms, transition carbides and evolving dislocation substructure have a high influence of strengthening mechanism of martensite microstructure. In first stage of tempering, these interactions as a function of temperature and time are not fully characterizated and it is required more research to provide understanding of martensite deformation, mechanical properties and to determinate the condition of tempering for a given application.

In previous works [1, 2], correlations between the internal friction and the microstructure of two steels containing 0.626 and 0.71wt.% carbon are proposed (A and B steels, respectively). These steels were quenched into water and tempered for 10 and 60 min at 423, 573 and 723K.

In both steels, the microstructure produced by the quenched was composed of a large amount of lath martensite, and small quantities of plate martensite and retained austenite. The martensite tetragonality was reduced with the increase of the tempering temperature by the precipitation of carbides from the segregated carbon and carbon in solid solution. The increase of the tempering time allowed the additional carbon segregation from the interstitial sites towards the dislocations.

In those works [1, 2], internal friction spectrum was decomposed into six peaks and thermal background. P1 at 215K, M1 at 225K, P2 at 235K, P3 at 260K, P4 at 380K and P5 at 428K, for 3Hz. P1was attributed to the interactions between edge dislocations and carbon atoms. P2 was related to the unpinning process of dislocations from carbides. P3 was related to the formation of kink pairs in the edge dislocations. P4 was associated to carbide precipitation.

P5 appears only in the sample tempered at 723K for 60 minutes of steel B, and there were not satisfactory explanation for the mechanism relaxation. M1 appears in tempered samples for 60 minutes and it is much broader than a single relaxation peak. This peak was considered as a superposition of P1 and P2. Simultaneous presence of P1 and P2 indicated that the pinning of the dislocations was produced by both segregated carbon and precipitates.

This paper presents the effects of the temperature and time tempering obtained during first stage of tempering on internal friction of a martensitic carbon steel. The steel was quenched into water and tempered at 340, 380, 420 and 460K for 10 min, and tempered at 380K for 1 and 20 hours. The internal friction was measured as a function of temperature from 300 to 600K.

Experimental

2.5mm×0.5mm×15mm sheets made of plain carbon steel containing 0.71wt.% carbon were used as test specimens. The chemical composition for steel is listed in Table 1. The specimens were heated at 1093K for 5min in a protected atmosphere for homogenization and then rapidly cooled into water (quenched), and tempered for 10 min at 340, 380, 420 and 460K. Additionally, samples were tempered at 380K for 1 and 20 hours.

Table 1. Chemical composition of steel.

Element	C	Si	Mn	Cr	Ni	S	P	Mo	Cu	Fe
Weigth %	0,709	0,209	0,685	0,294	0,037	0,005	0,019	0.016	0,009	-------

Conventional micro-Vickers hardness test as a macroscopic strength evaluation were performed with a load of 1kg. The samples were observed under scanning electron microscope (SEM). The evolution of martensite lattice towards body centered cubic during tempering was studied using X ray diffraction (XRD). The identification of phases was realized by scanning in the region 2θ = 20–100º. These measurements were performed using CuKα radiation ($\lambda = 1.542\times10^{-10}$ m).

The dilatometry measurements were performed using heating rates of 0.8 and 2 K/min in a temperature range from 300 to 700K. The equation 1 was used for the evaluation of activation energy. Where Q is the activation energy, T_i is the temperature (K) corresponding to the inflection point of the sample length vs. temperature curve, k_o is a constant, R is the gas constant and Φ is heating rate (K/min). For non isothermal heating experiments, the activation energy can be derived from the slop of the straight line obtained by plotting ln (T_i^2/Φ) vs. $1/T_i$ for different values of Φ [3].

$$\ln\left(\frac{T_i^2}{\Phi}\right) \approx \frac{Q}{RT_i} + \ln\left(\frac{Q}{Rk_o}\right) \qquad (1)$$

Internal friction measurements were performed at 3Hz with high vacuum, in a torsion pendulum by forced vibrations method, in a temperature range from 300 to 600K. Amplitude of deformation 3×10^{-6} and heating rate 0.8K/min were used. The internal friction spectrum has been deconvoluted into Debye peaks and a thermal background.

