

X-ray diffraction peak profile analysis of maraged Fe-Ni-Mn steels

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Abstract

X-ray diffraction peak profile analysis was used to identify changes in the lattice distortions during isothermal aging of Fe-10Ni-7Mn (wt. %) maraging steel. Integral peak breadths were analyzed using classical Williamson-Hall equation taking the elastic anisotropy into account. It was found that substantial lattice distortions rise during precipitation hardening which depend strongly on the specific Young's modulus of a given crystallographic direction. After normalizing by elastic stiffness, the magnitude of average lattice distortions increased reasonably in accordance with age hardening.

Keywords: X-ray diffraction, peak breadth, maraging, precipitation, lattice strain.

1. Introduction

Maraging steels are a class of carbon-free, iron-based alloys possessing ultrahigh strength in combination with good fracture toughness¹⁾. Strengthening of maraging steels is attributed to the formation of fine intermetallic compounds during aging treatment. At the early stages of aging treatment, coherent GP zones form in a supersaturated martensitic matrix which creates elastic strain energy due to the coherency strain or misfit dislocations depending on the size of precipitates²⁾.

During further aging, those precipitates grow in size leading to the maximum hardening then softening gradually after prolonged aging due to the overaging of precipitates and formation of reverted austenite. Moreover, dislocations annihilate during aging, and subsequently decrease in volume density³⁾. The precipitation reaction is to alter lattice distortions during aging of a maraging alloy thus changing the shape and integral breadths of X-ray diffraction peak profiles⁴⁾. The aim of this paper is to study the evolution of lattice distortions during aging of Fe-10Ni-7Mn (wt. %) maraging steel by means of X-ray diffraction peak profile analysis.

2. Experimental procedure

An Fe-10Ni-7Mn (wt.%) maraging alloy was prepared using a vacuum arc remelting furnace. Homogenizing treatment of the cast bar was performed at 1250°C for 24 hours in a sealed quartz tube. Homogenized specimens were then aged at 450°C for different

durations of 1, 6, 15, 30, 60 minutes followed by water quenching. X-ray diffraction was made using a Bruker-AXS D8-ADVANCE diffractometer with Cu-K_α radiation of $\lambda=0.1541\text{nm}$ at a step size of 0.005° per 5 seconds. Individual XRD peak profiles belonging to (110), (200), (211), (220) and (310) planes were extracted, then optimum linear combinations of Cauchy and Gaussian type functions were fitted to the peak profiles after background and α_1 - α_2 removal. Rachinger's correction was carried out for the resolution of the α_1 - α_2 doublet, accordingly⁵⁾.

The rest of instrumental broadening was recognized with a NIST SRM660a LaB₆ standard reference material, then subtracted from the diffraction peak profiles according to the Stokes formulation⁶⁾. Integral breadths (β) were calculated for the evaluation of the peak broadening. Microhardness measurements were carried out at different aging times using a load of 50g. Averages of three individual measurements were determined for a given aging condition.

3. Results and discussion

Fig. 1 shows X-ray diffractograms of the steels aged for different times at 450°C revealing the reflections belonging to the body-centered cubic (BCC) iron. Table 1 gives details of the corrected integral breadths (β) of the indicated diffraction lines.

It is found that the integral breadths change with aging time, in correlation with the age hardening due to the formation and growth of precipitations and due decreasing the dislocations density⁷⁾. A plot of β vs. K for a solution-annealed specimen is shown in Fig. 2 according to the classical Williamson-Hall equation given by⁸⁾:

$$\beta = \frac{1}{d} + 2\epsilon K \quad (1)$$

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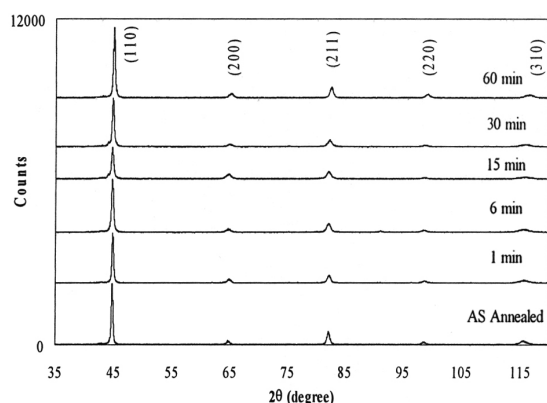


Fig. 1. X-ray diffractograms of the samples aged for different times at 450°C.

Table 1. Integral breadths of the indicated diffraction lines of the samples aged for different times at 450°C.

Aging time (min)	(110)	(200)	(211)	(220)	(310)
0 (solution annealed)	0.0279	0.0500	0.0420	0.0478	0.0641
1	0.0304	0.0624	0.0455	0.0518	0.0712
6	0.0379	0.0809	0.0603	0.0670	0.1072
15	0.0400	0.0838	0.0622	0.0717	0.1071
30	0.0403	0.0871	0.0628	0.0725	0.1127
60	0.0352	0.0756	0.0538	0.0614	0.0933

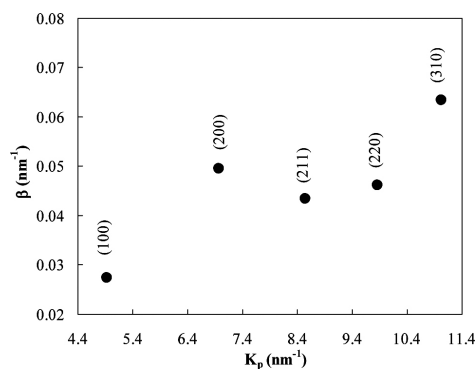


Fig. 2. A plot of integral breadth (β) versus diffraction vector of solution-annealed steel.

