STUDY OF MANGETIC PROPERTIES OF MARAGING 350 STEEL AS A FUCNTION OF GRAIN SIZE *

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Abstract

The magnetic properties of the maraging 350 steel were analyzed in four different grain sizes. The applications of maraging steels can be found in several fields such as aeronautics, super centrifuges, navy, etc. Thus, this kind of steel must combine excellent mechanical properties with good magnetic ones. Microstructure and grain size can affect these properties. In order to evaluate the influence of grain size in some magnetic properties such as coercive force, saturation magnetization, remanence magnetization and Magnetic Barkhausen Noise (MBN), four solution temperatures were selected to vary the grain size of the samples. The samples were also aged in two different conditions after solution annealing. Vibrating-sample magnetometer (VSM) and MBN were used to analyze the changings in the magnetic properties as the grain size increased. The results showed changing in these properties related to grain size, making it possible to choose the best heat treatment conditions for magnetic applications.

Keywords: Maraging steel; magnetic properties; grain size.

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1 INTRODUCTION

Maraging steels have been researched over the last few years both in experimental studies and in applications in the technological sectors due to their good applicability. This kind of steel is a low-carbon martensitic and the high strength and flexibility of maraging steels make it an ideal material for applications in various industrial sectors from high pressure vessels operating in critical processes, aeronautical components, submarine hulls to sports equipment [1]. Maraging steels possess also good magnetic properties and they can be used in ultra-high speed rotors for electric hysteresis motors once maraging steels are soft magnetic materials. For this purpose, the coercive force Hc must be low [1,2] and the saturation and residual induction must be high in fields with values in the range of 20–60 A/cm and the squareness ratio must be at least 0.7 [3].

Maraging steels present a soft and deformable martensitic structure when solution annealed due to the low carbon and high nickel content in the martensitic matrix [4]. Increasing the solution annealing temperature, grains will grow. This can affect the mechanical and magnetic properties [5,6].

In the aging condition (400 ºC – 650 ºC), Maraging steels are hardenable and this effect can be attributed to the precipitation of some intermetallic compounds in the martensitic matrix, mainly Ni3(Mo, Ti) and Fe2Mo [7,8]. When the only requirement is the high strength, the alloy is aged in the temperature range of 450 ºC to 550 ºC, but when the magnetic properties are taken into account, the alloy is aged in the temperature range of 550 ºC to 650 ºC [9] . During aging of maraging steels, the formation of austenite can occur as the Ni3Ti and Fe2Mo dissolve partially in the matrix and depending on their nickel content, the austenite formed at high aging temperatures can be retained at room temperature [10].

The microstructure and intermetallic phases can also influence the magnetic properties of maraging steels. Aging heat treatments between 500 °C and 700 °C can promote reverted austenite by diffusion controlled decomposition of martensite [11]. The composition of the maraging steels can also contribute in their magnetic properties [12–14].

Vibrating-sample magnetometer (VSM) and Magnetic Barkhausen Noise (MBN) are two important techniques to study magnetic properties of steels. The advantage of these techniques is that they are nondestructive and economical techniques and can be easily used in samples of various sizes (in case of MBN) under several external conditions. They are also sensitive to several parameters which affect the domain configuration as grain size, composition, surface conditions, hardness, residual stress and magnetic field strength.

In this research, magnetic properties of two Co-containing 18Ni 300 maraging steels were evaluated as a function of the grain size. In order to vary the grain size, four solution annealing temperature were chosen: 840, 950, 1050 and 1150 ºC. The samples were solution treated for 1 h. Two aging conditions were also selected: 480 ºC/3h and 560 ºC/1h. The MBN and hysteresis loop in two marging steels (300 and 350) were analyzed and discussion of the results took into account the grain size variation and a possible formation of austenite during aging [10].

2 DEVELOPMENT

2.1 Materials

The material used was the commercial Co-containing 18Ni maraging 350 steel. Its chemical composition is presented in table 1.

Alloys	Ni	Co	Mo	Ti	Al	Fe
350	17.65	11.65	4.69	1.44	0.065	Bal.

Table 1. Chemical composition (wt%) of the maraging 350 steel.

The as received condition of this material was a solution treatment at 820 ºC for 1 h. The material had a disc shape with 14.00 cm in diameter and 1.28 cm in thickness.

