Materials Physics and Mechanics 42 (2019) 183-190 Received: October 23, 2018

ARTIFACTS OF STRESS RELAXATION TECHNIQUE TO FIT RECOVERY ACTIVATION PARAMETERS FOR LOW CARBON STEELS

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Abstract. The paper compares model predictions of recovery kinetics of low carbon steels with activation parameters determined by various experimental techniques. It is concluded that the stress relaxation method overestimates the recovery rate because the creep of stressed specimens is ignored. To avoid such errors, it is recommended to use an alternative method of double loading. The more realistic (lesser) rate of recovery with the related activation parameters complies with independent data of mechanical tests.

Keywords: steels, recovery, modeling, stress relaxation

1. Introduction

Recrystallization of cold rolled automotive steels in annealing has a significant effect on their final structure and mechanical properties. This stimulates experimental studies of this phenomenon and evokes attempts to model it [1-3]. However, its numerical modeling is seriously complicated due to the concurrent recovery process that consumes the same thermodynamic driving force i.e. specific energy of lattice dislocations accumulated in the previous deformation. Moreover, this is the case both before and during recrystallization since the latter propagates gradually while admitting the recovery to go on within the remaining material part [4,5]. Therefore, to predict the recrystallization effects, accurate allowance for the recovery is necessary; in particular, related activation parameters (energy Q_a and volume V_a) should be determined with appropriate experimental techniques. The present paper reconsiders them in order to exclude artifacts in determination of these parameters and, hence, to improve the modeling of recovery kinetics.

The pioneering study of recovery in α−iron of high purity at temperatures 300 to 500°C after a low strain of 5% undertaken by Michalak [6] revealed a notable growth of the recovery degree in terms of flow stress reduction when increasing the annealing temperature; in particular, at 500°C this degree approaches about 80%. An IF−steel (0.002%C, 0.1%Mn, 0.023%Ti, 0.007%Nb (wt.%)) cold rolled to the thickness reduction of 80% and then treated at temperatures 500 to 625°C has been investigated by means of XRD technique [7]. According to the obtained results, the recovery of this material at 500°C develops much slower than in pure α−iron and does not exceed 40%.

Experimental study and numerical modeling of the kinetics of recovery in low carbon steels were performed in [8-11]. The stress relaxation technique, implemented on Gleeble thermo-mechanical simulators, has been employed in [8,11] in order to register the recovery

http://dx.doi.org/10.18720/MPM.4222019_4

kinetics. To this end, after the given compression deformation at some selected temperature, the specimen length is fixed whereas the gradually diminishing external stress displays the apparent softening presumably related to relaxation phenomena in the material. According to [8] the recovery degree of investigated steel (0.19%C, 0.445%Si, 1.46%Mn, 0.03%Al) after the strain of 15% exceeds 70% at 500°C and reaches about 90% at 550 to 600°C. However, the so strong softening of steel, different from pure iron, does not seem realistic and necessities consideration of possible experimental artifacts. In particular, the permanently applied compressive stress could result in some non-stationary creep so that an apparent softening is partly due to the weakening of specimen constraint rather than an internal recovery process. The same remark is also relevant to findings of work [11] where the stress relaxation in steel (0.05%C, 1.5%Al, 1.0%Mn, 0.4%Si) at 700 and 750°C approached 80% regardless of previous true strains (0.12 and 0.60).

To simulate quantitatively the recovery kinetics in annealing of the considered steels, the authors of [8,10,11] made use of the model [12] by Verdier, Brechet and Guyot (VBG), which employs two experimentally fitted parameters related to the underlying rearrangement of dislocations that results in reduction of their density. One of them is the recovery activation energy Q_a and another is the activation volume V_a suggested to be proportional to the average length of movable dislocation segments and the cross section area of dislocation core [5,13]. It is worth noting that the VBG model with Q_a and V_a fitted to different experimental data [8,10,11] predicts rather various recovery kinetics hardly consistent with each other and, hence, complicates selection of the most adequate and practicable model. In other words, the employed experimental methods do not ensure appropriately accurate determination of Q_a and V_a.

