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## **Materials**

### Microstructural evolution and tensile properties of low-carbon steel with martensitic microstructure during warm deforming and annealing

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**Abstract:** For preparing large-scale nano-grained and ultrafine-grained steel sheets by warm rolling and annealing, the effects of deforming temperature on both the flow stress and the microstructure evolution of 09MnNiD steel with lath martensitic microstructure were studied by warm-compression test and transmission electron microscopy (TEM) observation. Thereafter, the steel with the lath martensitic structure was multi-pass warm-rolled and then annealed. TEM results indicate that nano-grained and ultrafine-grained steel sheets are formed by warm rolling at 400°C and annealing at 400-600°C. In comparison with the as-warm-rolled specimen, the tensile strength at room temperature changes a little when the rolled samples are annealed below 450°C, and the tensile strength is greatly lowered as the annealing temperature increases to above 550°C.

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Key words: warm deformation; martensite; flow stress; nanomaterials; mechanical properties

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#### 1. Introduction

Severe plastic deformation (SPD) has gained worldwide interest because of its possibility of directly converting conventional metals to ultra-fine grained and nano-grained bulk materials [1]. Materials with ultra-fine grains, especially nanograins, possessing promising superior mechanical properties, such as high strength and high toughness, have potentially important applications. Recent researches show that equal channel angular press (ECAP) [1-4], high pressure torsion (HPT) [1, 5], repetitive corrugation and straightening (RCS) [6], accumulative roll-bonding (ARB) [7], and cold rolling of lath martensite steel [8-12] can be used for fabricating the ultra-fine and nano-grained materials. However, above-mentioned methods all encounter difficulties for use in the practical industrial production of large-sized structural steel.

Although severe cold rolling and low-temperature annealing have been proved to be feasible and efficient for large-scale industrial production, only a few researches were reported on the fabrication of ultra-fine grained and nano-grained steel sheets by cold rolling and low-temperature recrystallization of lath martensite [8-11]. The main reason is that the cold rolling lath martensite experiences high resistance for plastic deformation, which limits the dimensions of slabs and increase the production cost. To reduce the resistance to plastic deformation of lath martensite for large-scale bulk steel, the effects of warm rolling and annealing on the mechanical properties and microstructure of as-quenched martensite are studied. For as-quenched martensite, the rolling temperature can be increased to reduce the deformation resistance and do not deteriorate its effect of grain refinement. By multi-pass warm rolling, the rolling reduction of more than 70% can be obtained. In addition, nano-grained

or ultra-fine grained steel sheets by further low-temperature annealing are prepared. The above results will be very helpful for the practical industrial fabrication of ultra-fine or nano-grained steels.

#### 2. Experimental

Low carbon steel 09MnNiD (09 steel), normally used for pressure container, was used in this study. The chemical composition of the steel (wt%) was C 0.075, Si 0.29, Mn 1.40, Cr 0.12, Mo 0.18, Ni 0.63, P 0.016, S 0.0063, and balance Fe. To obtain fully lath-martensitic structure, the slab with the dimension of 10 mm  $\times$  100 mm  $\times$  270 mm was austenitized at 940°C for 30 min in a salt furnace and then quenched in salt-water. The flow-stress examination was carried out on a Gleeble-3500 thermo-mechanical simulator. Cylindrical specimens with the size of  $\phi$ 8 mm  $\times$  12 mm were used for the flow-stress examination. The samples were heated to each testing temperature for 5 min and compressed at a strain rate of 0.01 s<sup>-1</sup>.

Slabs were multi-pass warm-rolled at  $400^{\circ}$ C, and the final relative reduction in height is about 70%. The rolled specimens were annealed at 400, 500, and 550°C for 60 min, and 600°C for 10 and 20 min, respectively. Further, room temperature tensile test was performed on a Gleeble-3500 thermo-mechanical simulator on the warm-rolled and annealed specimens with the gauge dimension of  $2.8~\text{mm} \times 10.0~\text{mm} \times 25.0~\text{mm}$ . The microstructure of the samples was characterized by transmission electron microscopy (TEM) on a JEM-2010 microscope (JEOL) at 200 kV. Thin foils for TEM observation were prepared by twin-jet electro polishing to electron transparency.

#### 3. Results and discussion

# 3.1. Effect of deformation temperature on the flow stress and microstructure change during deformation

The true strain-true stress curves of 09 steel measured by the Gleeble-3500 thermo-mechanical simulator at three different temperatures are shown in Fig. 1.

All the three flow curves present single peak shape, which is usually caused by softening effect of dynamic recrystallization. The peak stress  $(\sigma_p)$  of lath martensite is 1367 MPa at the test temperature of 300°C. The  $\sigma_p$  decreases as the test temperature increases and the peak stresses are 1050 MPa at 400°C and 634 MPa at 500°C, respectively. Fig. 1 indicates that the increase of the deformed temperature greatly reduces the resistance of the steel with lath martensitic microstructure. This result is consistent with most results from warm deforming steel with the starting microstructure of ferrite or ferrite plus pearlite [12-13]. Nevertheless, the increase in temperature will result in the change in morphology of martensite, and deteriorate the effect of grain refinement from martensite phase transformation as well [14].

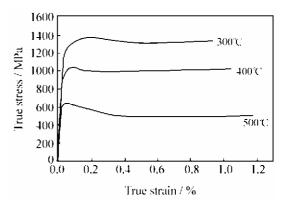
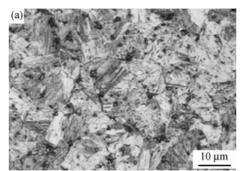


Fig. 1. True stress-true strain curves of martensite 09 steel warm compressed at 300, 400, and 500°C with a relative reduction in height of about 70%.

Fig. 2 shows the optical micrographs (OM) of the microstructures in samples before and after warm rolling at 400°C. The lath martensite microstructure was obtained in the quenched sample, as shown in Fig. 2(a). After warm rolling, deformation bands were formed in the sample (Fig. 2(b)). Microstructure comparisons illustrated that there was an obvious morphology change of lath martensite structure in the samples that underwent flow stress test at 300, 400, and 500°C, as shown in Fig. 3. Fig. 3(a) is the TEM image of the as-quenched lath martensite sample with the average lath width of about 280 nm, showing that the lath



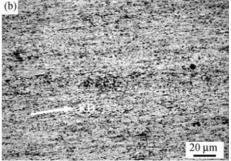


Fig. 2. Optic micrographs of samples before (a) and after (b) warm rolling at 400°C.

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