2.2 Heat treatments

Four solution annealing temperature (840 ºC, 950 ºC, 1050 ºC and 1150 ºC) were selected in order to obtain samples with different grain sizes. The solution annealing heat treatments were carried out for 1 h in a muffle furnace followed by an air cooled to room temperature. After the solution treatment, the samples were divided in two groups: one for the aging treatment at 480 ºC for 3 h and the other at 560 ºC for 1 h. After aging, the samples were air cooled. The dimensions of the samples were in average 1.5 cm × 1.0 cm × 1.5 mm.

2.3 XRD characterization

For the samples of the second group (aged at 560 ºC for 1 h), X-ray diffraction (XRD) measurements were carried out in order to detect any reverted austenite. The samples were sanded up to 400 mesh (SiC paper). The measurements were carried out using a Philips® X'Pert Pro diffractometer with a radiation of CoKα (0.1789 nm). The 2θ angle ranged from 45º to 104º. XRD was not carried out for the first group of samples. According to Santos et al, aging at 480 ºC up to 50 h does not form reverted austenite [15].

2.4 Growth of grain size

After the heat treatments (solution annealing followed by aging), the samples were sanded using SiC paper up to 2500 mesh, washed in distilled water and blow dried followed by polishing using alumina (Al_2O_3) of 1 and 0.05 µm to achieve optical quality of the images after etching. In order to reveal the prior austenite grain, an electrolytic etching of 10 V with stepped time in an aqueous solution of 20% chromic acid (H_2CrO_4) was used. An Optical Microscopy (MO) using a Zeiss Microscope model Axio Imager M2m was used to obtain the images of prior austenite grains. The grain sizes were computed using the software ImageJ following the recommendations of the ASTME112-96 [16]. In order to reveal the martensitic structure of the steels, a chemical etching with HNO_3 (4%) in methanol was used.

The samples were immerged in this reagent up to 10 s and the images were obtained by a Philips® XL–30 Scanning Electron Microscopy (SEM).

2.6 Vibrating Sample Magnetometer (VSM)

In order to measure the magnetic properties (magnetic remanence, magnetic saturation and coercive force) of the studied alloy, the VSM technique was used. The samples heat treated in all solution conditions combined with the two aging conditions were used for this test. Disks of 3.00 mm diameter and thickness in the range of 0.05 e 3.00 mm were manufactured. The measurements were carried out using an EGG-PAR model 4500 vibrating sample magnetometer (VSM). Measurements were performed at room temperature with maximum applied field of 5 kOe (400 kA/s), with 1 ms time constant and a total measurement time of 30 min. After obtaining the hysteresis graphs, the curves were corrected for demagnetization fields using the equations suggested by Chikazumi et al [17].

2.7 Magnetic Brakhausen Noise (MBN)

Magnetic Barkhausen Noise was measured on the samples for all aging treatment conditions. The sample sizes were 0.98 mm x 0.87 mm x 0.20 mm. The samples were sanded up to 220 mesh. Then they were sprinkled with alcohol and dried with a drier. The control of the excitation and processing variables and the acquisition of MBN signals were performed by the BarkTech module, developed entirely in the Dynamics and Instrumentation Laboratory of the Department of Mechanical Engineering of POLIUSP (Ladin-PME-EPUSP). The MBN measurements were carried out using a probe composed of an electromagnetic yoke which produces an alternating magnetic field on the sample, and also a coil to detect the MBN signal. The coil response is proportional to a time-variable field B that is produced using an alternating field H with frequency f. The change generated in the magnetization induces electrical impulses in the coil. MBN is isolated using amplification and the use of band pass filters. The results of these measurements were expressed as RMS (Root Mean Square) and represent the square root of the MBN signals over time. The frequency of sampling was 350 kHz, the Number of measurements on each sample was 35000, Frequency of excitation was 10 Hz, the Amplitude of excitation was 2 V and 11 Repetitions per sample.

2.8 Results and discussion

2.8.1 Study of the prior austenite grain size growth

Figure 1 shows the prior austenite grain sizes for the maraging 350 steel treated at several solution annealing temperatures and aged at 480 ºC for 3 h. For the first two solution annealing temperature (840 ºC and 950 ºC), the grain size is not so different. The grains for these two samples presented sizes of 89.9 and 106.8 µm, respectively (Figure 1a and 1b). For the grains to grow, a movement of dislocations must happen [18]. Martensitic structure can also be seen inside the prior austenite grains. The precipitates like Ni3Ti, TiC and other phases can act as barrier preventing the grain growth. The sample solution annealed at 1050 ºC presented a grain size of 182.4 µm

(Figure. 1c). The last precipitate to dissolve in the matrix is TiC and this carbonate is responsible to hold the grain until the solution annealing temperature reaches the value of approximately 1100 ºC. After this temperature, the grain is free to move. The sample solution annealed at 1150 ºC obtained the largest grain size (374.6 µm) among the samples of the maraging 350 steel (Figure.1d). Above this temperature, only the austenite phase is presented. That is the reason for the grains of this sample reached so large values of grain size. The same behavior was found for the samples aged at 560 ºC/ 1 h.

