

Kinetics of Martensite Reversion to Austenite during Overaging in a Maraging 350 Steel

Leandro Gomes de CARVALHO,^{1)*} Ronald Lesley PLAUT,¹⁾ Nelson Batista de LIMA²⁾ and Angelo Fernando PADILHA¹⁾

1) Escola Politécnica da Universidade de São Paulo-EPUSP, São Paulo, Av. Prof. Mello Moraes 2463, 05508-030, Brazil.

2) Instituto de Pesquisas Energéticas e Nucleares – IPEN, São Paulo, Av. Prof. Lineu Prestes 2242, 05508-000, Brazil.

(Received on September 26, 2018; accepted on January 16, 2019)

The present work has studied the kinetics of partial reversion of martensite into austenite, which occurred during overaging between 520 and 600°C in a maraging 350 steel. The microstructural modifications were followed using optical microscopy, scanning electron microscopy, X-ray diffraction and ferritoscopy. The observations carried out using microscopy showed that the reverted austenite is formed at interface regions such as grain boundaries, boundaries of the packets and martensite laths boundaries of the martensitic structure starting at 520°C, while the reverted austenite observed inside the martensite lath was formed starting at 560°C. The maximum percentage volumetric fractions of retained austenite were 18%, 25% and 37%, respectively for 520, 560 and 600°C. The higher the aging temperature, the faster the volumetric fraction attained its plateau of maximum constant value. The activation energy for the kinetics of the austenite reversion process has been determined as being 332 kJ/mol. The Avrami constant for the kinetics of the reverted austenite at 520°C was close to 1, while at 560°C was close to 2. The values determined for the activation energy and of the Avrami constants have been discussed and correlated with the possible transformation mechanisms.

KEY WORDS: maraging steel; phase transformation; kinetics; reverted austenite.

1. Introduction

Maraging steels are a special class of high strength steels that are hardened by precipitation reactions induced by alloying elements that promote the formation of intermetallic phases in a martensitic structure, during the aging treatment.^{1–3)} After extended aging times or temperatures higher than 500°C, changes in the mechanical or magnetic properties of the maraging steels can occur due to the presence of austenite.^{1,2,4–9)} The yield stress and the ultimate stress can decrease due to the presence of the austenite, while the fracture strength can increase.^{1,6,8,9)} The coercive field tends to grow with the increase of the austenite proportion, while the magnetic saturation decreases due to the presence of this phase.^{4,5)}

The austenite reversion in the maraging steels is normally attributed to the local enrichment of nickel of the body-centred lattice of the matrix. This enrichment is normally associated with the dissolution of precipitates of the type Ni₃X (X = Mo, Ti) and to the formation of intermetallic Fe–Mo compounds during overaging (Fe₇Mo₆ or Fe₂Mo).^{1,6,10–12)} Several researchers observed that the formation of austenite and of the Fe₂Mo precipitates occurred approximately at the same time. They suggested that the nickel arises from the dissolution of the Ni₃(Mo,Ti)

and would be used in the formation of the austenite reversion and the molybdenum used in the formation of the Fe₂Mo.^{1,6,11,13,14)} Studies performed by Moshka and coauthors¹⁵⁾ and by Shmulevitsh and coauthors¹⁶⁾ showed that the Ni₃Mo precipitate is thermodynamically more unstable than the Ni₃Ti precipitate. In this way, it is more probable that the dissolution of the Ni₃Mo precipitate may give origin to the reverted austenite and to the Fe–Mo precipitates simultaneously.

Apart from the presence of thick precipitates with the extended times or higher aging temperatures, the reverted austenite can present morphological changes with the increase of temperature and of the overaging treatment time.^{6,11,13)} One of these is that the inter-lath austenite that nucleates on the martensite lath boundaries, while the other type of austenite, known as reverted intra-lath austenite, grows inside the martensite lath in the form of Widmanstätten plates with twins.^{6,11,13,17)} Hosomi and coauthors and Carvalho and coauthors also identified two mechanisms of martensite reversion to austenite during continuous heating rates. A first mechanism attributed to partitioning to solute rich and solute poor regions due to short range diffusion and another mechanism linked to shear-like martensitic transformation.^{7,18)}

Previous research works have shown that the volumetric fraction of austenite has an increasing behaviour with the time and aging temperature for samples treated at temperatures higher than 500°C or for extended aging times.^{1,4–6)}

* Corresponding author: E-mail: leandro.carvalho@usp.br
DOI: <https://doi.org/10.2355/isijinternational.ISIJINT-2018-610>

The following of the reverted austenite global kinetics can be realised through the determination of the volume fraction in overaged samples, through the analysis of the X-ray diffraction patterns or even through the changes in magnetic properties of the coercive field and the magnetic saturation, since austenite is a paramagnetic phase.^{4,5,19–22} Habiby and coauthors²³ also verified that there is a relationship between the eddy current (Foucault current) variation and the austenite volumetric fraction, which can be used to follow up the kinetics of the martensite reversion.

Despite the behavior of the reverted austenite volume fraction with time and temperature has been analyzed by Pardal and coauthors for the maraging 300 steel¹⁹ and by Peters and coauthors for some experimental maraging steels,²⁴ no data has been found for the maraging 350 steel. Furthermore, it has been verified that a large number of research work has been related to the kinetics of precipitation.^{25–29} However, there is a shortage of detailed studies related to the kinetics of the reverted austenite during overaging, especially for the maraging 350 steel. Thus, this work analyses the microstructural modifications due to overaging thermal treatments carried out in the temperature range of 520 to 600°C in a maraging 350 steel, both at a local level – through optical microscopy and scanning electron microscopy, as well as at a global level – through X-ray diffraction analysis and ferritometry. Also, determinations of the activation energy and the Avrami “n” constants for the austenite reversion kinetics, have also been performed.

2. Materials and Methods

Samples were prepared by cutting a disk of 10 mm in thickness from maraging 350 steel bars of 140 mm in diameter, supplied in the solution annealed condition. The chemical composition of the bars is given in **Table 1**.

This disk was cut into samples of approximately 5 mm × 10 mm × 10 mm for the aging experiments. Each sample was aged between 520 and 600°C at various times from 6 min (0.1 h) to 18 h in a muffle furnace.

The microstructural investigations were carried out using an optical microscope Olympus BX60M and a scanning electron microscope (SEM) FEI Quanta 450FEG at an operation voltage of 20 kV. Samples for SEM were mechanically polished and were etched with Nital 2% reagent or Marble reagent, while the samples for optical microscopy were etched with Marble reagent.

