International Journal of
MATERIALS RESEARCH

Zeitschrift für METALLKUNDE

History

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Progress in understanding the metallurgy of 18 % nickel maraging steels

Maraging steels based on iron–nickel martensite constitute a very important family of high-strength steels, which distinguishes itself by demonstrating an unparalleled combination of excellent fabricability and high strength and fracture toughness after heat treatment. Heat treatment of these steels has now been perfected to ensure consistently high levels of strength, ductility, and toughness in a variety of product shapes and sizes. Cobalt-free variants have been commercialized as part of efforts to save production costs. Further knowledge has been generated on 18 % nickel maraging steels regarding phases precipitating during aging, thermal embrittlement, thermal cycling and austenite reversion/retention and their effect on mechanical properties. The paper reviews the progress made over the last one and half decades.

Keywords: Maraging steels; Fe–Ni martensite; Heat treatment; Fracture toughness; Thermal embrittlement

1. Introduction

Maraging steels based on the iron–nickel–cobalt–molybdenum system have emerged as an outstanding family of materials with exceptional combinations of characteristics, making them the designers' choice for specialized applications [1]. These grades generally contain 18 wt.% nickel and are commonly designated as 18Nixxxx, where xxxx is the nominal strength value in MPa attained after final heat treatment. Their outstanding attributes include ultra-high strength coupled with high fracture toughness, excellent formability under hot and cold working conditions, and ease with which heat treatment can be carried out to attain the high strength condition. A number of low alloy steel compositions have been developed to achieve a combination of high strength and high toughness, but maraging steels have retained their dominant position in this respect [1, 2]. Figure 1 [1] shows the superiority of maraging steels in a plot of strength versus toughness. Thanks to the attractive hot and cold deformation characteristics, the material can be readily produced in a variety of mill forms and shapes. It is amenable to flow forming, making it possible to produce thin-walled tubes in a near net shaping manner. The material is commercially available in different strength variants – starting from 1400 MPa and reaching up to 2400 MPa. The inverse relationship between strength and toughness, well established for high-strength steels, also

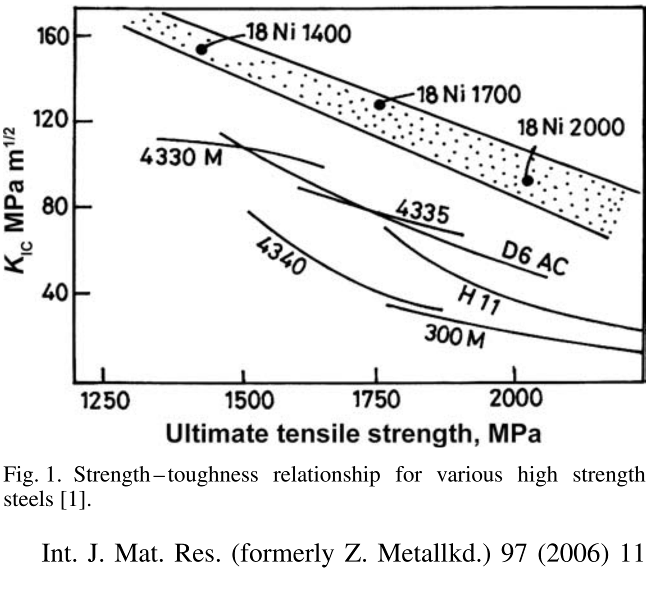

Fig. 1. Strength–toughness relationship for various high strength steels [1].

holds good within the family of 18 % Ni maraging steels; this can also be seen from Fig. 1, variants having higher strength showing lower fracture toughness. While the maraging steel variant 18Ni1700 is the most important grade in the series, the higher-strength variants 18Ni1900/2000 and 18Ni2400 are also used for specialized applications. There have also been efforts to develop Fe–Ni martensite-based maraging steel with a strength level of 2800 MPa. He et al. [3] recently reviewed these efforts.

The subject of Fe–Ni martensite-based maraging steel was reviewed by Floreen [4]. A number of papers on the subject appeared in the proceedings “Maraging steels: recent developments and applications” edited by R. K. Wilson in the year 1988 [5]. Since then there have been some more developments. The phenomenon of thermal embrittlement in maraging steels has been studied in greater detail, and a deeper understanding was reached on the mechanistic aspects and processing conditions to be adopted to prevent it from occurring. Improved heat treatments have been developed to attain high product quality in terms of strength, toughness, and ductility with a high degree of consistency. Cobalt-free maraging steel grades have been successfully commercialized. There has been an increased understanding of the decomposition reactions taking place in martensite and chemical composition/crystal structure of the phases precipitating during aging. The effect of austenite reversion on mechanical properties of maraging steels has been gone into in depth. In this paper an effort is made to review these various developments which have largely taken place in about the last one and half decades. While there have been a number of studies on these steels during this period dealing with corrosion, stress corrosion cracking, hydrogen embrittlement, surface modification and magnetic behavior, these aspects are not covered in the present review. The scope of the review is restricted to wrought grades of maraging steel containing 18 wt.% Ni; the review does not cover studies carried out on maraging steel compositions containing manganese or tungsten and stainless maraging steels. Maraging steels with 2800 MPa strength level are also not covered, as wt.% Ni in most of the compositions tried out is $\neq 18$ [3]. The focus is on studies relating to the effect of various heat treatments on structure, strength, and toughness.

2. Cobalt-free maraging steels

An important factor that has come in the way of extensive usage of maraging steels based on the Fe–Ni–Co–Mo system is the high cost. Cobalt is an expensive alloying element and is present in the range 8–12 wt.% in these steels, depending on the strength level, contributing importantly to the cost. Accordingly efforts were made to develop cobalt-free variants of 18 % Ni maraging steel with a comparable fracture toughness–strength combination. The development and commercialization of cobalt-free maraging steels, comparable to the standard cobalt-bearing grades with 1400, 1700, and 2000 MPa strength levels, was reviewed by Floreen and Bayer [6]. Lampson and Crownover [7] studied an 18 % Ni cobalt-free alloy with 3 % Mo and 1.4 % Ti and compared its performance with that of cobalt-bearing 18Ni1900 grade. Wilson et al. [8] reported development of a new cobalt-free maraging steel suitable for industrial application.

In cobalt-bearing grades Mo is present at a relatively high level. Cobalt decreases the solubility of Mo in Fe–Ni martensite, thereby promoting significant presence of Mo in the intermetallic strengthening precipitates. In cobalt-free grades of comparable strength, Mo is present at a lower level; Ti is present at a higher level and makes a dominating presence in the strengthening phase. The strengthening precipitates appearing in cobalt-bearing and cobalt-free grades during aging of martensite are reviewed in detail in Section 3.

Today cobalt-free 18 % Ni maraging steel grades are commercially available with different strength levels covering the range 1400–2000 MPa. The various advantages associated with cobalt-bearing 18 % Ni maraging steels – excellent mechanical properties, high degree of workability, easy heat treatment – are also available with cobalt-free maraging steels. He et al. carried out detailed studies on cobalt-free 18Ni2000 grade maraging steel and demonstrated that a yield strength level of 2050 MPa can be realized together with a fracture toughness level of 70 MPa with percent elongation of > 9 and percent reduction in area of > 50 by optimally choosing the aging parameters [9, 10]. Table 1 compares the chemical composition of cobalt-bearing and cobalt-free grades [11, 12]. Tables 2 and 3 compare the ty-

Table 1. Nominal chemical composition of cobalt-containing and cobalt-free maraging steel grades [10, 11].

Element	Cobalt-bearing 18Ni1400	Cobalt-bearing 18Ni1700	Cobalt-bearing 18Ni2000	Cobalt-free 18Ni1400	Cobalt-free 18Ni1700	Cobalt-free 18Ni2000
Nickel	18.5	18.5	18.5	18.5	18.5	18.5
Cobalt	8.5	7.5	9.0	Nil	Nil	Nil
Molybdenum	3.25	4.80	4.80	3.00	3.00	4.00
Titanium	0.20	0.40	0.60	0.70	1.40	1.85
Aluminum	0.10	0.10	0.10	0.10	0.10	0.10
Silicon	0.10 max	0.10 max	0.10 max	0.10 max	0.10 max	0.10 max
Manganese	0.10 max	0.10 max	0.10 max	0.10 max	0.10 max	0.10 max
Carbon	0.03 max	0.03 max	0.03 max	0.03 max	0.03 max	0.03 max
Sulfur	0.01 max	0.01 max	0.01 max	0.01 max	0.01 max	0.01 max
Phosphorous	0.01 max	0.01 max	0.01 max	0.01 max	0.01 max	0.01 max
Zirconium	0.01	0.01	0.01	–	–	–
Boron	0.003	0.003	0.003	–	–	–
Iron	Bal.	Bal.	Bal.	Bal.	Bal.	Bal.