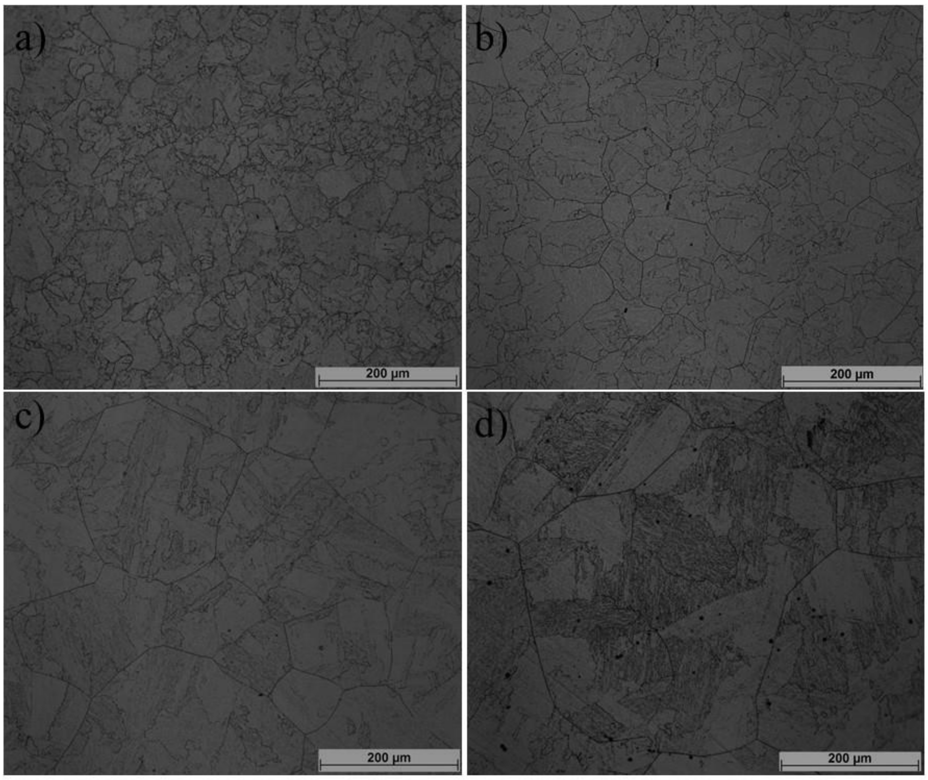

Figure 1. Optical micrograph (200 µm) of grain size for the samples solution treated at a) 840 ºC, b) 950 ºC, c) 1050 ºC and d) 1150 ºC for 1 h. All of them aged at 480 ºC/3 h.

The grain growth is exponential and the aging does not influence on the grain size as can be seen in Figure 2. For the first two grain sizes, the changing is not so considerable. After 1000 ºC, the grains increase considerably reaching the maximum size for this research at 1150 ºC. Martensitic blocks and laths can be seen in Figure 3. This structure was revealed after etching the surface of the samples with HNO_3 (4%) in methanol.

