Solid State Phenomena Vol. 114 (2006) pp 159-164
Online available since 2006/Jul/15 at www.scientific.net
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doi:10.4028/www.scientific.net/SSP.114.159

Evolution of Precipitate Coarsening Reaction In a Nanostructured Fe-Ni-Mn Maraging Alloy

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Keywords: Maraging: Precipitation: Coarsening: Nanostructure: Cold Rolling

Abstract. Fe-Ni-Mn maraging alloys show discontinuous coarsening of fct θ-NiMn precipitates along prior austenite grain boundaries (PAGBs) during isothermal aging. Heavy cold rolling of a solution annealed Fe-10Ni-7Mn (wt. %) maraging alloy and subsequent aging treatment at 773 K led to the formation of a nanostructured material. Hardness measurement and transmission electron microscopy were used to study the aging behaviour and microstructural evolution of the nanostructured alloy. At the early stage of aging, an ultrafine grained structure was observed with fine matrix precipitates. However, at later stages of aging, severe coarsening of precipitates was found. At that stage, a lamellar microstructure composed of finely precipitated ferrite laths and ultrafine ferrite grains were identified. Coarse fct θ-NiMn precipitates were identified at the ultrafine ferritic grain boundaries. Development of the coarsening reaction was found to increase the ultrafine grained ferritic regions with prolonged aging.

Introduction

Fe-Ni-Mn maraging alloys show substantial hardening during isothermal aging at 673 – 773 K [1]. The age hardening of these alloys has been found to be due to the precipitation of nanometer-sized particles of fct θ-NiMn intermetallic compound [2-7]. Fe-Ni-Mn maraging alloys are ductile in the solution annealed condition but suffer from severe embrittlement along prior austenite grain boundaries (PAGBs) after aging [3]. The intergranular failure of these alloys has been attributed to segregation of Mn [8], reversion to austenite particles [9] and precipitation of fct θ-NiMn intermetallic compound [10] at PAGBs. Recently, Hossein Nedjad et al. [11] found discontinuous coarsening of fct θ-NiMn precipitates at the grain boundaries resulting in the formation of a nano-scaled precipitate free zone (PFZ) along PAGBs in Fe-10Ni-7Mn (wt. %) maraging alloy. Meanwhile, localized straining and ductile rupture of the nano-scaled PFZ at PAGBs was proposed as the main source of intergranular failure.

Much research to investigate possible improvements in the mechanical properties of Fe-Ni-Mn maraging alloys has been reported. It has been found that alloying with Mo and Cr improves the fracture strength, but intergranular failure has not been prevented completely [12, 13, 14]. Hossein Nedjad et al. [15] investigated the effect of heavy cold rolling and subsequent aging treatment on the microstructure and mechanical properties of Fe-10Ni-7Mn (wt. %) maraging alloy. The process was found to produce a nanostructured material having an ultrahigh strength of about 2000 MPa.

The paper reports the effect of the isothermal aging time on the microstructure of heavily cold rolled and aged Fe-10Ni-7Mn (wt. %) maraging alloy. The formation of nanostructure at early stages of aging and the coarsening of precipitates at later stages of aging are reported.

Experimental Procedure

A vacuum induction melted and vacuum arc remelted material was encapsulated in a quartz tube and purged with argon after evacuation to 10^{-5} Torr. The chemical composition of the alloy is given in Table 1.

Fe	Ni	Mn	C	S	P	N	Al
Base	10.38	6.88	0.006	0.007	0.005	0.005	0.003

Table 1. Chemical composition of the alloy studied [wt. %]

Homogenizing was carried out at 1473 K for 172.8ks, 48hrs, followed by water quenching and cryogenic treating at 77 K for 1hr. Cold rolling to 85% was at room temperature followed by isothermal aging treatment at 753 K in a neutralized salt bath. Vickers Hardness measurement were made using a load of 9.8 N. Disc-shaped specimens of diameter of 3 mm and initial thickness 300 µm were cut using an electro discharge wire cutting machine and mechanically polished to a thickness of ca. 30 µm. Further thinning was carried out in a solution of CrO_3 (200 g), CH_3COOH (500 ml) and H_2O (40 ml) held at 285 K and using a voltage of 22-25 V on a twin jet TENOPUL-3 electropolishing machine. Transmission electron microscopy was carried out in a PHILIPS CM200-FEG microscope at 200 kV. All observations were made on TD sections of the rolled pieces.

Results and Discussion

Figure 1 shows the changes in hardness of the as-rolled alloy during isothermal aging at 753 K. This alloy has a hardness of about 310 HV in the as-rolled condition which increases to a maximum of about 585 HV after aging for 0.36 ks. Prolonged aging results in decreasing hardness.