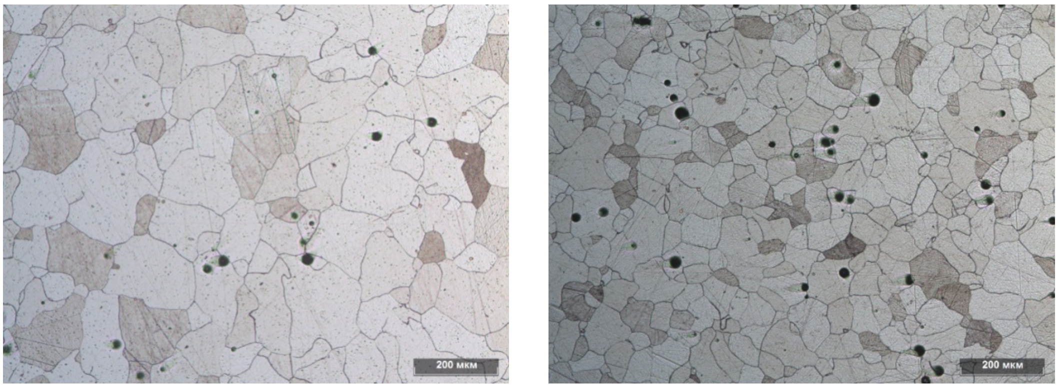
The present paper is aimed to determine the recovery activation parameters in a way free of experimental artifacts in order to provide reliable numerical modeling of the recovery in annealed automotive steels.

2. Investigated steels

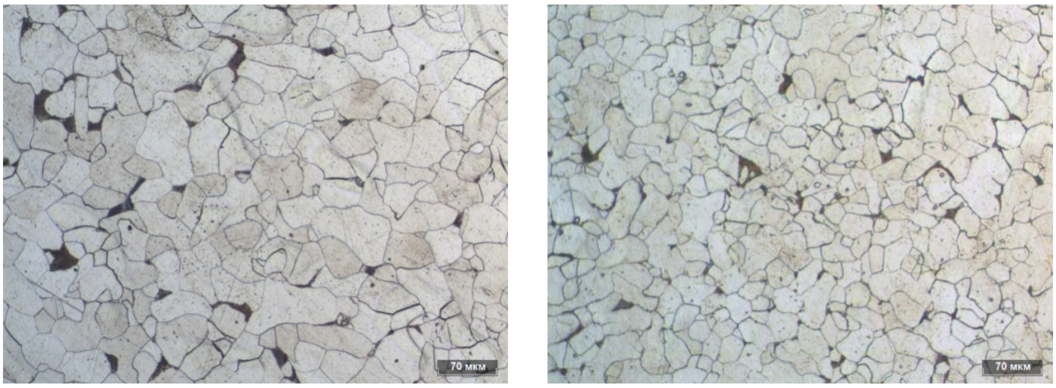
In this work the recovery kinetics is investigated with the stress relaxation technique and with the so called double loading technique. In the latter, after the first deformation the specimen is unloaded, undergoes recovery for a stated time lapse and then is deformed again so that comparison of the two loading diagrams enables evaluation of softening degree. Experiments by both methods are implemented on Ø10×15 mm specimens subject to the compressive stress in the unit HydraWedge of thermo-mechanical simulator Gleeble 3800. Under consideration are IF−steel (0.004%C, 0.14%Mn, 0.06%Ti) and 08ps steel (0.06%C, 0.17%Mn). Pieces of these steels taken at an intermediate stage of rolling in order to fabricate specimens for experiments on Gleeble 3800 had rather coarse grained ferrite structures different from those of final products. To improve this, 30 mm thickness of initial plates has been reduced to about 12 mm by hot rolling on a laboratory mill; then the plates have been cooled in air. Resulting average sizes of ferrite grains in IF and 08ps steels are 70 and 30 μm, respectively (Fig. 1).

3. Experimental procedures and results

Before the stress relaxation during an isothermal treatment of specimens with a fixed total deformation, they been heated with the rate 10°C/s to the stated temperature (250 to 600°C) and then compressed with the rate of 1 s^{-1} to the true strain of about 0.6. The obtained results (Fig. 2) evidence that in both steels stress relaxation degree increases with the annealing temperature from 20 to 40% (250°C) to about 80% (600°C). The so high values are close to those determined by the same method in [8,11]. In order to verify whether or not the employed technique provides a proper evaluation of the material recovery, an alternative "direct" method is then applied to the same steels in the cold rolled state.