Table 2. Nominal room temperature mechanical properties of cobalt-containing and cobalt-free 18Ni1700 maraging steels [10, 11]. Product size: 150 mm square.

Property	Cobalt-containing 18Ni1700	Cobalt-free 18Ni1700
Ultimate tensile strength (MPa) (Long)	1730	1790
Ultimate tensile strength (MPa) (Trans)	1723	1790
0.2 % Yield strength (MPa) (Long)	1695	1748
0.2 % Yield strength (MPa) (Trans)	1690	1748
% Elongation in $4.5\sqrt{A}$ (Long)	10.0	10.5
% Elongation in $4.5\sqrt{A}$ (Trans)	8.1	8.9
% Reduction in Area (Long)	46.7	56.1
% Reduction in Area (Trans)	30.3	45.8
Charpy V notch impact (Nm) (measured on small diameter bars)	27	34
Plane strain fracture toughness (MPa $m^{1/2}$)	Not available	104.0

Long: Longitudinal direction
Trans: Transverse direction
A: Cross sectional area

Table 3. Nominal room temperature mechanical properties of cobalt-containing and cobalt-free 18Ni2000 maraging steels. [10, 11]. Product size: 150 mm square.

Property	Cobalt-containing 18Ni2000	Cobalt-free 18Ni2000
Ultimate tensile strength (MPa) (Long)	1962	2049
Ultimate tensile strength (MPa) (Trans)	1952	2046
0.2 % Yield strength (MPa) (Long)	1915	1984
0.2 % Yield strength (MPa) (Trans)	1910	1997
% Elongation in $4.5\sqrt{A}$ (Long)	9.8	8.8
% Elongation in $4.5\sqrt{A}$ (Trans)	6.6	7.5
% Reduction in Area (Long)	43.9	45.3
% Reduction in Area (Trans)	28.4	38.2
Charpy V notch impact (Nm)(measured on small diameter bars)	23	20
Plane strain fracture toughness (MPa $m^{-3/2}$)	Not available	69.7 to 73.9 (data on two production heats)

Long: Longitudinal direction
Trans: Transverse direction
A: Cross sectional area

pical mechanical properties of the cobalt-free and cobalt-containing grades at strength levels of 1700 MPa and 1900 MPa, respectively [11, 12]. It can be seen that the cobalt-free grades compare well with the cobalt-bearing grades.

Both cobalt-free and cobalt-containing grades are processed through double vacuum melting: primary melting in a vacuum induction furnace and secondary melting in a vacuum arc furnace. In addition, close control is exercised at the stage of raw material selection. This way one ensures high levels of purity, close control of chemical composition and realization of an excellent combination of strength, ductility, and fracture toughness. In the following discussion the prefixes T and C will be used to the grade designation to distinguish between cobalt-free and cobalt-bearing grades.

3. Developments in understanding the precipitating phases

There has been substantial improvement with respect to our understanding of the early stages of transformation and nature of phases precipitating out of martensite in 18Ni maraging steels. This has been importantly due to a variety of sophisticated research instruments becoming available for studying decomposition and characterization of precipitating phases. The techniques used for carrying out these studies included transmission electron microscopy [9, 10, 13–20], atom probe field ion microscopy [21–27], Mössbauer spectroscopy [17, 28], small-angle X-ray scattering [15, 18], and small-angle neutron scattering [29]. However the various studies carried out have often lead to different conclusions on the nature of precipitating phases.

3.1. Cobalt-containing maraging steels

a) Relatively low aging temperatures

ω phase was observed by Lecomte et al. [14] in 67.4Fe–18.6Ni–8.9Co–4.8Mo alloy after aging at 420 °C, by Bouzid et al. [17] and Servant and Bouzid [29] in 70.4Fe–17.7Ni–8.8Co–3.0Mo after aging at 420 °C, 438 °C and 458 °C and by Tewari et al. [20] in 18Ni2400 after aging at 430 °C.

b) Relatively high aging temperatures.

C18Ni1700
In their review of 18 % Ni maraging steels, Decker and Floreen [1] stated that the precipitation hardening phase in C18Ni1700 is orthorhombic Ni_3Mo.

Vasudevan et al. [15,18] reported that aging for 3 h at 482 °C revealed η Ni_3Ti particles with a large amount of Mo substituting for Ti. Spherical Fe_2Mo precipitates were seen in the peak-aged condition with Ni and Co substituting some amount of Fe, and Ti substituting some amount of Mo. It is thus seen that Fe–Mo-based precipitates contribute importantly to the attainment of peak strength.

C18Ni1900/2000
While Spitzig et al. [13] reported precipitation of Ni_3Mo, Vanderwalker [16] and Sha et al. [21, 23, 24] reported precipitation of Ni_3Ti. Sha et al. showed that some of the Ni in Ni_3Ti precipitates is substituted by Fe and a small amount of Co and that some Mo is also present in the particles. For aging times of 2 h at 510 °C (peak aged condition) and 128 h at 510 °C, the Fe_7Mo_6 phase was observed by Sha et al. [21, 23, 24]. An unidentified tetragonal phase with the structure of the sigma phase was reported by Spitzig et al. [13].

C18Ni2400
Ni_3(Ti, Mo) precipitates were identified by Viswanathan et al. [19] and Tewari et al. [20]. Viswanathan et al. found Fe_2Mo precipitates in the peak-aged condition (3 h at 510 °C) [19].

It can be seen that there is a lot of variance in the results reported by different workers, even when the studies were carried out on the same grade. Based on the review of Decker and Floreen [1], the precipitation hardening phase in C18Ni1700 grade is orthorhombic Ni_3Mo. The work of Vasudevan et al. [15, 18] on the same grade reports the main precipitation-strengthening phase as Ni_3Ti. Spitzig et al. [13] report Ni_3Mo precipitates in C18Ni1900/2000. Sha et al. [21, 23, 24] reported precipitation of Ni_3Ti in the same grade. Vasudevan et al. [15] concluded that the discrepancy in reporting is a result of the close similarities between the structures and *d*-spacings of orthorhombic Ni_3Mo and hexagonal Ni_3Ti. While reflections of Ni_3Ti can be alternately indexed as Ni_3Mo, the diffraction patterns give a superior match with the hexagonal Ni_3Ti [15].

There is also disagreement among the results reported with reference to precipitation of Fe–Mo compounds in cobalt-bearing 18 % Ni maraging steels. Vasudevan et al. [15, 18] reported Fe_2Mo in C18Ni1700. Wanderwalker [16] and Spitzig et al. [13] have not reported any Fe–Mo-based precipitate after aging in 18Ni1900/2000, whereas Sha et al. [21, 23, 24] reported Fe_7Mo_6 in the same grade. Viswanathan et al. [19] report Fe_2Mo in C18Ni2400 after aging for 3 h at 510 °C. Precipitating phases like FeMo, Fe_2Mo, Fe_7Mo_6, Fe_2Ti have similar interplanar distances, leading to the difficulties in distinguishing them using electron diffraction [27].

The variations in chemical composition, aging temperature and time adopted by different workers contribute to the observed differences in precipitation behavior.

Based on the studies carried out by different workers, it can be concluded that (Ni, Fe, Co)$_3$(Ti, Mo) provides a comprehensive description of the precipitates. The relative proportion of Ti and Mo in the precipitates would depend on the chemical composition of the steel, with particular reference to the levels of Ti, Mo and Co. Thus there will be more Mo substituting Ti in C18Ni1700 compared to what happens in C18Ni2400.

The Fe–Mo-based precipitates seem to contribute significantly to the strength of the cobalt containing grades in the peak-aged condition. It appears, however, that the relative contributions to strengthening by (Ni, Fe, Co)$_3$(Ti, Mo) and Fe–Mo-based precipitates have not been quantitatively established. Further research is required to get this information.