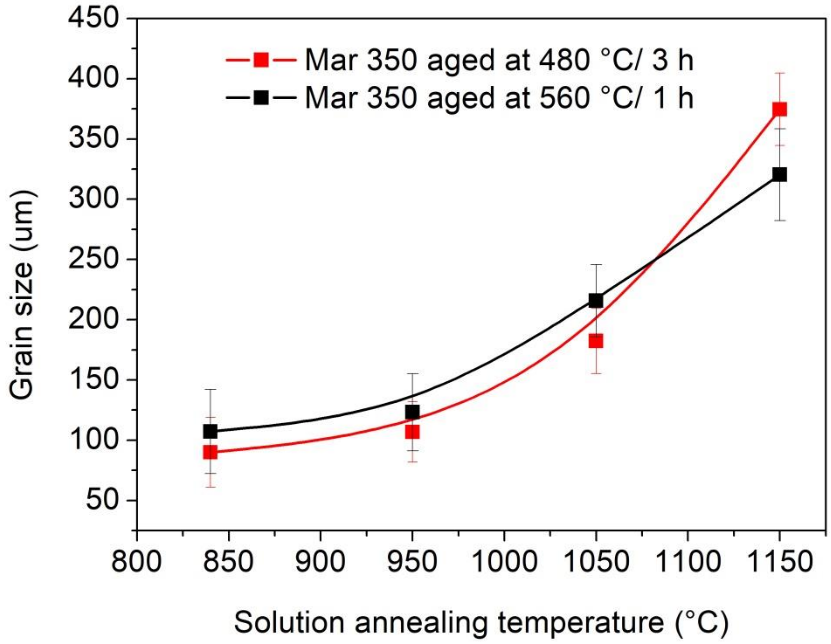

Figure 2. Grain size as a function of solution temperature for the two aging conditions.

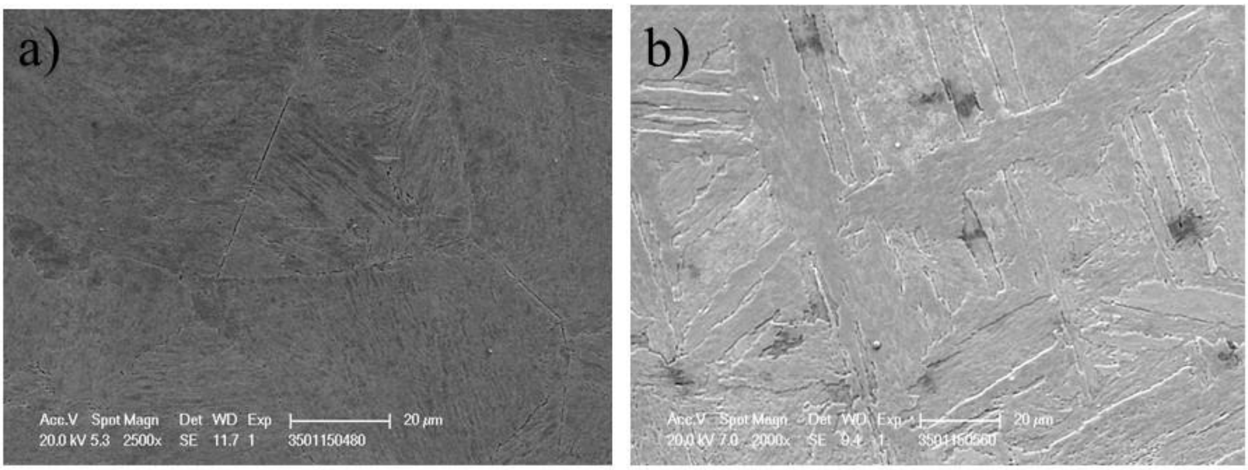

Figure 3. SEM of Martensite blocks and laths for the maraging 350 steel solution annealed at 1150 ºC for 1 h and aged at a) 480 ºC for 3 h and b) 560 ºC for 1 h (scale of 20 μm).

2.8.2 XRD analysis

XRD measurements were performed to detect any reverted austenite on the samples solution annealed at 840 ºC and 1150 ºC both aged at 560 ºC / 1 h (grain size extremes). Figure 4 shows the XRD patterns for these two conditions. Peaks of BBC structure were detected. These peaks correspond to the martensite phase (α'). No other peaks were detected. Aging above 500 ºC for long periods of time can promote the precipitation of reverted austenite as the precipitates such as $Ni_3(Ti, Mo)$ dissolve [19–21]. No diffraction peaks corresponding to reverted austenite nor the peaks of the intermetallic compounds were found meaning that the volume fractions of these phases can be below the detection limit of the technique used that is around 5 % [22]. Reverted austenite may exist for this condition but the content is too low to be detected. No reverted austenite is expected for the samples aged at 480 ºC/ 3 h as reported by Santos et al [15].

