ELSEVIER

Materials Science and Engineering A 490 (2008) 105–112

MATERIALS SCIENCE & ENGINEERING A

www.elsevier.com/locate/msea

Correlation between the intergranular brittleness and precipitation reactions during isothermal aging of an Fe–Ni–Mn maraging steel

S. Hossein Nedjad [a,*], M. Nili Ahmadabadi [b], T. Furuhara [c]

[a] *Faculty of Materials Engineering, Sahand University of Technology, P.O. Box 51335-1996, Tabriz, Iran*

[b] *School of Metallurgy and Materials Engineering, University of Tehran, P.O. Box 14395-731, Tehran, Iran*

[c] *Institute for Materials Research, Tohoku University, 2-1-1 Katahira, Aoba-ku, Sendai 980-8577, Japan*

Received 27 August 2007; received in revised form 25 December 2007; accepted 24 January 2008

Abstract

Evolution of the intergranular brittleness of an Fe–10Ni–7Mn (weight pct) maraging steel was correlated with precipitation reactions during isothermal aging at 753 K. Intergranular brittleness of the Fe–Ni–Mn steel raises after aging treatment which occurs catastrophically at zero tensile elongation in the underaged and peakaged steels. The intergranular failure is attributed to grain boundary weakening due to the formation of coarse grain boundary precipitates associated with solute-depleted precipitate-free zones during isothermal aging. Further, evidences of planar slip bands were found within the grains of a peakaged specimen loaded by tensile deformation. Those inhomogeneously deformed bands were identified to apply large strain localization in the soft precipitate-free zones at grain boundaries which is assumed to fascinate microcracks initiation at negligible macroscopic strains in the underaged and peakaged steels. During further aging, concurrent reactions including (i) overaging of matrix precipitates, (ii) spheroidization of grain boundary precipitates, (iii) growth of precipitate-free zone in width and (iv) diffusional transformation to austenite take place which increase tensile ductility after prolonged aging.

Keywords: Maraging steel; Intergranular brittleness; Grain boundary precipitation; Precipitate-free zone; Planar slip

1. Introduction

Iron–nickel–manganese martensitic steels show substantial age hardening but suffer from poor ductility after aging [1,2]. The Fe–Ni–Mn maraging steels exhibit intragranular dimpled ductile fracture in the solution-annealed condition which turns, by short isothermal aging, to premature intergranular brittle fracture, passing along prior austenite grain boundaries (PAGBs) [3]. Hereafter, the PAGBs will be refereed to as grain boundaries. Analogous to the temper-embrittlement of low alloy steels, Squires and Wilson [3] first suggested that intergranular failure of an Fe–12Ni–6Mn (weight pct) maraging steel arises from segregation of manganese at grain boundaries during isothermal aging at 573–773 K. Hereafter, all chemical compositions will be given in weight pct. Then Feng et al. [4] showed by Auger electron spectroscopy (AES) that nickel and manganese segregate at grain boundaries of an Fe–12Ni–6Mn steel during isothermal aging at 653 K. Later, Heo and Lee [5–7] reported a ductile–brittle–ductile transition in aged Fe–7Ni–8Mn and Fe–12Ni–6Mn steels which was well explained by successive manganese segregation and desegregation at grain boundaries during isothermal aging. Nevertheless, a few studies argued deleterious effect of manganese segregation on the intergranular failure of Fe–Ni–Mn steels. For instance, Suto and Murakami [8] identified that nickel and manganese concentrations at grain boundaries of an Fe–12Ni–6Mn steel never change in the ductile–brittle transition temperature and, consequently, criticized deleterious effect of manganese segregation in the intergranular failure! Alternatively, interaction of moving dislocations with disc-shaped precipitates at grain boundaries was suggested as a mechanism of grain boundary failure. Wayman and co-workers [9,10] did not find any evidences of second-phase particles or systematic correlation with manganese segregation at grain boundaries of embrittled Fe–20.8Ni–3.2Mn and Fe–20Ni–5Mn maraging steels. However, it has recently turned out that grain boundary precipitation is the main source of intergranular failure of Fe–Ni–Mn maraging steels. For instance, Mun et al. [11] reported precipitation of

* Corresponding author. Tel.: +98 412 345 9449; fax: +98 412 344 4333.
E-mail address: hossein@sut.ac.ir (S. Hossein Nedjad).

0921-5093/$ – see front matter © 2008 Elsevier B.V. All rights reserved.
doi:10.1016/j.msea.2008.01.070

face-centered cubic (fcc) austenite particles at grain boundaries of an Fe–7Ni–8Mn steel at early stages of aging at 723 K for which decohesion of austenite–ferrite interface was proposed to augment grain boundary fracture. Further, Lee et al. [12] identified closely spaced precipitation of a face-centered tetragonal (fct) θ-NiMn intermetallic compound at grain boundaries of an Fe–10Ni–5Mn steel at early stages of aging at 753 K. Meanwhile, Wilson [13] argued that manganese segregates at grain boundaries as an initial step in the formation of grain boundary precipitates and acts as a major embrittling element in the early stages of aging. Therefore, the source of grain boundary embrittlement in Fe–Ni–Mn maraging steels has remained controversial yet.