3.2. Cobalt-free 18 % Ni maraging steels

a) T18Ni1700

Ni_3Ti precipitates were seen by Vasudevan et al. [15], Vanderwalker [16], and Sinha et al. [30]. As per Vasudevan et al. [15] the precipitates are more correctly designated as (Ni, Fe,)$_3$(Ti, Mo) with significantly larger levels of Ti-to-Mo ratio in the cobalt-free version. No Fe–Mo-based precipitation was seen by any of these authors.

b) T18Ni1900/2000

Vanderwalker [16], Sha et al. [22, 25], and He et al. [9, 10] noticed Ni_3Ti precipitation. Fe_2Mo, FeMo and Fe_xTi precipitates have also been reported by Vanderwalker [16] after aging for 4 h at 510 °C. On aging at 440 °C and 480 °C, He et al. [9, 10] observed spherical precipitates with strong resistance to coarsening, the crystallography of which could not be identified. After 24 h and 360 h of aging at 510 °C, Sha et al. [21, 23, 24] reported Fe_7Mo_6 with some Fe substituted by Ni. The precipitation of Fe_7Mo_6 occurs at much longer aging times compared to what happens in the cobalt-bearing counterpart. The delayed appearance of Fe_7Mo_6 precipitates in T18Ni1900/2000 in comparison to C18Ni1900/2000 has been interpreted by Sha et al. [22, 25] as a result of Co in the matrix increasing the tendency for Mo to precipitate out of the matrix in the latter.

It is seen that there is considerable disagreement with reference to precipitation of other than Ni_3Ti type phases in cobalt-free 18 % Ni maraging steels. Sha et al. [25] reported Fe_7Mo_6 precipitates; this is, however, after much longer aging compared to their cobalt containing counterparts. No Fe–Mo-base precipitates have been reported by He et al. [9, 10] and Sinha et al. [30].

Even in the case of cobalt free maraging steels, (Ni, Fe, Co)$_3$(Ti, Mo) will be a comprehensive description of the particles. However, there will be less Mo substituting Ti in cobalt-free grades, compared to the situation in cobalt-bearing grades with comparable strength.

There are variations in chemical composition, aging temperature and time adopted by different workers and this contributes to the observed differences in precipitation behavior.

As regards cobalt free maraging steels, there are some important questions to be answered regarding phases contributing to strengthening in peak-aged condition and precipitation of Fe–Mo-based compounds in. The lower level of Mo, the absence of Co, and the relatively high stability of

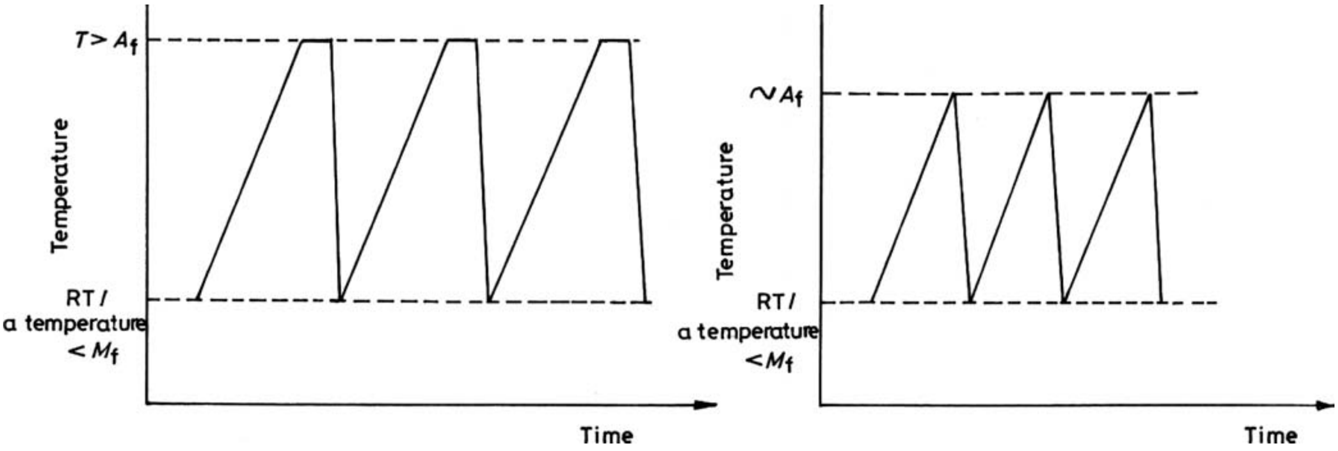

Fig. 2. Thermal cycling studies on maraging steel. (a) Cycling between RT and $T > A_f$ and (b) cycling between RT and ~ A_f.

(Ni, Fe, Co)$_3$(Ti, Mo) in cobalt-free grades mean that precipitation of Fe–Mo-based compounds should be a highly retarded reaction in these grades in comparison to their cobalt-containing counterparts.

4. Thermal cycling

Different workers have investigated the effect of repeated thermal cycling on the microstructure and mechanical properties of maraging steels. The studies can be broadly categorized into two groups – one dealing with cycling between ambient temperature (a temperature below M_f) and a temperature when austenite transformation gets completed during heating (A_f) and the other dealing with cycling between ambient temperature and a temperature in the austenitic phase field well above A_f. Figure 2 shows schematically the two types of thermal cycling.

4.1. Cycling between ambient temperature and ~ A_f

Goldberg [31] was the first to study the effect of repeated thermal cycling in C18Ni2000. Thermal cycling was carried out between ambient temperature and 825 °C. Repeated thermal cycling increased the amount of retained austenite and decreased the hardness after aging.

Viswanathan et al. [32] studied the effect of repeated thermal cycling in C18Ni2400. Cycling was carried out between ambient temperature and 750 °C. The volume fraction of austenite increased with increasing number of cycles, with maximum change occurring during the first cycle. Figure 3a shows the transmission electron microscope (TEM) micrograph of the steel after cycling for 5 times and aged for 3 h, the arrows showing fine austenite lamellae along the martensite lath boundaries. Figure 3b shows the TEM micrograph of the same steel after aging at 510 °C for 3 h without thermal cycling. Both yield strength and ultimate tensile strength decreased monotonically with increasing quantity of retained austenite; on the other hand there was a steady improvement of ductility and impact toughness [32].

Farooque et al. [33] and Qureshi and Farooque [34] studied the effect of repeated thermal cycling between room temperature and 800 °C in C18Ni2400. The heat treatment produced retained austenite in the microstructure.

When rapidly cooled from ~A_f, without allowing the austenite to attain equilibrium composition, the solute-rich austenite remains untransformed and the solute-depleted austenite transforms to martensite. Expectedly the rate of heating to ~ A_f and the hold time there influence the amount of austenite retained. The mechanism of formation of austenite in maraging steel during thermal cycling has been investigated by Lecompte et al. [14] and Bouzid et al. [35].

Microstructures containing retained austenite as a result of thermal cycling have not shown a better strength–toughness combination than that obtainable with peak-aged microstructures with equivalent strength. As such, these studies have had no industrial application. The subject is further discussed in Section 8.

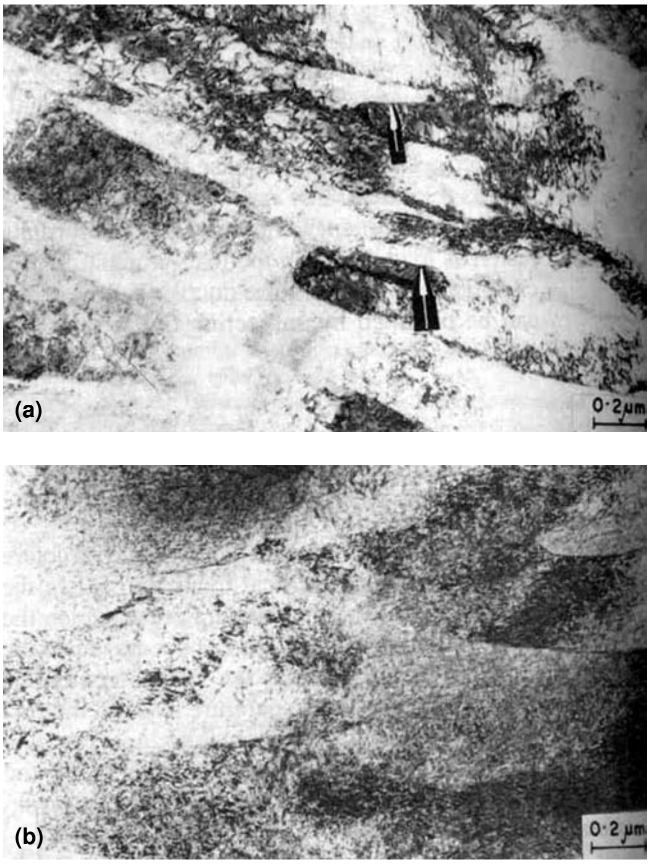

Fig. 3. TEM micrographs of steel C128Ni2400: (a) cycled for 5 times and aged at 510 °C for 3 h and (b) directly aged at 510 °C for 3 h after solution annealing [32].

