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Microstructural Analysis of Co-Free Maraging Steel Aged

Aline Castilho Rodrigues¹, Heide Heloise Bernardi¹, Jorge Otubo²

ABSTRACT: Maraging steels have low carbon content and are highly alloyed, having as main feature the ability to increase the mechanical strength after thermal aging. Therefore, the objective of this work is to analyze the effect of aging in a Co-free maraging steel, Fe – 0.014% C – 0.3% Mn – 3.9% Mo – 2.1% Cu – 0.19% Si – 11.8% Cr – 9.1% Ni – 1.0% Ti (wt), at different heat treatment times (10 min - 960 min) at constant temperature (550°C), after cold rolling up to 66% and 77% area reduction. Microstructures were analyzed by scanning electron microscopy (SEM - EDS) and mechanical properties by Vickers hardness measurements. The results showed a significant increase in the hardness of the material after aging heat treatment on the solution treated and deformed samples. The aging heat treatment which was harder about 650 HV was 550°C/60 min.

KEYWORDS: Maraging steel, Cold rolling, Hardness test Aging.

INTRODUCTION

Maraging steel was developed in the 1950's, to support the demand for a low density and temperature resistant steel (250°C – 300°C), for improving aircraft performance. This new material was created adding aluminum (Al) and titanium (Ti) in steel with nickel (Ni). In the 1960's, cobalt (Co) and molybdenum (Mo) were added into the composition of maraging steels. Clarence George Bieber, in International Nicky Company, studied the addition of these elements in the alloy and concluded that there is a significant increasing in mechanical resistance (Lopes, 2007; Méndez, 2000). The classification of commercial maraging steels, according to the percentage of nickel, is: 18Ni (250), 18Ni (300) and 18Ni (350) (Lopes, 2007).

Maraging steel received this name due to two words: martensite and aging, which mean martensite aged. Low carbon and alloyed, the maraging steels are capable of increasing their mechanical resistance and hardness after aging heat treatments. The treated solution state has a martensitic structure with high ductility and toughness, and it can be modified after aging heat treatments (Cardoso *et al.*, 2013a). Due to the ductility of martensite, the maraging steel allows cold mechanical forming, not requiring intermediate heat treatment. A mechanical processing technique that can be used in maraging steel is cold rolling.

During the cold rolling, plastic deformation and increase of hardness occur, therefore, there is an increasing of dislocation density and crystalline defects, hindering the movement between them. Due to the plastic deformation, there is an increase in tensile strength and yield point (Callister, 2012). On the other hand, these mechanical properties can also be improved by thermal aging, in which the formation of intermetallic precipitates occurs. In Table 1, the types of precipitates which may be formed

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Table 1. Precipitates hardeners formed during the aging of maraging steels (Padial *et al.*, 2000).

Phase	Crystalline Structure
Ni ₃ Mo	Orthorhombic
Ni ₃ Ti	Hexagonal ordered
Ni ₃ V	Hexagonal compact
Ni ₃ W	Orthorhombic
Fe ₂ (Mo, Ti)	Type hexagonal laves
FeMo	Tetragonal
FeTi	CsCl type cubic
Fe ₇ Mo ₄	Hexagonal
R (Mo-Co-Cr)	Hexagonal rhombic
X (Fe-Cr-Mo)	Body-centered cubic

on maraging steels, depending on the alloy composition, are shown (Padial *et al.*, 2000).

Steel with martensitic structure, when subjected to a heat treatment at a constant temperature, suffers a growth of intermetallic precipitates uniformly in the matrix (Carvalho *et al.*, 2013). The precipitate size is a contributing factor to the increased mechanical resistance of maraging steels. The smaller the precipitates and the more coherent with the matrix, the greater the hardness of the alloy, since there is less space between the particles; furthermore, they act as barriers to movement of dislocations (Padial *et al.*, 2000).

