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# Analysis of mechanical property distribution in multiphase ultra-fine-grained steels by nanoindentation

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## Abstract

The strength characteristics of microphases in ultra-fine-grained steels were analyzed using nanoindentation and AFM. It was found that there were fine ferrite grains (1–2  $\mu\text{m}$ ) formed by a strain-induced dynamic transformation in ultra-fine-grained steels. They had equiaxed and polygonal grain shape, and higher hardness and elastic modulus than coarse ferrite transformed statically. Strengthening factors of strain-induced dynamic transformation ferrite were analyzed in terms of cementite particles and dislocation density. © 2001 Acta Materialia Inc. Published by Elsevier Science Ltd. All rights reserved.

*Keywords:* Ultra-fine-grained steels; Nanoindentation; Hardness; Strain-induced dynamic transformation ferrite

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## Introduction

The nanoindentation technique has been used to evaluate the mechanical properties of various micromaterials. The load–depth curve can be derived from the depth-sensing indentation technique. Nanoindentation also has been used to probe mechanical properties in the sub-micron regime of multiphase materials, since this technique applies ultra-low loads (on the  $\mu\text{N}$  scale). For example, the effects of aging in the austenite and ferrite phases have been analyzed in duplex stainless steels [1], and changes in local mechanical properties across a grain boundary have been observed [2]. In these approaches, atomic-force microscope(y) (AFM) were used to identify the microphase and the grain boundary. Indenter positioning and residual indentation observation, in this case, can be performed within the interior of the grain, which has size less than 1–2  $\mu\text{m}$ .

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In this study, the nanoindentation technique is applied to analyze the strength properties of ultra-fine-grained steels. Ultra-fine-grained steels, whose ferrite grain sizes are approximately 1–2  $\mu\text{m}$ , were developed in order to increase both the strength and ductility of structural steels. Fabricated by isothermal multi-pass rolling at high temperature, they are composed of various microphases such as ferrite, pearlite and martensite. In particular, ultra-fine-grained steels have two kinds of ferrite: the fine-grained strain-induced dynamic transformation ferrite (SIDTF) obtained in the austenite temperature region and the coarse ferrite transformed statically during cooling. The ferrite grain refinement makes the ultra-fine-grained steels stronger than conventional structural steels. But the contributions of microphases to the strength of ultra-fine-grained steels have not been studied. While each phase is expected to have different strength characteristics, the individual mechanical properties of each single phase are hard to obtain by conventional testing technique due to the small size. We thus used the nanoindentation technique combined with atomic-force microscopy to analyze these mechanical properties.

## Experimental procedure

Ultra-fine-grained steels were fabricated by isothermal multi-pass rolling of API X65 steel above the Ar3 transformation temperature and subsequent cooling at Pohang Iron and Steel Co., Ltd. The chemical composition of API X65 steel is shown in Table 1. The ultra-fine-grained steels formed by hot rolling and water quenching, had ferrite and martensite phases. The ferrites are divided by grain size into SIDTF and cooling ferrite. The SIDTF has a fine grain size of 1–2  $\mu\text{m}$ , a grain refinement due to a strain-induced dynamic transformation from austenite to ferrite during multi-pass rolling at high temperature. SIDTF is known to be very stable against coarsening, even at elevated temperature, because of the quite random orientations of the ferrite grains [3].

For the nanoindentation test,  $10 \times 10 \times 1 \text{ mm}^3$  specimens were machined from the rolled plate. The specimen surface was mechanically polished and etched in 2% nital. API X65 steels were used for comparison with experimental results for the ultra-fine-grained steels.