4.2. Cycling between ambient temperature and a temperature well above A_f

Saul et al. [36] were the first to demonstrate that reduction of the prior austenitic grain (PAG) size can be realized by cycling the maraging steel between ambient temperature and a temperature in the austenitic phase field well above A_f. The optimum value of the upper temperature for effective grain refinement is very sensitive to the alloy composition and must be determined for each alloy. It was 927 °C for C18Ni1700 and 1027 °C for C18Ni2000 [36].

Nakazawa et al. [37] studied the effect of thermal cycling on C18Ni2400, among other compositions, and established that remarkable grain refinement can be achieved by choosing appropriate cycling conditions without degrading the strength and toughness.

Sinha et al. [38] studied the effect of thermal cycling between room temperature and 925 °C on C18Ni1700 and demonstrated a major effect of grain size refinement. Their studies on C18Ni1700 and T18Ni1700 showed that just one cycle has a significant refining effect on PAG size [39]. Figure 4a shows optical micrograph of T18Ni1700 after solution annealing at 1200 °C for 60 minutes and water quenching. The grain size is in the range 400–450 μm. Thermal cycling of this material once between 875 °C and room temperature resulted in a grain size of 50 μm. Figure 4b shows the optical micrograph after thermal cycling.

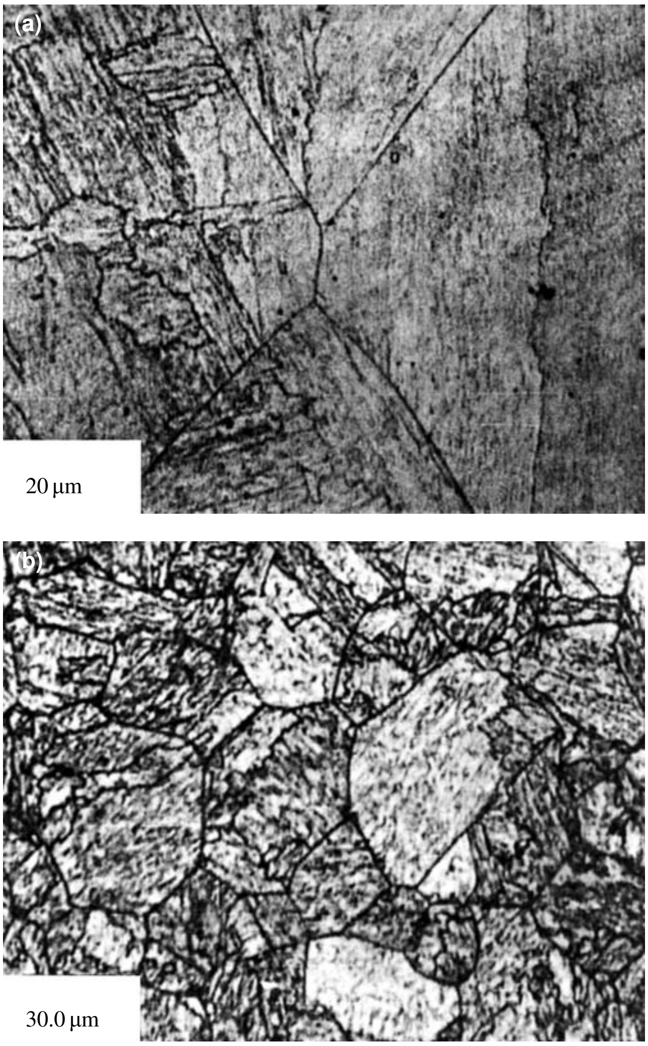

Fig. 4. Optical micrographs of T18Ni1700: (a) Solution annealed at 1200 °C and water quenched, and (b) material at (a) thermally cycled once between 875 °C and room temperature [29].

Grain refinement by thermal cycling in maraging steel has been attributed to new sets of austenitic grains forming on the defect structures introduced by α-to-γ shear transformation during the heating cycle [36, 38]. Fast heating rates promote this transformation. At relatively low heating rates diffusion-controlled transformation is an operating mechanism, in addition to the shear mechanism [40]. Taking into account that grain refinement is aided by shear transformation, the heating rate is an important parameter for industrial exploitation of thermal cycling for achieving grain refinement. The dependence of the mechanical properties of maraging steels on PAG size is reviewed in detail in Section 7.

The phenomenon of grain refinement by thermal cycling has been industrially exploited to produce massive maraging steel products with a fine grain size and optimum combination of mechanical properties of designers' interest, as brought out in a detailed manner in subsequent sections.

5. Thermal embrittlement of maraging steels

Maraging steels show marked degradation in toughness if they get exposed to temperatures in the range 780 to 950 °C while cooling from high temperatures (e. g. 1200 °C). This phenomenon has been termed thermal embrittlement (TE). It causes serious problems in processing components of thick sections of these steels [41]. TE has generally been characterized by a reduction in the Charpy impact energy [42–44]; Nes and Thomas [45] have shown that it also results in a decrease in ductility without impairing the strength. Ashida et al. [46] reported loss of ductility and toughness resulting from the precipitation of TiC on grain boundaries after a two-stage austenitising treatment (holding the austenite at 800 to 1000 °C after solution treatment at 1200 °C) of maraging steel. This treatment, as can be readily recognized, promotes TE. Sinha et al. [38] reported a decrease in plane strain fracture toughness (K_{IC}) due to TE in rolled rings of cobalt-bearing 18Ni1700 grade maraging steels.

Systematic investigations on the mechanistic aspects of TE in 18Ni–Co–Mo steels were reported during the 1970's by Kalish and Rack [42], Johnson and Stein [43], Maniar and James [47], Nes and Thomas [45], and Rack and Holloway [48].

Kalish and Rack [42] suggested that TE in these steels occurs because of segregation of Ti and C(N) at the prior austenite grain boundaries thereby reducing the interfacial energy and promoting intergranular fracture.

Johnson and Stein [43] studied the phenomenon using Auger electron spectroscopy. While agreeing with the concept of grain boundary segregation of solute atoms of Kalish and Rack [42], they believed that TE was a direct consequence of TiC/Ti(C, N) formation by the chemical reaction of Ti and C(N) at the grain boundaries.

Subsequent studies have confirmed that formation of Ti(C, N) at grain boundaries is essential to the occurrence of TE. Rack and Holloway [48] confirmed the role of TiC particles on prior austenite grain boundaries in causing TE. Nes and Thomas [45] concluded that the TE results from the heterogeneous decomposition of supersaturated Fe–Ti–C solid solution obtained during high-temperature solution treatment. Chandra-Holm et al. [49] showed that the presence of TiC particles at the PAG boundaries in a non-standard (containing 5.1 wt.% Ti) C18Ni1900 steel is very detrimental to the toughness and overrides any influence that the other microstructural features may have. More recently Sinha et al. [39] reported their detailed studies on TE in both Cobalt-bearing and Cobalt-free grades of 18 % Ni maraging steel. Their studies covered a wide range of intermediate holding temperatures (800 to 950 °C) during cooling from the austenitizing temperature of 1200 °C. They observed that TE is caused by precipitation of Ti(C, N) at grain boundaries from the supersaturated Fe–Ti–C solid solution obtained during high-temperature solution treatment. Cobalt-free grade T18Ni1700 is as much susceptible to TE as its cobalt-containing counterpart C18Ni1700.

Misra et al. [50] suggested that sulfur also plays a role in the embrittlement reaction and Ti_2CS could be an embrittling phase.

Most of the studies on thermal embrittlement have been carried out on samples homogenized at a high temperature in the range 1200–1300 °C in the austenitic phase field and cooled to a temperature in the embrittling range 800–950 °C. The fracture generally follows PAG boundaries, but material separation occurs by quasi-cleavage and local tearing, involving a certain amount of plastic deformation.