The alloying elements of maraging steel, such as molybdenum, titanium, nickel, cobalt, aluminum, and others, have a great influence on the mechanical properties of this alloy, in other words, they are responsible by precipitates hardeners (Carvalho *et al.*, 2013). However, recent studies (Hu *et al.*, 2008; Leitner *et al.*, 2011; Mahmoudi *et al.*, 2011; Mahmudi *et al.*, 2011; Nili-Ahmadabadi, 2008; Schnitzer *et al.*, 2010; Sha *et al.*, 2013) are being conducted in maraging steels without cobalt addition, in order to reduce the production costs of this alloy. Maraging steels are considered expensive due to their materials' preparation and expensive alloy elements such as nickel and cobalt processes. The elimination of the cobalt content and the substitution of nickel by cheaper elements, such as manganese, has been studied. Cobalt is used only to minimize the solubility of molybdenum. In the case of maraging steel studied, this element was replaced by chrome, which besides

improving the hardenability increases the corrosion resistance (Nili-Ahmadabadi, 2008).

Maraging steels are mainly applied in the aeronautical, aerospace, nuclear and military industries, as they have great advantages such as good weldability, high strength, high yield strength, high fracture toughness, they support high working temperatures, have good machinability, good formability, among others, with great applicability matrices and tools (Lopes, 2007).

High-tensile steels such as 300M and SAE 4340 have extensive application in aeronautics and aerospace industry, being mainly applied in the manufacture of structural components (Zang *et al.*, 2013; Boakye-Yiadom *et al.*, 2014).

Maraging steels are being investigated because of the possibility of replacing the commercial alloy steels, 300M and 4340 for example, widely used in aeronautical industry (Carvalho *et al.*, 2013). Thus, this study aims to examine the effect of aging heat treatment in non-commercial maraging steel (Co-free), in order to compare it to the 300M and 4340 steels.

EXPERIMENTAL

The maraging steel used in this work was produced by vacuum induction melting. The ingots was hot forged and then hot rolled until we obtained bars of 20x20 mm of dimension and chemical composition: Fe – 0.014% C – 0.3% Mn – 3.9% Mo – 2.1% Cu – 0.19% Si – 11.8% Cr – 9.1% Ni – 1.0% Ti (wt.%).

Its bar (20x20 mm) was separated into two parts resulting in two conditions:

- Condition I (C-I): bar dimension of 20 x 20 x 10 mm;
- Condition II (C-II): bars of 20 x 20 x 10 mm (~100 wt.), remelted (1 remelt) on an arc furnace, resulting in an ingot dimension of 16x10x95 mm.

Both conditions (C-I and C-II) were solution treated at 1050°C during 1 hour, quenched in water at room temperature and then cold rolled to a plate of 3 mm in thickness, resulting in 66 and 77 % of area reduction (AR) for C-I and C-II, respectively. The deformed samples (AR = 66 and 77 %) were cut in several parts and each part was annealed at 550°C for different times, ranging from 10 to 960 min.

Microstructural characterization was performed using a Scanning Electron Microscope (SEM) VEGA 3 TESCAN model, equipped with an energy dispersive spectrometer system (EDS). Vickers hardness

testing was performed using a Future-Tech FM-7000 microindenter with a load of 200 g/15s, in which the results correspond to the mean of ten values taken on each specimen. The micrograph analyses and the Vickers' hardness were undertaken in a longitudinal section of the samples (wire rolling direction).

RESULTS AND DISCUSSION

Figure 1 shows the hardness variation as a function of heat treatment time for maraging steel with AR's = 66 and 77 %. For solution treated (C-I and C-II) and deformed samples, the hardness values are represented at zero time in Fig. 1.

Solution treated samples (C-I and C-II) showed average hardness values of 250 and 300 HV, respectively. Comparing the solution treated to the deformed condition, it is observed an increase around 50 % in average hardness values ($HV_{66\%} = 425$

and $HV_{77\%} = 407$), corresponding to the effect of work hardening. About thermal aging, there is a significant increase in hardness at relatively low heat treating times (Fig. 1). For both area reductions, increased hardness around 200 HV occurs with only 10 min thermal aging. He *et al.* (2002) observed that the heat treatment at 470°C performed in a maraging steel with 18%wt. Ni, after forging, has achieved a 90% increase in hardness, with only 15 min of aging.

In the detail of Fig. 1, it is observed that the maximum hardness is obtained at 550°C/60 min for both ARs. On the other hand, the thermal aging at 550°C exhibits a decrease in hardness values after 240 min for deformed sample with AR = 66%, whereas a decrease it is observed after 90 min to condition AR = 77%. Therefore, it can be said that in smaller ARs, there is an increase in aging time of the alloy, delaying the onset of overaging. Based on the average hardness values, the processing in the matrix, resulting in a lath martensitic structure (Mahmoudi *et al.*, 2011; Hu and Wang, 2012), as shown in Figs. 2a and 2b. Co-free maraging steel is stable up to 16 hours of aging at 550°C.