X-ray diffraction analysis was carried out in heat treated samples at 520, 560, and 600°C for 1, and 18 hours. X-ray measurements were performed using a diffractometer X’Pert Pro, in step scan mode with a step size of 0.02°, time steps of 30 seconds and angular (2θ) interval 40°–100°. Radiation CuKα with wavelength 1.5405 Å was used with 40 kV and 40 mA. Spinner was used improving grain statistics per

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

Ni	Co	Mo	Ti	Al	S*	C*	O*	N*
18.16	11.92	4.81	1.22	0.074	30	30	8	2.0

*in ppm

measurement in X-ray diffraction patterns. The martensite and austenite lattice parameters, as well as the austenite volumetric fractions, were obtained with the aid of the Rietveld analysis using GSAS software.³⁰

Feritscope usually is applied to measure ferromagnetic phase content, especially in austenitic, duplex, and superduplex stainless steels.^{31,32} Feritscope measurements are based on magnetic induction method.³² In the present work, Fischer MP3 Feritscope was used to analyse the effect of aging heat treating on the kinetics of reverted austenite during overaging. It must be pointed out that the technical literature is somewhat confusing about the terms related to ferritometry. Throughout this paper, we will use feritscope when mentioning the equipment supplier, ferritometry when mentioning the equipment and ferritometry when mentioning the measuring technique.

3. Results and Discussion

This item of results and discussion has been subdivided into three main parts, namely: X-ray diffraction analysis and by ferritometry (3.1); microstructural analysis using optical microscopy and scanning electron microscopy (3.2); kinetic analysis of the reversion of martensite into austenite (3.3).

3.1. X-ray Diffraction and Feritscope Measurements for Maraging 350 Steel

The X-ray diffractogram presented in **Fig. 1** shows that according to temperature and aging time there is an increase in the austenite amount for the samples aged at 520, 560, and 600°C for 1 and 18 h. This can be seen (qualitatively), by comparing the areas of the austenite peaks $\gamma(200)$, $\gamma(220)$, $\gamma(311)$ and $\gamma(222)$ with the areas of the martensite peaks $\alpha'(110)$, $\alpha'(200)$, $\alpha'(211)$ and $\alpha'(220)$ for several heat treatment conditions. The values of the austenite volume fraction, austenite and martensite lattice parameters calculated from the diffractograms, based on the Rietveld method, are presented in **Table 2**.

In **Table 2** it may be observed that the austenite lattice parameter decreases with increasing aging temperature for isochronous treatments between 520 and 560°C for 1 h

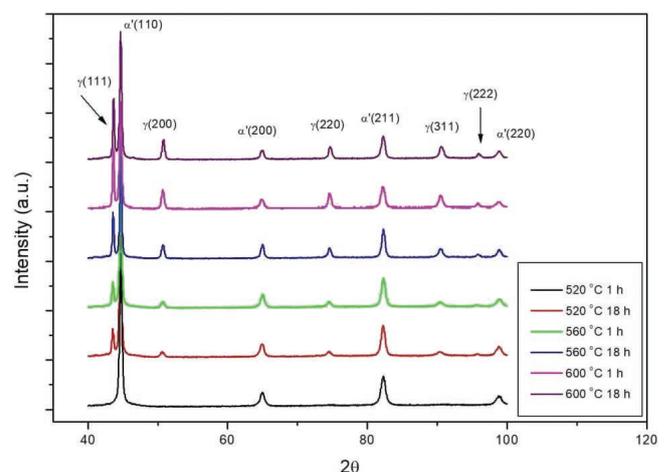


Fig. 1. X-ray diffractogram of samples in the as-received condition and aged at 520, 560 and 600°C for 1 and 18 h for the maraging 350 steel. (Online version in color.)

Table 2. Values of austenite volume fraction, austenite and martensite lattice parameter determined by the Rietveld method for the maraging 350 steel.

Heat treating	Austenite volumetric fraction (%)	Austenite lattice parameter (Å)	Martensite lattice parameter (Å)	Chi ²
520°C for 1 h	0.0 ± 1.0	–	2.8755 ± 0.0005	3.2
520°C for 18 h	14.0 ± 1.0	3.6051 ± 0.0005	2.8749 ± 0.0005	4.2
560°C for 1 h	17.0 ± 1.0	3.6042 ± 0.0005	2.8732 ± 0.0005	4.6
560°C for 18 h	30.0 ± 1.0	3.6020 ± 0.0005	2.8731 ± 0.0005	3.8
600°C for 1 h	36.0 ± 1.0	3.6041 ± 0.0005	2.8765 ± 0.0005	4.3
600°C for 18 h	34.0 ± 1.0	3.6002 ± 0.0005	2.8752 ± 0.0005	4.5

and 18 h in the Rietveld method. This fact occurs due to the change in chemical composition of the austenite with aging temperature, as has been observed by Pardal and coauthors¹⁹⁾ and by Li and Yin,¹¹⁾ which minimises the distortion of the lattice parameter. Furthermore, Pardal and coauthors¹⁹⁾ also observed the increase in the proportion of austenite in a maraging 300 due to the increase in temperature and aging time.

The calculated martensite lattice parameter presented a decrease with the increase in aging temperature up to 560°C, as seen from Table 2. That aspect has also been observed by Habiby and coauthors,³³⁾ in a maraging 350 steel for samples treated up to 650°C, and by Pardal and coauthors¹⁹⁾ in a maraging 300 for samples aged at 560 and 600°C. The decrease in the lattice parameter is due to the precipitation of substitutional atoms that were inside the martensite supersaturated lattice structure, decreasing the lattice distortion.³³⁾

Apart from the influence of the X-ray diffraction measurements, Fig. 2 shows that there is a significant effect of the time and aging temperature on the magnetic measurements performed with the ferriscope (m_f) used to follow the evolution of the kinetics of the martensite reversion, which may indicate that the proportion of austenite increases since austenite is a paramagnetic phase. In this manner, a relationship has been verified between the ferromagnetic measurements performed using the ferriscope and the volume fraction calculated by the Rietveld X-ray diffraction in the maraging 350 steel, as shown in Fig. 3.

The values of the determination coefficient R^2 of the adjusted second order polynomial function was 0.94. This polynomial function adjusted to the experimental data represents the relationship between the austenite volume fraction (V_v) and the ferriscope measurements (m_f), is given by Eq. (1).

$$V_v = -170.97 + 7.97 m_f - 0.08 m_f^2 \dots\dots\dots (1)$$

Pardal and coauthors⁵⁾ also observed that a second order polynomial function best adjusted to their experimental results for magnetic saturation and the volume fraction, which has been attributed to the changes in chemical composition of the austenite and of the martensite that influenced the magnetic properties. The result of Eq. (1) is a novelty of present work due to ferriscope measurements to be usually employed in the microstructure analysis of stainless steel.^{31,32)} Based on the polynomial coefficients of adjustment and the ferriscope measurements, it may be

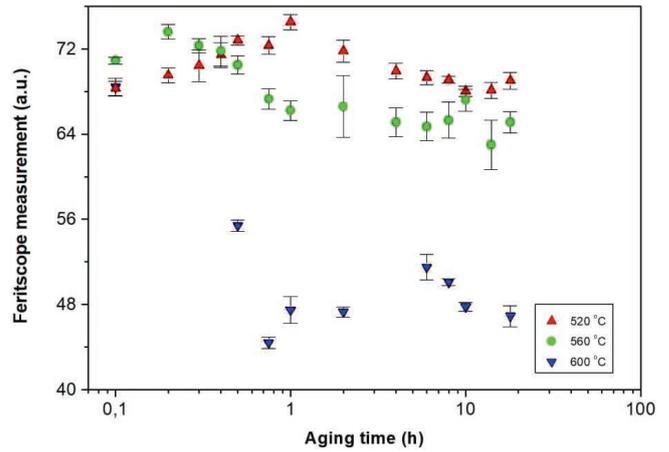


Fig. 2. Variation of ferriscope measurements as a function of aging time for 520, 560 and 600°C in a maraging 350 steel. (Online version in color.)