Instances of transcrystalline fracture have also been reported in embrittled maraging steels showing a relatively low level of K_{IC}. For example, Misra et al. [50] reported transcrystalline fracture in C18Ni1700 thermally embrittled to a K_{IC} level of 75–80 MPa $m^{1/2}$. They reported high aerial density of TiC(N)/Ti_2CS particles on the fracture surfaces.

Under industrial processing conditions, TE often occurs by dwelling of the hot worked structures in the embrittling range during cooling from the hot working temperatures. Under these conditions, it appears possible that the imperfections within the grains in the austenitic matrix become important sites for nucleation of titanium carbides/carbonitrides leading to fracture in a transcrystalline manner. It thus appears that TE in maraging steels cannot be explained solely in terms of precipitation of C/N at PAG's. Further, the presence of sulfur in the TiC/TiCN particles cannot be ruled out and this may influence the embrittlement process.

The role of carbon and nitrogen in causing embrittlement of the maraging steels has been clearly established. The minimum levels at which carbon and nitrogen have to be present in the steel to cause this embrittlement is worth examination. Maniar and James [47] reported that even a high purity version of 18 % Ni maraging steel might be thermally embrittled if subjected to very high temperature followed by exposure at an intermediate temperature where grain boundary precipitation is facilitated. Sinha et al. [39] reported TE in a cobalt-bearing steel at a carbon level of 50 ppm and a nitrogen level of 30 ppm. They reported a similar embrittlement in the cobalt-free steel studied by them where 60 ppm carbon and 30 ppm nitrogen were present. He et al. [51] stated that TE did not occur in the cobalt-free maraging steel containing 37 ppm carbon and 16 ppm nitrogen. Presence of carbon and nitrogen at low levels is indeed expected to bring down the proneness of the steel to TE, as schematically shown in Fig. 5. However Ti(C, N) precipitation is expected to occur only when a supersaturated Fe–Ti–(C, N) solution is exposed to temperatures in the range 780 to 950 °C during cooling. Such a treatment did not form part of He et al.'s [51] studies.

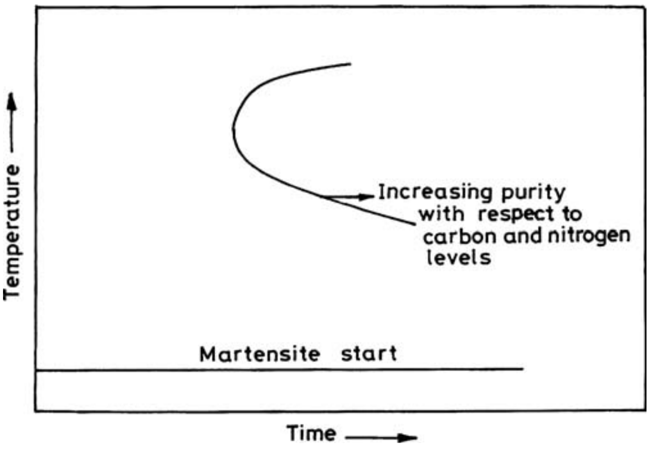

Fig. 5. TTT Diagram for Ti(C, N) precipitation at austenitic grain boundaries during cooling from high solution treatment or working temperatures (schematic).

It appears that TE is very much a problem to reckon with at carbon and nitrogen levels normally achieved, 50–100 ppm and ~30 ppm, respectively, in double vacuum melted maraging steels. Hot working and heat treatment practices have accordingly to be controlled to keep TE at bay.

6. Heat treatments to refine the PAG and to undo the TE

Sinha et al. [39] reported that no embrittlement was observed when the maraging steels were quenched from 1200 °C to room temperature and subsequently re-heated rapidly to an intermediate temperature in the range 780 to 950 °C. They attributed the non-occurrence of embrittlement when heat treated this way to the uniform precipitation of Ti(C, N) throughout the matrix on dislocations resulting from the martensite to austenite transformation. Way back in 1976, Nes and Thomas reported uniform precipitation of Ti(C, N) in C18Ni1700 quenched from 1300 °C to room temperature and subsequently reheated to 870 °C [45].

The effect of thermal cycling maraging steel between room temperature and a temperature lying well above A_f on grain refinement was reviewed in Section 4. It was also seen that substantial grain refinement occurs in the very first cycle and there is some more refinement as the cycling proceeds.

Sinha et al. [38] took advantage of the findings in the above two paragraphs to arrive at the solution to an important production problem. Maraging steel rings which were thermally embrittled suffered a large loss in toughness and there was excessive grain-boundary precipitation of second phase particles (TiC or Ti(C, N)). The toughness could be improved by solution treatment at 1200 °C followed by water quenching and cycling two times between 925 °C and room temperature. The solution treatment at 1200 °C took all carbon and nitrogen into solution in austenite and the subsequent cycling between 925 °C and room tempera-

Table 4. Effect of two-stage heat treatment on fracture toughness and ductility of 18Ni2400 grade maraging steel [51].

Heat treatment	Ultimate tensile strength (MPa)	0.2 % Yield strength (MPa)	% Elongation (4D)	% Reduction in area	Fracture toughness K_{IC} (MPa $m^{1/2}$)
820 °C Solution treatment + aging	2345 to 2365	2307 to 2350	5.0 to 6.5	25 to 31	34 to 38
950 °C + 820 °C Solution treatment + aging	2360 to 2410	2307 to 2355	8.0 to 8.5	32 to 38	41 to 42

ture ensured (i) a fine grain size and (ii) absence of carbonitrides at austenitic grain boundaries. This improved the fracture toughness of the rings [38]. The importance of the heating rate in the context of achieving grain refinement has been emphasized by Sinha et al. [39].

Two-stage/three-stage solution annealing treatments were developed for grades with strength levels up to 2400 MPa to undo the TE arising from cooling the maraging steel hardware from relatively high finishing temperatures. These treatments realize an excellent combination of strength, ductility, and toughness on a consistent basis [52, 53]. The first treatment comprises of soaking at 950 °C followed by water quenching. The 950 °C treatment causes a refinement of austenite grain size and delinking of grain boundaries and carbonitride precipitates. The fine grains formed after recrystallization at 950 °C will have boundaries relatively free from carbonitrides. The second stage in a two-stage treatment involves soaking at 820 °C followed by water quenching/air-cooling. Table 4 [53] illustrates the beneficial effect of the two-stage solution treatment over the single-stage treatment on the mechanical properties of C18Ni2400 in the aged condition. The remarkable improvement achieved with respect to ductility (% elongation and % reduction in area) and fracture toughness is obvious from Table 4.

Zechmeister et al. [54] showed that fracture toughness of maraging steel embrittled by grain boundary precipitation of titanium carbonitrides can be considerably improved by a sufficiently high amount of hot forming in the temperature range in which TE occurs. By destroying the grain boundary network of carbonitrides the hot working operation contributes to improvement in fracture toughness.

For products with a large cross-section, a three-stage heat treatment, consisting of soaking at 950 °C followed by water quenching, a second soak at 950 °C followed by water quenching and a third stage involving soak at 820 °C followed by water quenching/air cooling was developed [52, 53]. With this treatment, it was demonstrated that an excellent combination of strength and toughness could be realized even in heavy forgings and components with complex shapes (large variation in cross section from one location to another) [52, 53].

Sinha et al. [39] could get grain refinement in both C18Ni1700 and T18Ni1700 by heating from room temperature to a temperature as low as 800 °C in the austenitic phase field. Saul et al. [36] and Maki et al. [55] have reported that recrystallization in maraging steels is possible only when heating is done to temperatures above 900 °C. Saul et al. [36] emphasized that the optimum temperature for grain refinement is very sensitive to the alloy composition and must be determined for each alloy. Kuribayachi and Hariuchi [56] demonstrated that boron at a level of 30 ppm can suppress the recrystallization of the reverse transformed austenite and that 100 % recrystallization can be achieved only by increasing the solution treatment temperature to 950 °C.

Kuribayachi and Hariuchi reported that solution treatment under unrecrystallizing austenite conditions leads to refinement of martensite structures and, consequently, strengthening without any degradation of fracture toughness [56]. Sivasubramanian et al. brought out the importance of controlling the temperature for the final stage of solution annealing as close to 810 °C to realize high strength [57]. A higher temperature may result in coarsening of martensite packets and consequent drop in strength [57, 58].