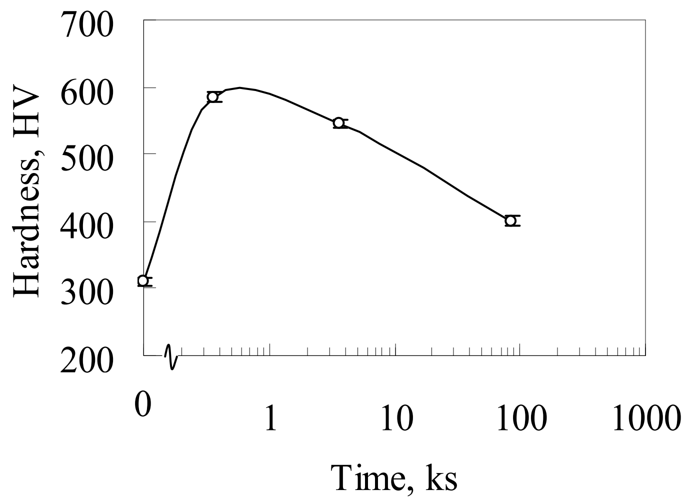

Fig. 1. Changes in hardness of the as-rolled alloy during isothermal aging at 753 K.

Figure 2 shows a bright field TEM micrograph and corresponding selected area diffraction pattern (SADP) of an as-rolled specimen. A typical deformed structure, consisting of elongated laths and shear bands, is indicated in Fig. 2 (a). The ring-like pattern in Fig. 2 (b) indicates ultrafine grain formation during heavy cold rolling.

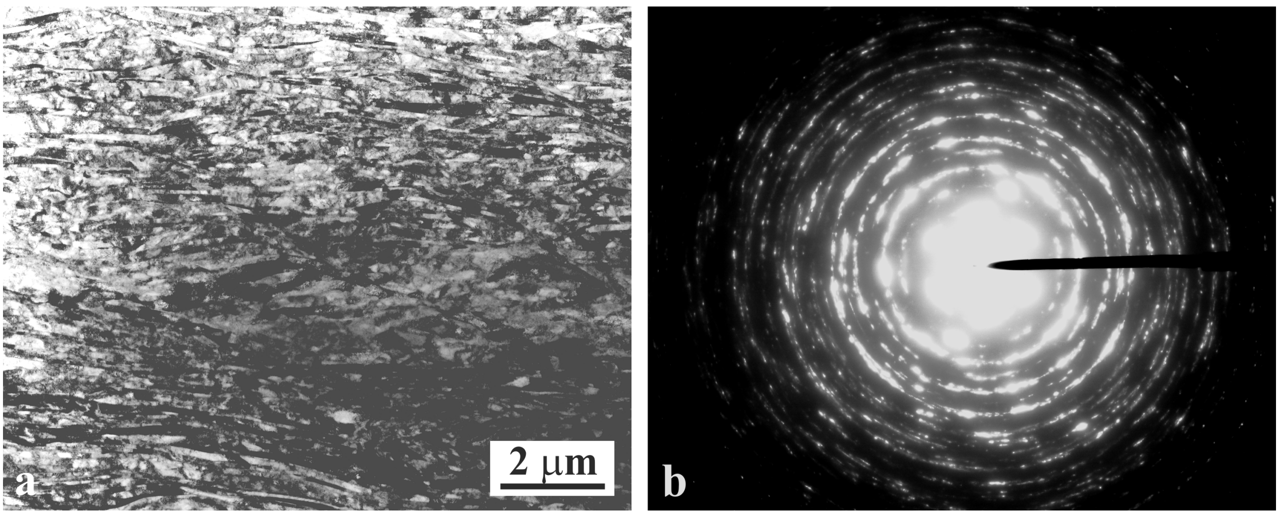

Fig. 2. (a) Bright field TEM micrograph showing deformed structure of the alloy after cold rolling for 85%; (b) SADP corresponding to (a).

Figure 3 shows a bright field TEM micrograph and corresponding SADP of a specimen aged for 0.36 ks. An ultrafine grained structure is seen in this specimen while coarse precipitates are found at grain boundaries (arrowed). A bright field TEM micrograph of a specimen aged for 3.6 ks is shown in Fig. 4 (a). An ultrafine grained area (A) is shown where the SADP obtained from an area of about 0.25 μm^2 in Fig. 4 (b) indicates large misorientations between grains in this region.

However, a ferrite band containing coarse precipitates is observed at the area B.

