Role of Pre-deformation in Age Hardening of a Niobium-microalloyed Steel


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The effect of pre-deformation on the age hardening of a niobium-microalloyed steel is investigated by virtue of mechanical testing in conjunction with microstructural observations. The steel is aged at three temperatures 600°C, 640°C and 680°C, respectively, after solution-treated at 1 200°C. The precipitation of Nb(C, N) is markedly accelerated by a pre-deformation of 15% prior to ageing, resulting in very fine particles being formed and a considerable increase in the strength of the steel with no large decrease in the ductility. Compared with the hot-rolled state, the yield strength of the deformed steel increases by about 280 MPa while that of the undeformed steel only by about 60 MPa after peak-aged at 640°C. Overall, the yield strength of the deformed steel may reach about 710 MPa with an elongation of about 19%.

KEY WORDS: niobium; microalloyed steels; precipitation; dispersion strengthening; mechanical properties.

1. Introduction

In microalloyed steels, the strengthening effect of microalloying additions is produced by the dispersion strengthening effect of fine carbonitride particles or by grain refinement, i.e., restraining of grain growth by the particles, or by a combination of these two effects. In order to hold a fine austenite microstructure, particles remaining undissolved in the austenite or particles precipitated during hot working are required. To produce very fine particles that are responsible for the dispersion strengthening, these particles must be freshly precipitated during or after transformation to ferrite. To achieve the desired metallurgical states, a detailed knowledge of the solubilities of microalloy carbides and nitrides are required along with a knowledge of their precipitation behaviour. Normally, TiN is extremely stable and can maintain without dissolution at high temperatures during reheating prior to rolling or during welding. Vanadium carbide has quite a high solubility in austenite even at temperatures as low as 1 050°C. Compared with vanadium carbide, niobium nitride and carbide have somewhat lower solubilities in austenite.

The combination of deformation and heat treatment is an important method of strengthening and toughening of microalloyed steels. For example, controlled rolling and cooling of microalloyed steels can refine the ferrite grain size, make the carbonitride precipitate dispersely and increase the amount of precipitation so as to raise the strength of the steels. It is seen from the Orowan mechanism of precipitation strengthening that if the hardening particles can be produced in a nanoscale rather than in a microscale with a large volume fraction, the strengthening effect will be substantially enhanced. This should therefore be a way to develop high strength microalloyed steels.

Nb(C, N) is stable at low temperatures in austenite but dissolves at higher temperatures, for example, during reheating before rolling. Nb(C, N) may precipitate in austenite under hot deformation (strain-induced precipitation) and the particles produced may resist grain growth and even dynamic recrystallization of the austenite. It is anticipated that pre-deformation prior to ageing after solution treatment can produce excess precipitation sites, such as dislocation cell walls and excess vacancies, in addition to some distortion energy, and thus promote phase transformation during ageing of a steel. As a result, the second-phase precipitation occurs rapidly with the particle size remaining extremely small, leading to a great increase in the strength of the steel without an apparent decrease in the ductility. It was the aim of the present work to investigate the effect of pre-deformation on the age hardening of a niobium-microalloyed steel.

2. Experimental Procedure

The experimental material was prepared by vacuum induction melting. The resulting ingot (50 kg) was hot rolled into plates 1 600 mm×320 mm×8 mm in size and then cut into samples 160 mm×32 mm×8 mm in size. The chemical composition of the steel is (wt%): 0.050C, 0.49Mn, 0.15Si, 0.123Nb, 0.011N, 0.0056S, and 0.007P with Fe in balance. All the samples were solution-treated at 1 200°C for 20 min and then quenched with an aqueous solution containing 10% NaCl. According to the solubility product, it may be estimated that at 1 200°C the solubility in austenite is about 0.11 wt% for Nb and 0.0095 wt% for N. After the solution treatment, some samples were cold rolled with a thickness reduction of 15%. The samples treated above were ma-
chined into smaller specimens 16 mm×16 mm×5 mm in size and aged for different periods of time at 600°C, 640°C and 680°C, respectively, in a salt-bath furnace and then cooled with the brine for use in different examinations.

The specimens were polished with the standard method in metallography for use in hardness measurements and optical microscopy observations. The Vickers hardness of the specimens was measured using an HV-50 hardness test machine with a load of 5 kg in order to determine the ageing kinetics. In the measurements, three readings were made for each condition and their mean value was taken as the measured result. For each ageing temperature, the specimen with the maximum hardness was examined using optical microscopy where the polished specimen was etched by a solution consisting of 4% nitric acid and 96% ethanol. Some specimens were also examined using transmission electron microscopy (TEM) equipped with an energy dispersive X-ray microanalysis system. The TEM thin foils were prepared by the standard dual-jet electropolishing technique with a solution consisting of 5% perchloric acid and 95% methanol. For tensile testing, the sheet-shaped specimens with a gauge length of 70 mm in terms of the standard relation $L = \frac{11.3\sqrt{A}}{t}$ ($L$ is the gauge length and $A$ is the cross section area) were machined and tested using a universal testing machine. In the tensile testing, three specimens were used and the mean value of data points obtained along with the standard deviation was taken as the result.

### 3. Results and Discussion

The ageing kinetics of the undeformed and deformed specimens is shown in Fig. 1. In the figure, the harness and ageing time corresponding to the peak are indicated for each curve. Clearly, the hardness initially increases with increasing ageing time and reaches a peak value at a certain time. After that, the hardness decreases with further increasing ageing time. Over the three ageing temperatures considered, 680°C is the most favourable as the ageing can obtain the largest peak hardness at this temperature, especially for the pre-deformed specimens.

