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Observation of simultaneous increase in strength and ductility by grain refinement in a Fe-34.5Mn-0.04C steel

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Abstract. Fine grained Fe-34.5Mn-0.04C steel samples with fully recrystallized grain sizes of 3.8 to 2.0 µm were prepared by cold rolling followed by annealing at temperatures of either 650 °C or 800 °C. It is found that a simultaneous increase in both strength and ductility can be obtained by grain refinement, leading to an observation that the best combination of strength and ductility occurs in the sample with the finest recrystallized grain size.

1. Introduction

Grain refinement is an important microstructural design approach for strengthening of metals. However, grain refinement induced strengthening is normally associated with decrease in tensile ductility due to reduced work hardening capability. It has been reported, for example, that an abrupt decrease in tensile uniform elongation occurs in both Al and interstitial free steel as the grain size is reduced down to about 1 µm [1]. However, recent studies on high Mn austenitic TWIP steels have shown that the reduction in tensile uniform elongation is less sensitive to grain refinement, and that rather larger tensile uniform elongation is still achievable in fine grained samples with grain sizes in from a few micrometers to the sub-micrometer range [2-5]. For example, a high uniform elongation of 48 % was observed for a Fe-31Mn-3Al-3Si steel sample with a grain size of 1.8 µm [2]. In such a fine grain size deformation twinning was significantly suppressed, leading to a suggestion that the high uniform elongation is related to limited dynamic recovery due to the low stacking fault energy, which enhances work hardening. More interestingly, a simultaneous increase in both yield strength and uniform elongation was observed by Koyama et al. [6] in a binary Fe-33Mn austenitic alloy as the grain size was reduced from 3.8 µm to 1.2 µm. Under these conditions in this alloy the yield strength was increased from 200 MPa to 420 MPa, while the uniform elongation was increased from 35% to 45%. The stacking fault energy for this steel was about 50 mJ/m², which is higher than the upper limit of 30-45 mJ/m² for the occurrence of deformation twinning [7,8]. No deformation twins or deformation-induced martensite were observed in the tensile deformed ultrafine grains. Instead
numerous stacking faults were found, which were considered to be responsible for the observed enhancement in the uniform elongation [6].

In a recent study [9], a fine grained (3.8 µm grain size) Fe-34.5Mn-0.04C steel was produced that showed excellent work hardening and tensile ductility over a wide range of temperature from room temperature down to -180 °C. This steel showed a yield strength of 274 MPa and a uniform elongation of 45%, which are comparable to, and in even slightly higher than, the corresponding values reported for the Fe-33Mn alloy in [6]. Similarly no deformation twinning or martensite formation were observed in the tensile-deformed sample, and the deformation was dominated by dislocation mechanisms. This raises a question as to whether a simultaneous increase in both strength and uniform elongation can be obtained in the Fe-34Mn-0.04C alloy if the grain size is refined to below 3.8 µm. In this study, several samples with grain sizes in the range 3.8-2.0 µm were produced by cold-rolling followed by controlled annealing, and tested. The results show a clear improvement in both strength and tensile ductility with reduction in grain size.

2. Experimental

The steel used in this study had a nominal composition of Fe-34.5Mn-0.04C. An ingot was produced from a vacuum induction furnace, and subsequently forged in the temperature range of 800–1100°C to form a 13 mm thick plate. The plate was consequently cold rolled to a thickness reduction of 90 % using a laboratory rolling mill with a roll diameter of 230 mm. No edge cracking occurred during the course of cold rolling.

It was established in a previous study [9] that after 90 % cold rolling an annealing treatment at 800°C for 1 hour generates a recrystallized grain size of about 3.8 µm. The same heat treatment was applied in this study to prepare a similar grain size. To produce finer grain sizes, the 90 % cold rolled sheet was subjected to annealing treatments at a lower temperature of 650˚C for either 5, 10, or 30 minutes.

Tensile specimens with gauge dimensions of 10 mm in length, 5 mm in width and 1.3 mm in thickness were machined from the rolled and annealed sheet such that the tensile direction was parallel to the rolling direction (RD). Note that the dimensions of the tensile specimen are different from those used for the low temperature tensile testing in the previous study [9], in particular the cross section area of 5 × 1.3 mm² used in this study is much smaller than the value of 12.5 × 1.3 mm² used in [9]. Tensile tests were carried out at room temperature at a strain rate of 10⁻³ s⁻¹.

Microstructural characterization was performed by means of optical microscopy, scanning electron microscopy (SEM) coupled with electron backscatter diffraction (EBSD), and transmission electron microscopy (TEM). Thin foil specimens for the TEM observations were prepared by twin-jet electro-polishing in a solution of 100 ml HClO₄ + 900 ml CH₃COOH at -15°C. All microstructural observations were carried out on the longitudinal section containing the RD and normal direction (ND).