IF−steel



08ps

Fig. 1. Microstructures of the investigated steels after industrial rough rolling (left) and after additional hot rolling in laboratory conditions (right)

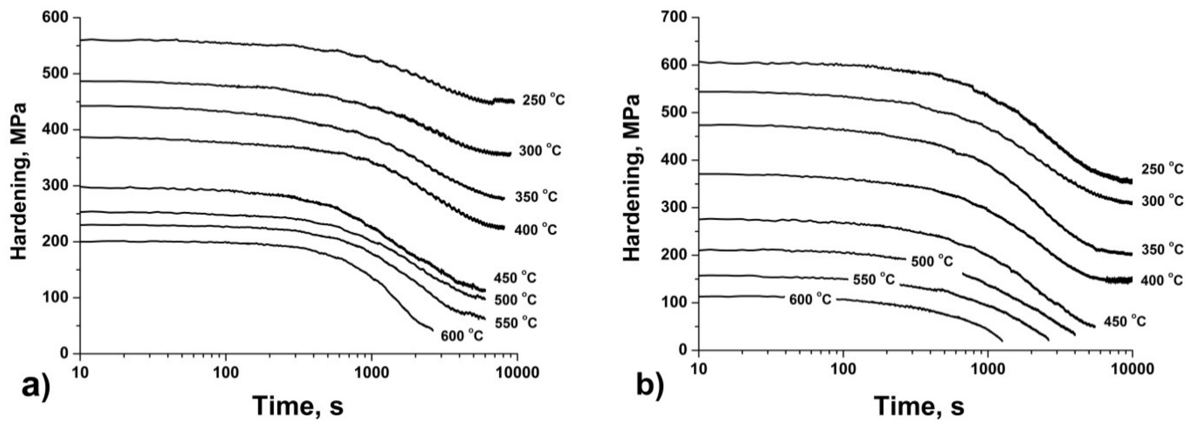


Fig. 2. Diagrams of stress relaxation of IF (a) and 08ps (b) steels at various temperatures

Standard tensile specimens for mechanical testing at room temperature have been cut from steels IF and 08ps cold rolled to the thickness reduction of 57 and 58%, respectively. Corresponding increments of yield stress for these steels relative to their previous hot rolled states are 330 and 465 MPa. These reference values can be used to normalize absolute magnitudes of softening in annealing at various temperatures in the range 300 to 550°C for six hours and at 550°C for five hours. The annealing treatment has been made in the Nabertherm furnace, and tensile test machine Zwick/Roell Z100 has been used to determine mechanical properties of both the cold rolled and annealed steels. Respective values of relative softening are represented in Fig. 3.

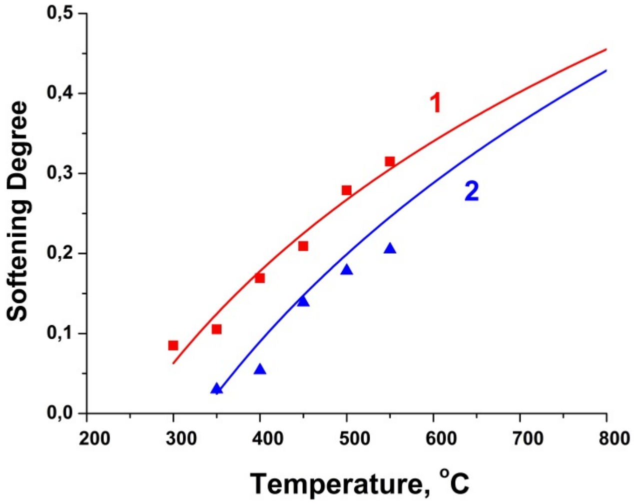


Fig. 3. Maximum softening degrees depending on the annealing temperature for cold rolled IF (1) and 08ps (2) steels

Note that the applied annealing times notably exceed durations of stress relaxation in Fig. 2 and, accordingly, are sufficient to complete this phenomenon. The plots of Fig. 3 indicate that the softening degree at the maximum temperature 550°C approach only about 30 and 20% for IF and 08ps steels, respectively. Even when roughly extrapolating the plots to 800°C, these degrees will not exceed 50%. In all, comparison of the considered results to the data of Fig. 2 indicates that the material softening degree evaluated by the stress relaxation method [8,11] is always overestimated. As previously mentioned, this can be due to the specimen creep developing under compressive loads in parallel with the softening as such.