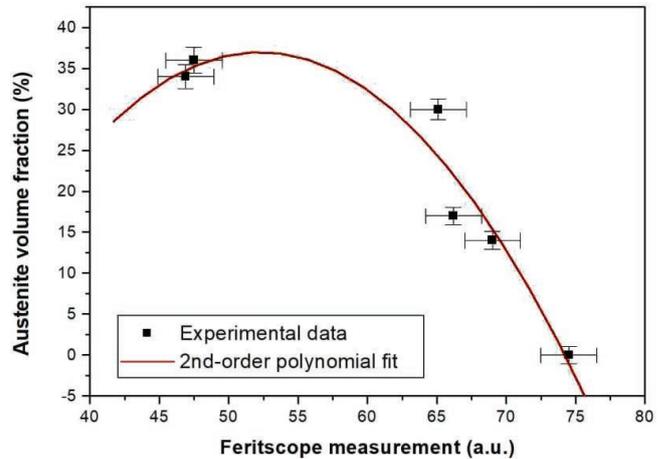


Fig. 3. Relationship between ferriscope measurements and the austenite volume fraction determined by X-ray diffraction for a maraging 350 steel. (Online version in color.)

possible to obtain the austenite volume fraction as a function of aging time, as shown in Fig. 4.

From Fig. 4 it may be observed that the aging temperature strongly influences the kinetics of the reverted austenite. The higher the aging temperature, the faster the volume fraction attains a steady plateau, which may be observed when comparing the behaviour of the austenite volume fraction as a function of aging time for 520, 560 and 600°C. Such an asymptotic behaviour has also been observed in overaged samples of maraging 300 and 400 steels.^{19,34,35)}

In Fig. 4 it also may be observed that the maximum reverted austenite volume fraction for 520, 560 and 600°C are respectively 18%, 25% and 37% for the analysed maraging 350 steel. Pardal and coauthors¹⁹⁾ showed that the maximum austenite volume fraction for a maraging 300 steel for samples treated at 560 and 600°C were, respectively 29.7% and 41%. Thermodynamic calculations performed by Guo and Sha³⁵⁾ also indicated that the maraging 250 steel presented an equilibrium austenite volume fraction of 41% for 427°C, 47% for 482°C and 55% for 538°C. This is could be linked with the influence of chemical composition of maraging steels on thermodynamic stability of phases. Peters²⁴⁾ also showed that for a Fe-18%Ni alloy had a retarding

effect of cobalt on the austenite formation kinetics during overaging. These facts show an influence of the chemical composition on the retained austenite kinetics in maraging steels for isothermal treatments. Previous dilatometric studies comparing the 300 and 350 maraging steels also showed the influence of the chemical composition of these two types on the martensite reversion during continuous heating.¹⁴⁾ Furthermore, the influence of chemical composition on the martensite reversion, the influence of titanium content on mechanical properties of maraging 300 steel was also

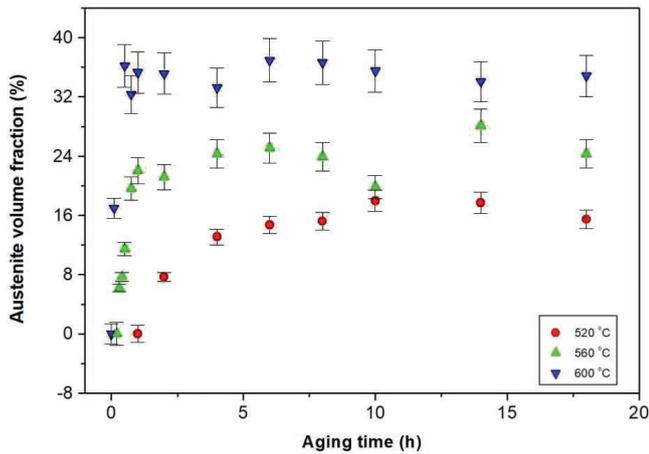


Fig. 4. Variation of austenite volume fraction determined by feritscope measurement and polynomial fitting as a function of aging time and temperature for maraging 350 steel. (Online version in color.)

observed by Hosseini and Arab.³⁷⁾

3.2. Microstructure Analysis After Overaging for Maraging 350 Steel

Figure 5 shows that the reverted austenite comprises the lighter areas of the micrograph obtained after etching with the Marble reagent, while the martensite lath is associated with the darker areas. From the comparative analysis of the micrographs, it may be stated qualitatively that there is an increase in the volume fraction with the increase of aging time in samples treated at 600°C, specially when comparing Figs. 5(b), 5(c) and 5(d) in relation to **Fig. 10(a)** (see also Fig. 4).¹⁹⁾

Figure 6(a) presents the micrograph of maraging 350 steel aged at 520°C for 18 min which indicates that reverted austenite is not formed in the regions of lath boundaries. This fact evidences that exists an incubation time for martensite to austenite transformation, which was observed for X-ray diffraction measurements and feritscope (see Figs. 1 and 4). This behaviour could be linked with the stability of the intermetallic phases formed during aging.^{6,15)} Figures 6(b), and 6(c) show that inter-lath austenite, identified as γ_{inter} , is nucleated and grows on lath martensite boundaries in samples aged at 520°C for 2 and 4 hours.¹³⁾ The values of austenite volume fraction determined by feritscope measurement and polynomial fitting were 0%, 7.9% and 13.3%, respectively, for samples aged at 520°C for 18 min, 2 h and 4 h (see Fig. 4).