Hossein Nedjad studied an Fe–10Ni–7Mn maraging steel [14]. Intergranular embrittlement, grain boundary precipitation behavior and age-hardening precipitates of this steel during isothermal aging at 753 K have been reported already [15–18]. This paper is aimed to correlate the grain boundary embrittlement and precipitation reactions during isothermal aging treatment.

2. Experimental procedure

An Fe–10.35Ni–6.88Mn–0.006C–0.007S–0.005P–0.005N–0.003Al steel weighing 6 kg was prepared in a vacuum induction melting furnace under 10^{-2} mbar using electrolytic iron, electrolytic manganese and pure nickel shots. Bars weighing 200 g were cut from the ingot and remelted under argon gas in the water-cooled copper mold of a vacuum arc melting furnace. Remelted bars were encapsulated in quartz tubes under argon gas after evacuation to 10^{-5} mbar. Homogenizing treatment was performed at 1473 K for 173 ks followed by water quenching. Cold rolling to 85 pct reduction was carried out at room temperature followed by solution annealing treatment at 1223 K for 3.6 ks in a vacuum furnace, water quenching and subzero treatment at 77 K for 3.6 ks. Sheet-type tensile test pieces of 1 mm thickness, 2.5 mm width and 8.5 mm gage length were cut according to JIS Z2201 from a solution-annealed steel and aged for various times at 753 K in a neutralized salt bath. Tensile tests were carried out using a Shimadzu universal machine at a cross-head speed of 1 mm/min at room temperature. Fractography of broken tensile test pieces was performed by a scanning electron microscope. Auger electron spectroscopy studies of the solution-annealed and aged steels were carried out by a Physical Electronics[1] PHI 680 scanning Auger microscope operating at a voltage of 10 kV and current of 10 nA. For transmission electron microscopy, disc-shaped specimens of diameter 3 mm and initial thickness 300 μm were cut using an electro-discharge wire cutting machine, then manually polished to a thickness of ca. 30 μm. For observation of deformed structure, rectangular specimens of dimension 0.3 mm × 1 mm × 3 mm were cut from a broken tensile test piece close to the fracture tip by an electro-discharge wire cutting machine, then manually polished to a thickness of ca. 30 μm. Further thinning of thin foils was accomplished electrochemically in a solution of CrO_3 (200 g), CH_3COOH (500 ml) and H_2O (40 ml) at 285 K using a TenuPol[2]-3 instrument. Transmission electron microscopy was carried out with a PHILIPS[3] CM200-FEG microscope operating at 200 kV.

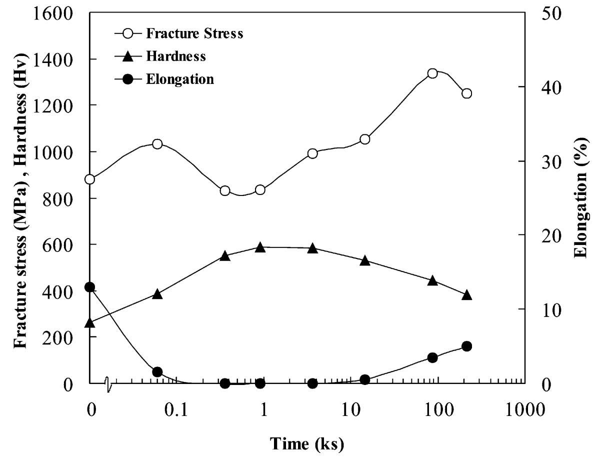

Fig. 1. Changes in the hardness, fracture stress and tensile elongation of the studied steel *vs.* isothermal aging time at 753 K.

3. Results

Fig. 1 shows changes in the hardness, fracture stress and tensile elongation of the studied steel *vs.* isothermal aging time at 753 K. Hardness increases by increasing aging time up to a maximum of 585 Hv at 3.6 ks then decreases at later stages of aging. Fracture stress turns to increase at initial stages which, unexpectedly, decreases after short aging to a minimum at 0.36 ks. Then it increases at intermediate stages of aging and eventually decreases at later stages of aging. Tensile elongation decreases drastically at early stages of aging to zero, remains zero at intermediate stages and eventually increases with aging time at later stages of aging, resuming to a maximum of about 5 pct at prolonged aging times. Tensile test pieces aged at initial and intermediate stages were broken suddenly before yield point. Overaged specimens were also broken suddenly, but in a midway between yield strength and ultimate tensile strength. Fig. 2(a) shows a scanning electron micrograph of a broken solution-annealed tensile test piece indicating an intragranular dimpled ductile fracture. Scanning electron micrographs of specimens aged for 0.36 (underaged), 3.6 (peakaged) and 86.4 (overaged) ks are shown in Fig. 2(b), (c) and (d), respectively. Those broken tensile test pieces exhibit intergranular fracture with prevailing secondary cracks propagated along grain boundaries as indicated by dashed lines in Fig. 2(c). It is found out that the proportion of intergranular fracture increases qualitatively at early stages of aging and decreases after prolonged aging. High-magnification scanning electron micrographs illus-

[1] Physical Electronics is a trademark of Physical Electronic Inc., Chanhassen, MN.

[2] TenuPol is a trademark of Struers A/S, Denmark.

[3] PHILIPS is a trademark of Philips Electronic Instruments Corp., Mahawh, NJ.