

The extent and mechanism of nanostructure formation during cold rolling and aging of lath martensite in alloy steel

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Abstract

Transmission electron microscopy was used to study microstructural evolutions during cold rolling and isothermal aging of an Fe–10Ni–7Mn (wt.%) maraging steel at 753 K. The steel shows lath martensite after homogenizing treatment which evolved by cold rolling to a typical deformed structure, consisting of lamellar dislocation boundaries and shear bands. Ultrafine-grained regions were found frequently in the isothermally aged steels. The process of ultrafine grain formation during isothermal aging is attributed to extended recovery to be correlated with precipitate coarsening reactions. At later stages of aging, recrystallization-assisted discontinuous coarsening of grain boundary precipitates was identified at lamellar boundaries, giving rise to a banded structure consisting of ferrite bands and precipitation hardened laths. It was demonstrated that partially-nanostructured multi-phase steel is fabricated by cold rolling and aging of lath martensite.

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1. Introduction

Fabrication of nanostructured metallic materials has been realized by severe plastic deformation (SPD) techniques such as equal channel angular pressing, torsion straining under high pressure, accumulative roll bonding, etc. [1]. As pointed out in an early study [2], it has been recently reported that cold rolling and annealing of lath martensite in low carbon steels specifically gives rise to ultrafine grain formation as in the SPD routes [3,4]. The ability to fabricate nanostructured steels by conventional cold rolling and annealing treatment is overwhelming.

The process of ultrafine grain formation during SPD mainly encompasses dislocation motions and dislocation cells rotation upon repetitive shear straining [1]. Although similar mechanism has been assumed for cold rolling, but the specific ability of cold rolling in producing nanostructured steels is attributed to specifications of starting microstructure before deformation, i.e., lath martensite [5]. Lath martensite in steels has a hierarchical multi-scale substructure including packets, blocks and laths

[6–8]. In fact, a preliminary austenite grain is effectively refined during martensitic transformation down to a few hundreds of nanometers for lath widths. Further, individual laths are highly dislocated in the order of heavily cold deformed metals [9]. Consequently, it is proposed that the refined substructure and high density of dislocations in lath martensite accelerates evolution of deformed structure during cold rolling. Nevertheless, details of cold-rolled lath martensite and the mechanism of ultrafine grain formation during subsequent annealing have not been well understood yet.

A unique family of high-performance steels, namely maraging steels, has been explored on the basis of iron-nickel lath martensite. Those steels show superior formability in the solution-annealed martensitic condition due to their extra-low carbon content, but acquire ultrahigh strength along with good fracture toughness after aging of lath martensite [10]. Many applications of those steels in aerospace, military and production tooling are reported frequently. In this paper, the ability of ultrafine grain formation by cold rolling and aging treatment is examined for an Fe–Ni–Mn maraging steel. The steel shows typical lath martensite and it is aimed to understand the extent and mechanism of nanostructure formation during cold rolling and isothermal aging treatment.

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2. Experimental procedure

A vacuum induction melted and vacuum arc remelted Fe–10.35Ni–6.88Mn (wt.%) maraging steel was encapsulated in a quartz tube purged with argon after evacuation to 10^{-5} Torr. Homogenizing was carried out at 1473 K for 172.8 ks followed by water quenching and cryogenic treating at 77 K for 3.6 ks. Cold rolling to 85% thickness reduction was carried out at room temperature followed by isothermal aging treatment at 753 K in a neutralized salt bath. Disc-shaped specimens of diameter of 3 mm and initial thickness of 300 μm were cut using an electro discharge wire cutting machine and mechanically polished to a thickness of ca. 30 μm . Further thinning was performed in a solution of CrO_3 (200 g), CH_3COOH (500 ml) and H_2O (40 ml) held at 285 K with a twin jet TENOPUL-3 electropolishing machine. Transmission electron microscopy was carried out in a PHILIPS CM200-FEG microscope operating at 200 kV. All observations were made on TD planes of the rolled strips.