During ageing, the hardness peak of an age hardening steel generally indicates the size of hardening particles (second-phase) reaching a critical size with a sufficient quantity. When the particle size is very small, the particles are coherent with the matrix and dislocations can cut through them. In this scenario, the strength of a precipitation hardening steel increases with increasing particle size. With increasing particle size, the particles gradually change to be, to some extent, incoherent with the matrix and therefore it is getting difficult for the dislocations to move by the cutting mechanism. After the particles grow to a certain size during ageing, the dislocations can no longer cut through the particles and their movement will be based on the Orowan mechanism. As a result, the strengthening mechanism would change to the Orowan mechanism (usually the particle is incoherent with the matrix) from the cutting mechanism (usually the particle is coherent with the matrix). Thus, this particle size is the above-mentioned critical size. According to the Orowan mechanism, the strengthening effect decreases with further increasing particle size. Therefore, the peak ageing time is the favorable ageing time for the age hardening steel. It is seen from Fig. 1 that both ageing temperature and pre-deformation have a great influence on the peak ageing time. When the ageing temperature is lowered to 640°C from 680°C, the peak ageing time for the undeformed specimens is increased by more than two orders of magnitude, while only by one order of magnitude for the deformed specimens. The ageing can attain the peak within much shorter time for the deformed specimens than for the undeformed ones. All the above demonstrates that the pre-deformation can considerably facilitate the precipitation of second-phase. As described in the introduction section, the above effect should mainly arise from the pre-deformation-induced excess precipitation sites, such as dislocation cell walls and excess vacancies, which can promote the second-phase precipitation. This effect is particularly evident at low ageing temperatures where the thermal activation for precipitation is weak so that the excess precipitation sites created by the deformation can play a leading role. For instance, at 640°C, it just needs 5 min for the ageing to reach the peak for the deformed specimens but this process needs 2 h for the undeformed specimens. Since the peak-ageing time is too short at 680°C (15 s for the deformed specimens and 50 s for the undeformed one), it is difficult to control the material heat treatment in engineering practice. Also, since the hardening effect is too small for the specimens aged at 600°C, it is not practically significant to use this ageing temperature in engineering applications. For these reasons, we just examined the mechanical properties of the specimens peak-aged at 640°C, which are shown in Fig. 2. The values for the hot-rolled specimens are also plotted for comparison. The yield strength of the undeformed specimen is about 60 MPa higher than that of the hot rolled one and the elongation is reduced by about 5%. However, the yield strength of the deformed specimen

![Fig. 1. Typical ageing kinetic curves of (a) undeformed and (b) deformed specimens.](image)
is about 280 MPa higher than that of the hot rolled one and the elongation is only reduced by about 8%. Overall, the yield strength of the deformed specimen may reach about 710 MPa with an elongation of about 19%. As with the yield strength, the ultimate tensile strength of the deformed specimen is also far higher than that of the undeformed one. Therefore, the strength of the steel is considerably enhanced by the pre-deformation.

Figure 3 shows typical transmission electron micrographs for the undeformed and deformed specimens that were peak-aged at 640°C. Evidently, the particles in the deformed specimen are much finer and more dispersed with a more amount than those in the undeformed one. Clearly, most of the particles in the deformed specimen are smaller than 40 nm. The EDX analysis on the particle only shows Nb, N and C signals (see Fig. 4), demonstrating that the precipitate is mainly Nb(C, N). Of course, contamination may partially contribute to the C signal, but this does not exclude a qualitative indication of C involved in the precipitate. It should be these fine Nb(C, N) particles that are mainly responsible for the above differences in ageing kinetics and mechanical properties between the deformed and undeformed specimens. Of note is that with the TEM used in this work it is difficult to observe very small particles due to its limited spatial resolution. Consequently, there could be many very small particles being not exhibited in the TEM micrographs.

Figure 5 shows typical optical micrographs for the undeformed and deformed specimens that were peak-aged at 640°C. Clearly, some recrystallization has occurred in the undeformed specimens with some granular ferrite being formed. Nevertheless, the microstructure in the deformed specimens is still the tempered sorbite maintaining the orientation of lath martensite. As a consequence, the structure strengthening may also have some contribution to the
strength increase in the deformed specimens. However, this type of strengthening would not be strong because there is only some small difference in matrix microstructure between the undeformed and deformed specimens (see Fig. 5). Of course, some dislocation strengthening may also be involved because the pre-deformation may create some excess dislocations although the amount of deformation is small.

It is worth mentioning that compared with the undeformed specimens the elongation of the deformed specimens is only reduced by about 3% when peak-aged at 640°C but the yield strength is increased by about 220 MPa, showing that the precipitation enhancement by pre-deformation can dramatically increase the strength without damaging the ductility apparently. This implies that the toughness of the steel is also improved by the pre-deformation as it depends on both the strength and the ductility.

4. Conclusions

The effect of pre-deformation on the age hardening of a niobium-microalloyed steel has been studied. During ageing at different temperatures after solution treatment at 1200°C, the precipitation of Nb(C, N) is enhanced substantially by a pre-deformation of 15% prior to ageing, leading to very fine particles being formed. Compared to the hot-rolled state, the yield strength of the pre-deformed steel may increase by about 280 MPa with no large decrease in ductility after peak-aged at 640°C. Overall, the yield strength of the deformed specimen may reach about 710 MPa with an elongation of about 19%. The dispersion strengthening is mainly responsible for the strength increase.

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REFERENCES