In products with a large cross-section, it appears, there is a limitation on the heating rate that can be achieved in industrial practice, thus, one may not achieve adequate grain refinement after a single stage 950 °C heat treatment. A second treatment at 950 °C may be helpful in achieving the needed refinement of the microstructure. Even when heating rates are high, as reviewed in Section 4, more grain refinement occurs in the second and subsequent cycles. The final leg of solution treatment at 820 °C, where no recrystallization of austenite may be taking place, it is believed, contributes to refinement of martensite structures and consequent strengthening without any degradation of fracture toughness, as reported by Kuribayashi and Horiuchi [56].

Table 5. Heat treatment – microstructure – inspectability in ultrasonic testing interrelationship in 280 mm diameter C18Ni2400 billets [55].

Heat treatment after forging	Microstructure	Signal to noise ratio
820 °C anneal	Relatively coarse microstructure (grain size ASTM 2–3)	< 2 dB
820 °C anneal + 950 °C anneal	Relatively fine grained microstructure (grain size ASTM 5)	8–10 dB
820 °C anneal + 950 °C anneal + 820 °C anneal	Fine grained microstructure (grain size ASTM 6–7)	10–12 dB

Solution treatment at 800/820 °C may not always be adequate to recrystallize the deformed austenite. For example, in commercially produced maraging steels, small quantities of boron (≈ 20 ppm) are intentionally added. This can push up the austenite recrystallization temperature to 950 °C. Since recrystallization is an essential prerequisite for refinement of grains and creating new grain boundaries without decoration by carbonitride particles, it becomes necessary to soak at an adequately high temperature for PAG refinement by recrystallization to occur. One can thus explain the choice of 950 °C for the first (in the case of two-stage) and for the first and second (in the case of three-stage) stages of solution treatment.

The refinement of microstructure resulting from a two-stage/three-stage solution treatment as opposed to single-stage treatment also dramatically improves the inspectability of the material in ultrasonic testing (UST), as illustrated by Table 5 for a 280 mm diameter billet of C18Ni2400 [59]. After the single stage conventional treatment at 820 °C, the grain size was coarse, 2–3 on the ASTM scale, and ultrasonic inspectability was poor with signal to noise (*S*/*N*) ratio of −2 dB. A second stage treatment at 950 °C resulted in substantially refined grain size, 5 on the ASTM scale and major improvement in inspectability, *S*/*N* ratio becoming 8–10 dB. A third stage treatment at 820 °C led to still finer grain size (6–7 on the ASTM scale) and further improvement in inspectability (S/N ratio reaching 10–12 dB level). Considering that the material is used for critical aerospace applications, improved inspectability during UST is of great significance.

7. Effect of solution annealing temperature/PAG size on martensite morphology and mechanical properties

The effects of solution annealing (SA) temperature and the resulting PAG size on microstructure and mechanical properties of different grades of 18 % Ni maraging steels have been investigated by many workers. Different conclusions were reached for different grades and there is some lack of clarity. PAG size was found to increase monotonically with increase in SA temperature in both cobalt-bearing [60, 61] and cobalt-free [51, 61] grades. The situation could be different if one is starting with as-hot-worked material. The work of Misra et al. [50] showed that recrystallization of the deformed grains of the as-hot-worked structure may not take place or may not be complete at a relatively low solution temperature. A relatively high solution temperature would be necessary to complete recrystallization. There may be a drop in grain size following the recrystallization. In such a situation an increase in SA temperature would result in a drop in PAG size. In order to stay away from this confusion, the following discussion has been limited to research where data on functional dependence of PAG size on SA temperature is also available.

7.1. Martensite morphology

Results of different workers with reference to the dependence of martensite morphology on SA temperature/PAG are at variance. He et al., based on their studies on T18Ni2000 grade, found no change in martensite morphology over the SA temperature range 800–1200 °C [51]. It was a blocky martensite consisting of laths with width of 0.2–1.0 μm over the wide grain size range 20–450 μm. Other workers found a change in the martensite morphology with incrase in SA temperature/PAG size. For example, Goldberg [65] observed in C18Ni1900 that fine PAG size was associated with blocky (massive) martensite, whereas irregular shaped bands or stringers were seen with coarse PAG size. Rack [64] working with C18Ni2400 and Sinha et al. [66] who studied T18Ni1800 reported a change from blocky martensite to stringer martensite starting at 850 °C and completing at 1000 °C, and the corresponding PAG size ranges were 35–50 μm and 100–130 μm, respectively.

7.2. Strength

a) Cobalt-bearing grades

Floreen and Decker [62] studied 18.4 %Ni–7.0 %Co–4.5 %Mo–0.22 %Ti (C18Ni1600) maraging steel and found that with an increase in the SA temperature over the range 760 °C to 1260 °C the yield strength (YS) showed a declining trend (from 1682 MPa at 760 °C to 1530 MPa at 1260 °C) in the solution-treated + aged (STA) condition. The ultimate tensile strength (UTS) showed a similar trend. The PAG size was ASTM 6–8 for SA temperatures in the range 760–982 °C, ASTM 4 for SA temperature 1150 °C and ASTM 0 for ST temperature 1260 °C.

For C18Ni2400 Tuffnel and Cairns [63] reported that in the STA condition the yield and ultimate tensile strength drop sharply with increase in the SA temperature in the range 760–980 °C.

Saul et al. [36] studied the effects of PAG size on the yield and ultimate tensile strength of the C18Ni1700. There is a small but measurable drop in strength with increase in PAG size. However, the variation of PAG size in their studies was not purely a result of varying the SA temperature.

Rack [64] studied C18Ni2400 found that the relationship between the PAG size and yield strength in STA conditions obeys the Hall–Petch equation.

Zechmeister et al. [54] reported an increase in the yield and ultimate tensile strength values in C18Ni2400 with decrease in PAG size, when there was no decoration of the grain boundaries with carbides; a Hall–Petch relationship was obeyed.

b) Cobalt-free grades

The results obtained by different workers have shown considerable disagreement. The study of Sarma and Sinha [61] on C18Ni1800 showed that both yield and tensile strengths in the STA condition are independent of the PAG size. Studies of He et al. [51] on T18Ni2000 covered variation of the SA temperature over the range 800–1200 °C and a corresponding increase in PAG size from 20 to 450 μm. The yield strength in the STA condition dropped with increase in PAG size, the relationship obeying the Hall–Petch equation.

We can attempt an explanation for the disagreement among the different results in the STA condition starting with the expression used by He et al. [51] for the aged yield strength (σ_y):

$$\sigma_y = \sigma_m + \sigma_{gs} + \sigma_{d,s} + \sigma_s + \sigma_p$$

where σ_{m}, σ_{gs}, $\sigma_{d,s}$, σ_{s} and σ_{p} are the contributions to the yield strength due to the matrix, the grain size, dislocations and substructure, solid solution strengthening, and precipitation strengthening, respectively.

He et al. [51] found no change in martensite morphology in T18Ni2000 over the SA temperature range 800–1200 °C, corresponding grain size range being 20–450 µm. This means that $\sigma_{d,s}$ is independent of the PAG size. Goldberg [65], Rack [64], and Sinha et al. [66] found a change in the martensite morphology with increasing SA temperature/PAG size. Their studies indicate that $\sigma_{d,s}$ may indeed be a function of SA temperature/PAG size. It is possible that the differences in chemical composition of the steels studied by different workers contribute to different functional relationships between martensite morphology and SA temperature/PAG size. This will result in differences in dependence of $\sigma_{d,s}$ on SA temperature/PAG size.

Precipitation reactions are also influenced by the dislocations and the substructure of the martensite matrix in the solution-treated condition; therefore, if there is a change in the nature of martensite with SA temperature/PAG size, there will be an effect of SA temperature/PAG size on σ_{p}. Further, with progress of precipitation, the dislocation and substructure would be affected; i. e. with σ_{p} building up, $\sigma_{d,s}$ would be affected.

The chemical composition, with particular reference to the levels of cobalt, molybdenum, and titanium, would have an important effect on σ_{p} and σ_{s}. As discussed in Section 3, the nature of precipitating phases and the kinetics of precipitation can sensitively depend on the chemical composition. Added to this are differences in aging temperatures and aging times adopted by different workers.

The different functional relationships reported between SA temperature/PAG size and yield strength by different workers are believed to arise from the effects discussed above.

7.3. Ductility

a) Cobalt-bearing grades

Floreen and Decker [62] found in their studies on C18Ni1600 that ductility values (% elongation and % reduction in area) in the solution-treated and aged condition largely remain constant over the SA temperature range 760–1150 °C (12–13 % in 25.4 mm and 53–58 %, respectively) but show deterioration after solution treatment at 1260 °C (10 % and 44 %, respectively). It may be recalled that the PAG size was 4 and 0 on the ASTM scale after solution treatment at 1150 and 1260 °C, respectively.

