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Tensile properties and internal friction study of dislocation movement in iron–copper system as a function of copper precipitation

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Abstract

Tensile properties and internal friction of thermally aged (at 773 K) Fe–1 wt% Cu alloys are measured as a function of copper precipitation in the temperature region between 100 and 600 K. Two regimes have been clearly identified in terms of yield stress at low temperatures below 250 K. It was found that in the over-aged samples the yield stress decreases much more abruptly with the temperature than in the specimens annealed at shorter time. The internal friction spectra show the existence of similar features in all samples, which indicates that no drastic change of the microstructure occurs during aging. These results suggest that the lack of softening at low temperatures originates principally from thermally-activated kink-pair formation and gliding processes, controlled by the size and/or density of Cu-precipitates.

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1. Introduction

The effects related to copper precipitation are known to influence the embrittlement susceptibility of pressure vessel steels (RPVS) in nuclear reactor [1]. Under irradiation, the growth of copper precipitates is enhanced which causes the increase of the friction stress in RPVS. Since copper precipitates represent strong obstacles to dislocation motion, the irradiation induces hardening of RPVS, and consequently a rise of the ductile to brittle transition temperature [2–11]. A similar hardening effects are observed in the case of thermal aging experiments of super-saturated Cu–Fe model alloys [6]. Because of that, the model alloys are widely used to analyze the effects of copper precipitation to the mechanical properties. The study of various binary iron–copper alloys [12] showed that even small concentrations of copper a little higher than the solubility limit (maximum concentration of solute dissolvable in solvent) are capable of producing a large increase in yield stress upon thermal aging. The peak hardening is found to depend on the copper content and aging temperature [12]. Before reaching the peak hardening, the copper precipitates form obstacles to dislocation motion since their shear modulus is smaller than that of iron, and hardening occurs

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[13]. In the over-aging regime, when thermal aging is continued past the peak hardening condition corresponding to precipitate diameters larger than 5–6 nm [14], the alloy becomes softer. While the kinetics of copper precipitation and the induced hardening are well studied up to the peak hardening [6], the over-aged phase is less understood, due to lack of a complete description of incoherent copper precipitation and its interaction with dislocations.

Here, an analysis of the influence of copper precipitation and temperature on dislocation movement in the over-aged regime is presented, by performing tensile tests and internal friction measurements in aged Fe–1 wt% Cu alloys.

2. Experimental

The Fe–1 wt% Cu model alloy was prepared by melting low carbon steel in air, and adding the corresponding weight percent of pure copper. After cooling down, the block was heated up to 1350 K (1050 °C) and hot rolled four times. The chemical composition of the material is presented in Table 1.

The various stages of copper precipitation were achieved by a thermal aging process, which consists of time dependent heat treatments at 773 K (500 °C) in argon atmosphere, and subsequent fast quenching into iced water. The duration of the heat treatments varied form 0.1 to 430 h.

The tensile tests were performed on an Instron 1341 machine, in a temperature region between 100 and 600 K, with a constant crosshead speed of 0.1 mm min^{-1} which corresponds to a strain rate of $1.4 \times 10^{-4}\ \mathrm{s}^{-1}$. The tensile specimens are 24 mm long, with a cylindrical gauge section of 2.4 mm in diameter, and 12 mm in length.

The internal friction measurements were performed in an inverted torsion pendulum operating at around 1.5 Hz, in the temperature range between 90 and 400 K. The vibrational amplitude was about 10^{-4} and the heating/cooling rates were about 1 K min^{-1}. The measurements have been performed in He atmosphere.

3. Results

In Fig. 1, the yield stresses of different aged Fe–1 wt% Cu samples and of pure iron are presented as a function of test temperature. By increasing the temperature, all samples exhibit a decrease of the yield stress due to the increase of dislocation mobility caused by thermally activated dislocation–diffusion processes. An athermal ‘plateau’ is reached at temperatures higher than approximately 250 K, in each case the yield stress value depending on the ageing duration of the material. Below the peak hardening regime, corresponding to a heating time of 15 h in Fig. 1, the copper precipitates form obstacles to dislocation motion, since their shear modulus is smaller than that of iron. This induces an increase in the dislocation pinning strength and causes hardening of the model alloy. If the flow stress is represented by the following expression:

$$\sigma = \sigma_0 + \sigma^*(\dot{\varepsilon}, T), \tag{1}$$

the hardening can be understood [15] as an increase of the elastic limit of the athermal part of the flow stress, σ_0. The second term corresponds to the short range, intrinsic lattice type contribution (also