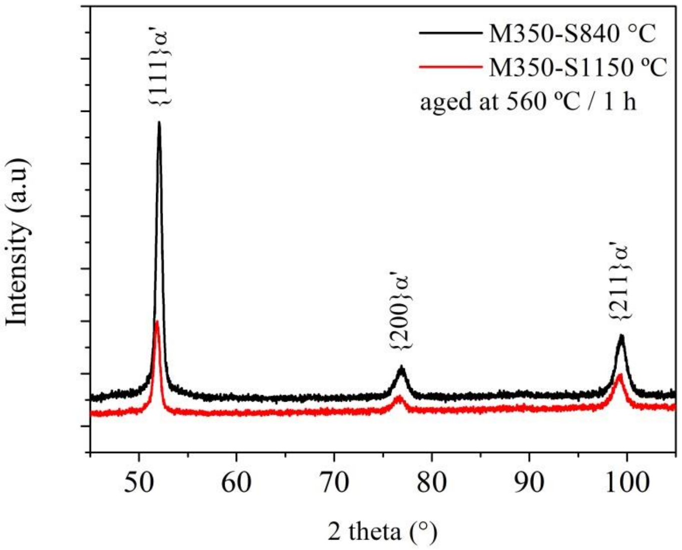

Figure 4. XRD for the samples solution annealed at extreme temperatures and aged at 560 ºC for 1 h.

2.8.3 VSM analysis

One of the parameters affected with grain size was the remanence magnetization as can be seen in the graph of Figure 5a. For the smallest grain size, Maraging 350 showed the highest remanence magnetization value in the two aging conditions.

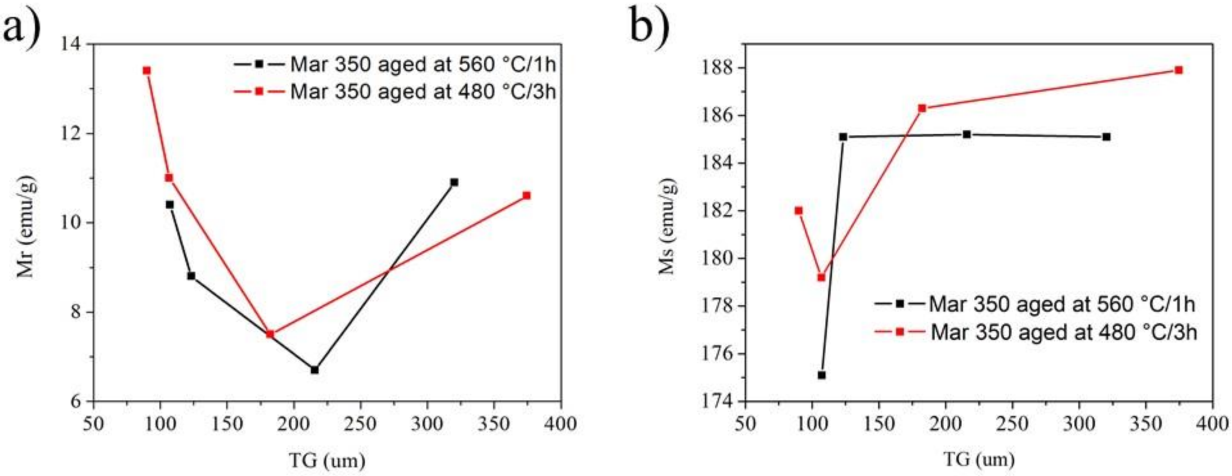

Figure 5. Remanence magnetization (a) and saturation magnetization (b) as function of grain size.

The smaller the grain size, more capacity the Maraging steel has to retain the magnetism. The remanence magnetization falls among the first three grain sizes (89,9 μm, 106,8 μm and 182,4 μm). This corresponds to the solution temperatures between 840 ºC and 1050 ºC. After the grain size of 182,4 μm, the remanence magnetization curve rises again for both aging conditions. Crystalline defects act as a barrier to the movement of magnetic domains. As the grain boundary is considered to be a defect in the crystalline lattice, the more grain boundaries the material possesses (the smaller the grain size), the greater the resistance to the movement of magnetic domains. On the other hand, the larger the grain, the more magnetic domains will be inside the grain, indicating that the material will also retain a little more magnetization as can be observed for the larger grain sizes in both aging conditions. This also explains why the saturation magnetization is higher for the larger grains (Figure 5b). More domains inside the grains require more magnetization to reach the saturation.

The variation of the coercive force with grain size is shown in Figure 6 for both aging conditions. The considerable variation of the coercive force is noticed when the two aging conditions are compared. The samples aged at 560 ºC/1 h presented higher values for the coercive force for all the grain sizes when compared with the other aging condition (480 ºC/3 h). Magnetic hardening increases as the aging temperature increases. Maraging steel is a soft magnetic material and its magnetic hardening is a disadvantage in its application. These results are in agreement with the results of Tavares et al [1] for the maraging 350. The authors studied the same magnetic properties for a larger range of aging temperatures and drew the same conclusions.