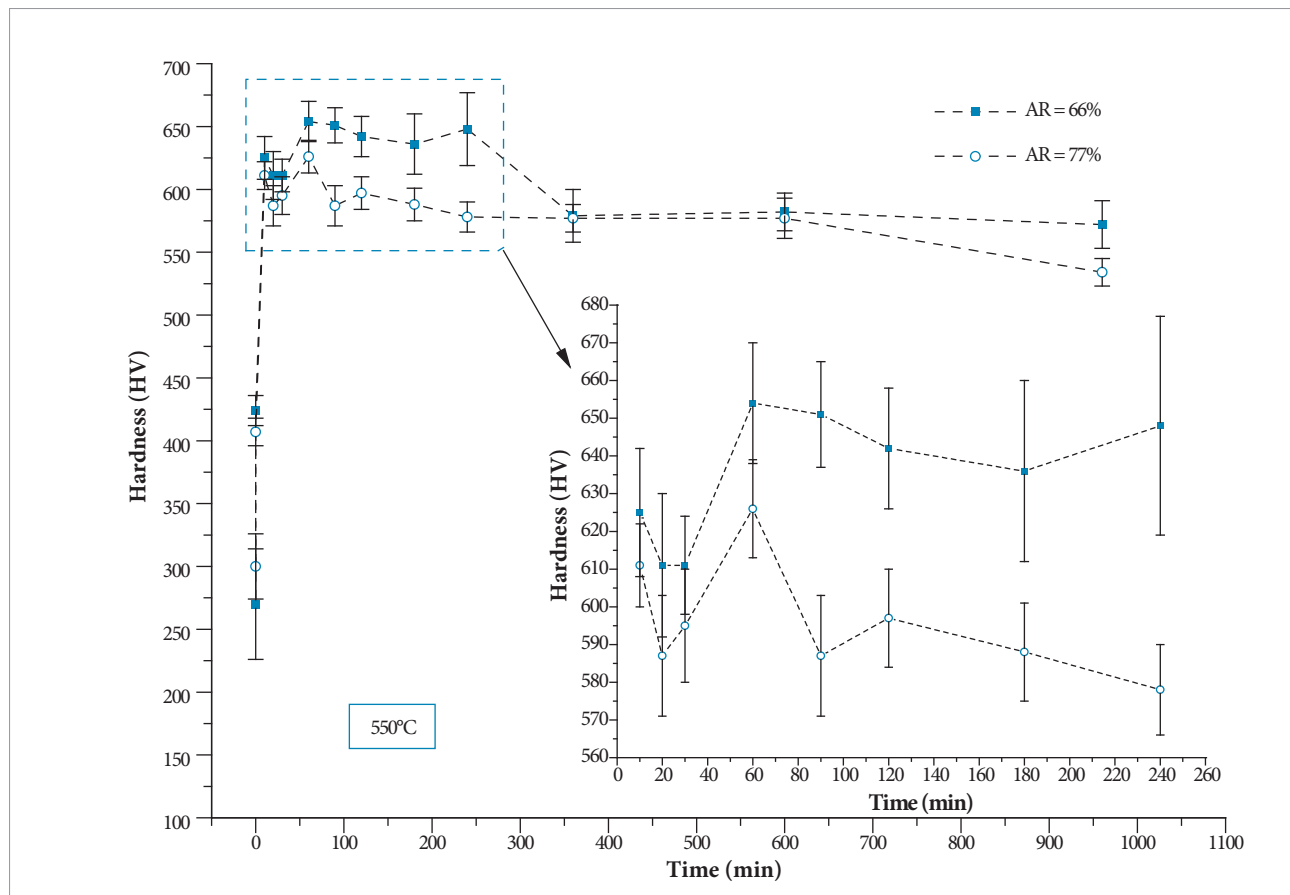


Figure 1. Annealing behavior of the maraging steel deformed with AR's = 66% and 77%.