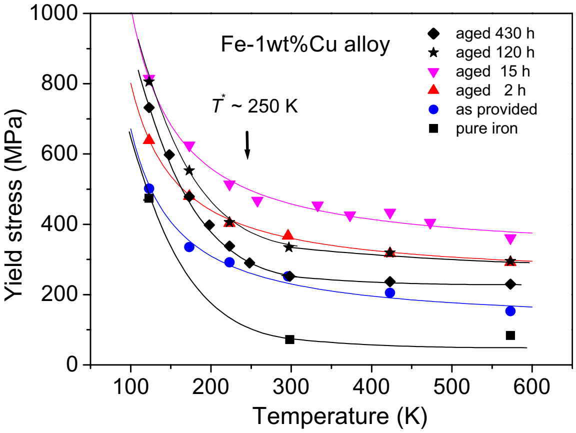

Fig. 1. Yield stress as a function of test temperature for the as-provided and aged Fe–1 wt% Cu model alloy. The points correspond to the experimental data and full lines represent a guide for an eye.

Table 1
Impurity composition of the Fe–1 wt% Cu model alloy

Element	Mn	P	S	Al	Cr	Ni	Cu	C	N	V	W	Pb	Zr
Fraction (ppm)	270	110	20	–	145	255	9500	35	58	20	180	36	30

known as the Peierls contribution) to the yield stress, and it can be expressed as,

$$\sigma^* = \sigma_p\left(1 - \frac{T}{T_c}\right)^m, \tag{2}$$

where σ_p is the Peierls constant, m describes the shape of the Peierls barrier (typically $m \cong 2$), and T_c is the critical temperature above which the Peierls contribution vanishes:

$$T_c = \frac{H_p}{k_B \ln(\dot{\varepsilon}_0/\dot{\varepsilon})}. \tag{3}$$

The $\dot{\varepsilon}$ is the strain rate, and H_p is the activation enthalpy for thermal activation over the Peierls barrier. At and above the peak hardening regime, which is approximately represented by the behavior of the 15 h aged sample, the precipitates start playing the major role in dislocation movement, and two types of behavior are observed. It is found that the hardening rate differs for the samples aged below the peak hardening regime and for the over-aged samples. In another words, the yield stress does not vary as a function of temperature in the same way in all samples. In order to illustrate this in more detail, the yield stress as a function of aging time for two test temperatures, 125 and 435 K is plotted in Fig. 2. At 435 K, the over-aged samples exhibit softening with increasing aging. This is caused by the ability of dislocations to bow. In this situation, the copper precipitates are well separated in space and become incoherent softer obstacles to dislocation movement (phase transformation to 9 R and thereafter to fcc structure [9]). Because of that, dislocations release their energy related to the elastic distortion and softening of the material occurs. However, at low temperatures, see Fig. 2, the over-aged model alloy samples remains hard, indicating that the thermal energy is not large enough to activate dislocation glide. Bowing of dislocations can be described via a kink-pair formation process which is strongly temperature dependent [16]. Therefore, the behavior of the yield stress of the over-aged samples should be explained by the second term in Eq. (1). According to Seeger's model [16], the two types of thermal behavior (the over-aged and solid solution regimes) can be regarded as having different kink-pair activation energies. In order to analyze the origin of this enthalpy change caused by aging in more detail, the yield stress of pure iron is also plotted in Fig. 1. It is evident that a similar change of the kink-pair activation energy is required in order to understand the change of the yield stress behavior between pure iron and the as provided Fe–1 wt% Cu model alloy. Moreover, the decrease rate of the yield stress as a function of the test temperature in over-aged samples seems to resemble the pure iron case, which indicates that over-aged samples tend to approach the behavior of pure iron. To estimate the change of the kink-pair activation energy, the internal friction measurements are performed.