Results and Discussion

Fig. 1 shows hardness measurements. In samples tempered for 10 minutes, the hardness decreases when the tempering temperature is increased above 380K. The hardness was not affect significantly with the increases of time tempering from 10 to 60 minutes. However, the samples tempered for 20 hours have the lowest hardness. At low temperatures, the hardness decreases more significantly with the tempering time than the tempering temperature.

Fig. 2 shows the XRD results of the quenched sample. Only peaks corresponding to the martensitic (α′) and austenite (γ) are visible. The same reflections were present in all tempered samples. This is agreed with the beginning of austenite transformation at temperatures above 460K. The evolution of martensite is analyzed by the evolution of (200) peak. The method used is the measurement of martensitic doublet (200) and (002) [4]. The amounts of interstitial carbon of samples quenched and tempered at 340K were estimated to be 0.34wt% by XRD. This suggests than the carbon atoms are segregated to dislocations during quenching and there is not additional carbon segregation during tempering at 340K. The carbon content diminishes when the tempering temperature is increased from 340 to 380K. The rest of samples have the same value (0.05wt%). The carbon content evaluation by XRD is not accurate to more than 0.05wt.% carbon due to the uncertainty involved in locating the peak position of a broad peak [4].

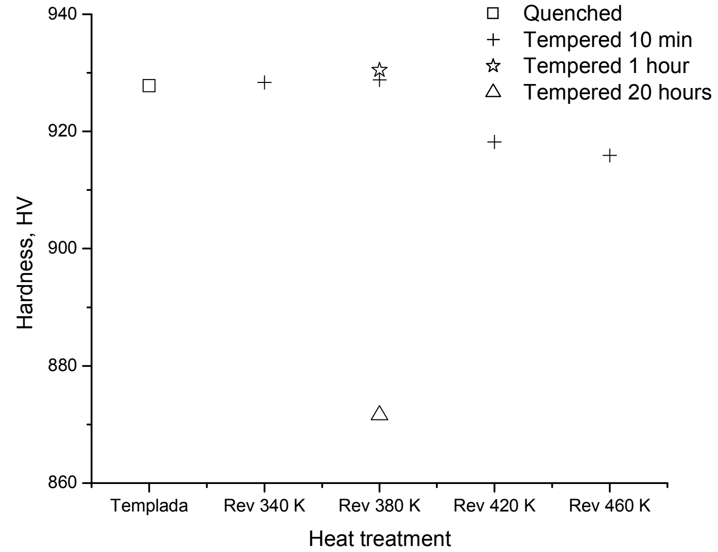

Fig. 1. Tempering intensity dependence of the micro-Vickers hardness.

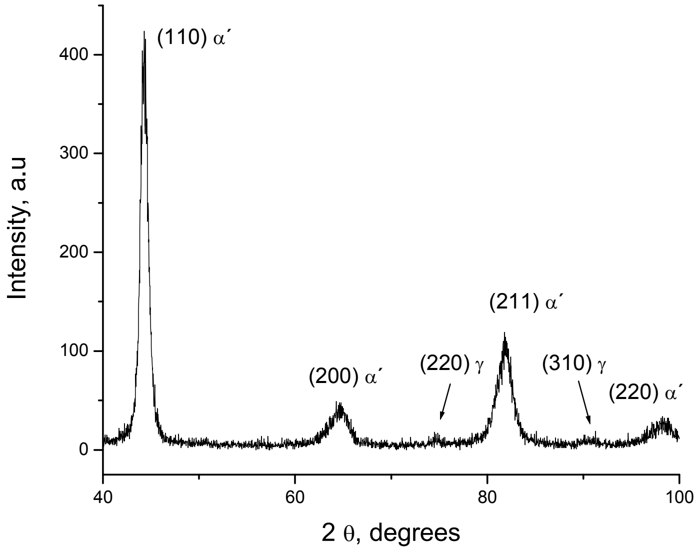

Fig. 2. Diffractogram of the quenched samples.

