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Splitting phenomenon in the precipitation evolution in an Fe–Ni–Al–Ti–Cr stainless steel

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Abstract

The evolution of precipitation and mechanical properties of an Fe–Ni–Al–Ti–Cr stainless steel was studied during ageing at 525 °C. Atom probe tomography and transmission electron microscopy were applied to follow the microstructural evolution. An initial hardening reaction, which is remarkable in terms of extent, is reported to be caused by the formation of complex multi-component clusters. They are composed mainly of Ni, Al and Ti. After ageing to peak hardness (3 h), splitting of these clusters into spherical and elongated particles was observed. Based on the chemical composition, the spherical precipitates were identified to be of type NiAl B2, and the elongated particles were associated with the η-phase (Ni_3(Ti, Al)). Both types of precipitates contribute to the strength of the material.
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Keywords: Maraging steel; Atom probe tomography; Precipitates

1. Introduction

The era of maraging steels started in the 1940s, when Bieber [1] was working on iron–nickel magnetic alloys. He had already noted that some of his Fe–28Ni–4Ti–4Al alloys could be significantly hardened by heat treatment. Those first and further works led to the development of the well-known maraging steels based on the Co–Mo hardening system, namely the so-called 18Ni (2 0 0), 18Ni (2 5 0) and 18Ni (3 0 0) alloys. However, owing to the tremendous jump in cobalt prices in the period 1978–1980, the development of cobalt-free grades was promoted. These cobalt-free grades generally featured inferior properties compared with cobalt-containing grades, but their properties appeared sufficient for typical applications of maraging steels.

The precipitation behavior and strengthening mechanisms in cobalt-free grades have been studied extensively, employing a variety of characterization techniques. It has been shown that maraging steels containing Ni and Al are strengthened by the formation of the ordered β′-NiAl-phase [2–5]. It is generally thought that their formation proceeds via solute-rich clusters within the martensitic matrix. Out of these nuclei, coherent NiAl precipitates are formed. They show a spherical shape, and they are uniformly distributed within the matrix [6].

In contrast, it has been reported that strengthening in Ti-containing maraging steels is caused by the precipitation of the hexagonal η-phase (Ni_3Ti) [7–10] exhibiting an orientation relationship to the martensitic matrix of Burgers type [10–13]. In the literature, some debate exists on the mechanism of the formation of the η-phase. In most studies found in the literature, heterogeneous nucleation on dislocations is proposed. The subsequent growth takes place via pipe diffusion [14–16]. Others advance the theory that, first, the formation of coherent zones on dislocations in the martensitic matrix takes place, which act as nucleation sites for the η precipitates [10,14]. There is also a discrepancy in

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the dominating strengthening mechanism in this kind of maraging steels. While the majority of the studies attributed strengthening to the formation of Ni_3Ti, others proposed an additional contribution from some B2-type ordering of Fe and Ni atoms in the matrix [10].

However, Gemperle et al. [17] reported that, when Si is supplemented in Ti-containing maraging steels, the so-called G-phase exhibiting the chemical composition $Ti_6Si_7Ni_{16}$ forms during ageing. Depending on the chemical composition of the alloy, the G-phase ($Ti_6Si_7Ni_{16}$) and the η-phase (Ni_3Ti) can precipitate either simultaneously or separately [17]. The shape of the Ni_3Ti-phase was thought to be rod-like, and the G-phase shows a spherical morphology. Recently, atom probe tomography (APT) was used to follow the precipitation sequence of an Fe–Ni–Al–Ti–Cr alloy containing ~1.1 at.% Si during ageing at 525 °C [18]. In that work, an undefined precursor phase was observed during the early stages of ageing. This precursor phase causes strengthening up to peak hardness and acts as nuclei for the formation of precipitates when further ageing is applied. Out of these nuclei, both the spherical G-phase and the rod-shaped η-phase are formed independently. The observed splitting phenomenon is reported to affect the mechanical properties detrimentally.

However, the improvement and development of maraging steels requires a fundamental understanding of the role of various alloying elements on the evolution of precipitation during ageing. The studies described above leave some open questions, especially in the Ti-containing grades, e.g., how do the precipitates nucleate during ageing.

