Ultrafine grained steels managing both high strength and ductility

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Abstract. Mechanical properties of the ultrafine grained (UFG) steels having mean grain sizes much smaller than 1 µm were systematically shown from the original experimental data. The UFG steels performed very high strength that reached to 2-4 times of the starting materials having conventionally coarse-grained structures. On the other hand, the uniform tensile elongation was commonly limited within a few percents when the materials have single-phased UFG structures. The limited uniform elongation was explained in terms of early plastic instability. On the basis of the understanding, the ways to manage both strength and ductility in the UFG steels were indicated. The effectiveness of making the UFG structures multi-phased was confirmed.

1. Introduction
Grain refinement has been always one of the most important subjects in microstructure control of structural metallic materials. However, the minimum mean grain size that has been conventionally achieved in bulky materials is around 10 µm. In case of commercial steels, the champion value has been 5 µm in the steel plate thermomechanically processed by so-called controlled rolling [1]. On the other hand, it has become recently possible to fabricate the steels with ultrafine grained (UFG) structures of which mean grain size is smaller than 1 µm, at least in laboratory scales [2]. One of the ways to obtain the UFG steel is thermomechanical processing under critical conditions, where phase transformation from heavily deformed austenite to ferrite is basically used. For example, grain sizes around 1 µm have been achieved in carbon steels through heavy one-pass deformation at low temperatures around 500°C in undercooled austenite region followed by rapid cooling [3]. The other route to realize the UFG structures is severe plastic deformation above logarithmic equivalent strain of about 4 [4]. The SPD can be applied not only to steels but also to most of workable metals and alloys, and the UFGs in 100 nm dimensions or even nanocrystals have been obtained in various kinds of metals and alloys [4].

The UFG materials perform the strength 2-4 times higher than that of the conventionally grain-sized materials [4,5]. On the other hand, however, the UFG materials usually exhibit limited ductility (especially limited uniform elongation) [4,5]. This imbalance between strength and ductility is the biggest issue of the UFG materials for practical application in the future. If the issue can be overcome, the UFG materials are quite attractive, because high strength can be achieved in simple chemical compositions without special alloying elements. The mechanical properties of the UFG materials have not yet been systematically clarified. In the present paper, strength and ductility of the UFG ferritic
steels are systematically shown from the experimental evidences, and the way to manage both strength and ductility in the UFG steels is discussed.

2. Experimental
A Ti-added ultralow-carbon interstitial free (IF) steel was highly deformed by the accumulative roll-bonding (ARB) process, in order to obtain an UFG structure. The ARB is a kind of SPD process using rolling deformation, which was originally developed in the present author’s group [4,6]. Figure 1 illustrates the principle of the ARB process. Two pieces of the plain IF steel sheet 1 mm thick, 30 mm wide and 300 mm long were stacked after degreasing and wirebrushing the contact surfaces. The stacked sheets were spotwelded at the top and tail ends, held in an electric furnace set at 500°C for 600 s, and then roll-bonded by 50% one-pass rolling using a two-high rolling mill with a roll diameter of 310 mm without lubrication. The roll-bonded sheet was cut into two and the above mentioned procedures were repeated up to 7 cycles. Because von Mises equivalent strain for 50% rolling is 0.8, the total strain applied to the material by 7 cycles of the ARB is 5.6. The ARB processed sheets were annealed at various temperatures ranging from 400 °C to 800 °C for 1.8 ks to change the grain size.

Another thermomechanical processing [7,8] was applied on a plain low-carbon steel (JIS-SS400) to fabricate another kind of UFG structure. The detailed procedures of the process were shown in the previous article [7]. The sheet of the SS400 having fully martensitic structure was obtained by austenitization and water-quenching. The martensite sheet was conventionally cold-rolled by 50% total reduction and annealed at various temperatures from 200 °C to 700 °C for 1.8 ks. By annealing under appropriate conditions, the UFG structures composed of UFG ferrite with mean grain size of 100 nm and nano-carbide dispersing within the UFG ferrite matrix uniformly can be formed [7,8]. This is a simple but novel process to obtain multi-phased UFG structure in carbon steels [7].

Microstructures of the obtained UFG steels were observed on the sections perpendicular to the transverse direction (TD) of the sheets by means of scanning electron microscopy (SEM), electron back-scattering pattern analysis in a field-emission type SEM (FE-SEM/EBSP) and transmission electron microscopy (TEM). Mechanical properties of the specimens were measured in tensile test at room temperature and initial strain rate of 8.4 x 10^{-4} s^{-1}. Tensile specimen with gage width of 5 mm and gage length of 10 mm (1/5 miniature of JIS-5 specimen) was used for the tensile test. An extensometer was attached to the specimen in the tensile test, in order to measure exact displacement.