3. Results

An optical micrograph of a homogenized steel is shown in Fig. 1a, demonstrating packets and blocks formed within a prior

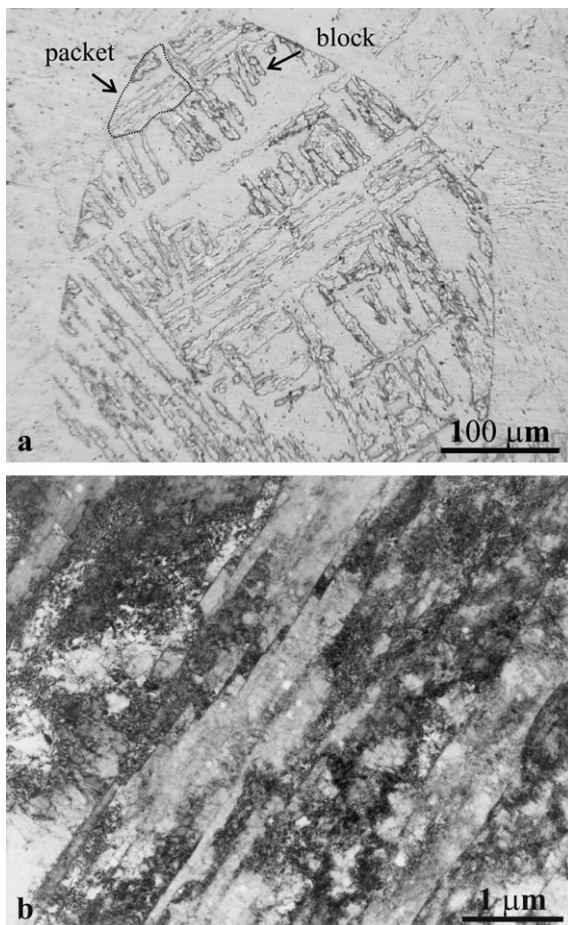


Fig. 1. (a) Optical micrograph showing substructure of lath martensite in the studied alloy and (b) bright-field transmission electron micrograph showing narrow laths of high dislocation density.

austenite grain. A bright-field transmission electron micrograph of a set of parallel laths formed within a block is shown in Fig. 1b which indicates narrow width and high dislocation density of individual laths. This is the starting microstructure to be subsequently cold-rolled for 85% reduction. Fig. 2 shows bright-field transmission electron micrographs of a cold-rolled steel along with steels cold-rolled and aged for 0.36, 3.6 and 86.4 ks at 753 K. In the cold-rolled steel (Fig. 2a), a typical deformed structure consisting of lamellar dislocation boundaries and shear bands is identified. After aging for 0.36 ks (Fig. 2b), a microstructure consisting of ultrafine grains and elongated cells is observed. Fig. 2c shows microstructure of a steel aged for 3.6 ks after cold rolling. Two distinct regions are present; (i) a grain-refined region in the lower part of the micrograph and (ii) a lamellar region, in the upper part of the micrograph, composed of precipitation hardened laths and ferrite bands within which coarse precipitates have been embedded. After aging for 86.4 ks (Fig. 2d), a multi-phase structure consisting of equiaxed ferrite grains, coarse precipitates and overaged laths has been realized. High-magnification transmission electron micrographs of the aforementioned steels are shown in Fig. 3. Elongated and equiaxed dislocation cell blocks are found in the cold-rolled steel. However, colonies of equiaxed ferrite grains with a size of about 100 nm (encircled) are found in the aged steels. Selected-area electron diffraction patterns (SADPs) of a cold-rolled steel and a steel cold-rolled and aged for 0.36 ks are shown in Fig. 4. The SADPs were made using large and small area-selecting apertures in order to qualitatively evaluate local and long range misorientations. Ring-like patterns are found for both of the cold-rolled and aged steels using large apertures (Fig. 4a and c). However, it is identified that the circumferences of individual rings become more completed in the aged steel which may indicate long range misorientations of the aged steel. Using small apertures, SADP of the cold-rolled steel (Fig. 4b) shows mainly $\langle 111 \rangle_{\text{bcc}}$ patterns rotated for a few degrees, but a rather complicated SADP (Fig. 4d) is observed for aged steel which may arise from a few randomly-oriented grains.

4. Discussion

It is found out that highly-alloyed lath martensite in an Fe–Ni–Mn steel evolves to a typical deformed structure by heavy cold rolling. The deformed structure shows lamellar dislocation boundaries and shear bands as found, for example, in an interstitial-free steel cold-rolled for 90% reduction [11]. In a typical deformed structure two types of boundaries are distinguished, i.e.: (i) geometrically-necessary dislocation or lamellar dislocation boundaries which are usually high angle boundaries and (ii) incidental dislocation or dislocation cell boundaries which are mainly low angle boundaries [12,13]. SADPs obtained using a small aperture from the present deformed structure is similar to that observed in metals deformed for one or two passes by equal channel angular pressing [14]. Therefore, more deformation would be necessary to evolve present structure to be comparable with conventional SPD metals which are deformed at least for four passes by equal chan-

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