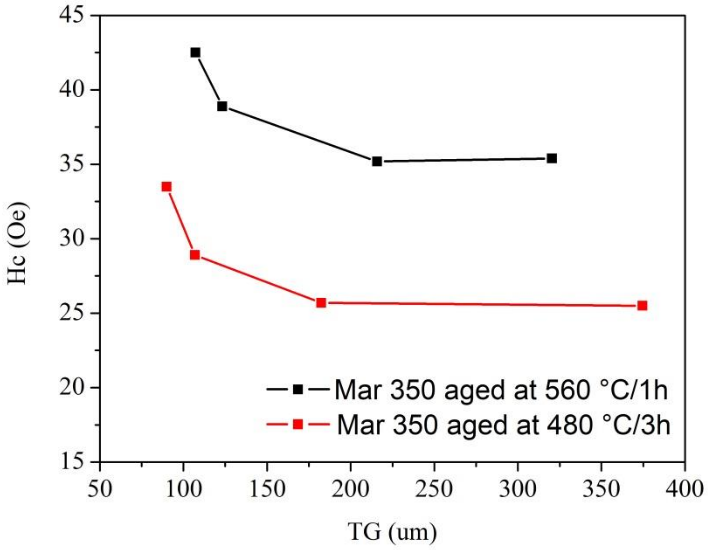

Figure 6. Coercive force for the two aging conditions in Maraging 350.

The MBN is shown in Figure 7. No significant relationship between the MBN and the grain size can be confirmed but the influence of aging condition can be noticed on the graphs. The samples aged at 560 ºC for 1 h presented the lowest values for the MBN. This can be attributed to any non-magnetic phase precipitated for this aging condition, once that the MBN signal decreased when compared with the other aging condition. The exception is for the sample solution annealed at 950 ºC and aged at 480 ºC/3h. The decrease of MBN for this condition is not clear yet. Maybe not only the grain sizes are influencing the MNB but also the precipitates. Although not shown in the XRD diffractogram (Figure 4), this can be an indicative of the precipitation of a non-magnetic phase as reverted austenite for the aging condition of 560 ºC/1 h. For this condition, its content can be less than 5 % and the XRD does not detect its peak, but MBN can detect any non-magnetic phase when the signal is measured.

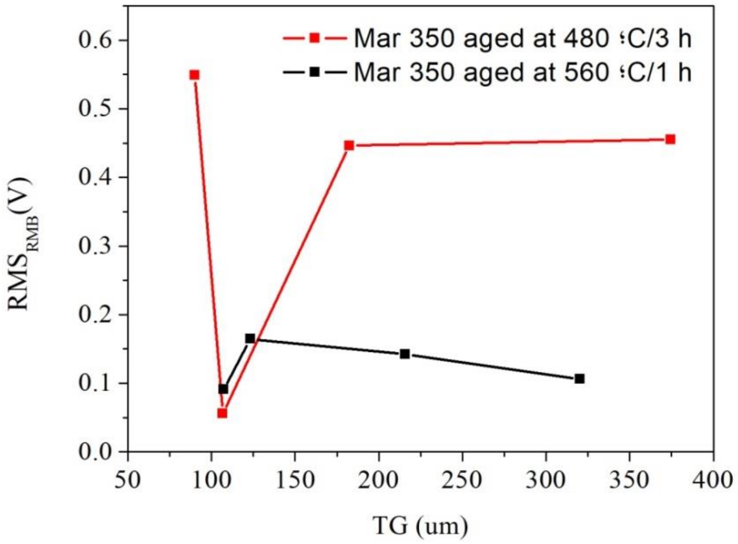

Figure 7. MBN for the maraging 350 aged at 480 ºC for 3 h and 560 ºC for 1 h.

Future works in this field can confirm the effect of the precipitates on the MBN considering the variation of grain size for Maraging steels.

5 CONCLUSION

The grain growth of the maraging 350 is exponential. After the solution temperature of 1050 ºC, the grain increases considerably. Aging after the solution annealing treatments does not affect the grain size. Magnetic properties as remanence and saturation magnetization are also influenced by the grain size once that these properties presented a similar behavior for the two aging conditions showed in the graphs. As the aging temperature increases, the coercive force also increases indicating a magnetic hardening of the material. The aging conditions affect also the MBN, indicating a possible non-magnetic phase precipitated along the microstructure.

Acknowledgments

The authors would like to thank to CAPES for the financial support and also to the Federal Fluminense Univeristy where the Barkhausen measurements were carried out.

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