3. Results and discussion

3.1. Microstructure

Figure 1 shows the appearance in the optical microscope and in the TEM of the 90% cold rolled sample. A banded structure, with the band direction parallel to the rolling direction is seen from the optical image (figure 1a). The band widths vary from about 1 µm to about 10 µm. Some of the bands show somewhat wavy features that are associated with localized shear deformation. TEM observation (figure 1b) shows the formation of a fine scale deformed lamellar structure, with the lamellar boundaries approximately parallel to the RD. The spacings between the lamellar boundaries (figure 1b) are much finer than the band widths seen on the optical image (figure 1a). Measurements of the lamellar boundary spacings along the ND showed a spread from a few nanometers to about 150 nm, with an average value of about 40 nm. In addition to the deformed lamellar structure, nanotwin
bundles and localized shear bands were also observed. The nanotwin bundles had an area fraction of about 18%. Such fine scale heterogeneities in the deformed microstructure may have an effect on the local variation of stored energy, and therefore on the nucleation of recrystallization. However they showed little effect on the homogeneity of the obtained recrystallized microstructure over large areas.

Figure 1. (a) Optical image and (b) TEM image showing the deformed microstructure of the Fe-34.5Mn-0.04C steel cold rolled to 90% thickness reduction.

The 90% cold-rolled sample was annealed under four different conditions, as described above, to produce four different grain sizes. Figure 2a shows an EBSD orientation map obtained from the sample annealed at 650°C for 5 minutes. It is seen that the grain structure is rather uniform, although there remains about 2% of unrecrystallized deformed material (indicated by DS in figure 2a). All the other three samples, which were annealed at 650°C for 10 minutes, 650°C for 30 minutes and 800°C for 60 minutes, were fully recrystallized. An example EBSD image obtained from the sample annealed at 650°C for 10 minutes is shown in figure 2b to illustrate the formation of a uniform recrystallized grain structure. The mean grain sizes were measured from the EBSD maps for the four annealed samples and the results are given in table 1. The same grain size of 3.8 µm as obtained in the previous study [9] was reproduced after annealing at 800°C for 60 minutes. Annealing at 650°C for 5, 10 and 30 minutes produced three finer, but nevertheless similar, grain sizes of 2.0, 2.1 and 2.2 µm (table 1). Considering the presence of 2% unrecrystallized microstructure after annealing at 650°C for 5 minutes and the similar recrystallized grain sizes after annealing at 650°C for up to 30 minutes, it seems that a grain size of 2.0 µm is the minimum grain size that can be obtained from annealing of the 90% cold rolled Fe-34.5Mn-0.04C steel.

3.2. Tensile behaviour
Figure 3 shows engineering stress-strain curves for the four samples with different grain sizes. Grain refinement from 3.8 µm to about 2.0 µm resulted in two striking changes. Firstly, a simultaneous increase in both strength and tensile ductility is seen, and secondly, a transition from continuous yielding in the 3.8 µm grain sized sample to a discontinuous yielding in the three finer grain sized samples takes place. These observations are similar to those reported for the Fe-33Mn alloy as the grain size is refined from 3.8 µm to 1.2 µm [6]. Note that the change in the yielding behavior is associated with a significant increase in the yield strength, e.g. from 256 MPa to 405 MPa corresponding to a grain size change from 3.8 to 2.2 µm. Also note that the lower yield strength is very sensitive to the grain size change. A decease of grain size from 2.2 µm to 2.0 µm leads to an increase of the lower yield strength from 405 MPa to 466 MPa. It should be pointed out that the
occurrence of discontinuous yielding is in general undesirable for practical applications of the steel. To remove the discontinuous yielding, an annealed sample with a 2.0 µm grain size was further deformed by a slight cold rolling (about 2% thickness reduction) and tensile tested. It was found that not only did this result in the flow behavior becoming continuous, but also a further increase in the yield strength, indicating a possibility to further improve the mechanical properties of the steel.

**Figure 2.** EBSD maps showing the microstructure of the Fe-34.5Mn-0.04C steel cold rolled to 90% and then annealed at 650°C for (a) 5 minutes and (b) 10 minutes.

**Figure 3.** Tensile stress-strain curves for samples with different recrystallized grain sizes.
Table 1. Mean grain sizes and tensile properties of the four samples investigated.

| Sample          | Mean grain size (µm) | σ (Yield strength) (MPa) | σ (0.2%) (MPa) | UTS (MPa) | Uniform elongation (%) | Total Elongation (%) |
|-----------------|----------------------|--------------------------|----------------|-----------|------------------------|----------------------|
| 650°C, 5 min    | 2.0                  | 466                      | 640            | 32.6      | 39.6                   |                      |
| 650°C, 10 min   | 2.1                  | 444                      | 635            | 35.6      | 39.4                   |                      |
| 650°C, 30 min   | 2.2                  | 405                      | 620            | 37.5      | 38.8                   |                      |
| 800°C, 60 min   | 3.8                  | 256                      | 550            | 32.5      | 34.1                   |                      |

4. Summary
Fe-34.5Mn-0.04C steel samples with fully recrystallized fine grain sizes of 3.8 to 2.0 µm were prepared by cold rolling followed by an annealing treatment either at 650 °C or 800 °C. Investigation of the mechanical properties through tensile testing revealed a simultaneous increase in both strength and ductility occurred with grain refinement in the present steel. Based on the experimental observations it was concluded that the sample with the finest recrystallized grain size exhibits the best combination of strength and ductility.

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