Nes and Thomas [45] worked with C18Ni1700 and reported after solution treatment at 1300 °C, reheating to 870 °C and aging for 3 h at 480 °C, very poor ductility (1.8 % elongation and 9.5 % reduction in area), even though the material was not in a thermally embrittled condition. It appears that the large PAG size after solution treatment at 1300 °C persisted even after 870 °C treatment and led to the poor ductility. Rack [67] reported that increasing PAZ size in C18Ni2400 results in a decrease in ductility.

The author's own studies on C18Ni1700 revealed that solution treatment at 1150 °C followed by solution treatment at 820 °C yielded a coarse-grained microstructure, and after aging some of the ductility values were very low, getting into the unacceptable range.

b) Cobalt-free grades

Sinha et al. [66] reported in T18Ni1800 that the ductility in the STA condition decreases with increasing SA temperature/PAG size.

He et al. [51] studied the ductility in T18Ni2000 in the STA condition and found that there was practically no change in ductility up to 1100 °C; thereafter the % elongation and % reduction in area dropped catastrophically from 10.4 to 5.0 % and 50.0 to 13.4 %, respectively. The rate of grain growth was found to be very high when the solution treatment was carried out at > 1100 °C.

It can be seen that there is a broad agreement among the results published by various workers on the effect of SA temperature/PAG size on ductility. Relatively high SA temperatures can lead to very fast grain growth resulting in a catastrophic drop in ductility.

7.4. Toughness

The results obtained by different workers have shown considerable disagreement. This is illustrated by studies on cobalt-free grades. The study by Sinha et al. [66] and Sarma and Sinha [61] on T18Ni1700 showed that fracture toughness in the STA condition decreases with increasing SA temperature. The decrease in fracture toughness was attributed to a change in martensite lath morphology from blocky to stringer type. Studies of He et al. [51] on T18Ni2000 showed that the fracture toughness in the STA condition increases with increasing SA temperature over the range 800–1200 °C with martensite morphology remaining the same throughout. Figure 6 shows the differences.

There is considerable difference in the chemical composition of the cobalt-free maraging steels studied by Sinha et al. [30] and He et al. [9, 10] as shown in Table 6. This could have led to the difference in the dependence of the martensite morphology on SA temperature/PAG size reported by them, as discussed in Section 7.1. It appears that this difference contributes to the disagreement shown in Fig. 6 between the results of the two sets of workers. The differences shown in Table 6 between the aging conditions used by the two sets of workers for generating the data in Fig. 6 might also have had a contribution. It is hence not possible to make generalized observations on the effect of SA temperature/PAG size on K_{IC}.

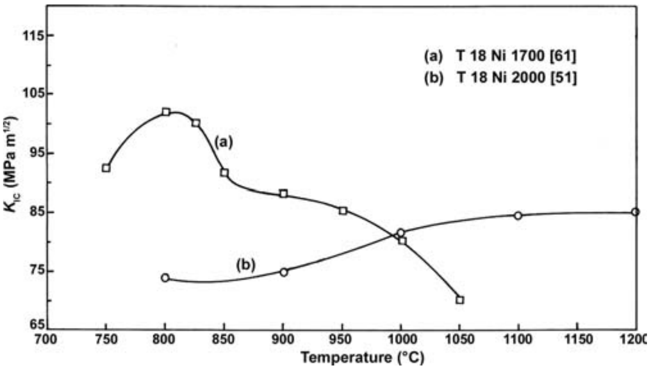

Fig. 6. Variation of fracture toughness with SA temperature.

Table 6. Studies on cobalt-free maraging steels by He et al. [8, 9] and Sinha et al. [29]: differences in chemical composition and aging parameters.

Researchers	Chemical composition (wt.%)			Aging temperature (°C) and time (h)
	Nickel	Molybdenum	Titanium	
He et al.	18.9	4.1	1.4	480, 12
Sinha et al.	17.1	2.25	1.9	480/500, 3

There is need to do further research to study the interrelationships among chemical composition, processing/solution treatment, and morphology of martensite and mechanical properties.

The phenomenon of thermal embrittlement comes into picture when relatively high SA temperatures are used with slow cooling (air cooling in large sections) following thereafter. This is borne out by the studies of Misra et al. [50] and Sarma and Sinha [61]. The subject of thermal embrittlement was discussed in detail in Section 5.

From an engineering point of view, one often needs to work with a material with an optimum combination of strength, ductility and toughness. He at al., after scanning the results obtained on T18Ni2000 grade over the SA temperature range 800 – 1200 °C in this respect, concluded that 800 °C with an associated PAG size of 20 μm leads to optimum performance. It is important to have finer grains for improved fatigue and environmental strength [68]. Yasunaka et al. [69, 70] suggested that the grain size must be reduced to prevent unstable low-stress failures. Rack [64] reported that coarse-grained, high-strength maraging steels could fail catastrophically. In summary, there is advantage in adopting a solution treatment that would yield a relatively small PAG size if an optimum combination of strength, ductility and toughness is to be achieved.

8. Effect of austenite in the microstructure on mechanical properties

Considering the well-accepted effect of retained austenite to increase toughness in martensitic stainless steels [71], several attempts were made to improve toughness in maraging steels by bringing in some amount of austenite into the microstructure through overaging or through thermal cycling.

Overaging: The intermetallic precipitates formed during aging are not the stable equilibrium phases in the sense that prolonged aging would lead to the formation of equilibrium austenite and ferrite. The response to austenite reversion depends on the alloy composition. Since nickel, one of the major alloying elements, is an austenite stabilizer, the reversion tendency depends on whether the alloying elements enrich or deplete the matrix with respect to nickel.

Thermal cycling: When standard production practice is followed, < 2 % retained austenite is present in maraging steels in the solution-treated condition. Increased levels can be realized at room temperature after thermal cycling as discussed in Section 4.

Diverse conclusions have been arrived at as to how the presence of austenite modifies the fracture toughness and strength.

Kenyon [72] expressed the view that reversed austenite may be detrimental to toughness; deformation under load is concentrated in the softer austenite, which therefore reaches its critical strain for fracture at an early stage.

Carter [73] found a beneficial effect of reversed austenite on fracture toughness of 16.3Ni-12.87Co-4.98Mo-0.78Ti maraging steel, toughness monotonically increasing and UTS and YS values monotonically decreasing with increasing reversed austenite content.

While verifying the possible effect of reverted austenite exercising crack blunting and thereby enhancing fracture resistance, Rack and Kalish [74] found that toughness was not altered by varying the amount of reverted austenite in C18Ni2400. They found evidence that the beneficial effect of reverted austenite may be counteracted by the brittle nature of the matrix in which overaged particles serve as excellent crack nucleation sites.

Pampillo and Dixton [75] studied reversion of austenite in C18Ni1400. During the early stages of aging (0.3 h of aging at 565 °C), reverted austenite precipitating along martensite lath boundaries improves toughness. After overaging (39 h at 565 °C), reverted austenite precipitated at PAG boundaries and there was a drop in toughness. The intermetallic precipitate particles that grew in size after overaging and the precipitation of reverted austenite at PAG boundaries were stated to be responsible for the decrease in toughness. The authors found that the yield strength increases with increasing volume fraction of reverted austenite.

Moriyama and Takaki [76] reported that a small amount of reverted austenite (2 – 3 vol.%) in C18Ni1700 does not reduce the hardness so much, but suppresses crack growth during fatigue loading; the maximum length of the non-propagating crack becomes longer in the presence of reverted austenite. The existence of reverse austenite also improves fracture toughness.

Sinha et al. [30] studied T18Ni1700 grade and reported poor toughness in the overaged condition; they concluded that the formation of reverted austenite and coarsening of precipitates was responsible for the poor toughness.

Viswanathan et al. [77] studied the effect of reversed austenite formed by overaging in C18Ni2400 on its mechanical properties. Reversed austenite caused a decrease in yield strength, ultimate tensile strength, and ductility. Impact toughness is higher at the initial stages of overaging (when % austenite is 13), but prolonged overaging (austenite content of 22 – 24 %) induced severe embrittlement. The authors concluded that the embrittlement is associated with the coarsening of intermetallic precipitates formed during the early stages of aging.