The backscattered electron images that are shown in Figs. 6(d), 6(e) and 6(f) indicate that reverted austenite is

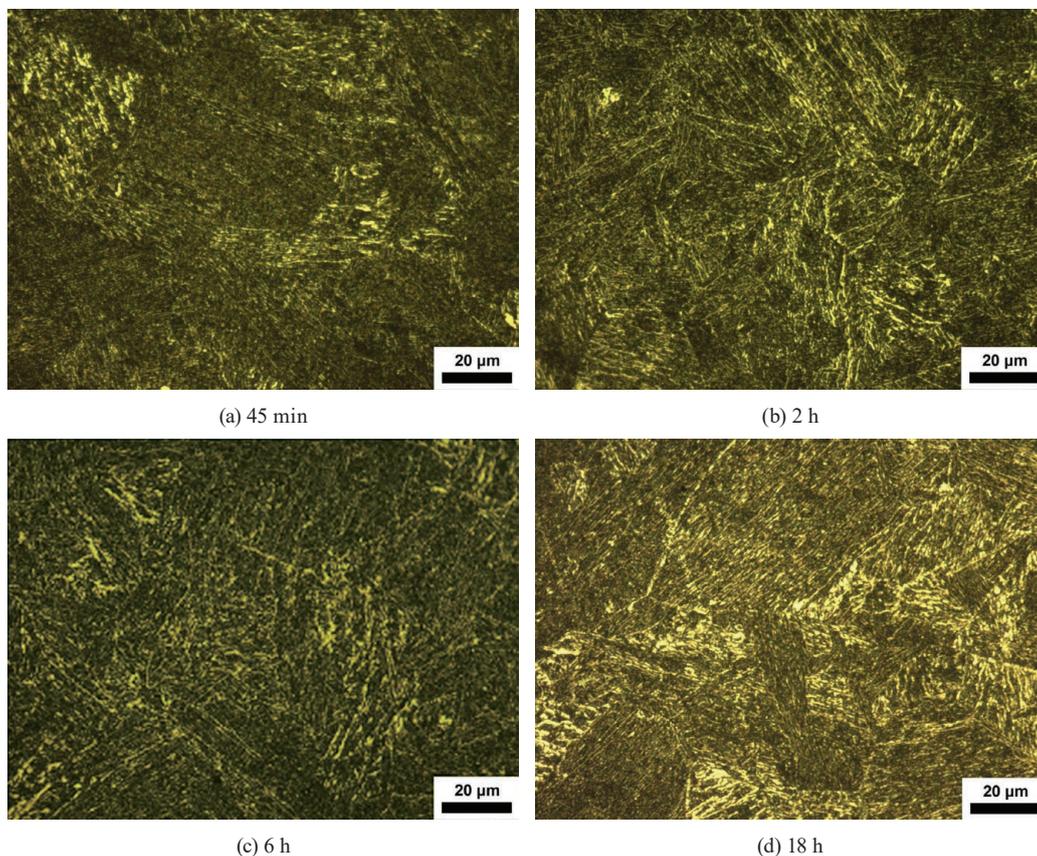


Fig. 5. Microstructure of the maraging 350 steel revealed by the Marble reagent and obtained in an optical microscope for samples aged at 600°C for 45 min (a), 2 h (b), 6 h (c) e 18 h (d). (Online version in color.)

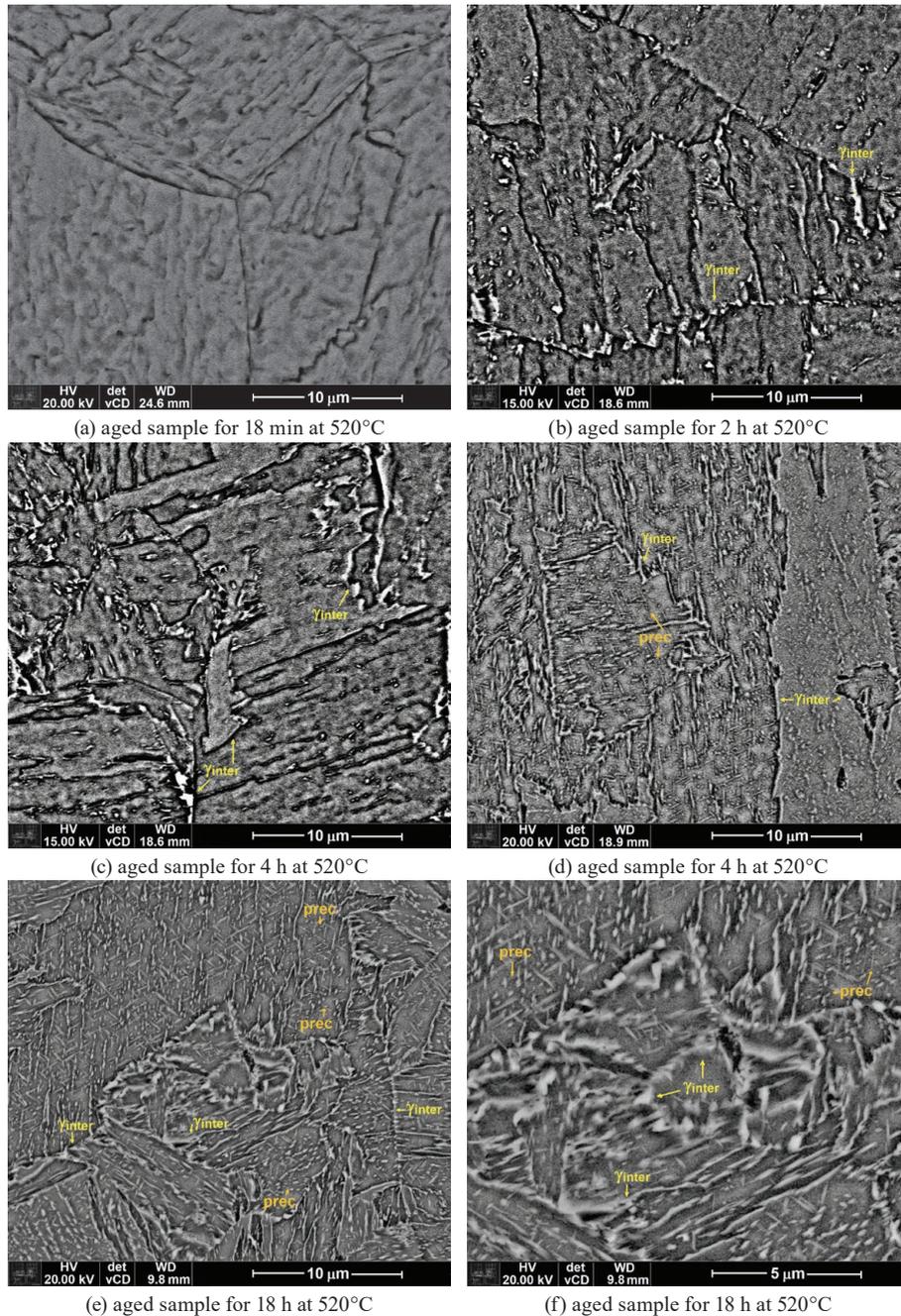


Fig. 6. Backscattered electron image of the maraging 350 steel using 2% Nital in samples aged at 520°C for 18 min, 2 h, 4 h, 10 h, and 18 h. (Online version in color.)

formed in the regions of lath boundaries and martensite packages, for samples aged at 520°C for 10, and 18 h, which is represented as γ_{inter} in these figures. No significant change has been observed on the austenite volume fraction for these samples, once these values reach a plateau of 18% (see Fig. 4). Moreover, inside the martensite laths, we may observe the presence of phases with a smaller size relative to the austenite, presenting ellipsoidal, cylindric and spheroidal morphologies, which could indicate the presence of the phases- Ni_3Ti , Ni_3Mo and Fe_2Mo , identified as prec in Figs. 6(d), 6(e) and 6(f).^{12,13,15)} It was also observed that the precipitates coarsened when comparing micrographs of samples aged at 520°C for 10 h and 18 h.