In order to avoid artifacts of the stress relaxation approach to the recovery evaluation while making use of the convenient Gleeble 3800 machine, an alternative double loading method has been applied to the same materials as follows. Specimens are heated with the rate of 10°C/s to the temperature of deformation (400 to 550°C for IF−steel, 400 and 500°C for 8ps steel) compressed with the rate of 1 s^{-1} to the true strain of 0.6, unloaded and kept at the same temperature for a stated time interval and then compressed again with the same rate to the true strain of 0.2. To analyze absolute softening due to the recovery for time *t* between two deformations, the current hardening magnitude relative to the first yield stress $\sigma_{0.2}^{(1)}$ is calculated as:

$$\Delta\sigma(t) = \sigma_{0.2}^{(2)}(t) - \sigma_{0.2}^{(1)}. \tag{1}$$

This is plotted in Fig. 4 so that $\Delta\sigma(0) = \sigma_{\max} - \sigma_{0.2}^{(1)}$, where $\sigma_{\max}$ is the flow stress at the end of the first deformation; $\sigma_{0.2}^{(1)}$ and $\sigma_{\max}$ have been averaged over all tests. According to these data, the maximum relative softening $\Delta\sigma(t)/\Delta\sigma(0)$ at 500°C remains within 15 and 20% for IF and 8ps steels, respectively, that is significantly lesser than previous estimates with the stress relaxation technique.

It is worth noting in Fig. 4 that the softening of IF−steel is monotonous and mostly expires for 1000 to 2000 s, whereas a stage of softening for about 2000 s in steel 08ps is followed by some hardening during about 10^4 s; then this material slowly softens again. This behavior of steel 08ps is due to dissolution of cementite situated in perlite colonies and between ferrite grains that is quite possible at temperatures above 400°C. Specifically, free carbon atoms form dislocation atmospheres responsible for the considered hardening, and the following softening is due to the eventual diffusion rearrangement of such atmospheres. Formation of the latter in the annealed steel has been confirmed by presence of yield drops on diagrams of its loading at room temperature.

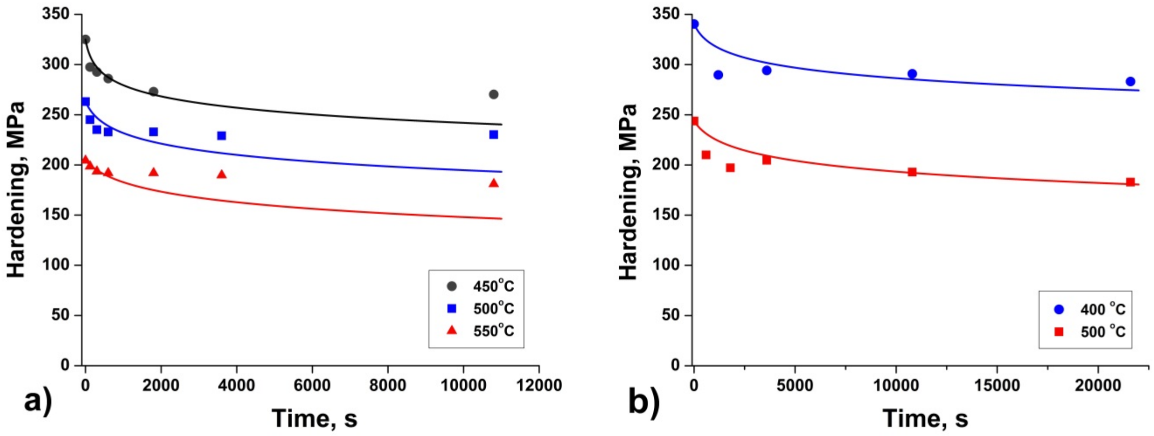


Fig. 4. Reduction of hardening relative to the reference (hot rolled) state when annealing IF (a) and 08ps (b) steels at various temperatures. Continuous dependences are provided by VBG model fitted to respective sets of experimental points