Fig. 3 shows the dilatometry curves for the quenched sample and samples tempered for 10 minutes. Three stages could be distinguished during the measurements. First stage is characterized by the precipitation of epsilon carbide. This tempering phenomenon occurs between 350 and 420K and results in a reduction of the tetragonality of the martensite lattice. Second stage is characterized by transformation of retained austenite between 480 and 510K. The point of inflection at about 480K indicates the beginning of this stage. In third stage, cementite precipitation occurs between 510 and 600K. Stages II and III are similar in all samples. However, when the tempering temperature is increased the stage I is disappeared.

Fig. 4 shows the dilatometry curves for the samples tempered at 380K. In these samples, the same three stages of tempering are observed: precipitation of epsilon carbide, transformation of retained austenite and cementite precipitation. When the tempering time is increased the stage I is disappeared. The activation energy of the different process was determinate from equation 1. The activation energies from stages II and III are similar for all samples. The activation energy from stage I increases with increasing of the temperature tempering, from 73KJ/mol for quenched sample to 82KJ/mol for sample tempered at 460K. In samples tempered at 380K, the activation energy was not affect significatively with the increase of time tempering from 10 and 60 minutes. However, the samples tempered for 20 hours have the highest activity energy (102KJ/mol). This is agreed with the lowest carbon content in solid solution when the intensity tempering is increased.

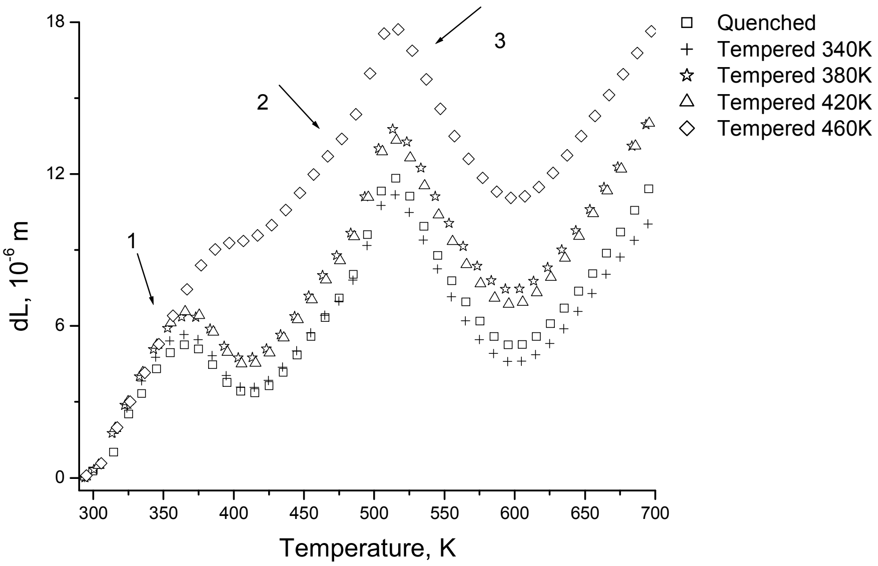

Fig. 3. Dilatometer curve for samples quenched and tempered for 10 minutes.

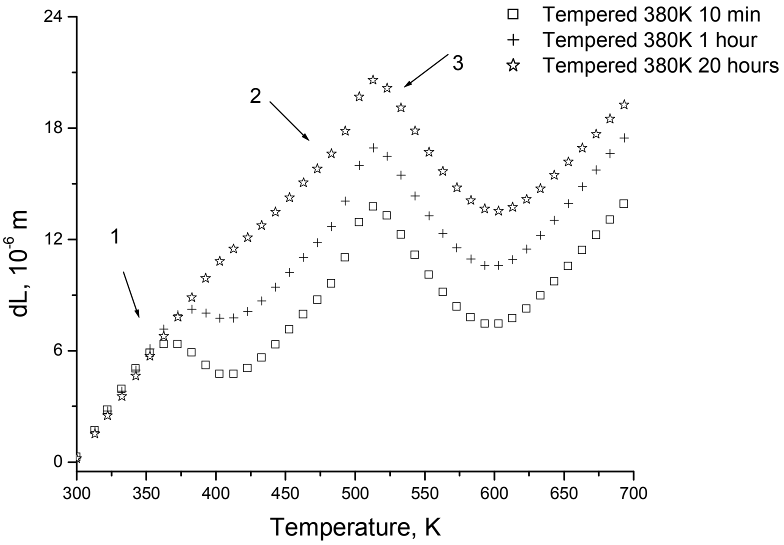

Fig. 4 Dilatometer curves for tempered samples at 380K.