Thus, the present study focuses on the precipitation evolution in Si-free Fe–Ni–Al–Ti–Cr maraging steels. APT and transmission electron microscopy (TEM) were used to follow solute distribution, precipitates and microstructure.

2. Experimental

The steel grade investigated is a commercial Ti-containing maraging steel of type Fe–Ni–Al–Ti–Cr. This stainless steel grade is alloyed with Ni, Al and Ti to form nanometer-sized precipitates and with Mo to improve the stainless properties. The chemical composition of the steel investigated is listed in Table 1. It should be noted that all further compositions presented in this paper are given in atomic per cent. For the experiments, the maraging steel was solution annealed at 850 °C for 0.5 h, followed by air cooling. Ageing was performed at 525 °C for 0.25, 3 and 10 h, followed by air cooling. The samples to be investigated were cut from the bulk material using a diamond saw. To follow the effect of ageing on mechanical properties, Rockwell hardness tests (HRC) were performed.

Table 1
Chemical composition of the maraging steel investigated.

	Fe	Cr	Ni	Si	Ti	Al	Mo
wt.%	Bal.	12.12	9.05	0.05	0.35	0.7	2.0
at.%	Bal.	12.96	8.57	0.1	0.41	1.44	1.16

All material conditions were investigated by APT and selected ones by TEM. In order to prepare tips for the atom probe investigations, small rods with cross section $0.3 \times 0.3\ mm^2$ were cut out of the aged bulk material. Subsequently, the tips were prepared in two steps: first, by electropolishing of the small rods in a layer of 15% perchloric and 85% acetic acid solution topped over a dielectrical liquid called galden, which is a perfluoropolyether from Solvay Solexis; and secondly, using an electrolyte of 2% perchloric acid in butoxyethanol [19]. The atom probe analyses for all samples were conducted on a 3DAP™-X from Oxford Nanoscience. The measurements were performed at a temperature of ~60 K with a pulse fraction of 15% under ultra-high vacuum ($<10^{-10}$ mbar). The reconstruction procedure and the analysis were conducted with IVAS 3.4.1™ software from Imago Scientific Instruments.

TEM investigations were performed on a Philips CM12 equipped with a Si (Li) EDS-detector from EDAX. For TEM investigations, specimens 3 mm in diameter were stamped out of the material. The small discs were mechanically polished to a thickness of ~100 μm. Subsequently, these discs were thinned to electron transparency using the electrolytic double jet technique (Struers Tenupol-5). The electrolyte used was a mixture of ethanol, butylcellosolve, perchloric acid and distilled water (Struers Electrolyte A-2).

3. Results

3.1. Hardness measurements

Fig. 1 shows the evolution of the hardness of the investigated steel grade as a function of ageing time at an ageing temperature of 525 °C. It can be seen that, in the as-quenched state, the material exhibits a relatively soft mechanical behavior. In this condition, the hardness is comparable with what was obtained for similar steel

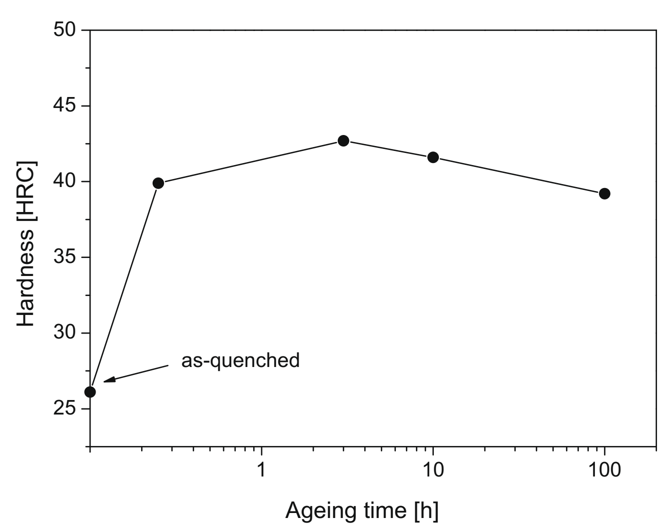

Fig. 1. Hardness as a function of ageing time at 525 °C of the alloy investigated.

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