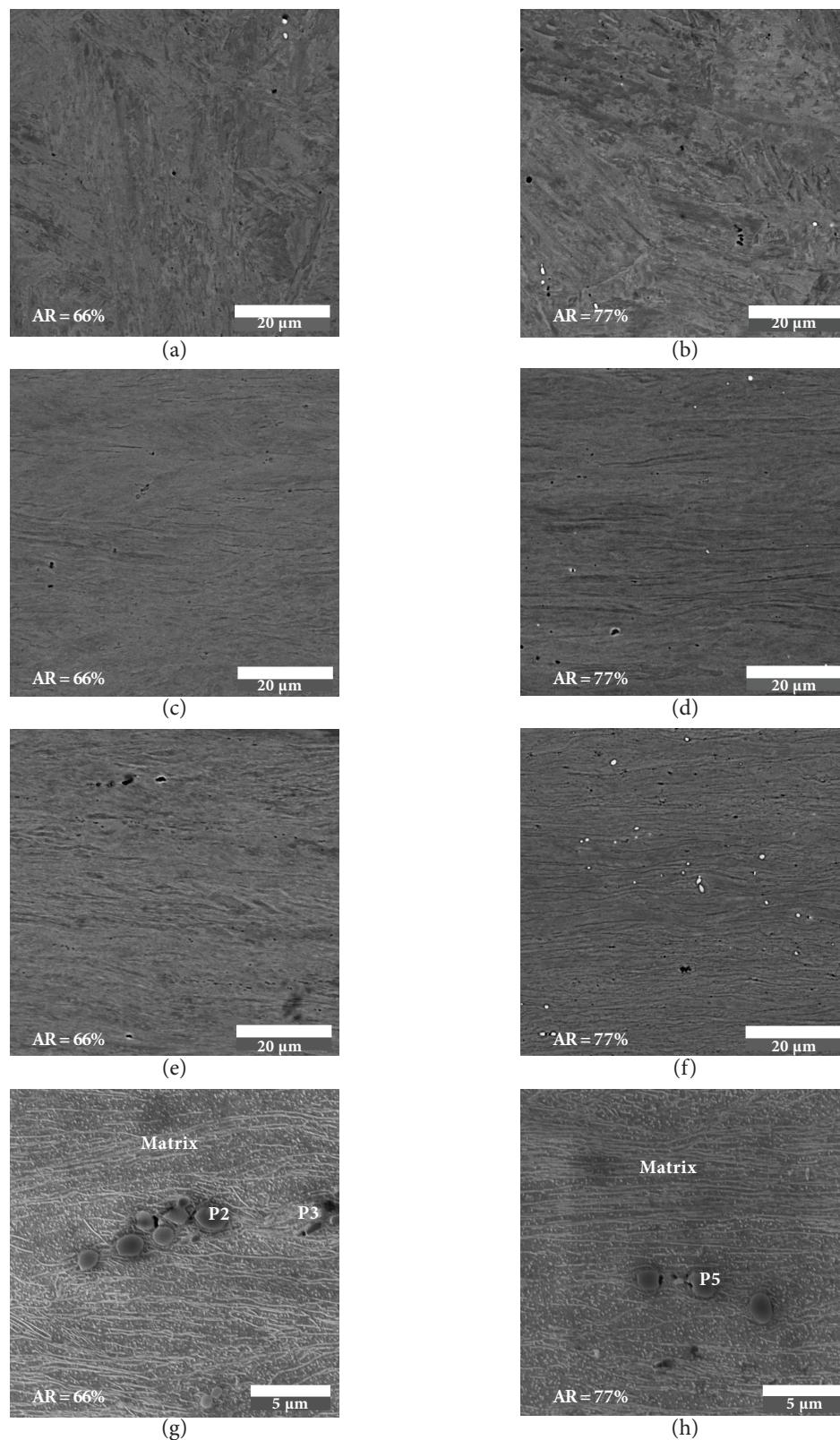


Figure 2. Microstructures of maraging steel under the conditions (a); (b) solution treated; (c); (d) deformed; (e); (f) aging at 550°C/1h (SEM-BSE) and (g); (h) aging at 550°C/16h (SEM-SE).

