



Full length article

Dynamic strain ageing in iron alloys: The shielding effect of carbon



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ABSTRACT

Dynamic strain ageing in iron due to various interstitial (carbon) and substitutional (nickel, silicon, chromium, aluminum) solute atoms has been studied by *in situ* straining in a transmission electron microscope. The effect of carbon in solid solution is characterized by two different carbon-dislocation interactions in the jerky flow and serrated flow temperature domains, and by a low mobility of screw dislocations controlled by a “high-temperature Peierls mechanism”. Substitutional atoms in solid solution can either move the domain of dynamic strain ageing to higher temperatures or not, depending on their chemical affinity for carbon. The results are interpreted by a shielding effect of mobile carbon atoms, inhibiting the interaction between dislocations and substitutional solute atoms.

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1. Introduction

Due to their interaction with mobile dislocations, solute atoms can improve the strength and the creep resistance of alloys at intermediate and high temperatures. However, the dislocation-solute interaction at intermediate temperatures also results in the dynamic-strain ageing (DSA) effect and associated plastic instabilities (Portevin-Le Châtelier effect) which substantially decreases the ductility. In body-centered-cubic (BCC) iron, the most well-known DSA effect is due to the dynamic interaction between mobile dislocations and solute carbon (or nitrogen) atoms, in the so-called “blue-brittleness” temperature range of 100–300 °C [1–3].

In spite of its technological importance, the exact origin of DSA in ferritic steels remains poorly understood. One can distinguish DSA due to interstitial atoms (carbon, nitrogen) [1–4] and DSA due to substitutional atoms [4–7]. In both cases, three temperature domains can be defined:

- Low temperatures where mobile dislocations interact with fixed solute atoms. In the case of substitutional solutes, this results in the solid-solution strengthening which has been recently investigated by *in-situ* straining experiments in a transmission electron microscope (TEM) [8].

- Intermediate temperatures where solute atoms become sufficiently mobile to move to the nearest dislocations and pin them. This results in the DSA effect characterized by stress instabilities, a high strain-hardening, a low strain-rate sensitivity, and a low ductility. For interstitial solutes, this temperature domain can be divided in two sub-domains, with irregular jerky flow at the lowest temperatures, and more unstable serrated flow above.
- High temperatures where solute atoms are mobile enough to follow gliding dislocations. The yield stress decreases with increasing temperature, the strain hardening decreases and the ductility increases.

The transitions between these three domains vary as a function of applied strain-rate and corresponding dislocation velocity, which allows one to define activation energies related to the kinetics of dislocation-solute interactions.

These activation energies cannot however be easily interpreted. Indeed, both transitions (beginning and end of DSA, i.e. lower and higher temperature limits respectively) should a priori be controlled by the diffusion of the active solutes, i.e. both activation energies should be equal to the solute diffusion energy. In reality, these activation energies are in a ratio of nearly two [1–5], which shows that the diffusion mechanisms at the beginning and end of DSA are different.

In the case of interstitial solutes, the activation energy at the jerky-serrated flow transition is equal to that at the beginning of DSA, and half that at the end of DSA. These different activation energies have been partly explained for interstitial carbon in iron

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[9]. Indeed, the activation energy at the beginning of DSA is close to that of carbon diffusion [1–3], which can be justified by the low density of interstitial carbon that must travel over rather long distances in order to pin dislocations. Under such conditions, the activation energy at the end of DSA must involve something more, attributed to the nucleation of kink-pairs on screw dislocations [9]. This point will be discussed further in this paper, and the jerky-serrated flow transition will be interpreted.

In the case of substitutional solutes, DSA often takes place at higher temperatures than in the preceding case. The activation energy at the end of DSA then corresponds to the diffusion energy of the substitutional solute involved, which can be interpreted by a conventional diffusion-controlled glide mechanism (where no kink-pair nucleation is necessary, contrary to the case of interstitial carbon). Under such conditions, considering that the density of solute atoms (usually larger than 1%) is so high that only short-range diffusion motion is necessary to lock dislocations, the activation energy at the beginning of DSA (about half the activation energy of solute diffusion) could be interpreted by short-range pipe diffusion. On the other hand, recent studies of FeCr alloys conclude that DSA is controlled by the same diffusion mechanisms of interstitial carbon or nitrogen as in binary FeC alloys [6]. These different hypotheses will also be discussed in what follows.

The above analysis shows that DSA due to interstitial and substitutional solutes exhibit some common properties, but also differences. This article is thus devoted to the comparison between the two types of DSA, their combination when both interstitials and substitutionals are present, and the interpretation of the different activation energies involved.