Fig. 5 presents the spectrum of internal friction and elastic modulus of the quenched sample. High temperature background of internal friction is considered as increasing exponentially with the temperature. The spectrum has two peaks P1 and M1. P1 appears about at 380K and it is associated to the precipitation of epsilon carbide. This peak was considerate as a Debye peak with the temperature and activation energy from dilatometry.

M1 is asymmetrical and much broader than a Debye peak. The elastic modulus has a step about 380K, which is associated to the peak P1. At temperatures around M1 multiples steps appear which suggest the superposition of various relaxation peaks. In the temperature zone of M1, the tempering proceeds by a sequence of structural changes, including recovery of the dislocation structure, dissolution of epsilon carbide, precipitation of cementite and decomposition of retained austenite. Tkalcec and et al [5] reported a broad and asymmetrical internal friction peak at 540K in quenched samples of 1.26 wt.% carbon, which is deconvoluted into two Debye peaks at 420K and 540K, and it is attributed to the Snoek-Köster relaxation. Martin and et al [6] confirms than this peak is a thermally activated relaxation.

We considered than M1 can be attributed principally to the dislocation relaxation process, and this one is considered as a superposition of two Debye relaxation peaks P2 at 420K and P3 at 480K. P2 and P3 can be associated to the interaction of the thermally activated dislocations with carbide epsilon and cementite, respectively. With the increasing temperature, the dislocations are mobile and become easier to move under applied stresses. Initially, epsilon carbide starts to migrate following the moving dislocations. When the temperature is higher than about 420K, two different processes begins, by one hand the rearrangement of epsilon carbide exert a dragging force against the movement of dislocations and reduces the internal friction, which results in the formation of P2. But by other hand dissolution of epsilon carbide stars and the mobility of dislocations is increased, which contributes to the formation of P3. As it is know, the transition of epsilon carbide to cementite is preceded by the dissolution of epsilon carbide. The decreases of internal friction at temperatures higher than about 480K can be attributed to the dragging effect of cementite. Thus, the right part of a relaxation peak P3 is produced by the pinning of the dislocations by cementite.

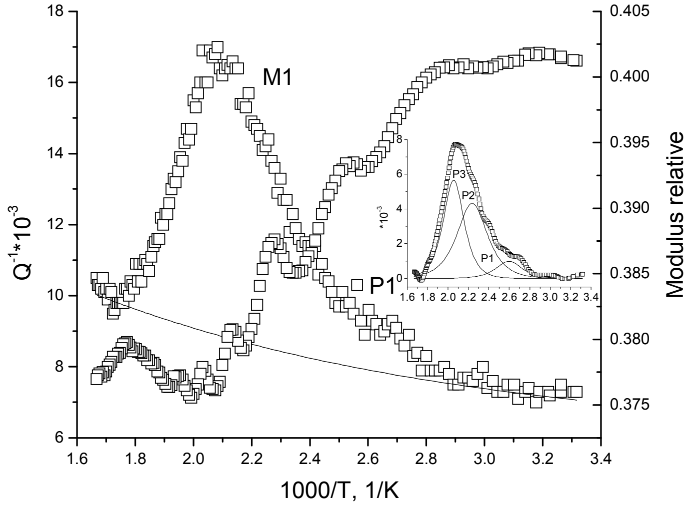

Fig. 5 Temperature dependent internal friction curves of quenched sample.

Figure 6 shows the spectrum of internal friction of the samples tempered for 10 minutes and elastic modulus of sample tempered at 460K. In all samples P1 and M1 appear, and the spectrum is decomposed into three peaks: P1, P2 and P3. The height of P1 diminishes when the temperature tempering is increased, which is associated to the lowest carbide precipitation.