Figure 7 shows the presence of inter-lath austenite arranged in a well-defined direction, which indicates an orientation relationship between the austenite and the mar-

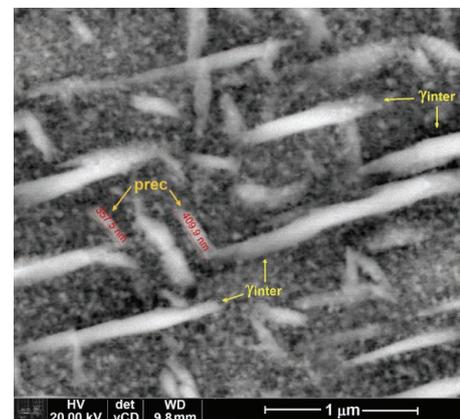


Fig. 7. Backscattered electron image of the microstructure of the maraging 350 steel using 2%Nital reagent in a sample aged at 520°C for 18 h. (Online version in color.)

tensite.¹³⁾ Further, in Fig. 7 we may observe the presence of phases of a nanometric size with ellipsoidal and cylindrical morphologies.^{6,13,15)}

Figures 8(a)–8(f) show also that the temperature and aging time affect the kinetics of the austenite formed on the martensite lath boundaries and the martensite packages in samples aged at 560 and 600°C. An explanation for such a behaviour is that an increase in temperature from 560°C to 600°C accelerates the diffusion rate of the alloying elements, leading to larger volumetric fractions for higher aging temperatures. Moreover, the extended aging times lead to larger austenite volume fractions¹⁹⁾ (see Fig. 4). Yamada and coauthors also show that austenite formed during overaging in Fe–Ni–Co–Mo alloys had higher nickel

and concentrations when compared with base composition of these alloys.³⁸⁾

Figures 9(a) and 9(b) illustrate the inter-lath formation of austenite starting from the reverted martensite in sample aged at 560°C for 18 h. By comparing the micrographs obtained for the sample aged at 520°C for 18 h (see Fig. 6(f)) and the sample aged at 560°C for 18 h, it may be observed that the austenite formed in the inter-lath martensite of the samples overaged at 560°C is thicker than the formed at 520°C. Even further, Figs. 9(a) and 9(b) show the formation of austenite needles of the Widmanstätten type in the interior of the martensite laths (identified as γ_{intra}), as well as the presence of smaller intermetallic precipitates.^{6,12,13)}

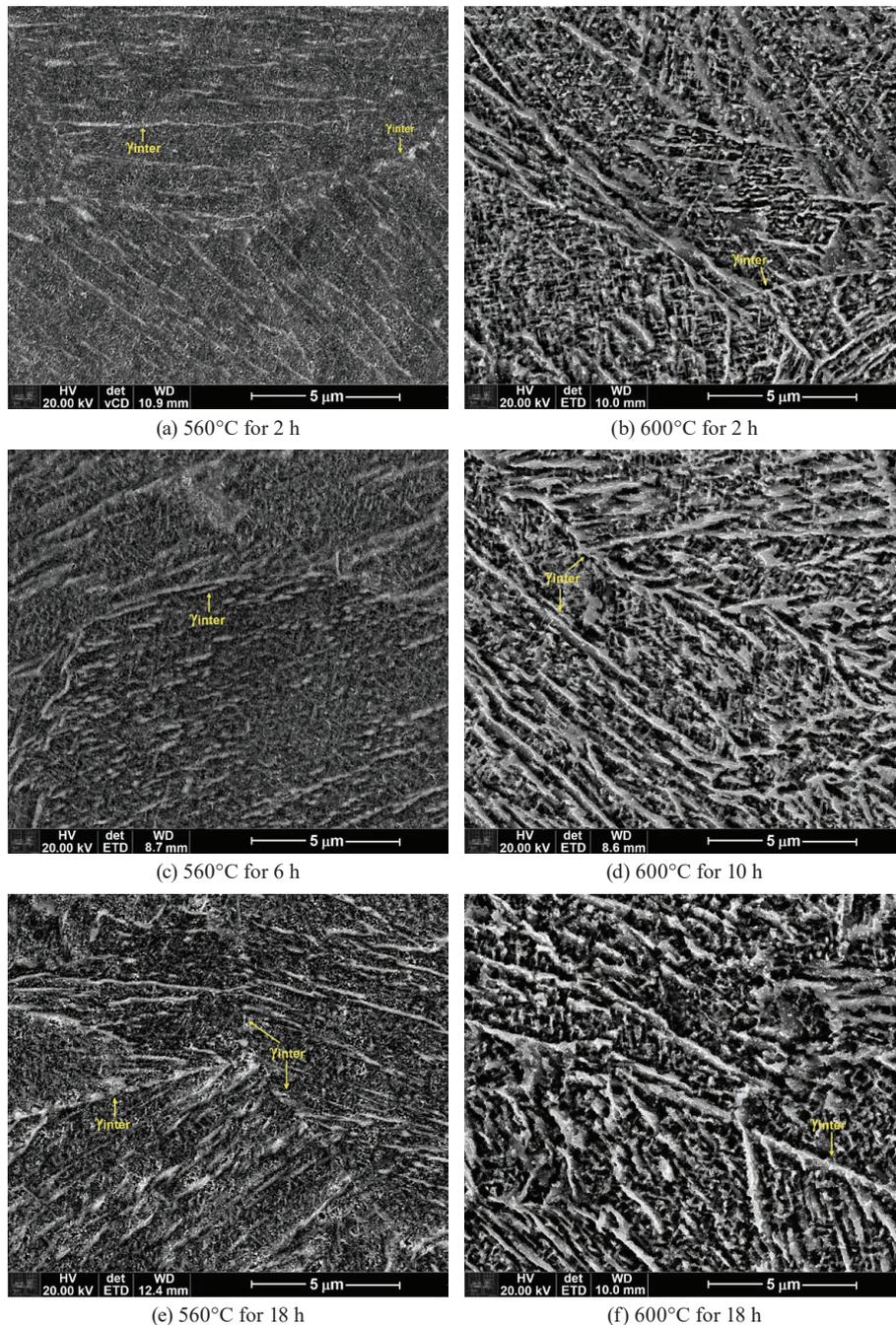


Fig. 8. Secondary electron images of the microstructure of the maraging 350 steel using the Marble reagent in samples aged at: 560°C for 2 h (a), 600°C for 2 h (b), 560°C for 6 h (c), 600°C for 6 h (d), 560°C for 18 h (e) and 600°C for 18 h (f). (Online version in color.)

