



Structural and magnetic characterization of martensitic Maraging-350 steel



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ABSTRACT

A solubilized Maraging-350 steel used in the nuclear industry, as received from the manufacturer, was finely characterized by X-ray diffraction (Rietveld refinement), Mössbauer spectroscopy and magnetization techniques. For these characterizations, samples were prepared with exceptional care regarding any possible modification of the physical properties of the steel due to mechanical work done on the original piece during specimen preparation. The results showed that the steel is martensitic, although evidence of a crystallographic distortion from the cubic symmetry usually attributed to the martensite was found. It was also revealed that the atomic configurations of the iron nearest neighbors may be assembled in three groups, according to the hyperfine magnetic field at the iron sites. Magnetic minor loops displayed a soft magnetic material with the coercive field, residual induction and loop area obtained as a function of the maximum applied field in the loop obeying peculiar behaviors. The measured properties represent key information for a suitable control for the aging of Maraging-350 and, consequently, for the design of ultracentrifuges used in the isotope enrichment of nuclear fuel.

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1. Introduction

Maraging steels constitute a class of ultra-high strength and magnetic steels that have several applications, from sportive equipments to aeronautic components. They are also applied as high velocity rotors, used in hysteresis motors for ultracentrifuges. Therefore, they are strategic materials for the nuclear industry. This family of low carbon steels has as main alloy elements Ni (18 wt%), Co, Ti and Mo and, sometimes, Al or Cr [1–3]; it is divided in subclasses – 200, 250, 300, 350 and 400 [3,4]. Particularly, Maraging-350 has the largest amount of cobalt and titanium, which are responsible for increasing the temperature formation of reversed austenite [4].

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 [2–5]. After that, the steel is cooled to room temperature

(RT), in the time of minutes or 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, thermal treatments in the 480 °C–650 °C temperature range induce changes in the local chemical composition and, even, favors the intermetallic compounds precipitation. The tribological, mechanical or magnetic properties may be modified, accordingly to several studies [3,6–8].

A large number of results, regarding the structural and magnetic properties of the martensitic state of the Maraging-350 steel, have been reported previously. According to these studies, it is generally accepted that the respective martensite phase stabilizes with the cubic space group *Im*–*3m*, with a lattice parameter of 2.880(2) Å [7,9–11]. The alloys elements are not crystallographically ordered, as indicated by Mössbauer spectroscopy, and a typical pattern shows a superposition of different hyperfine magnetic fields acting on iron sites [12–14]. The subspectral areas and magnitude of these hyperfine magnetic fields were traced as a function of the aging time.

Measurements of the macroscopic magnetic properties have revealed that this steel is a soft ferromagnetic material, and some

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magnetic parameters – e.g., the residual induction and coercive field – show considerable scattering [15,16]. The spread in these magnetic data may be attributed to the “mechanical history” of the samples characterized, invariably different in each investigation previously conducted. In this sense, it is largely recognized that the structural and magnetic properties are very sensitive to cold or hot rolling, extrusion or other mechanical processes that may easily modify the intimate properties of the as-cast alloy. The magnetic hardening of the steel is one of the most striking effects [15].

The aim of this investigation was to characterize structurally and magnetically a *least possibly mechanically altered* sample of a Maraging-350 steel, solubilized and stabilized in the martensitic state. This was done, first, by carefully preparing the specimens for characterization by X-ray diffraction, Mössbauer spectroscopy and magnetometry. Then, numerical analyses were applied to experimental results more acutely or extensively than usual. As will be shown, this methodology led to new insights on the Maraging-350 steel physical properties. This will provide key information for the processing of this material useful for the nuclear industry.

2. Experimental details

The raw material characterized in this investigation was a commercial Maraging-350 steel (i.e., a forged billet), made by a Brazilian steel company. Table 1 presents the chemical composition of the steel, as informed by the manufacturer.

The original piece was disk-shaped, with 2.0 cm tall and 13.5 cm in diameter, cut from an as-fabricated cylinder of solubilized Maraging-350. The disk was carefully cut in the middle, in a precise metallographic cutter with a diamond saw, forming two semi-disks of nearly the same size. Then, slices (≤ 1 mm) were taken in the same way (i.e., by slow cutting and cooling the abrasion region) from the rectangular face of the semi-disk. Each one of these slices was divided in four nearly squared plates of ~ 2 cm² frontal area. Two of the squared plates were manually sanded on one surface with sandpaper 100, 200 and 400 and, then, used for characterization by X-ray diffraction. The other pair of plates was further sanded until they reached ~ 50 μ m thickness and were used for Mössbauer spectroscopy. Finally, a small disk ~ 3 mm in diameter was cut from one of the thinnest plates and used for magnetization measurements.

