



## Discontinuous yielding induced by the addition of nickel to interstitial-free steel

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Received 3 February 2014; revised 7 March 2014; accepted 7 March 2014

Available online 20 March 2014

Interstitial-free steels containing 1–3% Ni were tensile tested. A clear yield point and yield point elongation were observed in each steel, even though they contained no solute carbon or nitrogen in the ferrite matrix. The mechanism could be explained by grain refinement strengthening enhanced by the addition of Ni, which is derived from the increase in Hall–Petch coefficient caused by grain boundary segregation.

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**Keywords:** Interstitial-free (IF) steels; Grain boundary strengthening; Hall–Petch effect; Yield phenomena

The yield point in iron is a phenomenon that has been investigated for several decades because of its industrially important problems, such as the stretcher strain in press forming and the Lüders propagation during tensile deformation. In particular, the mechanism of discontinuous yielding has been discussed on the basis of dislocation theories, and the existence of solute carbon was considered to be one of the dominant factors to cause discontinuous yielding. Cottrell and Bilby [1] reported that the yield point phenomenon in steel is triggered by the release of dislocations locked by the solute carbon atmosphere (Cottrell atmosphere). On the other hand, the present authors reported that the Hall–Petch coefficient,  $k_y$ , significantly increases with increasing solute carbon content up to 60 ppm in ferritic steel although the friction stress is not greatly increased in this carbon content range [2]. The mechanism of the increase in  $k_y$  has also been discussed in terms of grain boundary segregation of carbon atoms, and it has been suggested that discontinuous yielding is caused by the emission of dislocations from the grain boundary and that the carbon atoms present stabilize the dislocation emission site at the grain boundary [3]. In other words, the increment of the upper yield stress by the addition of

carbon is mainly due not to solid solution strengthening by solute carbon but to the enhanced contribution of grain refinement strengthening. If this idea is true, the yield point should appear when the value of  $k_y$  was enlarged by the addition of some substitutional elements which do not form a Cottrell atmosphere at ambient temperature. Morrison and Leslie have previously reported [4] that Ni and Si enlarge  $k_y$  in interstitial-free (IF) steels. In this study, the yielding behavior during tensile testing was investigated by using Ti-added IF steels containing 1–3% Ni to clarify the mechanism of discontinuous yielding in polycrystalline steel. The results of this research could provide important information about the contribution of solute interstitial elements in the discontinuous yielding phenomenon.

The specimens used in this study are three kinds of IF steels with different amounts of Ni prepared with a vacuum induction melting furnace:

Fe–0.99Ni–0.081Ti (1Ni), Fe–1.98Ni–0.048Ti (2Ni) and Fe–3.02Ni–0.076Ti (3Ni) (mass%).

A sufficient amount of Ti was also added to each steel to fix the carbon and nitrogen as Ti(C,N). Ingots of 80 mm<sup>3</sup> were hot-rolled to 4 mm<sup>3</sup> at 1493 K. After descaling by surface grinding, the steel plates were cold-rolled to 80% thickness reduction. The cold-rolled steel sheets were then annealed for 1.0 ks in the ferrite single phase region from 933 to 1098 K to control the grain size. Finally, all specimens were annealed at

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873 K for 3.6 ks to ensure that the Ti(C,N) was fully formed. In order to obtain a larger grain size, the steel plates were cold-rolled to 70% thickness reduction and then subjected to recrystallization treatment at 953 to 1073 K for 1.0 ks. The steel sheets were then cold-rolled to a further 10% thickness reduction and annealed again in the ferrite single phase region for 43.2 ks for grain growth to occur. The microstructure was observed by optical microscopy and scanning electron microscopy using electron backscattered diffraction and a Cs-corrected STEM/TEM (JEM-ARM 200F) equipped for energy-dispersive X-ray spectroscopy (EDS). The specimens for scanning transmission electron microscopy (STEM) were prepared by a dual-beam FIB machine (FEI Quanta 3D 200i). The grain size was evaluated by the nominal grain size obtained by the quadrature method [5]. It was confirmed that the ferrite grain size is in the range between 14 and 105  $\mu\text{m}$  and specimens have equiaxed ferrite grains without texture. Tensile tests were carried out at the initial strain rate of  $1.0 \times 10^{-3} \text{ s}^{-1}$  for sheet test pieces standardized as JIS13B. The yield stress was evaluated at 0.2% proof stress. In addition, an aging index (AI) test was performed to confirm whether carbon and nitrogen exist in solid solution or not. The AI testing was conducted as follows: tensile testing at room temperature to 10% elongation, aging at 373 K for 3.6 ks, then tensile testing again at room temperature.