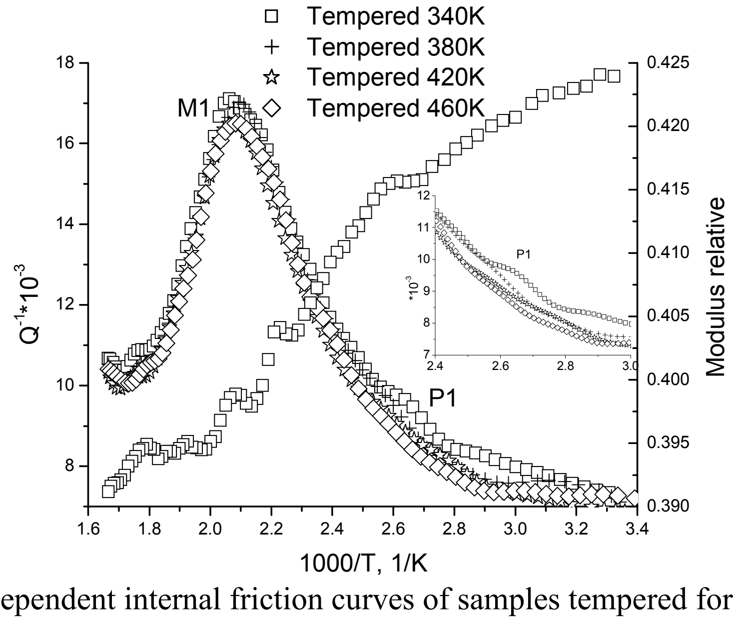

Fig. 6 Temperature dependent internal friction curves of samples tempered for 10 minutes.

The step of elastic modulus about 380K is lowest when the temperature tempering is increased. The steps at temperatures around M1 suggest the superposition of various relaxation peaks.
Figure 7 shows the spectrum of internal friction of the samples tempered at 380K for 1 and 20 hours, and elastic modulus of sample tempered at 380K. The spectrum is decomposed into three Debye peaks: P1, P2 and P3. P1 is lowest when the time tempering is increased. The step of elastic modulus about 380K is lowest when the time tempering is increased. The difference of temperature between peaks P2 and P3 is increased when the temperature and time tempering is highest. This can be associated to the transition from epsilon carbide to cementite. The transition can be more slowly when the intensity of tempering is increased.

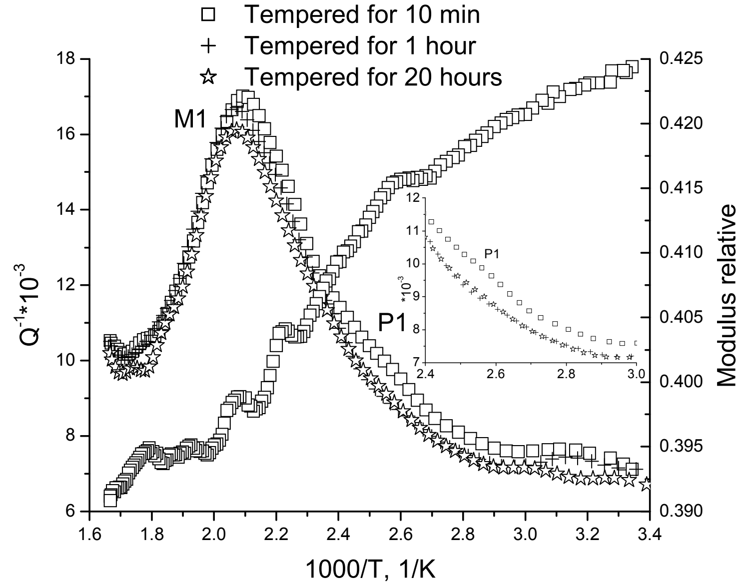

Fig. 7 Temperature dependent internal friction curves of samples tempered at 380K.

Conclusions

The increasing of the time and temperature tempering of martensitic carbon steel increases the proportion of epsilon carbide, and thus the carbon content in solution is reduced. An internal friction peak P1 appears at 380K, and its height is lowest when the intensity of tempering is increased. Thus, this peak can be used for the evaluation of the precipitation of epsilon carbide. A broad peak M1 appears at 480K. This peak is decomposed into two Debye peaks: P2 at 420K and P3 at 480K. P2 can be associated to the interaction between dislocations and epsilon carbide and P3 can be associated to the interaction between dislocations and cementite.

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