He et al. [10] studied the formation of austenite during overaging and its effect on mechanical properties of

T18Ni2000. Overaging at an intermediate aging temperature of 480 °C for 50 h led to formation of 9 % austenite at martensite lath boundaries, a drop in strength and an increase in toughness. Overaging at 540 °C, on the other hand, led to formation of austenite at PAG boundaries, martensite lath boundaries followed by formation inside laths resulting in serious drop in strength with no significant increase in toughness.

The morphology of reverted austenite is decided by the aging temperature and time [19, 78]. Reverted austenite with different morphological features was identified by Viswanathan et al. [19, 77] and He et al. [10]. Micrographs a, b and c in Fig. 7 illustrate the Widmanstatten, recrystallized and globular morphologies respectively, of reversed austenite observed by Viswanathan et al. [77] in overaged C18Ni2400. Morphology of austenite is also expected to influence the mechanical properties of maraging steels [79].

In the cobalt-containing grades, it has generally been agreed that when metastable Ni_3(Ti, Mo) precipitates dissolve and Fe–Mo-based precipitates appear there occurs a localized Ni enrichment leading to the formation of reversed austenite in the microstructure [4, 31, 33, 74, 80]. In the cobalt-free grades, Sinha et al. [81] and He et al. [10] concluded that austenite reversion arises from nickel enrichment resulting from dislocations and other defect structures. At longer overaging times, He et al. observed austenite reversion arising from dissolution of precipitates. Austenite reversion during overaging is retarded in cobalt-free grades, compared to that in their cobalt-bearing counterparts [25, 81].

The studies referred to above are based on generating austenite by overaging. Viswanathan et al. [32], on the other hand, introduced austenite in C18Ni2400 by thermal cycling between room temperature and 750 °C. With increasing content of retained austenite the toughness increases, but the tensile and yield strength values decrease.

Antolovich et al. [82] introduced austenite into C18Ni2000 through a non-standard heat treatment (thermal cycling) and found that this resulted in an improvement in fracture toughness.

One can attempt to understand the disagreement between the toughness results reported by different workers by noting that two reactions are taking place simultaneously during overaging. One is the formation of an increasing amount of the soft austenite in the microstructure with its expected crack blunting effect; the other is the coarsening of precipitate particles, facilitating easy crack nucleation at the particles. Austenite reversion is expected to result in improvements in the toughness of the maraging steel, if there is no substantial coarsening of precipitate particles to facilitate easy crack nucleation. The chemical composition of the steel and the aging temperature are expected to influence the kinetics of austenite reversion and the coarsening of precipitate particles significantly. There are indeed significant differences in the chemical composition of the steels investigated by different workers. Further, there are differences in the temperatures and times used by different workers for overaging; this is expected to influence the morphology and volume fraction of reverted austenite and in turn the mechanical properties of maraging steel.

While examining the effect of austenite on the mechanical properties of maraging steel, a distinction should also be made between retained austenite formed by thermal cycling and reverted austenite formed by overaging. The two types of austenite may differ in terms of the morphology and fineness of distribution. Resistance to fracture offered by them could hence be different. The quantitative depen-

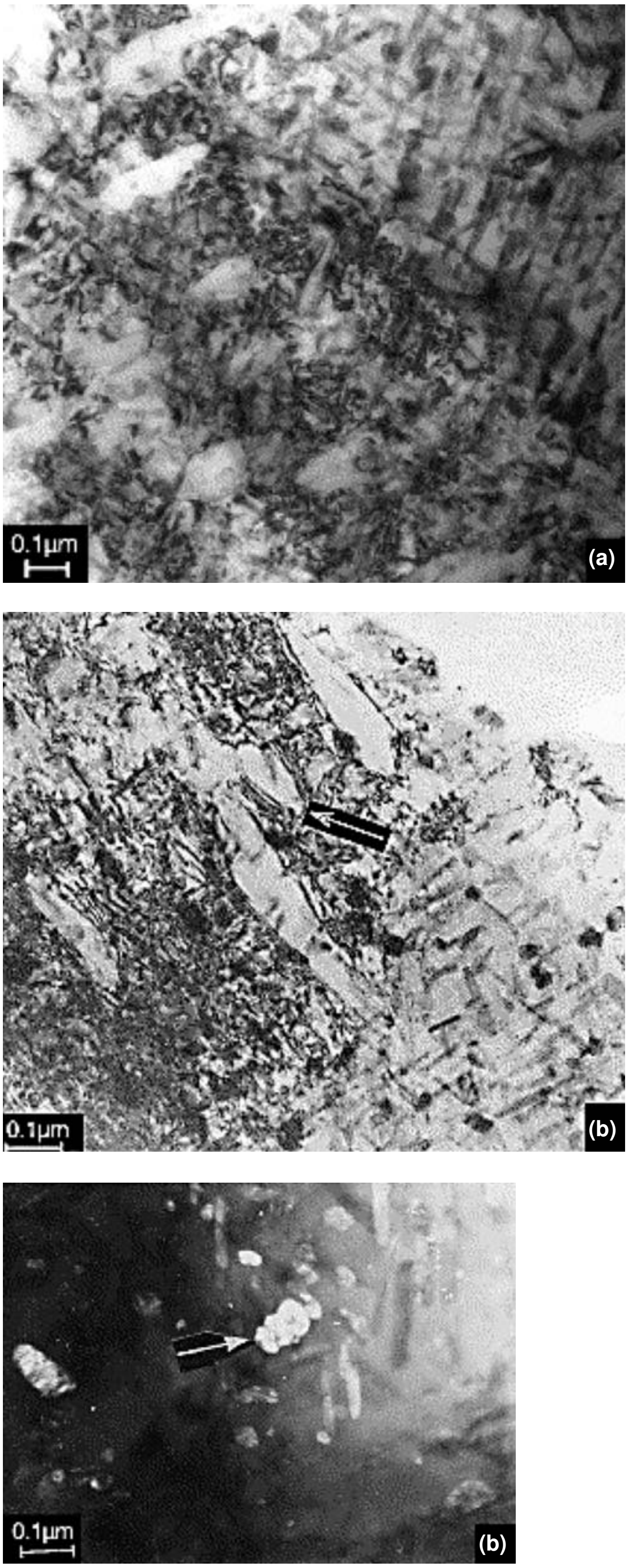

Fig. 7. Transmission electron micrographs of C18Ni2400 aged at 640 °C for 8 h showing: (a) Widmanstätten austenite, (b) recrystallized austenite, and (c) globular austenite.

dence of mechanical properties on austenite morphology is an interesting area for further research.

It appears that significant toughness improvement without a drastic drop in yield strength is realized at relatively low levels of reversed austenite, finely distributed along martensite lath boundaries. However, it remains difficult to recommend specific overaging treatments to improve the fracture toughness on a reproducible basis without significant loss in yield strength. Instead, lower strength variants of maraging steels in the peak-aged condition can give equally good strength–toughness combinations. There has, thus, been no driving force to pursue the approach of developing optimum overaged conditions to exploit improved toughness of maraging steels. Consequently, maraging steels are not being used for critical applications in the overaged condition.

It has been clearly established in the case of cobalt-containing maraging steels that austenite reversion occurs by dissolution of metastable $Ni_3(Ti,Mo)$ precipitates and the appearance of Fe–Mo-based compounds leading to localized Ni enrichment. In cobalt-free grades, the $Ni_3(Ti, Mo)$ precipitates are relatively more stable and Fe–Mo-based compounds do not precipitate or appear very belatedly. Under the circumstances, nucleation at dislocations and lattice imperfections appears to be an important mechanism for austenite reversion.

9. Conclusion

With reference to realizing maraging steels based on Fe–Ni martensite with outstanding combinations of mechanical properties – strength, ductility and toughness – and with a high degree of consistency from batch to batch, there has been a phenomenal success. Significant advances have been made in our understanding of precipitation reactions in martensite. Response of maraging steels to thermal cycling has been well studied and commercially exploited. The heat treatments available today are able to completely take care of variations in the preceding hot working phase of manufacture and consistently lead to an indeed high level of mechanical properties, even in large section sizes. The phenomenon of austenite retention/reversion has been widely researched both in terms of microstructure and mechanical properties. The cobalt-free maraging steels have become a commercial reality and several studies on these materials carried out in the recent years are bound to help in further optimizing the operating/application regimes.

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(Received January 20; accepted June 17, 2006)

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