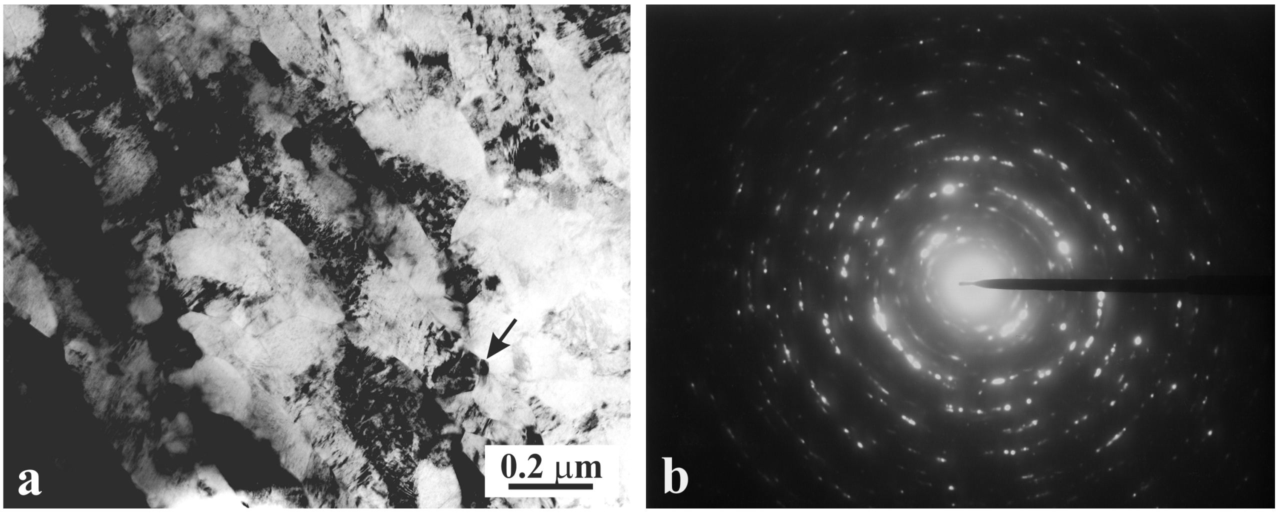

Fig. 3. (a) Bright field TEM micrograph showing ultrafine grain formation in a specimen aged for 0.36 ks; (b) SADP corresponding to (a).

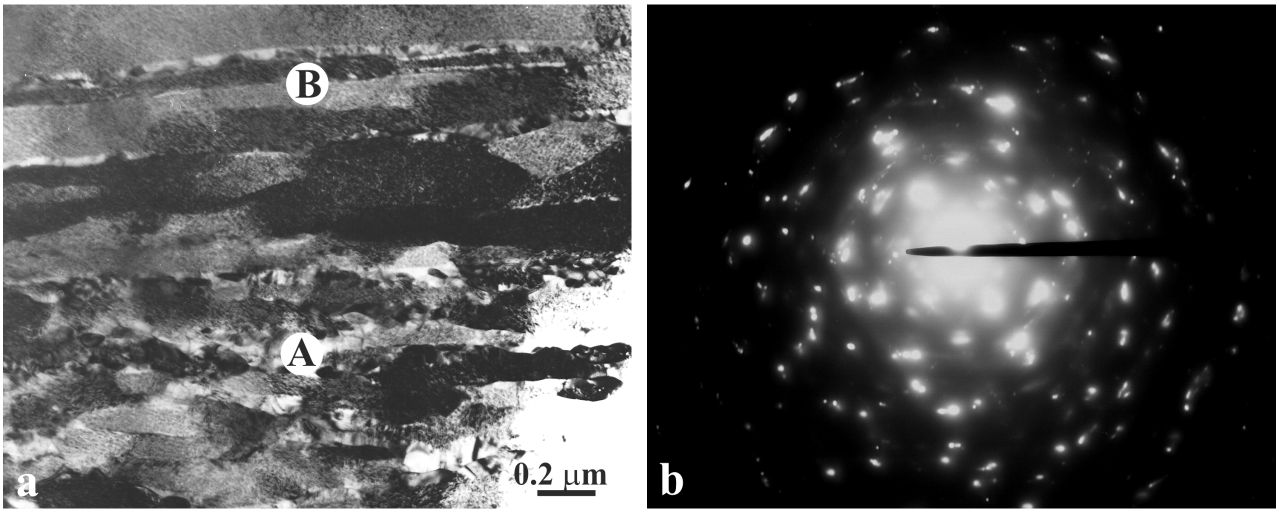

Fig. 4. (a) Bright field TEM micrograph showing microstructure of a specimen aged for 3.6 ks; (b) SADP corresponding to the area (A).

Figure 5 shows a bright field TEM micrograph of a specimen aged for 86.4 ks indicating nano-crystalline (NC) ferrite grains, elongated ferrite laths (α) and coarse precipitates (arrowed).