Figure 1 shows the Hall–Petch relationship at 0.2% proof stress in Ni-bearing IF steels. The data of the IF steel without Ni [6] are also plotted in this figure. The stress at  $d^{-1/2} = 0$ , extrapolated by the least-squares method, could be regarded as the friction stress ( $\sigma_0$ ). The friction stress increases almost linearly with an increase in Ni content, indicating that the solid solution strengthening in ferrite steel is proportional to the Ni content in the range of 1–3% Ni content. On the other hand, the slope in this figure, the so-called Hall–Petch coefficient ( $k_y$ ), enlarges with increasing Ni content. In particular, it is noted that the value of  $k_y$  in 3Ni steel ( $k_y = 350 \text{ MPa } \mu\text{m}^{1/2}$ ) is more than twice that in IF steel without Ni ( $k_y = 150 \text{ MPa } \mu\text{m}^{1/2}$ ). These results mean that the addition of Ni contributes not only to solid solution strengthening but also to grain refinement strengthening associated with the increase in  $k_y$ . Figure 2 shows the effect of nickel under 0.2% proof stress and friction stress in ferritic steels with the grain size of

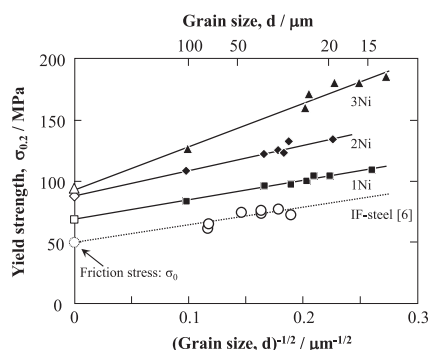


Figure 1. Effect of nickel on Hall–Petch relation in IF steels.

20  $\mu\text{m}$ . It was confirmed that the slope at 0.2% proof stress is almost the same as that ( $32 \text{ MPa mass}^{-1}$ ) reported by Morrison and Leslie [4], Kranzlein et al. [7] and Rees et al. [8]. Since, the true solid solution strengthening should be represented under friction stress, the 0.2% proof stress shows both solid solution strengthening and grain refinement strengthening. In the other words, the difference between the 0.2% proof stress and friction stress corresponds to the contribution made by grain refinement strengthening. The pure solid solution strengthening due to Ni is expressed as  $\sim 15 \text{ MPa mass}^{-1}$ . This result suggests that the effect of alloying elements on the Hall–Petch coefficient as well as solid solution strengthening must be evaluated separately when considering the yield strength of polycrystalline metals, even if the grain size is controlled to be same.

Figure 3(a) shows the stress–strain curve of specimens with the same grain size of 20  $\mu\text{m}$ . As mentioned above, the yield stress increases with increasing Ni content, but it should be noted here that a clear yield point appears in the Ni-bearing IF steels. In particular, a fairly large yield point elongation appears in 3Ni steel. The open marks in the elastic deformation region shows the friction stress that was estimated from the result shown in Figure 1. The difference between yield stress and friction stress corresponds to the amount of grain refinement strengthening. On the other hand, Figure 3(b) shows the result of the AI test for the 3Ni steel. If solute interstitials are retained in the ferrite matrix, the yield stress should be enhanced after aging due to strain aging. However, no change in the flow stress is evident after aging. This indicates that both carbon and nitrogen have fully been fixed as Ti(C,N), and that the discontinuous yielding observed in Ni steels is not caused by solute interstitial elements. In other words, the existence of solute interstitial elements is not an essential condition for the occurrence of discontinuous yielding; rather, that is a substantial phenomenon observed in steel strengthened by grain refinement strengthening.

It is generally recognized that the pile-up theory [9,10] can explain the yielding phenomenon in polycrystalline metals. In this theory, yielding occurs when the dislocations are emitted from the grain boundary when the stress concentration at the grain boundary exceeds a critical stress required to activate the dislocation source

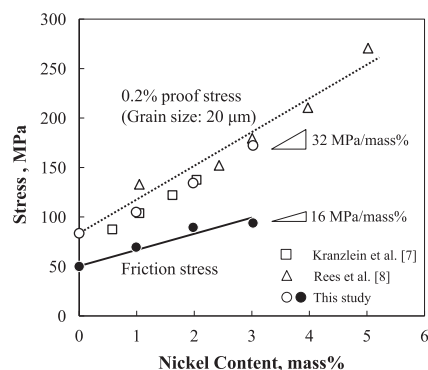


Figure 2. Effect of nickel on friction stress and 0.2% proof stress in IF steels.

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