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Aging behavior and mechanical properties of maraging steels in the presence of submicrocrystalline Laves phase particles

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ABSTRACT

Cold rolling and annealing of homogenized Fe–Ni–Mn–Mo–Ti–Cr maraging steels resulted in the formation of submicrocrystalline Fe_2(Mo,Ti) Laves phase particles. Optical and scanning electron microscopy, X-ray diffraction, tensile and hardness tests were used to study the microstructure, aging behavior and mechanical properties of the annealed steels. The annealed microstructures showed age hardenability during subsequent isothermal aging at 753 K. Ultrahigh fracture stress but poor tensile ductility was obtained after substantial age hardening in the specimens with 2% and 4% chromium. Increasing chromium addition up to 6% toughened the aged microstructure at the expense of the fracture stress by increasing the volume fraction of retained austenite. The Laves phase particles acted as crack nucleation sites during tensile deformation.

1. Introduction

Maraging steels are a group of martensitic steels possessing ultra-high strength along with good fracture toughness. Conventional 18 wt.% Ni maraging steels are very costly in part because of expensive alloying elements such as Ni and Co. Maraging steels have found many applications in aerospace, military and production tooling [1]. Extensive research has been carried out to develop low-cost maraging steels through the substitution of nickel and cobalt by cheaper elements like manganese. For example, cobalt-free 18Ni maraging steels were developed in the early 1980s. Fe–Ni–Mn ternary alloys show good age hardenability comparable to 18 wt.% Ni maraging steels [1–4]. However, Fe–Ni–Mn maraging steels suffer from intergranular fracture after aging [4,5]. Early studies suggested that segregation of manganese atoms at prior austenite grain boundaries during isothermal aging is responsible for the grain boundary embrittlement of Fe–Ni–Mn maraging steels [4,6]. Recently, Hossein Nedjad [7] identified discontinuous coarsening of FCT θ-NiMn precipitates at grain boundaries during isothermal aging of an Fe–10Ni–7Mn (wt.%) maraging steel.

Great effort has been exerted to toughen economical Fe–Ni–Mn maraging steels that suffer from catastrophic intergranular brittleness [8–10]. Further alloying with molybdenum [11], titanium [12], tungsten [13] and chromium [14] has been explored as a possible means of improving mechanical properties. Among those alloys, an Fe–9Ni–5Mn–5Mo (wt.%) steel showed improved tensile properties [11]. Further alloying resulted in greater improvement in the tensile properties of an Fe–9Ni–5Mn–5Mo–1.5Ti–3Cr (wt.%) maraging steel [14]. Subsequently, further chromium addition up to 3.8 wt.% resulted in ductile grain boundary fracture with moderate tensile strength but poor tensile elongation [15]. Although many attempts were performed to improve the mechanical properties of Fe–Ni–Mn maraging steels, the favorable properties

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have not yet been actually realized. Therefore, further investigation is needed to develop economical ultrahigh strength steels.

Based on the conventional method for fabricating maraging steels, hot deformation and subsequent heat treatment processes are often carried out on cast ingots [16]. In an alternate processing method, after long-term homogenization in a vacuum atmosphere, the cast ingots were cold rolled at ambient temperature. Afterward, solution-annealing process was preformed. In consequence of this procedure, more homogenous and refined microstructures were obtained. During laboratory-scale processing of a series of Fe–Ni–Mn–Mo–Ti–Cr maraging steels, the latter method was used in the present study, mainly due to local feasibility and the instrumentation difficulties of hot deformation.

In contrast to previous experiences with 18Ni and Fe–Ni–Mn maraging steels [8,17], it was found that solution annealing augments precipitation of molybdenum and titanium-enriched Laves phases in the cold rolled steels, instead of forming precipitate-free, supersaturated lath martensite. Also, the Laves phase formation occurs frequently in a wide range of highly alloyed tooling and stainless steels [18].

The objective of this paper is to report the formation of Laves phase and mechanical properties evolution during aging of Fe–Ni–Mn–Mo–Ti–Cr maraging steels processed via cold rolling and solution annealing.

