Study on Internal Friction of Dislocation Density in Bake-Hardening Steel

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ABSTRACT: This study investigates internal friction of dislocation density in bake-hardening steel. The experiment was conducted as follows: pre-deformation being set at 2%, 5% and 10% with the baking set at 170℃×20min. The curve of internal friction and the configuration of transmissivity were also observed. It can be concluded that with the increase of the pre-deformation value, the Snoek peak of the experimental steel decreased. Moreover, both of the SKK peak and the dislocation density grew at first and then dropped. It is noted that while the pre-deformation was set at 5%, its SKK value and the dislocation density reached their respective peak value, whereas both decreased while the pre-deformation at 2% and 10%. This study investigates the changing mechanisms of dislocation density in bake-hardening steel in relation to different degree of pre-deformation.

1. Introduction

Low level of dislocation density would be massively produced after the processes of hot-rolling, cold-rolling, annealing and smoothing of the bake-hardening steel. Nevertheless, after the deformation processes such as stamping and prestraining, there would be a sharp increase of the dislocation intensity in the ferrite matrix. Subsequent to the baking time-effect of 150~200℃, the thermal activation energy would rise in the free carbon atom, which would speed up the diffusion of dislocation. It would also be segregated into the fundamental dislocation point and the pinning dislocation point where which would form the Cottrell atmosphere [1,2]. Dislocation intensity would directly affect the BH value in baking hardening. This being so, understanding its effect on bake-hardening steel would be crucial to further investigate its core mechanisms. The measurement of dislocation intensity has been widely mentioned in scientific literature. In sum, the common methodologies include: positron annihilation[3], X-Ray diffraction[4] and internal friction[5,6].

The static information of the micro-structure of materials could be analyzed through adopting the methodologies of positron annihilation and diffraction. However, the error in point measurement and the transitional point of solid defects could not be detected. The objective of this study is to demonstrate that internal friction could be adopted as a more effective methodology comparatively, especially for measuring the destruction test and for obtaining the data of changing in micro-structure. More significantly, internal friction is an effective way to receive the kinetics parameters in defect polarization. The frequency measurements of the spectrum can fully cover the eigen-frequencies...
occurring in the slow migration process of the defects. Moreover, the addition of stimulus signal would become an alternating stress and the responding signal be strain. Therefore, internal friction is another effective and important way to study the defects relaxation process and the changes in micro-structure. In short, this paper adopts TEM and internal friction to analyze the changes of dislocation intensity after bake-hardening.

2. Experimental material and method

Low carbon bake hardening steel in annealing was maintained as the main experimental material. Table 1 listed its chemical composition as follows:

|   | C     | Si    | Mn     | P     | S     | Al    | N    |
|---|-------|-------|--------|-------|-------|-------|------|
|   | 0.19-0.21 | 0.01-0.13 | 0.20-0.25 | 0.011 | 0.007 | 0.019 | 0.002 |

The gauge length of 20mm as standard tensile specimen was used to be tested for the experimental material. The following procedure was utilizing UTM5305 to perform various pre-deformation on axial tension. The adjustment of the degree of pre-deformation was: 2%, 5% and 10%, with 5mm/min being the tensile speed. 101-1 oven was the baking equipment, and the baking temperature was set at 170°C×20min.

The BH value was tested in accordance with GB/T24174-2009, and was analyzed by the means value of the 5 sample experiments. The average value of these 5 samples was obtained and observed. The micro-structure of the samples was observed through ZEISS optical microscope, and the dislocation morphology by JEM-2100 TEM. The internal friction measurement was conducted by MFP-1000 multifunctional internal friction instrument. The size of internal friction sample was: 50mm×1mm×1mm. The heating rate was set at 3°C/min. The room temperature was set at 700°C. The strain amplitude was 20×10⁻⁶. The frequency testing was: f=1. A corresponding fitting software was adopted to reflect and reveal the nature of internal friction. The testing methods include: annealing state, pre-deformation after annealing and baking pre-deformation after annealing.

3. Discussion of experiment results

3.1 The curve of different pre-deformation and after-baking

In Fig. 1 (a) to Fig. 1(d), the baking temperature was set at 170°C and baking time 20 minutes. The pre-deformation rate was: 0%, 2%, 5% and 10%. In each graph, the respective curve was the record of the real internal friction curve.
From these 4 graphs, it can be observed that in Fig. 1(a), the Snoek peak reached the highest before pre-deformation. There was no occurrence of the SKK peak and no dislocation of the free migration. In Fig. 1(b), (c) and (d), SKK peak occurred after pre-deformation which demonstrated the massive free migrating dislocation. After the pre-deformation at 2%, 5% and 10%, the common findings were that the SKK peak reached its maximum and then decreased.

3.2 Appearance observation of dislocation organization
In order to observe the dislocation configuration at various pre-deformation after baking, a TEM analysis was conducted on the experimental materials. The baking temperature was set at 170℃ and the baking time 20 minutes. The pre-deformation value was set at 2%, 5% and 10%. The experimental results were shown in Fig. 2 (a) to Fig. 2 (d).
From Fig. 2 it can be observed that under the same physical circumstances of baking temperature at 170°C and the baking time of 20 min., the dislocation density reached its maximum at the pre-deformation set at 5%. While the pre-deformation was changed from 2%→5%→10%, the dislocation density increased initially but decreased subsequently. At 2% of pre-deformation, there would be an interaction movement of the dislocation in the crystal, and thus forming a disorganized and uneven distribution in which a dislocation tangle was produced. With the increase to 5% of the pre-deformation, a massive dislocation would occur which would form a dislocation net, and more dislocation would be tangled in it. It should be noted that the distribution would form an even-distributing dislocation net. With the continuous increase of the pre-deformation at 10%, the dislocation density decreased and the dislocation tangle still occurred. This was due to the dislocation reaction in the spaces between the dislocation and thus the two phenomena canceled each other out.

4. Conclusion
The experimental steel materials were set at 2%, 5% and 10% pre-deformation. Combining its internal friction curve and the TEM analysis, it can be observed that with the increase of pre-deformation, the Snoek peak decreased. Both the SKK peak and the dislocation density increased at first but decreased afterwards. At 5% of pre-deformation, both the SKK peak and the dislocation density reached its maximum. At 2% and 10% pre-deformation, both decreased correspondingly. This experiment adopted internal friction to study the changing mechanisms of bake-hardening steel at 2%, 5% and 10% pre-deformation after baking.

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