Let us compare in more detail the data on recovery of investigated steels at 500°C (Fig. 4). In steel 08ps, the initial stage of quick recovery is longer and the related recovery degree is higher. Besides, unlike IF−steel where the softening is practically completed at the stress of about 230 MPa, steel 08ps approaches the lower stress of 175 MPa. It is worth noting that such a difference is the case despite the pinning of dislocations by carbon segregations in 08ps. The observed stagnation of recovery in IF−steel at notably higher stresses evidence for presence of rather strong barriers impeding dislocation rearrangements. Presumably, the effect is due to Ti−C complexes (steel contain 0.06% of Ti) which result in high tetragonal distortions of crystal lattice interacting with both the edge and screw dislocations. Unlike the atmosphere pinning that weakens in annealing because of carbon redistribution, the considered effect is insensitive to an isothermal treatment insofar as the considered complexes are thermodynamically stable. Thus, the chemical composition can notably affect the kinetics of steel recovery.

3. Modeling results

In order to evaluate relevance of our findings to the recovery phenomenon, it is expedient to reconsider them in terms of the popular VBG model [12]. The latter employs the following expression for the rate of softening:

$$\frac{d\Delta\sigma}{dt} = -\frac{64\Delta\sigma^2\nu_D}{9M^3\alpha^2E(T)}\exp\left(-\frac{Q_a}{RT}\right)\sinh\left(\frac{V_a\Delta\sigma}{kT}\right), \tag{2}$$

including Debye's frequency ν_D (~$2*10^{12}$ s^{-1}), energy Q_a and volume V_a of recovery activation, Taylor’s factor M suggested to be 2.7 for ferrite, dimensionless $\alpha \approx 0.33$, absolute gas constant R, Boltzmann's constant k, absolute temperature T, and the Young modulus E. Temperature dependence of the latter:

$$E(T) = 2.11\times10^{11}\left[1-\frac{T-300}{1989}\right](\text{MPa}), \quad (3)$$

is compiled from [14]. The main parameters Q_a and V_a of Eq. (2) should be fitted to appropriate experimental data. However their simultaneous fitting, particularly to limited data of real accuracy, often leads to a great variation of related modeling results. To avoid this issue we fix Q_a by taking for it the activation energy E_{SD} of self-diffusion in α–iron. According to [15] this energy is equal to:

$$E_{SD}(T) = 236.5 + \Delta E_{SD}(T)\,(\text{kJ/mol}), \quad (4)$$

where $\Delta E_{SD}(T)$ is the temperature dependent contribution of magnetic effects. Experimental data on this contribution, as shown in Fig. 5, can be accurately approximated by Boltzmann's function: $\Delta E_{SD}(T) = a + b\big(1+\exp\big((T/T_C - c)/d\big)\big)$, where parameters fitted to experimental data [15] are equal to: a = - 11.895 kJ/mol, b = 65.984 kJ/mol, c = 0.934 and d = 0.128, respectively; T_C is Curie's temperature.

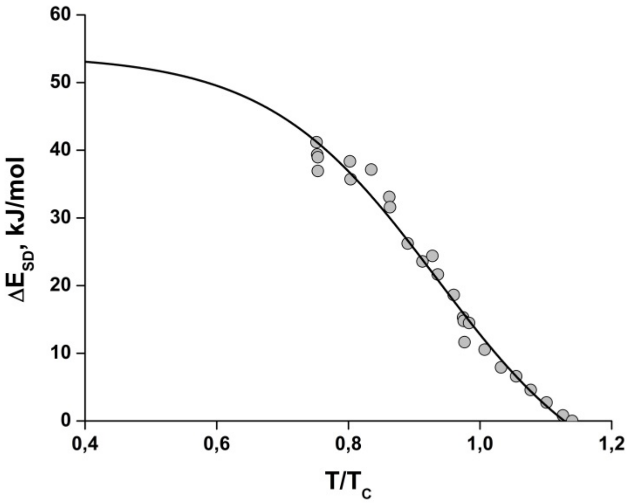


Fig. 5. Dependence of the magnitude contribution of $\Delta E_{SD}(T)$ on ratio T/T_C (T_C is Curie's temperature). The presented experimental data [15] are approximated using Boltzmann's function

Thus V_a remains the only parameter of Eq. (2) to be fitted to experimental data represented in Fig. 4. We have implemented the fitting to the total data of both steels thus neglecting the rather weak effect of their difference in chemical composition. The resulting value of V_a is $5.23*10^{-28}$ m^3, i.e. 31.7b^3 where b is the Burgers vector magnitude; this result satisfactorily complies with [5,10,12].