where d and ϵ are grain size and lattice strain, respectively. And K is the diffraction vector given by $K=(2\sin \theta/\lambda)$. In this equation, the integral breadth of the X-ray diffraction peak profile belonging to an arbitrary plane with Miller index (hkl) is attributed to the grain size and the lattice distortions. With an appropriate linear fitting of data, grain size and lattice microstrain could be obtained⁹⁾. However, it is found that the (200) and (310) diffraction lines have more integral breadths than neighboring diffraction lines resulting in the so-called anisotropic broadening phenomenon which is known due to the anisotropy in elastic constants. Table 2 gives the elastic modulus proposed for the studied steel. The problem of strain anisotropy would be, however, overcome by taking

the effect of elastic constants anisotropy into account using an alternative model of the classical Williamson-Hall equation as given by¹⁰⁾:

$$\beta = \frac{1}{d} + 2 \frac{\sigma}{E_{hkl}} K \quad (2)$$

Table 2. Elastic module proposed for different directions of the studied steel¹⁰⁾.

direction	<110>	<200>	<211>	<220>	<310>
E (GPa)	183	104	183	183	123

Fig. 3 shows changes of β versus $\frac{K}{E_{hkl}}$. Accordingly, the slope of a given linear regression gives the prevailing stress amplitude. The average lattice stress could be converted to lattice strain using an average value of Young's modulus according to the Hook's law subsequently. Fig. 4 shows changes of the average lattice strain derived from the stress amplitudes in combination with microhardness of the samples aged for different times. It is found out that the lattice strain increases with increasing aging time in correlation with the hardness. Precipitation behavior of Fe-Ni-Mn steel has been studied in detail already. So far, Suzuki¹¹⁾ proposed the zone formation due to a miscibility gap as a hardening mechanism in Fe-Ni-Mn alloys and confirmed the possible spinodal decomposition by thermodynamic calculations. The GP zones at initial stages of aging have been suggested to be bcc β -NiMn phase. Precipitation of fct θ -NiMn ($L1_0$), fcc Ni_3Mn ($L1_2$), and bcc NiMn (L) has been reported at later stages of aging in Fe-Ni-Mn steels¹²⁻¹⁴⁾. Therefore, it is assumed that in the initial stages of aging of the present steel, Ni- and Mn-enriched GP zones form, which is believed to hold coherent interfaces with bcc iron matrix. During the growth of the GP zones, coherency strain arises in response to the accompanied volume change. During further aging, those GP zones are thought to transform into fct NiMn precipitates in-situ where a coherency loss could take place essentially. Further aging goes toward over aging which reduces lattice strain by oriented growth of plate-like precipitates. Therefore, the maximum strain observed after aging for 30 min is thought to correspond to the transition from GP zones to semi-coherent NiMn precipitates. This transition point was found to precede maximum hardening conditions. Resistance of fine precipitates at initial stages of aging against cutting thoroughly by moving dislocations determine the hardness which later turns to dislocation by passing via formation of dislocation loops around precipitates. The maximum hardness is thought to be correlated with the conditions of dislocation by passing. Therefore, it is assumed that the maximum strain in the present study is accompanied by dislocations cutting precipitates thoroughly while maximum hardness is

obtained with a time delay when precipitate grows in size and becomes resistant against cutting after which dislocation looping governs deformation mechanism.

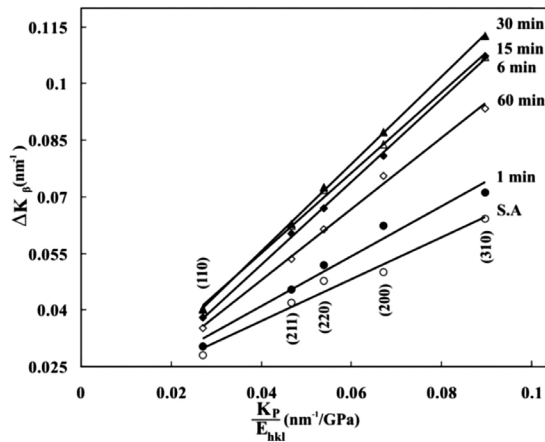


Fig. 3. Plot of integral breadths versus normalized diffraction vector for samples aged for different times at 450°C.

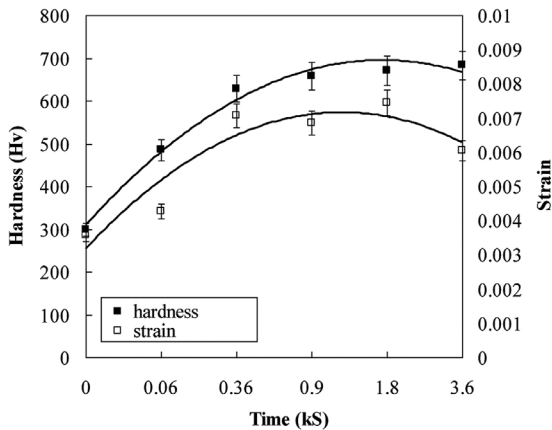


Fig. 4. changes in the lattice strain and micro hardness of the studied steel during isothermal aging at 450°C.

4. Conclusion

It was shown by X-ray diffraction peak profile analysis that the lattice strain increases in accordance with microhardness during aging of Fe-Ni-Mn maraging alloy. Changes in the lattice strain obey the hardening pattern reasonably.

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