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Maraging-350 steel: Following the aging through diffractometric, magnetic and hyperfine analysis

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ABSTRACT

Plates of solution annealed Maraging-350 steel were submitted to aging under an inert atmosphere, varying the time and temperature. The aged samples were characterized by X-ray diffraction and Mössbauer spectroscopy. The results revealed that the aging treatments induced the reversion of austenite, in amounts that vary with the time and the temperature of the heat treatment. The lattice parameters of the martensite and austenite phases, as well as their hyperfine parameters, were obtained at all aging conditions. No intermetallic compounds were identified in any of the aged samples, but a poorly crystallized phase fraction, the consequence of an incomplete martensite ⇒ austenite reversion transformation, was observed for some samples. The tetragonal distortion from cubic symmetry presented by the martensite in the solution annealed steel was not eliminated after aging.

1. Introduction

Maraging steels constitute a class of ultra-high strength and magnetic steels that have several applications, ranging from sporting equipment to aeronautic components. They also have applications as high velocity rotors and are used in hysteresis motors.

Heat treatments are applied for tempering and hardening of Maraging steels, aiming to provide these alloys with desirable mechanical or magnetic properties. Knowing the final structural and magnetic state of a steel, heat-treated at specific conditions of time and temperature, is essential for the intended application. This is especially important for the Maraging steels.

This family of low-carbon high alloy steels has as main alloying elements Ni (18 wt%), Co, Ti and Mo and, sometimes, Al or Cr [1,2]. They are divided into sub-classes – 200, 250, 300, 350 and 400, according to their yield strength (in ksi) [3]. Particularly, Maraging-350 has the second highest content of cobalt – first is Maraging-400 – and the highest content of titanium, which are responsible for increasing the formation temperature of reversed austenite [3].

The metallurgical routine of its fabrication involves, after the proper fusion alloying, a solubilization step – usually performed at 820 °C, for 1 h – to dissolve the alloy elements in the iron austenite matrix [2,4]. After that, the steel is cooled to room temperature (RT), in time varying from minutes to a few hours. This temperature path leads to a martensitic transformation of which the final product is a metastable structure, with the alloy elements forming an extended solid solution in iron, supposedly crystallized in the *bcc* structure.

Further, heat treatments in the 480–650 °C temperature range – a process of so-called *aging* – induce changes in the local chemical composition and may even favor the precipitation of intermetallic compounds. The tribological, mechanical or magnetic properties may be thus modified, according to several studies [4–6]. A large number of results, regarding the structural and magnetic changes that occurred as a result of an aging treatment, have been reported previously [7–10].

The aim of this study was to identify the aging effects – structural and magnetic – on the Maraging-350 steel, starting from samples solution annealed and stabilized in the martensitic state. This investigation was carried on with extreme care in the preparation of the specimens for characterizations in order to avoid or, at least, to minimize mechanical effects commonly observed from cutting, sieving or grinding the steel. This procedure is essential when searching exclusively for thermal effects; otherwise, mechanically induced transformations may mask the appropriate results. We took the same precautions in the description of a *virgin* (i.e. not aged) steel sample [7].

Here, the aging effects were followed through diffractometric and hyperfine analyses of samples aged under specific conditions (i.e., time and temperature) usually applied in the aging of the

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Maraging-350 steel. The phase(s) present in the heat-treated samples were identified by X-ray diffraction and Mössbauer spectroscopy. Both characterization techniques were conducted using new approaches, concerning the mathematical models for fitting experimental curves. The methodology applied led to a more precise description of the physical and chemical properties of a heat-treated Maraging-350 steel. This shall provide key information for the processing of this useful material.

2. Experimental details

The raw material used in this investigation was a commercial Maraging-350 steel (i.e., a forged billet), made by a Brazilian steel company. Details on the steel composition and about the preparation of samples for the characterizations may be found elsewhere [7]. In the present study, pairs of plates – a thin one ($\phi \leq 100$ μm) and a thick one ($\phi \approx 1$ mm) – taken from the solubilized specimen were simultaneously submitted to heat treatments, i.e., each of the pairs was annealed at 480 °C, 580 °C and 650 °C, for periods of 3 h, 6 h and 12 h, under an argon atmosphere. After each heat treatment, the plates were polished again, with the same care taken as before. The thin plates were reduced to a final thickness of ~50 μm.

The thick and thin plates were used for X-ray diffraction and transmission Mössbauer spectroscopy, respectively. Other basic details about the characterization techniques, such as equipment and numerical analysis, may also be found in Ref. [7]. In the present investigation, a cobalt tube (not a copper tube as in [7]) was used for the X-ray measurements, aiming to minimize the effects of the fluorescence, which is significant when the copper radiation is used.

The diffractograms were refined by the Rietveld method (FullProf program), considering the $I4$ and $Fm-3m$ space groups, attributed respectively to the martensite (α') and austenite (γ) phases, eventually present in the samples characterized. The $I4$ space group was chosen for martensite since, according to our earlier studies, this phase presents a tetragonal distortion [7]. For the refinements, both phases were considered to have the same nominal sample composition.

