

## Addressing the issue of precipitates in maraging steels – Unambiguous answer

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### ABSTRACT

Despite several decades' long study, the identity of the precipitating phases responsible for the strengthening of maraging steels is still not clear. In the current work, this issue was extensively investigated using experimental and theoretical approaches. First, in-depth characterization of the precipitates in C250 steel and the precipitation order were performed through a combination of various Transmission Electron Microscopy (TEM) methods. In parallel, thermodynamic calculations were used for the prediction of the phase content at equilibrium. Then, in order to isolate the effects of the different precipitates, model alloys were cast and aged. It was shown that the phases responsible for the strengthening during the initial stages of aging are Ni<sub>3</sub>Mo and Ni<sub>3</sub>Ti. In the over-aged (close to equilibrium) condition, the steel consists of martensite, reverted austenite, Ni<sub>3</sub>Ti, and Fe–Mo phases. This conclusion was found to be in perfect agreement with thermodynamic calculations. The formation of Ni<sub>3</sub>Mo at early stages of aging, despite its calculated lowest driving force for formation, was attributed to a low barrier for formation.

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## 1. Introduction

Maraging steels are low-carbon nickel-rich martensitic steels characterized by a unique combination of high strength and good fracture toughness. The most common steels of the 18 Ni grades are C200, C250, C300, and C350, where numerical designation represents the yield strength (in KSI) of the steel grade [1–3].

The exceptional mechanical properties of the steels stem from the strengthening of a soft martensitic matrix by intermetallic precipitates of nanometer size, formed in the following path [4]: upon cooling from solution temperature, the Ni-rich austenite matrix transforms to soft lath martensite with high dislocation density [1]. Subsequent ageing at temperatures between 450 °C and 550 °C leads to precipitation of intermetallic phases [5]. Despite numerous studies on this subject, there is still a discrepancy among the different reports regarding the type of precipitates formed in these steels [6]. In the review by Decker et al. [2], the authors attribute the hardening in C250 steels to the appearance of Ni<sub>3</sub>Mo precipitates. Vasudevan et al. [5,7] contradicted this finding, reporting that strengthening of C250 steel results from the combined presence of Ni<sub>3</sub>Ti (initially) and

Fe<sub>2</sub>Mo precipitates (at longer times). In a series of studies carried out by Sha et al. [4,8–12], combining atom probe characterization with thermodynamic calculations, they concluded that the driving force value gives a good measure of the sequence of precipitation. Atom probe measurements indicated the formation of Ni–Ti rich precipitates. Since the highest driving force was obtained for the Ni<sub>3</sub>Ti phase, it was concluded that this phase is the first to precipitate from the BCC matrix. Fe<sub>2</sub>Mo<sub>6</sub> was reported to appear at advanced stages of aging. This result is in line with that of Vasudevan et al. [5,7] and contradicts Decker et al. [2]. Thus, it is still not clear which precipitates are responsible for strengthening and the order of precipitation.

Identification of the precipitates is hampered due to the limitation of the experimental methods. Two of the used methods for such studies are Transmission Electron Microscopy (TEM) and Atom Probe Tomography (APT) [13–18, for example]. However, TEM's resolution is impaired while studying maraging steels, due to strong magnetic effects caused by the martensitic matrix. In addition, the similarity in interplanar distances, distinct orientation relationships with the matrix, and double diffraction phenomena impose substantial difficulties in distinguishing between the possible precipitating phases from electron diffraction patterns. On the other hand, the lack of crystallographic structure in APT measurements impedes the ability to fully identify the precipitate type solely on the basis of this technique.

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The purpose of current study is to shed light on the nature of the strengthening precipitates in C250 maraging steel and to gain a better understanding of the correlation between thermodynamic predictions and the sequence of precipitation. The precipitates were characterized by both analytical and conventional TEM, starting from very short aging times up to extensive over-aging, which is close to the equilibrium state.

## 2. Materials and methods

### 2.1. Thermodynamic calculations

For the thermodynamic calculations, the Thermo-Calc software package [19] was used. Thermo-Calc is designed for thermodynamic and phase diagram calculations for multi-component systems. The calculations are based on thermodynamic databases produced by expert evaluation of experimental data using the CALPHAD method [20,21].

In the current study the calculations were based on three databases:

- PKP, Kaufman binary alloys database [22].
- SSOL5, SGTE (Scientific Group Thermo-data Europe) Solutions database version 5 [23].
- TCFE7, TCS Steels/Fe-alloys database version 7 [24].

The PKP database is based on thermodynamic modeling performed in the 1970s and contains only binary interactions. It was used here as a reference, for comparison with the work of Sha [8,9] who used this database. SSOL5 and TCFE7 are modern and up-to-date databases that contain both binary and ternary interactions.