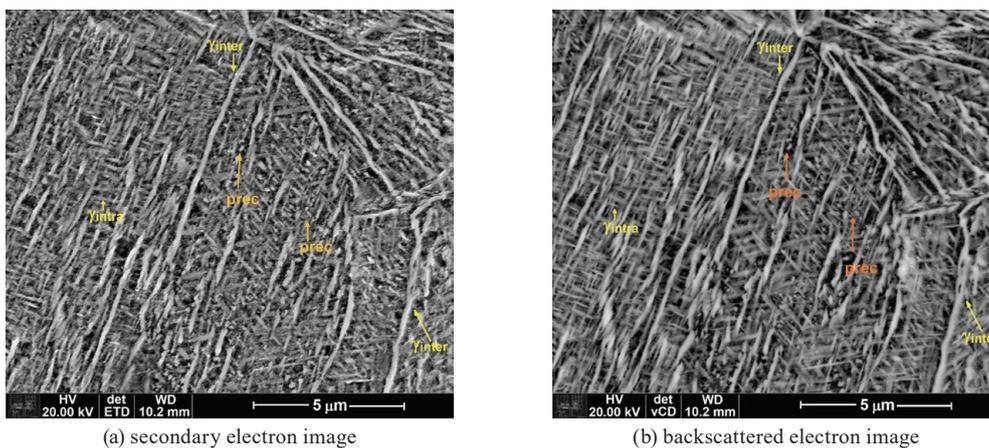


Fig. 9. Micrographs of the microstructure of the maraging 350 steel using 2% Nital reagent and obtained by scanning electron microscopy in a sample aged at 560°C for 18 h. (Online version in color.)

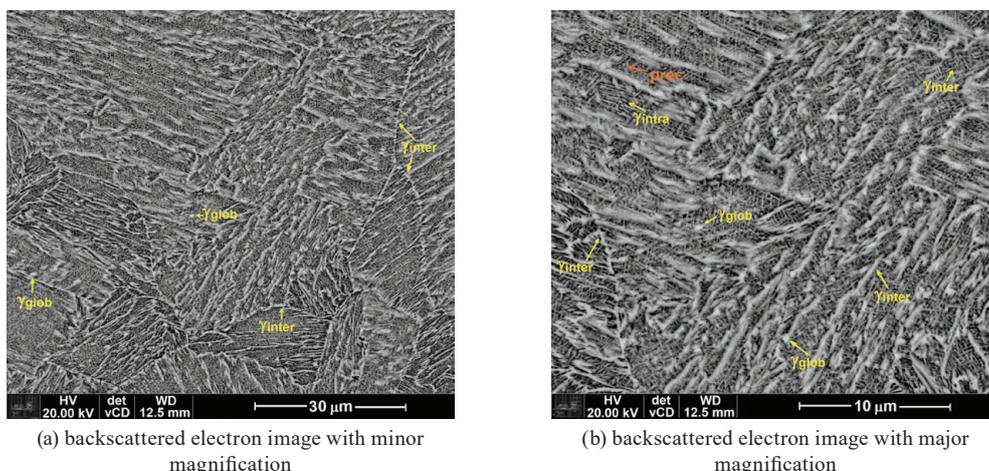


Fig. 10. Micrographs of the microstructure of the maraging 350 steel using 2% Nital obtained by scanning electron microscopy in a sample aged at 600°C for 18 h. (Online version in color.)

Figure 10 presents the micrographs obtained using back-scattered electrons for a sample aged at 600°C for 18 h. Figure 10(a) shows that reverted austenite can be observed with smaller magnification relative to the others. Figure 10(b) illustrates that the austenite located between the martensite laths is thicker than that formed at 520 and 560°C for 18 h (see Figs. 6(f) and 9). Even further, Figs. 10(b) shows that in the interior of the martensite laths the presence of austenite needles of the Widmanstätten type, as well as the presence of globular austenite between the martensite laths (identified as γ_{glob}).^{6,12,13)}

3.3. Kinetic Analysis of Reverted Austenite

One of the methods used to evaluate the precipitation apparent activation energy in maraging steels is based upon the determination of the time necessary to reach peak hardness for every aging temperature or for a given fixed fraction transformed, which follows an Arrhenius equation of the type:^{26–29)}

$$\ln t = \frac{Q}{RT} + \ln t_0 \dots\dots\dots (2)$$

where Q is the activation energy, R the universal gas constant, t and T are, respectively, the time and the temperature for which the peak hardness or the fixed transformed fraction is obtained during aging.

Schnitzer and coauthors³⁹⁾ applied this same equation to the study of the kinetics of reverted austenite in maraging PH 13–8 steels, by considering the time needed to reach a given volume fraction. The novelty of present work is the determination of activation energy of martensite reversion to austenite in Ni–Co–Mo–Ti maraging steels formed during overaging. In the present work, the time needed to attain an 18% volume fraction of austenite has been used, since that was the maximum value of the austenite proportion at 520°C. Based on the graph of Fig. 4 of the previous section it has been determined that the aging times needed to reach the 18% austenite volume fraction being 10 h, 0.7 h and 0.1 h, respectively for 520, 560 and 600°C.

Figure 11 shows that data followed well the model given by Eq. (2) for the determination of the activation energy, with a correlation of 0.998. Using linear regression, the activation energy for the reverted austenite has been evaluated, with a value of 332 ± 20 kJ/mol, which is quite close to the value determined by Guo and Sha of 342 kJ/mol³⁶⁾ for the martensite reversion in a maraging 250 steel using DSC. However, it is higher than the value reported by Schnitzer and coauthors³⁹⁾ for a maraging PH 13–8 (234 ± 20 kJ/mol). The activation energy for reverted austenite for maraging 350 steel also is about 20% above those determined for the diffusion of substitutional elements in ferrite. Such values are for nickel (245.8 kJ/mol),⁴⁰⁾ titanium (272 kJ/mol).

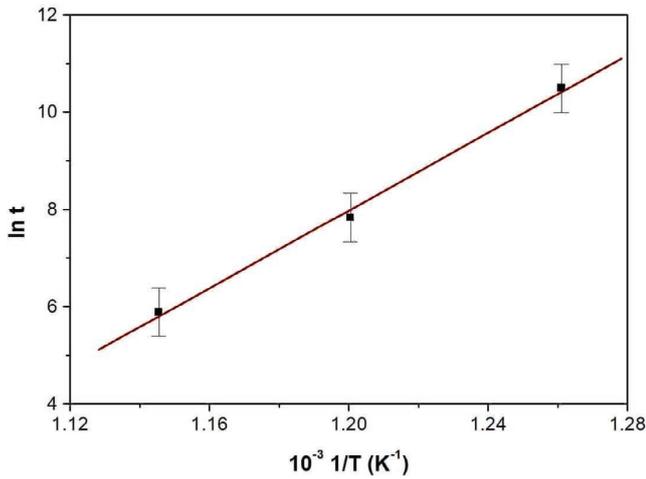


Fig. 11. Determination of the activation energy of reverted austenite using the Arrhenius-type equation. (Online version in color.)