In the nanoindentation test, a Berkovich tip was used as an indenter to evaluate hardness and elastic modulus [4]. In this situation, loading rate, maximum indentation load and surface-polishing method affect the experimental results. Primarily, the evaluated hardness may increase or decrease with decreasing maximum indentation load. Therefore optimum experimental conditions must be determined before testing. Various maximum indentation loads from 250 to 2000  $\mu\text{N}$  were employed. The hardness

Table 1  
Chemical compositions of API X65 steels (ultra-fine-grained steels)

Compositions	C	Si	Mn	Ti	Nb	V
%	0.1	0.26	1.5	0.016	0.046	0.053

values obtained were constant regardless of maximum indentation load. The same procedure was then applied at various loading rates of 50–1000  $\mu\text{N/s}$  and constant hardness values were obtained, regardless of loading rate. Thus all indentation tests in this study were made at 100  $\mu\text{N/s}$  up to a maximum indentation load of 1000  $\mu\text{N}$ . Finally, the work-hardening effect of mechanical polishing was considered by comparing the hardness values obtained by mechanical polishing and 2% nital etching with the one obtained by electro-polishing in 50% water and 50% perchloric acid solution. No difference between them was found.

Nanoindentation tests were made on the surface of specimen after indenter positioning by AFM. Several indentation mark arrays at sub-micrometer spacing were made. After indenting, the residual indentation, indented grain shape, and grain size were observed by AFM.

## Results and discussion

Nanoindentation hardness values were obtained in API X65 steels. All the ferrite phases has about 2 GPa hardness and a uniform hardness distribution. On the other hand, nanoindentation hardness values at grain boundary were different with the one of the grain interior. In Fig. 1, the obtained hardness increased slightly as the indentation approached the grain boundary. It could be thought that the grain boundary played a role of the barrier of dislocation movement. Also hardness at the grain boundary with the precipitate (circle in row 1) increased significantly. But hardness at the grain boundary without precipitate (circle in row 2) dropped significantly. This hardness drop could be thought to occur from the stress relief at the etching step of grain boundary.

The microstructure can be observed by optical microscope in hot-rolled and water-quenched ultra-fine-grained steels, but the shape and the size of the fine ferrite grains could not be determined accurately. An AFM was required for accurate indenter

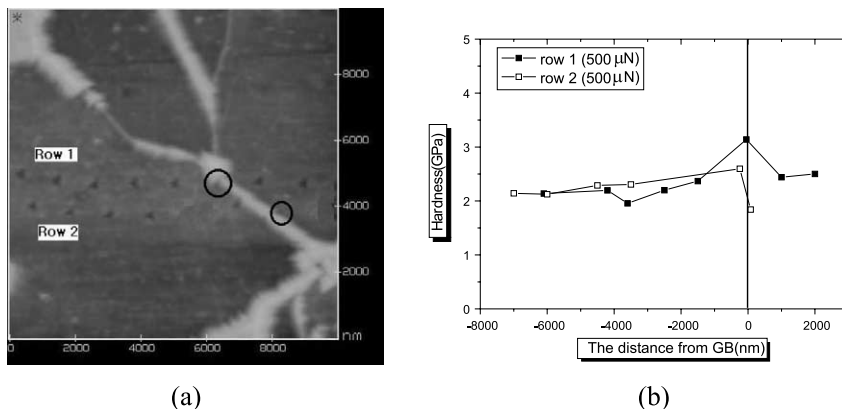


Fig. 1. The strength characteristics of grain boundary in API X65 steels: (a) the residual indentations near the grain boundary and (b) the hardness variation near the grain boundary.

positioning and observation of the ultra-fine ferrite. Hardness and elastic modulus were evaluated from indentation load–depth curve [4]. The obtained hardness distribution in ferrite phases differed from that of API X65. Hardness values of various ferrite phases differed with grain size and shape. The average hardness of the fine grain size of 1–2  $\mu\text{m}$ , classified as SIDTF, was 2.92 GPa above the 2.01 GPa value of coarse ferrite transformed statically. Additionally, they had different elastic modulus values: SIDTF has a higher elastic modulus than coarse ferrite.