Figure 2 shows the microstructure of the maraging steel at different conditions. It is observed for solution treated sample (C-I and C-II) that the solution heat treatment at 1050°C for 1h was effective in dissolving primary precipitates (formed during the early stages of maraging steel

After thermal aging at 550°C for 1 hour (Figs. 2e and 2f), it can be seen that the microstructure is similar to deformed samples in AR = 66 and 77 % (Figs. 2c and 2d). This heat treatment time showed to be effective in relation to increased hardness (Fig. 1), due to presence of fine precipitates dispersed in the matrix, which contribute to the increase of this mechanical property (Hu and Wang, 2012). However, the technique of scanning electron microscopy, used in order to analyze the microstructure of this alloy, is not efficient to identify them in the matrix.

Increasing the annealing time to 16 h (Figures 2g and 2h) promoted an increment in precipitation rate. These precipitates grew coarser in subsequent higher annealing times, but there still are fine precipitates elongated in the rolling direction, and few precipitates changed from elongate morphology to a spherical one. In this condition, it is observed some coalescence of precipitates having sizes of the order of 2 µm. Regardless of AR, the precipitates have similar morphologies. The EDS analysis (Table 2) showed that those precipitates, referred as P2 and P5, were depleted in Ni, and enriched in Mo and Ti (Figures 2g and 2h).

In the analysis performed on the matrix — points 1 and 4 in Figs. 2g and 2h, respectively —, compared to the nominal composition of the alloy, it can be said that the rolling processes or thermal treatments did not significantly alter the composition of the material in terms of Mo, Ti, Cr and Ni elements.

In relation to precipitates P2 and P5, a large increase of Mo and Ti was observed. This alloy contains low nickel, so it is not possible to stimulate the formation of Ni_3Mo or Ni_3Ti phases, which usually appear in the commercial maraging steels 250 and 350 (Padial *et al.*, 2000). The presence of cobalt element in commercial alloys promotes the formation of Ni_3Mo . On the other hand, the maraging steel study in this work does not have Co (Co-free) and low Ni content. However, the alloy phase formed in this alloy can be $Fe_2(Mo, Ti)$, $FeMo$, $FeTi$ or Fe_7Mo_4 (Padial *et al.*, 2000). The precipitate P3 features a larger enrichment of Ti in relation to P2 and P5, probably, the Ti-rich precipitates are carbides (TiC) or Fe_2Ti , $FeTi$ (Castanheira *et al.*, 2006). The TiC is formed by carbon, which is characterized as

Table 2. Chemical compositions of the constituent phases (wt. %).

Constituent	Mo	Ti	Ni	Cr
Composition	3.9	1.0	9.1	11.8
Matrix 1	4.6	1.2	9.2	12.1
Precipitate 2 (P2)	16.0	3.0	4.7	14.1
Precipitate 3 (P3)	7.6	12.5	6.4	9.9
Matrix 4	4.5	1.0	9.0	12.5
Precipitate 5 (P5)	14.5	2.6	5.4	14.8

an impurity of the material (Padial *et al.*, 2000). Furthermore, other techniques are necessary, for example, an X-ray diffraction to determine the type of precipitate formed.

The SAE 4340 and 300M steels are considered high-tensile steels, low-carbon and low-alloy steels, being used in the aerospace industry due to their excellent mechanical properties. Commercial maraging steels are being studied in order to replace the 4340 and 300M steels, especially in the aeronautical and aerospace industry. According to studies by Cardoso *et al.* (2013b), the SAE 4340 and 300M steels can achieve hardness values of 250 HV and 350 HV, respectively, after drawing back. However, the maraging steel studied with forming processes and appropriate heat treatment can achieve hardness values around 670 HV, i.e., almost double the amount reported by the 300M steel.

CONCLUSION

Based on this investigation, the following conclusions can be drawn:

- The cold forming contributes to mechanical hardening, however an effective increase of hardness is obtained after thermal aging;
- The hardness increased significantly with low time thermal aging at 550°C due to precipitation of the second phase particles. The maximum value obtained was of 650 HV at 550°C/60 min;
- Based on the average hardness values, the Co-free maraging steel is stable for up to 16 hours of aging at

550°C. It is possible to note a subtle decrease in the hardness values for both AR, due to coarsening of precipitates;

- Appropriate thermal aging promoted the precipitation of the second phase particles, rich in Mo and Ti.

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