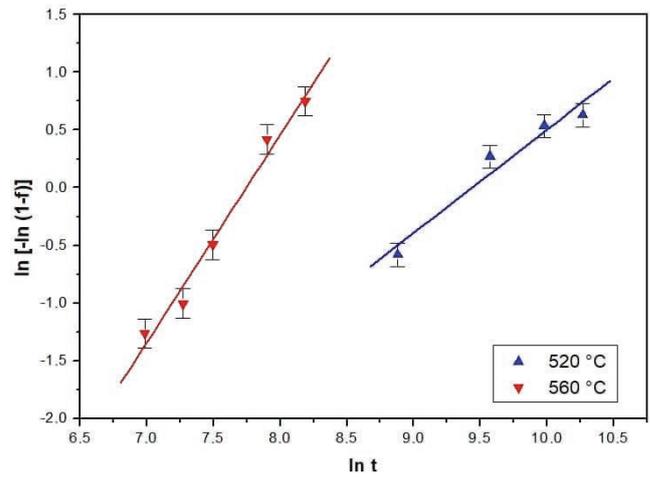


Fig. 12. JMAK model for the reverted austenite at 520 and 560°C in a maraging 350 steel. (Online version in color.)

mol)³⁶⁾ and for molybdenum (251 kJ/mol),⁴¹⁾ respectively. A possible explanation for this discrepancy is the presence of coarse precipitates in the martensitic matrix during overaging, which hinder the migration at the austenite/martensite interface.^{6,12,42)}

Another approach that could justify the behaviour described in the previous paragraph would be to consider the effect of the alloying elements on the microstructure and on the stability of the precipitates in the maraging steels containing cobalt. It is known that nickel and molybdenum act as strong austenite stabilisers, while titanium and cobalt act as martensite stabilisers in maraging steels containing cobalt.^{14,25)} Previous work has shown that the Ni₃Mo is a phase thermodynamically more unstable than the Ni₃Ti phase. Thus, the Ni₃Mo phase could have a primordial paper in the austenite nucleation due to local nickel enrichment of the martensitic matrix and in the formation of the Fe₂Mo or Fe₇Mo₆ phases.¹⁵⁾ Moreover, Li and Yin¹¹⁾ showed that the cobalt level in the austenitic phase is smaller than the base composition in the maraging steels. In this manner, austenite nucleating in the martensite would tend to expel cobalt to the interface region which could hinder the austenite/martensite interface mobility due to solute drag. This would agree with the mechanisms discussed by Hillert⁴⁴⁾ and by Hersent and coauthors,⁴⁵⁾ for the anchoring the migration of the interfaces in the presence of the solute.

Apart from the activation energy of the reverted austenite formed during overaging, another novelty of the present work is the analysis of possible transformation mechanisms with the help of the equation of John-Mehl-Avrami-Kolmogorov (JMAK) and your relationship with microstructural transformations. The kinetic isothermal of the phase transformation is usually described by a modified relation of the equation of John-Mehl-Avrami-Kolmogorov (JMAK):³⁶⁾

$$f = \frac{V_v}{V_{veq}} = 1 - e^{-kt^n} \dots\dots\dots (3)$$

In which, in the present work, *f* is the transformed volumetric fraction, *V_v* is the volumetric fraction of reverted austenite, *V_{veq}* is the maximum volumetric fraction (or at equilibrium of the reverted austenite), *k* is the speed con-

Table 3. Avrami *n*-constant and *k*-constant values for JMAK model at 520 and 560°C in a maraging 350 steel.

Temperature (°C)	<i>k</i> -constant (s ⁻¹)	Avrami <i>n</i> -constant	R
520	2.2 × 10 ⁻⁴	0.89 ± 0.14	0.98
560	8.7 × 10 ⁻⁷	1.80 ± 0.15	0.99

stant, and *n* is the Avrami constant.

Based on Eq. (3) the linearised equation is:

$$\ln[-\ln(1 - f)] = \ln k + n \cdot \ln t \dots\dots\dots (4)$$

From this equation and the experimental data, the graph in **Fig. 12** has been obtained.

Table 3 shows that the Avrami constant for the kinetics of the reversion of austenite at 520°C is close to 1. According to Christian,⁴⁶⁾ the value corresponding to 1 refers to the nucleation at grain boundaries after saturation. This is consistent with the inter-lath austenite formation observed in item 3.2 of the present work and by previous works.^{6,12,13)} Saturation would be a consequence of the presence of precipitates.^{6,12,13,15)}

The value of the Avrami constant for 560°C is close to 2. This could be related to the growth of the inter-lath austenite (started at that temperature), as observed in item 3.1. Christian⁴⁶⁾ suggested that the value of 2 could be related to the growth of all particles solely in one dimension starting from a small size with a constant nucleation rate, which is consistent with the growth of the Widmanstätten plates with twins inside the martensite laths.^{6,11,13,17)}

4. Conclusions

The experiments and analysis performed in a maraging 350 steel allowed the following conclusions:

- (a) The microstructural observations carried out using optical, and scanning electron microscopy showed that the reverted austenite is formed at interface regions, such as grain boundaries, package boundaries and lath boundaries of the martensitic structure at temperatures starting at 520°C. For the reverted austenite observed in the interior of the martensite lath, it started forming at 560°C.

(b) The kinetics of the reverted austenite is strongly influenced by the aging temperature. Higher aging temperatures, lead to the faster attainment of a constant plateau of a volumetric fraction, as noted by the austenite volume fraction as a function of aging time for 520, 560 and 600°C. The maximum volume fraction of reverted austenite for the maraging 350 steel that has been analysed was 18%, 25% and 37%, respectively for 520, 560 and 600°C.

(c) The value of the activation energy for the reverted austenite of 332 ± 20 kJ/mol, is about 20% higher than the activation energies for the diffusion of substitutional alloying elements such as nickel (245.8 kJ/mol), titanium (272 kJ/mol) and molybdenum (251 kJ/mol).

(d) The value of the Avrami constant for the kinetics of the reverted austenite at 520°C was close to 1, which is consistent with the nucleation and growth at grain boundaries after saturation of the inter-lath austenite. The Avrami constant for 560°C is close to 2, which could be related to the growth of the intra-lath austenite in the form of Widmanstätten plates with twins within the martensite lath, starting at this temperature. These plates would grow solely in one dimension, starting from a small size with a constant nucleation rate.