In ultra-fine-grained steels, both the SIDTF and the coarse ferrites could be detected by AFM, but another phase was observed. The variations of indentation load–depth curves are shown in Fig. 2. The unidentified phase had much lower maximum indentation depth than ferrite. Also, it had very high hardness compared with ferrite values. The hardness and elastic modulus of each phase are shown in Table 2.

Fig. 3 classifies these microphases in terms of the grain size, shape, and hardness. In ferrite phases, SIDTF has equiaxed and polygonal grain shape and fine grain size less than 2  $\mu\text{m}$ . The hardness value of SIDTF was 2.6–3.1 GPa higher than that of coarse cooling ferrite. The cooling ferrite has random grain shape and coarse grain size larger than 5  $\mu\text{m}$ . The hardness was similar to that of the ferrite phase in API X65 steels.

Although the unidentified phase can be identified as martensite from an optical microscope image, the unidentified phase in an AFM image needs to be assessed. The hardness of the unidentified phase was evaluated from an indentation load–depth curve. This phase had a unique island shape with 4–5  $\mu\text{m}$  grain size (different from ferrite phases in AFM and SEM images), as shown in Fig. 4. It had the same alloy

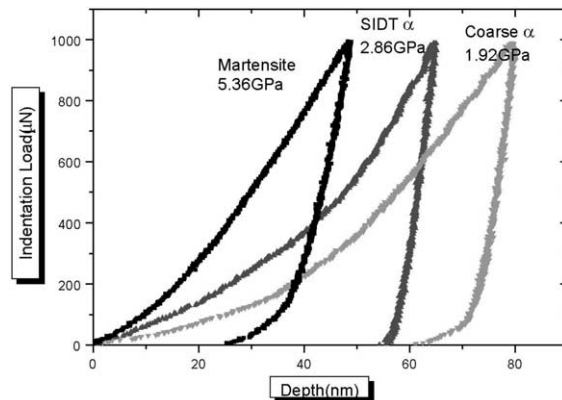


Fig. 2. Nanoindentation load–depth curves for various phases in ultra-fine-grained steel.

Table 2

Mechanical properties of ultra-fine-grained steels obtained from nanoindentation test

	SIDTF	CF	M
E	284	251	281
H	2.92	2.01	5.74

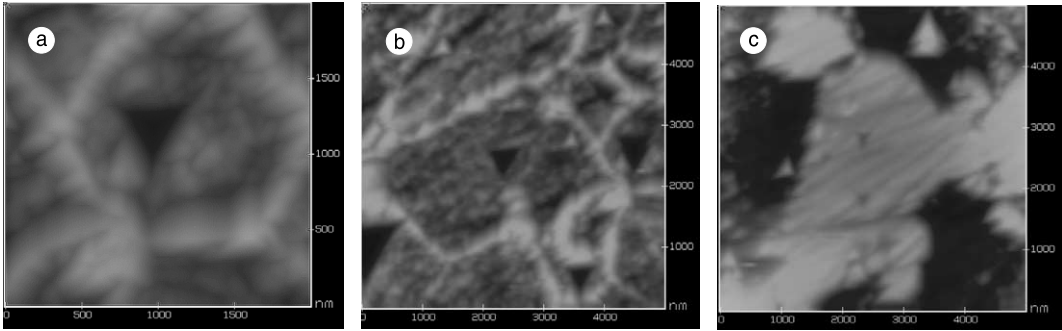


Fig. 3. Characteristics of various microphases in AFM images of ultra-fine-grained steels: (a) SIDTF, (b) coarse ferrite and (c) martensite.