Investigations into the annealing behaviour of heavily deformed Fe-C alloys indicated precipitation of iron carbide (Fe_3C) particles in the ultrafine grained ferritic matrix and grain boundaries. Further, the role of iron carbide precipitates in the stabilization of nanostructures has also been denoted [16]. It was found that precipitation and coarsening of fct θ-NiMn intermetallic compound takes place during isothermal aging of Fe-10Ni-7Mn (wt. %) maraging alloy [11]. A similar role between fct θ-NiMn and iron carbide precipitates can be established in the evolution of ultrafine grained structures in heavily deformed steels. Fe-Ni-Mn maraging alloys have a lath martensitic structure in the solution annealed condition. The possibility of ultrafine grain formation in lath martensite by cold rolling and warm annealing has been reported by Tsuji et al. [17]. However, lath martensite of maraging alloys is more ductile than Fe-C martensite which indicates a possibility of a higher degree of cold deformation. Further, accelerated development of the deformed structure and consequently the feasibility of ultrafine grain formation is expected for highly alloyed martensite in maraging steels.

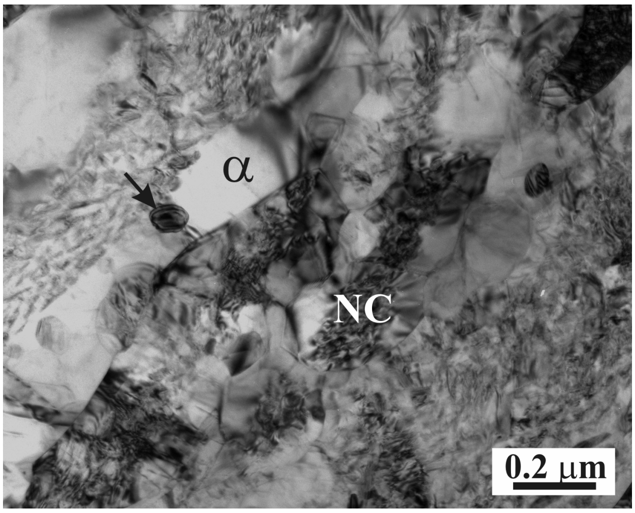

Fig. 5. Bright field TEM micrograph showing microstructure of a specimen aged for 86.4 ks. NC, α and arrow indicate nano-crystalline ferrite grains, a ferrite lath and a coarse fct θ-NiMn precipitate, respectively.

Discontinuous coarsening of fct θ –NiMn precipitates along with a nano scale precipitate free zone (PFZ) has been found to be a source of intergranular failure in Fe-10Ni-7Mn (wt. %) maraging alloy [11]. Coarsening of fct θ-NiMn precipitates was also found in the present work, but the continuity of PFZ disappeared. Therefore, the ultrahigh strength of this alloy [15] in comparison with the premature failure of conventional Fe-Ni-Mn maraging alloy, should be due to the discontinuity of the PFZ as a consequence of the nanostructure.

Considering the SADPs of the as-deformed specimen and specimen aged for 0.36 ks, it is concluded that the ultrafine grain formation in this alloy takes place during cold rolling. Further, precipitation takes place in these grains during aging. Therefore, the contribution of recovery and recrystallization during aging treatment in ultrafine grain formation in the early stages of aging is proposed as being insignificant. At later stages of aging, two types of coarsening were found, i) coarse precipitates have been embedded within ferritic band (Fig. 4: b) and ii) coarse precipitates at grain boundaries in the ultrafine grained area (Fig. 4: a). The mechanism of the coarsening reaction in type (i) is proposed as being discontinuous which could be assisted by recrystallization tendencies. In the case of second type, it is proposed that conventional coarsening takes place in the ultrafine grained area, i.e. matrix precipitates dissolve at the expense of grain boundary precipitates coarsening. It is proposed that the lattice diffusion of Ni and Mn atoms in nano-scaled grains is enhanced by a higher density of dislocations. High volume diffusivity and short diffusion distance are supposed to facilitate coarsening reactions at the aging temperature. Detailed studies on the precipitation behaviour and misorientations between NC ferrite grains will be published elsewhere.

Conclusion

The annealing behaviour of heavily cold rolled Fe-10Ni-7Mn (wt. %) maraging alloy during isothermal aging was investigated. An ultrafine grained structure was observed at the early stages of aging which is proposed as being inherited in the deformed structure. However, the formation of nanocrystalline ferrite and coarsening of fct θ-NiMn precipitates were found at later stages of aging.

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High Pressure Technology of Nanomaterials
doi:10.4028/www.scientific.net/SSP.114

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