The X-ray diffractograms were measured at room temperature, using CuK α radiation ($\lambda = 1.5418$ Å), in a conventional diffractometer, in the θ – 2θ Bragg-Brentano geometry. The 2θ range was from 30° up to 90° , with increments of 0.02° and a counting time of 6 s per step.

Transmission Mössbauer spectra were taken at RT, from a constant acceleration spectrometer, with a $^{57}\text{Co(Rh)}$ source of 25 mCi of activity, using as absorbers the foils described above. The numerical fits were made considering a Lorentzian line shape and applying the criterion of the minimum chi-squared. The equipment was calibrated with metallic iron (α -Fe) at RT.

The magnetization – $M(\text{emu/cm}^3)$ – vs. applied magnetic field – H_{ap} (Oe) – curves were taken at RT, in a vibrating sample

magnetometer (VSM) with 10^{-5} emu sensitivity. Starting with the sample at zero magnetization, minor loops were extracted until the magnetic saturation of the sample was reached. Magnetic induction – $B(H)$ – as a function of the true magnetic field (i.e., $H = H_{\text{ap}} - N_d M$) was calculated, considering the demagnetizing factor ($N_d = 0.33$) of the specimen used in the magnetic measurement.

3. Results and discussions

3.1. X-ray diffractometry

Fig. 1 shows the diffractogram for the Maraging-350 steel. The diffractometric profile is characteristic of a monophasic sample and corresponds to the martensite phase, as expected for the solubilized steel. In comparison with other diffractograms published earlier, no extra peaks could be identified, which means that no other phase is present in significant amount (i.e., in the resolution limits of the diffraction technique). The FULLPROF program [17] was applied to refine the crystalline structure by the Rietveld method, considering the $Im-3m$ space group, as would be expected for the martensitic maraging. A pseudo-Voigt shape function was used to fit the experimental data. The data refined were the lattice parameter, the peak shape and isotropic thermal parameters. The lattice parameter obtained is $a = 2.8836$ Å, a value that is consistent with those obtained by others for martensitic Maraging-350 [7,9–11]. However, a close inspection in the more significant region of the diffractogram shows that the peaks [200] and [211] are slightly displaced from their expected angular positions. Both are shifted to left – [200] (more) and [211] (less) – which suggests that, contrary to what is commonly accepted, the martensitic phase of Maraging-350 stabilizes with another structure (i.e., non-cubic system) or, at least, a distortion from this structure must be considered. A rigorous search for the space group that best refines the diffractometric profile and, consequently, reveals the right crystalline structure for this material is currently being conducted and will be published elsewhere.

3.2. Mössbauer spectroscopy

The Mössbauer spectrum for the Maraging-350 steel is shown in Fig. 2. It clearly reveals a magnetic material – consistent with

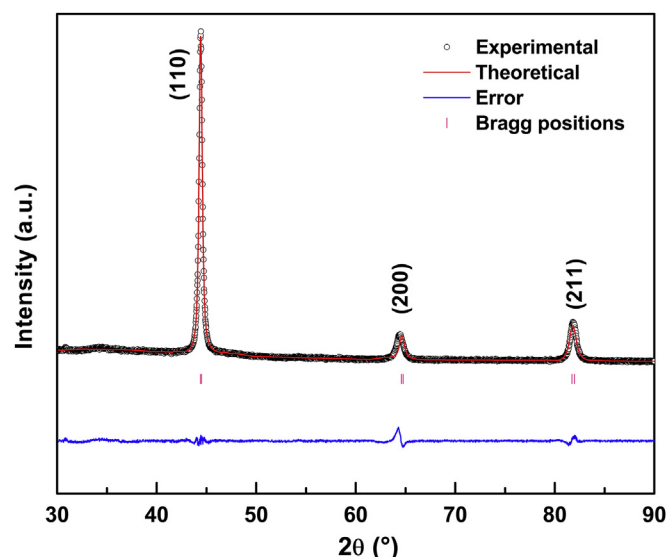


Fig. 1. Refined diffractogram for the Maraging-350 steel.

Table 1
Chemical composition of the Maraging-350 steel.

Alloys elements	WT%
Nickel	18.5
Cobalt	12.0
Molybdenum	4.5
Titanium	1.5
Al, Si, Cr, Mn	~ 0.1
C, S, Ca, Zr, P	< 0.1

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