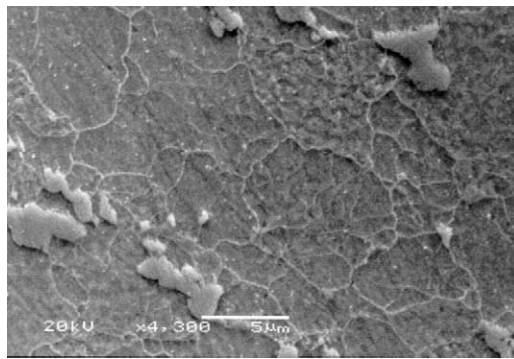


Fig. 4. SEM image of the unidentified phase in ultra-fine-grained steel.

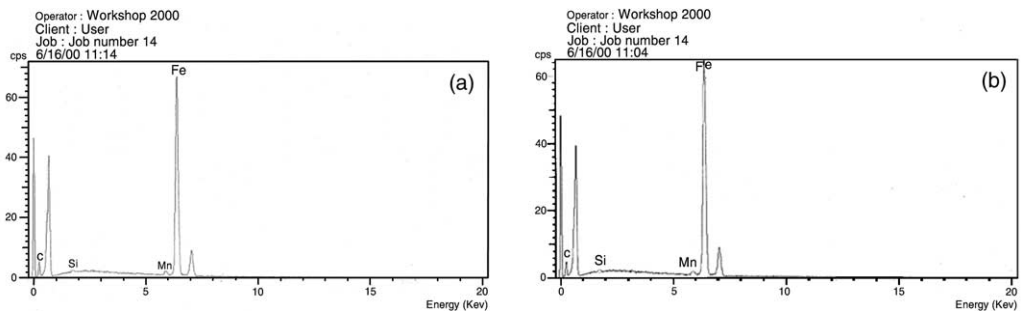


Fig. 5. The comparison of alloys contents between ferrite and unidentified phase by EDS test in ultra-fine-grained steels.

content as ferrite phase from the EDS test as shown in Fig. 5. Consequently, from the size of phase, alloy contents and hardness value, the unidentified phase was determined to be martensite. The average hardness of martensite was about 5.74 GPa.

In ultra-fine-grained steels, fine and equiaxed ferrites with 1–2  $\mu\text{m}$  grain size (SIDTF) are formed that have planar, high-angle grain boundaries separating individual ferrite

grains [5]. SIDTF was found to have high strength compared with that of normal coarse ferrites in this study. The strengthening mechanisms are expected to be the presence of cementite particles in SIDTF grain, the change in chemical composition, and dislocation density.

First, hard cementite particles play a role in strengthening SIDTF. There are cementite particles of size 0.1–0.2  $\mu\text{m}$  at grain boundary between individual SIDTF grains and also much smaller cementite particles inside the SIDTF grain. Cementite particles are formed by the precipitation of supersaturated carbon in SIDTF ferrite during cooling [3,6]. The rate of phase transformation of SIDTF is very rapid, and carbon diffusion is not fast enough for para-equilibrium between austenite and ferrite. SIDTF ferrite is thus supersaturated with carbon, and cementite particles form during cooling that act as a barrier to dislocation movement and consequently strengthen SIDTF. The change in chemical composition also strengthens SIDTF. The elastic modulus of SIDTF is higher than that of coarse ferrite. It may have the higher carbon content.

Finally, dislocation density affects the hardness of SIDTF. Deformation is suppressed in materials with low mobile dislocation density. It is known from previous work that SIDTF has a low dislocation density because it does not undergo any form of deformation once formed [5]. So the hardness of SIDTF is higher than that of coarse ferrite.

## Summary

From the nanoindentation test, all the ferrite phases of API X65 steels had the uniform hardness values of 2 GPa, but the hardness values at grain boundary were different with the existence of the hard precipitation particles. In ultra-fine-grained steels, SIDTF with very fine grain size (1–2  $\mu\text{m}$ ) had higher hardness (2.92 GPa) than 2.01 GPa of coarse ferrite transformed statically. It was analyzed that the presence of cementite particles and very low dislocation density in the SIDTF grain increased the strength of SIDT ferrites. Martensite was found to have island shapes and the hardness of 5.74 GPa.

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