This study relies on *in situ* straining experiments in a TEM, which give the geometrical features and the kinetics of dislocation motion as a function of temperature. This technique has already been used to study DSA in TiAl, FeAl [10], FeC [9], and Zr [11]. The present experiments have been carried out in iron containing three different amounts of carbon, and in FeNiC, FeSiC, FeCrC and FeAlC alloys.

2. Experimental

In situ straining experiments have been carried out in a JEOL 2010HC TEM, using a high-temperature straining holder designed in our institute. The movies have been recorded by a Megaview III video camera working at 25 images per second, and analyzed frame by frame. Microsamples were rectangles with an electro-polished thin-edged hole, glued with a high-temperature cement on two rings adapted to the straining holder (a former version of the high-temperature holder is described in Ref. [12]).

Supplementary data related to this article can be found online at <http://dx.doi.org/10.1016/j.actamat.2016.04.018>.

Experiments as a function of the carbon concentration have been made in

- i) Ultra-pure iron containing less than 0.3 ppmwt of C, O, and N, provided by J.P. Peyrade [13],
- ii) Electrolytically pure iron containing 3–4 ppmwt of C, less than 2 ppmwt each of S, N, and Cr, and less than 5 ppmwt each of O and P, and
- iii) FeC same as ii) but with 50 ppmwt of C.

Alloys ii) and iii) were provided by the Ecole des Mines of St Etienne. The three above FeC alloys are hereafter designed as i) Fe-1 ppmat C, ii) Fe-15 ppmat C, and iii) Fe-230 ppmat C.

Other experiments were carried out in Fe5%Cr, Fe14%Cr, Fe1%Ni, Fe1.5%Si (percentages in weight) with the same amounts of other elements and carbon as in the electrolytically pure iron [8,14].

These alloys are hereafter designed as FeCrC, FeNiC and FeSiC. Lastly, two FeAlC alloys have been provided by Arcelor Mittal, with 8%wt (17%at) of Al, and respectively 20 ppmat and 130 ppmat of carbon.

All alloys have a grain size of the order of 100 μm , and an initial dislocation density lower than 10^8 cm^{-2} .

3. Results

3.1. Iron with interstitial carbon

3.1.1. Fe-16 ppmat C

The results published in Ref. [9] were focused on the comparison of *in situ* experiments and *post mortem* observations above 250 °C. Here we show new results of *in situ* experiments above 20 °C. At increasing temperatures, the kinetics of dislocations can be described as follows:

- At 20 °C, dislocations have a slow and steady motion controlled by a nearly athermal Peierls mechanism on screws described in Ref. [15].
- At 100 °C, dislocations start to move in bursts, as shown in Fig. 1. This means that no dislocation motion is observed upon straining during several minutes, until a source is unlocked and emits many dislocations in less than one frame of the video (40 ms). The thick slip trace at the foil surface, which has appeared in this short time, is more easily seen in the intermediate picture Fig. 1b showing the difference between the initial and final states of the observed area. The rapidly moving dislocations have escaped on the right side, but one can notice small movements of neighboring dislocations characterized by pairs of black/white contrasts.
- At 200 °C, the bursts are much more violent than at 100 °C, as shown in Fig. 2. Indeed, the intermediate difference-image Fig. 2b shows a high density of slip traces with many directions, which have appeared suddenly like an avalanche. As discussed in Refs. [9], dislocations tend to lock themselves in the pure screw orientation, as shown in Fig. 2a. These straight screws appear in black contrast in the intermediate difference-image, which means that they have been unlocked during the burst.
- At 250 °C and above, the straight screw dislocations start to glide slowly and viscously, and their motion seems to be controlled by a “high-temperature Peierls mechanism” similar to that already observed at and below 20 °C. They form dipoles by cross-slip at super-jogs, as shown in Fig. 3. The average screw velocity is 7 nm/s. This unexpected resurgence of a low-temperature Peierls mechanism at 250 °C and above has been confirmed by *post-mortem* observations and discussed in Ref. [9]. It will be discussed again in Section 4.1. Avalanches of rapidly gliding dislocations are however superimposed on the slow and viscous motion of screws, as long as the latter is too slow at account for the imposed strain-rate, i.e. below 300 °C.

3.1.2. Fe-230 ppmat C

The same sequence of events as above has been observed, however with slightly different temperature limits:

- At 20 °C, the motion is already in bursts as shown in Fig. 4a–d. Indeed, new almost vertical slip traces appear frame after frame in the zone limited by the arrows. This zone progressively extends to the right as expected from the propagation of a Luders band at the micro-scale. As in Fe-16 ppmat C at 100 °C, the

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