2. Experimental Procedure

Three heats of Fe–Ni–Mn–Mo–Ti–Cr alloys with different additions of chromium, Specimen 1 (2% Cr), Specimen 2 (4% Cr) and Specimen 3 (6% Cr) were prepared in a vacuum arc melting furnace using electrolytic iron, manganese, chromium, pure nickel shots, ferromolybdenum and ferrotitanium brickets. The size of the ingots is 150×40×8 mm. Melting and solidification were carried out in the water-cooled copper mold of the vacuum arc melting furnace after primary evacuation at 10^{-4} mbar and then purged with argon at 300 mbar. Table 1 gives the chemical compositions of the alloys prepared. Bars cut from the cast ingots were encapsulated with titanium foils in quartz tubes under argon gas and then homogenized for 173 ks at 1423 K. Afterwards, the specimens were quenched in water. The homogenized bars were cold-rolled for 50% thickness reduction at ambient temperature, annealed for 3.6 ks at 1323 K in a vacuum furnace, and subsequently quenched with water. Sheet-type tensile test pieces of 2 mm thickness, 6.25 mm width and 25 mm gage length were cut according to ASTM A370. Aging treatment was carried out for various time periods at 753 K in a neutralized salt bath. Tensile tests were carried out using a universal tensile test machine at a cross-head speed of 1 mm/min at ambient temperature. Changes in hardness during aging were measured in the Rockwell C scale. The hardness tests were repeated five times per condition. X-ray diffraction(XRD) was carried out using Cu-K_{α} radiation of 0.15410 nm wavelength in a Bruker AXS D8-ADVANCE instrument at a step size of 0.02° scanned for 1 S. Optical and scanning electron microscopy were used for microstructural and fractographic studies.

Table 1 – Chemical compositions of the three studied specimens (in wt.%).

Steel	Ni	Mn	Mo	Ti	Cr	C	P	S	Fe
Specimen 1	8.58	6.39	4.29	1.36	1.75	0.025	0.007	0.023	Bal.
Specimen 2	8.75	6.26	5.07	1.39	4.51	0.030	0.007	0.022	Bal.
Specimen 3	8.89	6.55	4.68	1.54	6.33	0.025	0.007	0.024	Bal.

3. Results and Discussion

Fig. 1a, b and c show, respectively, optical micrographs of the solidification microstructures of Specimen 1, Specimen 2 and Specimen 3. Specimens 1 and 2 exhibit packets of lath martensite which is typically found in maraging steels. However, in combination with lath martensite, retained austenite and odd particles were found in the inter-dendritic regions. In Fig. 1a and b the gray structure represents lath martensite and the brighter structure (γ) is retained austenite. Arrowheads show the odd particles. From the structure of the particles, it can be concluded that they were formed due to the positive microsegregation of the alloying elements during solidification.

Specimen 3 represents a needle-like dendritic microstructure as shown in Fig. 1c. In comparison to the specimens with lower Cr, Specimen 3 shows higher inter-dendritic microsegregation of alloying elements. Therefore, it can be concluded that increasing chromium content in the present steels beyond 4 wt.% drastically augments inter-dendritic microsegregation.

Optical micrographs of Specimens 1 to 3 after undergoing the thermomechanical procedure (homogenization, cold rolling and annealing treatments) are shown in Fig. 1d, e and f, respectively. This thermomechanical history provides solution-annealed martensite in 18Ni and Fe–Ni–Mn maraging steels [8,17]. On the contrary, in the present steels, it can be observed that the lath martensite has disappeared completely and has been replaced by a uniform dispersion of submicrocrystalline second phase particles in a matrix without characteristic martensite morphology. The second-phase particles were distributed heterogeneously by increasing chromium content in Specimens 1 to 3. Furthermore, some regions of Specimen 3 showed no submicrocrystalline second-phase precipitation. A secondary electron scanning micrograph of the annealed Specimen 2 steel is shown in Fig. 2, demonstrating the formation of submicrocrystalline second-phase particles. The energy dispersive X-ray spectrum analysis of the second phase particles indicated that those precipitates contain high molybdenum (12 wt.%), titanium (25.5 wt.%) and iron (35.9 wt.%) concentrations.

X-ray diffractograms obtained from the solidification microstructures of the studied steels are given in Fig. 3a. They show diffraction lines belonging to the body-centered cubic (BCC) iron and face-centered cubic (FCC) iron which verify the formation of martensite and retained austenite, respectively. The relative intensities of BCC and FCC diffraction lines change with chromium content. The FCC diffraction lines represent higher intensities in Specimen 3. Therefore, it