The time dependences of softening, determined when substituting the above-considered parameters in Eq. (2) and shown in Fig. 4 by continuous curves, prove to be satisfactorily close to experimental points. The average relative error of the modeling does not exceed 4% for both steels, whereas the most error at the longest annealing time of IF−steel reaches about 20%. Note that the corresponding absolute deviation within 40 MPa is comparable to the experimental stress error of about 15 MPa. Thus, with all simplifications of the employed model kept in mind, even the maximum deviation of calculated stress from its experimental counterpart seems to be acceptable.

Let us compare predictions of softening kinetics at 500°C by VBG model with the above-considered activation parameters and those fitted by other authors to the data of stress relaxation experiments; under consideration are IF−steel (0.004%C, 0.14%Mn, 0.06%Ti) as well as steels (0.19%C, 0.445%Si, 1.46%Mn, 0.03%Al) [8] and (0.05%C, 1.5%Al, 1.0%Mn, 0.4%Si) [11]. Owing to the difference of these materials in chemical composition and the previous strain degree, related softening processes start at different stresses. Therefore, to reveal clearly the effect of activation parameters in application of Eq. (2), it is expedient to employ in modeling a virtually unique stress at t = 0. We take for this the initial stress in our stress relaxation experiment on IF−steel as shown in Fig. 6, where the meaning of vertical axis is similar to that of Fig. 4. This figure, also represents the results of modeling with Q_a and V_a as determined in the present work by the double loading and in [8,11] by the stress relaxation technique. Evidently, in the latter case the softening rate is notably overestimated as compared to those based on the former technique, more relevant as free of such artifacts as creep of stressed specimens.

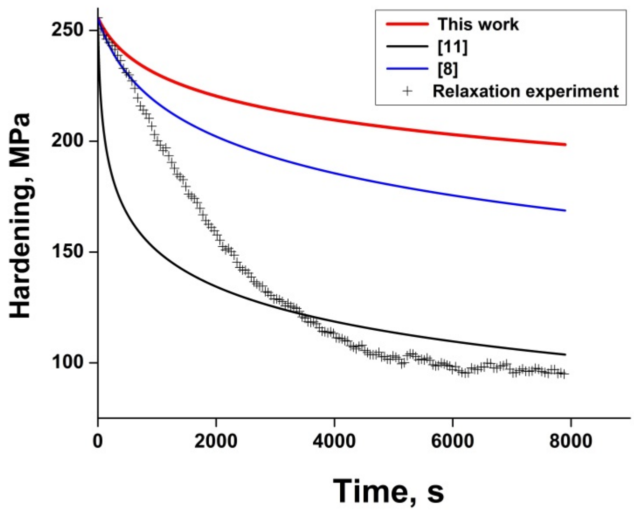


Fig. 6. Results of the modeling of recovery at 500°C with activation parameters determined in this work and in [8] and [11]. For reference, an experimental diagram of stress relaxation of IF−steel at the same temperature is shown

4. Conclusions

To sum up, the obtained results evidence that Q_a and V_a determined in stress relaxation experiments generally lead to excessive rates of recovery in modeling this phenomenon. In order to avoid such errors while making use of the up-to-date Gleeble simulators, it is recommended to apply an alternative double loading method. As confirmed by independent mechanical tests, parameters based on this method provide the more accurate modeling of recovery kinetics. Moreover, this approach saves satisfactory accuracy even if the unique activation energy of self-diffusion is used for Q_a so that only V_a is fitted to experimental data.

Acknowledgments. *This work was supported by grant from Russian Science Foundation (project No. 17-19-01178).*

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