2 Mechanical Properties of UFG Steel with Ferrite Single Phase
Figure 2 shows the stress-strain curves of the IF steel ARB processed by various cycles. The strength of the sheet greatly increases by just 1 cycle of the ARB, while the tensile elongation significantly decreases. The strength rises up with increasing the ARB cycle and the tensile strength reaches to 900 MPa after 7 ARB cycles which is 3.2 times higher than that of the starting sheet (280 MPa). On the other hand, the elongation of the sheets shows nearly constant value after 1 ARB cycle. Especially, the uniform elongation is limited within a few %. These are typical mechanical properties of the SPD materials having single phase [6,9,10].
A TEM microstructure of the IF steel ARB processed by 6 cycles is shown in Fig.3. The ARB processed materials typically reveal the UFG microstructure elongated along the rolling direction (RD) of the sheets. The mean grain thickness in Fig.3 was 210 nm. It has been already confirmed that the elongated UFGs are mostly surrounded by high-angle grain boundaries and the ARB processed sheets are uniformly filled with the elongated UFGs throughout thickness [11-14]. The material having such a microstructure performs very high strength but limited uniform elongation (Fig.2).

When the ARB processed IF steel is annealed at various temperatures, a continuous change of the microstructures happens, which is called continuous recrystallization [5,15,16]. Figure 4 shows TEM microstructures of the IF steel ARB processed by 5 cycles and then annealed at various temperatures for 1.8 ks. Through these processes, the bulky sheets of IF steel having various mean grain sizes ranging from 0.2 µm to 20 µm can be obtained, which are useful to test the mechanical properties of the UFG materials systematically.
Stress-strain curves of the IF steel with various mean grain sizes, which were fabricated by the ARB and annealing process, are shown in Fig.5. The stress-strain behaviors changes as the mean grain size decreases. It is obvious that the flow stress, especially the yield stress, significantly increases with decreasing the grain size. On the other hand, the elongation, especially the uniform elongation, suddenly drops when the grain size becomes smaller than 1 µm. Uniform elongation in tensile test is determined as the strain at which macroscopic necking initiates. Macroscopic necking is usually explained by plastic instability. Equation (1) indicates the well-known Considère criterion for plastic instability.

\[
\sigma \geq \left( \frac{d\sigma}{de} \right)
\]  

(1)

According to this equation, necking initiates when the flow stress (left-hand term) becomes equal to the strain-hardening rate (right-hand term). The plastic instability is verified in the ultrafine grained IF steel with various mean grain sizes in Fig.6. In Fig.6, the dotted lines are the true stress-strain curves while the solid lines strain-hardening rate obtained by differentiating each stress-strain curve. The point at which two curves meet, i.e., the plastic instability point, coincided well with the uniform elongation of these specimens actually measured. That is, the limited uniform elongation in the UFG ferritic steel is understood in terms of plastic instability. As is shown in the stress-strain curves (Fig.5), grain refinement significantly increases the yield strength of the material. On the other hand, strain-hardening is not enhanced by grain refinement, but it rather decreases with decreasing the grain size. As a result, plastic instability happens at very early stage of tensile test in the UFG ferritic material, resulting in limited uniform elongation. This is an inevitable nature of the UFG materials having single phase.

### 3 Mechanical Properties of Multi-Phased UFG Steel

Understanding on the basis of plastic instability also tells us how to improve the ductility of the UFG materials. If the strain-hardening (right-hand term in eq.(1)) is enhanced by some means, it is expected that the plastic instability is delayed even in the UFG material. One of the ways to enhance strain-hardening is to disperse fine second phase(s) within the UFG ferrite matrix uniformly. Figure 7 is a TEM microstructure of the low-C steel (SS400) processed in the latter thermomechanical treatment (Martensite-method) [7]. The starting sheet having as-quenched martensite structure was
conventionally cold-rolled by 50% and then annealed at 500°C for 1.8 ks. The specimen shows a kind of UFG structure. It is noteworthy that a number of carbides with nano-meter sizes uniformly precipitate within the UFG ferrite matrix. This is because the as-quenched martensite was a supersaturated solid solution of carbon. Consequently, the structure resulted from the Martensite-method is a multi-phased UFG structure composed of nearly equiaxed UFG ferrite having mean grain size of 100 nm and nano-carbides uniformly disperse within the UFG ferrite.

The multi-phased UFG steel fabricated by the Martensite-method actually managed both high strength and adequate ductility. Stress-strain curves of the low-C steel Martensite-processed are shown in Fig.8. Strain-hardening is clearly enhanced and the specimen annealed at 550°C showed tensile strength of 850 MPa, uniform elongation of 10% and total elongation of 10% at the same time. The standard tensile strength of this material is 400 MPa. The result clearly shows that dispersing fine precipitates is effective to manage both strength and ductility in the UFG steels.

4 Summary

Mechanical properties of UFG steels were shown based on the author’s original experimental data. UFG steels perform very high strength but limited uniform tensile ductility when they have single phase structure. The limited uniform elongation was understood in terms of early plastic instability, which is an inevitable feature of the UFG single-phased materials. It was also indicated at the same time that dispersing nano-carbides within the UFG ferrite matrix is an effective way to improve the uniform elongation of the UFG steels. Steels have various options of multi-phased structure, such as, ferrite + cementite, ferrite + martensite, ferrite + bainite, and so on. It was clearly shown in the present paper that the studies of the UFG steels should be focused on “multi-phase” in future.

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