Acknowledgments

The authors are grateful to the CNPq (Brazil) and to the CTMSP (São Paulo, Brazil).

REFERENCES

- B. Rohit and N. R. Muktinatalapati: *Mater. Sci. Technol.*, **34** (2018), 253.
- M. N. Rao: *Int. J. Mater. Res.*, **11** (2006), 1594.
- W. Sha and Z. Guo: *Maraging Steels: Modeling of Microstructure, Properties and Applications*, Woodhead Publishing Limited, Cambridge, (2009), 216.
- S. S. M. Tavares, M. R. da Silva, J. M. Neto, J. M. Pardal, M. P. C. Fonseca and H. F. G. Abreu: *J. Alloy. Compd.*, **373** (2004), 304.
- J. M. Pardal, S. S. M. Tavares, M. C. Fonseca, M. R. da Silva, J. M. Neto and H. F. G. Abreu: *J. Mater. Sci.*, **42** (2007), 2276.
- U. K. Viswanathan, G. K. Dey and V. Sethumandhavan: *Mater. Sci. Eng. A*, **398** (2005), 367.
- L. G. de Carvalho, R. L. Plaut, M. A. Martorano and A. F. Padilha: *Characterization of Minerals, Metals, and Materials 2015*, ed. by J. S. Carpenter *et al.*, Springer, Cham, (2015), 3.
- C. S. Carter: *Metall. Trans.*, **2** (1971), 1621.
- Y. He, K. Yang and W. Sha: *Metall. Mater. Trans. A*, **36** (2005), 2273.
- R. F. Decker and S. Floreen: *Maraging Steels: Recent Developments and Applications*, ed. by R. F. Wilson, TMS, Warrendale, PA, (1988), 1.
- X. Li and Z. Yin: *Mater. Lett.*, **24** (1995), 239.
- U. K. Viswanathan, G. K. Dey and M. K. Asundi: *Metall. Trans. A*, **24** (1993), 2429.
- M. Farooque, H. Ayub, A. U. Haq and A. Q. Khan: *J. Mater. Sci.*, **33** (1998), 2927.
- L. G. Carvalho, M. S. Andrade and A. F. Padilha: *Mater. Res.*, **16** (2013), 740.
- O. Moshka, M. Pinkas, E. Brosh, V. Ezersky and L. Meshi: *Metall. Mater. Sci. Eng. A*, **638** (2015), 232.
- M. Shmulevitch, L. Meshi, M. Pinkas and R. Z. Shneck: *J. Mater. Sci.*, **50** (2015), 4970.
- N. Atsmon and A. Rosen: *Metallography*, **14** (1981), 163.
- K. Hosomi, Y. Ashida, H. Hato, R. Atagi, K. Ishihara and H. Nakamura: *Tetsu-to-Hagané*, **64** (1978), 595 (in Japanese).
- J. M. Pardal, S. S. M. Tavares, M. P. C. Fonseca, H. F. G. Abreu and J. J. M. Silva: *J. Mater. Sci.*, **41** (2006), 2301.
- L. N. Belyakov, V. L. Nikol'skaya and S. S. Ryzhak: *Met. Sci. Heat Treat.*, **10** (1968), 438.
- F. Habiby, A. Ul Haq and A. Q. Khan: *Mater. Des.*, **13** (1992), 259.
- M. Ahmed, A. Ali, S. K. Hasnain, F. H. Hashmi and A. Q. Khan: *Acta Metall. Mater.*, **42** (1994), 631.
- F. Habiby, T. N. Siddiqui, S. H. Khan, A. U. Haq and A. Q. Khan: *NDT&E Int.*, **25** (1992), 145.
- D. T. Peters: *Trans. Am. Soc. Met.*, **61** (1968), 62.
- D. T. Peters and C. R. Cupp: *Trans. AIME*, **236** (1966), 1420.
- J. M. Pardal, S. S. M. Tavares, V. F. Terra, M. R. da Silva and D. R. dos Santos: *J. Alloy. Compd.*, **393** (2005), 109.
- P. P. Sinha, K. T. Tharian, K. Sree Kumar, K. V. Nagarajan and D. S. Sarma: *Mater. Sci. Technol.*, **14** (1998), 1.
- V. K. Vasudevan, S. J. Kim and C. M. Wayman: *Metall. Trans. A*, **21** (1990), 2655.
- U. K. Viswanathan, G. K. Dey and M. K. Asundi: *Metall. Trans. A*, **24** (1993), 2429.
- A. C. Larson and R. B. Von Dreele: GSAS, LANSCE, Los Alamos, (1994), 231.
- M. A. Martorano, C. F. Tavares and A. F. Padilha: *ISIJ Int.*, **52** (2012), 1054.
- D. M. Escriba, E. Materna-Morris, R. L. Plaut and A. F. Padilha: *Mater. Charact.*, **60** (2009), 1214.
- F. Habiby, T. N. Siddiqui, H. Hussain, A. Ul Haq and A. Q. Khan: *J. Mater. Sci.*, **31** (1996), 305.
- A. G. Reis, D. A. P. Reis, A. J. Abdalla, A. A. Couto and J. Otubo: *Defect Diffus. Forum*, **371** (2017), 73.
- T. B. J. Alves, G. C. S. Nunes, L. F. S. Tupan, P. W. C. Sarvezuk, F. F. Ivashita, C. A. S. de Oliveira and A. Paesano Jr.: *Metall. Mater. Trans. A*, **49** (2018), 3441.
- Z. Guo, W. Sha and D. Li: *Mater. Sci. Eng. A*, **373** (2004), 10.
- S. R. E. Hosseini and H. Arabi: *ISIJ Int.*, **52** (2012), 147.
- K. Yamada, M. Igarashi, S. Muneki, H. Okada and F. Abe: *ISIJ Int.*, **42** (2002), 882.
- R. Schnitzer, R. Radis, M. Nöhrer, M. Schober, R. Hochfellner, S. Zinner, E. Povoden-Karadeniz, E. Kozeschnik and H. Leitner: *Mater. Chem. Phys.*, **122** (2010), 138.
- J. Kučera and K. Stránský: *Mater. Sci. Eng.*, **52** (1982), 1.
- S. Huang, D. L. Worthington, M. Asta, V. Ozolins, G. Ghosh and P. K. Liaw: *Acta Mater.*, **58** (2010), 1982.
- F. Zhu, Y. F. Yin and R. G. Faulkner: *Mater. Sci. Technol.*, **27** (2011), 395.
- M. Ahmed, I. Nasim and S. W. Husain: *J. Mater. Eng. Perform.*, **3** (1994), 248.
- M. Hillert: *Acta Mater.*, **52** (2004), 5289.
- E. Hersent, K. Marthinsen and E. Nes: *Metall. Mater. Trans. A*, **44** (2013), 3364.
- J. W. Christian: *The Theory of Transformations in Metals and Alloys*, 2nd ed., Pergamon Press, Oxford, (1975), 584.