3. Results and discussions

3.1. X-ray diffraction

Fig. 1 shows the diffractograms for some selected samples. Refined lattice parameters ($a=b$ and c) and phase molar fractions are presented in Table 1, which includes data for all samples characterized. The diffractometric profile of the 480 °C/12 h sample (Fig. 1a) is analogous to that of the solution annealed steel (see Fig. 1 of Ref. [7]). It is characteristic of a monophasic sample, corresponding to the martensite structure. As for the as-received steel – and for the samples annealed at 480 °C for 3 h and 6 h (diffractograms not shown) – no extra peaks could be identified, which means that no secondary phases have precipitated in the sample in significant amounts (i.e., within the resolution limits of the X-ray diffraction technique).

In contrast, the diffractogram of the 580 °C/12 h aged sample (Fig. 1b) shows peaks of a minor phase, which by its structure (*fcc*) may be identified as belonging to austenite. Indeed, this phase is recurrently found in aged or even just annealed solution Maraging-350 steels [1,3,8,11]. Austenite is also (is not) present in the 580 °C/6 h (3 h) aged sample (diffractograms not shown).

The lattice parameters of the martensite phase change slightly from one sample to other. As a general trend, a decreases whereas

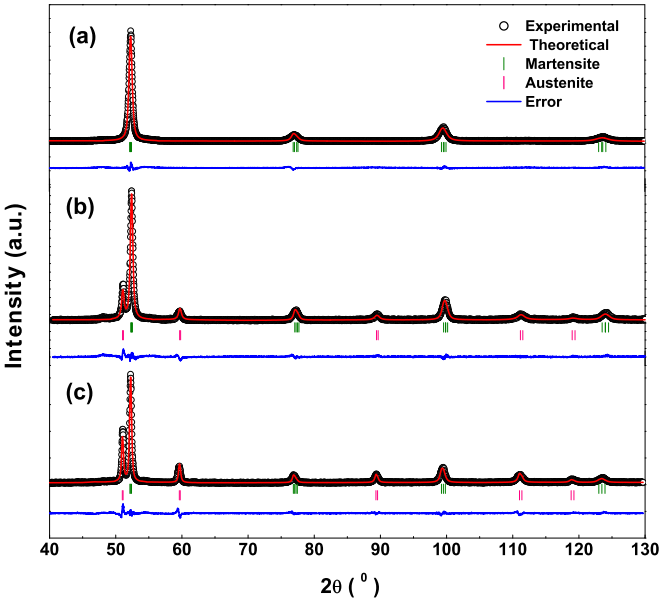

Fig. 1. Refined diffractograms for the 480 °C/12 h (a), 580 °C/12 h (b) and 650 °C/12 h (c) aged Maraging-350 samples.

Table 1
Lattice parameters and molar fractions of the phases identified in the as-received and aged steel samples.

	Sample Phase		$a=b$ (Å)	c (Å)	Phase fraction (%)
As-received		Martensite	2.8842 (2)	2.8688 (4)	100
		Austenite	–		–
480 °C	3 h	Martensite	2.8812 (3)	2.8604 (4)	100
		Austenite	–		–
	6 h	Martensite	2.8796 (2)	2.8611 (4)	100
		Austenite	–		–
	12 h	Martensite	2.8794 (3)	2.8608 (4)	100
		Austenite	–		–
580 °C	3 h	Martensite	2.8670 (5)	2.8569 (9)	91.5 (4)
		Austenite	3.5962 (6)	–	8.5 (1)
	6 h	Martensite	2.8736 (2)	2.8605 (4)	88.4 (2)
		Austenite	3.5995 (5)	–	11.6 (1)
	12 h	Martensite	2.8720 (2)	2.8587 (4)	74.0 (2)
		Austenite	3.5965 (2)	–	26.0 (1)
650 °C	3 h	Martensite	2.8739 (3)	2.8652 (6)	57.7 (1)
		Austenite	3.5964 (2)	–	42.3 (1)
	6 h	Martensite	2.8754 (5)	2.8703 (9)	65.7 (2)
		Austenite	3.5975 (2)	–	34.3 (1)
	12 h	Martensite	2.8789 (2)	2.8665 (4)	63.7 (2)
		Austenite	3.5992 (2)	–	36.3 (1)

c is more or less constant, as the temperature raises for isochronical aging treatments.

The evolution with aging time of the tetragonal distortion may be perceived from Fig. 2. The c/a ratios for the 650 °C and 580 °C temperatures initially grow and then further decrease, both converging after 12 h of heat treatment to ~0.9955.

In Ref. [7], we argued that the distortion in the as-solubilized state may be attributed to the different atomic sizes of the alloy elements present in the steel composition. Now, it is clear that heat treatments, which may produce precipitation of titanium or molybdenum (i.e., the elements with the largest atomic radii) compounds, do not eliminate the distortion from the cubic symmetry. The variation of the c/a ratio for the 480 °C condition, although also slight, reveals that some atomic re-arrangement takes