The calculations were made for the composition of maraging C250 steel at 510 °C and atmospheric pressure. An equilibrium calculation was performed that considers all possible phases in the

**Table 1**  
Nominal chemical compositions [in wt%] of the studied alloys.

	Ti	Co	Mo	Ni	Fe
C 250	0.5	10	5.5	18	Bal.
M-type	–	9.26	4.17	15	Bal.
T-type	2.13	9.18	–	15.34	Bal.

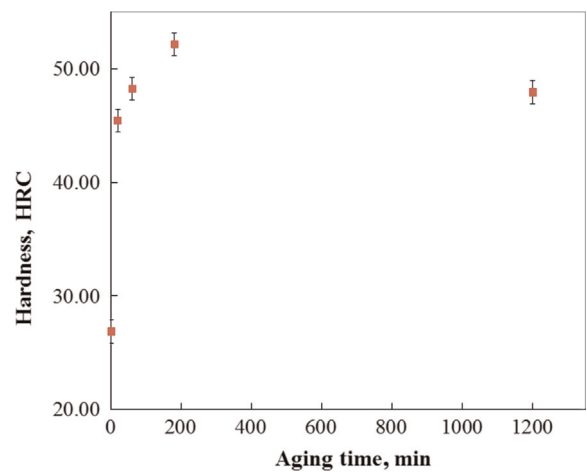
**Table 2**  
Driving force ( $\Delta G/RT$ ) for precipitation of possible phases in C250 steel at 510 °C.

Phase	Composition	Crystal structure	PKP	SSOL <sub>5</sub>	TCFe <sub>7</sub> [19]
Martensite( $\alpha$ Fe)	Mostly Fe	BCC $a=2.482 \text{ \AA}$	0	0	0
Austenite( $\gamma$ Fe)	Mostly Fe	FCC $a=2.023 \text{ \AA}$	0	0	0
Ni <sub>3</sub> Ti	Ni <sub>3</sub> Ti	Hexagonal $a=5.092 \text{ \AA}$ $c=8.297 \text{ \AA}$	–0.21	0	0
$\mu$ phase	Fe <sub>7</sub> Mo <sub>6</sub>	Rhombohedral $a=4.751 \text{ \AA}$ $c=8.988 \text{ \AA}$ $\gamma=120^\circ$	0 Fe <sub>3</sub> Mo <sub>2</sub>	0 Fe <sub>0.36</sub> Mo <sub>0.46</sub> ~FeMo	0 Fe <sub>0.46</sub> Mo <sub>0.54</sub> ~FeMo
Laves phase	Fe <sub>2</sub> Mo	Hexagonal $a=4.755 \text{ \AA}$ $c=7.767 \text{ \AA}$	–0.87 ~FeTi	–0.08 Fe <sub>0.66</sub> Mo <sub>0.33</sub> ~Fe <sub>2</sub> Mo	–0.08 Fe <sub>0.66</sub> Mo <sub>0.33</sub> ~Fe <sub>2</sub> Mo
Ni <sub>3</sub> Mo	Ni <sub>3</sub> Mo	Orthorhombic $a=4.224 \text{ \AA}$ $b=4.448 \text{ \AA}$ $c=5.064 \text{ \AA}$	–1.39	–1.25	–1.09

system. The results include not only the set of stable phases but also the driving forces for the formation of metastable phases. The driving force is defined here as  $(-\Delta G/RT)$ , where  $\Delta G$  is the Gibbs energy difference due to the formation of the phase. Hence, the values are contra-intuitive. A phase that is at equilibrium has zero driving force [10], and the driving force for a phase that is predicted as non-existing at equilibrium has a negative value. Since Ni<sub>3</sub>Mo phase was not included in the TCFE<sub>7</sub> database, the parameters set for the Ti–Ni–Mo ternary system were taken from Santhy and Kumar [25] and added to the database.

### 2.2. Studied material and experimental methods

Experiments were carried out on commercial C250 steel and two model alloys: Fe–Ni–Co–Mo (marked as M-type) and Fe–Ni–Co–Ti (marked as T-type). The composition of the model alloys was set to produce precipitation of only one type of precipitate, Mo-based in the M type (i.e., no Ti was added to the alloy) or Ti-based in the T type (i.e., no Mo was added to the alloy). The detailed compositions of the studied alloys are given in Table 1. The model alloys were prepared from high purity (99.995%) shots of the elemental ingredients, mixed to the desired compositions, arc melted five times under high purity (99.999%) argon atmosphere, solution treated at 1130 °C for two hours, and aged at



**Fig. 1.** Hardness of C250 steel as a function of time of aging at 510 °C.

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