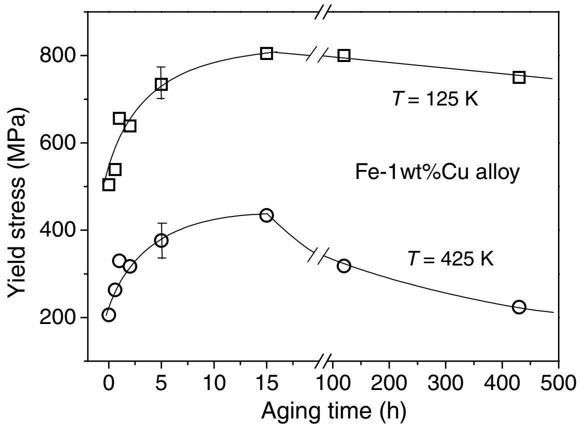

Fig. 2. Yield stress as a function of aging time in the thermal and athermal regimes.

The internal friction coefficient, Q^{-1}, as a function of the temperature and aging is shown in Fig. 3. The spectra show the existence of similar features in all samples, which indicates that no drastic change of the microstructure occurs during aging. The results obtained in the case of non-deformed, and room temperature shear-deformed samples (the degree of deformation was about 10%) are presented and indicated by the dotted and full thick lines, respectively. The internal friction of the non-deformed samples exhibit the Snoek peak at about 310 K [17], associated with the relaxation process of carbon interstitial atoms. Upon aging, the intensity of the Snoek peak decreases due to the increase of carbon trapping mainly by the precipitates. In the deformed samples three peaks are found in the deconvoluted spectra at about 220, 260 and 310 K, see Fig. 3. The 220 and 310 K peaks can be assigned to the Snoek–Köster relaxations [18], corresponding to the modified β- and γ-relaxation processes that are typical for the pure α-iron, respectively [19,20]. They result from the change of the kink-pair generation in screw dislocations, characterized by the Burgers vectors $a_0\langle 111\rangle/2$, due to the presence of carbon

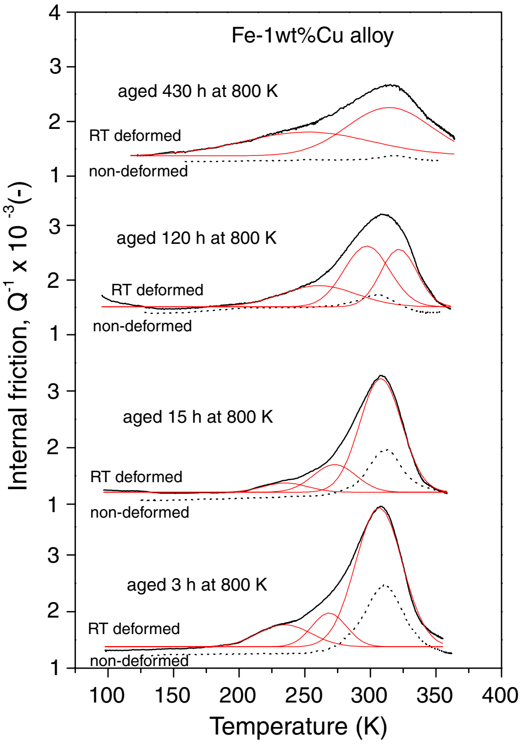

Fig. 3. Internal friction coefficient as a function of temperature for the Fe–1 wt% Cu alloy. Thick and dotted lines represent the room-temperature deformed and non-deformed spectra, respectively. Thin lines correspond to the deconvoluted internal friction peaks of the deformed samples.

interstitials. All peaks are abruptly shifted towards higher energies by increasing the aging time above the peak hardening, in a nice correspondence with the abrupt change of the hardening rate observed in the tensile tests. The energy of the middle peak (260 K) is about 0.75 eV and is lower than the both the kink-pair activation energy of pure iron (~0.9 eV) and the Snoek–Köster (carbon) relaxation process (0.85 eV). Thus, this feature could be related to the kink-pair activation process being modified due to the interaction between dislocations and copper impurities.

4. Conclusion

Tensile properties and internal friction of an Fe–1 wt% Cu alloy are measured as a function of copper precipitation in the temperature region between 100 and 600 K. Upon aging, different temperature dependencies of the yield stress are observed, and attributed to the change of the kink-pair activation energy. This effect originates from the dislocation movement influenced by the subtle interplay between the temperature and the size of precipitates. A similar change of the activation energy of dislocation movement is required to understand the change of the yield stress behavior between pure iron and the as-provided Fe–1 wt% Cu model alloy.

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