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Analysis of the precipitation behaviour in a high-speed steel by means of small-angle neutron scattering

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Abstract

Small-angle neutron scattering (SANS) and energy-filtered transmission electron microscopy (EFTEM) were employed to characterise the precipitation behaviour in the high-speed steel HS6-5-2 during tempering. With SANS the evolution of the size distribution of precipitates during isothermal tempering at 590 °C was investigated. The SANS results are discussed assuming that three populations of precipitates can be distinguished during tempering at 590 °C. The smallest population with sizes around 1 nm is supposed to be carbon-rich clusters. Intermediate-sized particles between approximately 1 and 10 nm might be either MC and/or M_2C carbides, which cannot be distinguished by SANS. In addition, the ratio *A* of magnetic and nuclear scattering intensity was used to gain information on the composition of the precipitates. The *A*-ratio is discussed assuming a substitution of iron in the precipitates by carbide forming elements with increasing tempering time. Finally, the correlation of the results obtained by SANS with those achieved by EFTEM is presented.

Keywords: High-speed steel; Precipitation behaviour; Small-angle neutron scattering

1. Introduction

During the various stages of the heat treatment of high-speed steels different precipitation reactions occur. The microstructure of high-speed steels in the as-quenched stage consists of blocky primary carbides in a martensitic matrix. A small volume fraction of proeutectoid and/or autotempered carbides is also likely to be present because of precipitation during quenching. Subsequent triple tempering leads to precipitation of fine secondary hardening carbides which strengthen the martensitic matrix. During tempering (aging) the proeutectoid and autotempered carbides coarsen or dissolve. Depending on steel composition and tempering temperature the secondary hardening carbides are of the types MC and/or M_2C in various volume fractions [1–3]. The secondary hardening carbides precipitate as small disks or needles obeying defined orientations with respect to the martensitic matrix [4]. The effect of overaging results in the precipitation of M_6C and $M_{23}C_6$ at martensite plate boundaries and prior austenite grain boundaries. This reaction occurs on the expense of the secondary hardening carbides [3].

Extensive studies concerning the tempering and overaging behaviour of high-speed steels have been conducted [1–3,5]; however, the knowledge is still limited. The smallness of the carbide precipitates makes it difficult to characterise them even by direct microscopy, like transmission electron microscopy (TEM) or atom probe field ion microscopy (APFIM). Moreover, both methods are restricted to a relatively small sample volume and, therefore, it is very time consuming to obtain representative size distributions by TEM or APFIM.

As modelling of precipitation reactions as well as mechanical properties requires information on type, volume fraction and size distributions of the precipitates, complementary methods have to be applied. Small-angle neutron scattering (SANS) is a suitable technique for characterisation of

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precipitation reactions especially at the early stages when precipitates are too small for TEM as it was shown, e.g., for maraging steels [6]. Due to the ferromagnetic nature of the martensitic matrix, additional information with regard to the chemical composition of the precipitates can be gained from the ratio of the magnetic and nuclear small-angle scattering intensity.

In this work, SANS was used to study the precipitation reactions in the steel grade HS6-5-2 during isothermal tempering at 590 °C. In order to exclude possible influences of cooling and heating between the tempering steps only single tempering was carried out. The SANS results are compared with results derived from energy-filtered transmission electron microscopy (EFTEM).

2. Experimental

2.1. Sample preparation

The nominal chemical composition of the high-speed steel grade HS6-5-2 is listed in Table 1. Samples with a diameter of 25 mm and a thickness of 1 mm were used for SANS investigations. All samples were austenitised at 1210 °C for 10 min and subsequently quenched in oil. Afterwards, single tempering was carried out at 590 °C for 10, 20, 60, 180 and 540 min.

2.2. SANS

SANS was employed to analyse the evolution of the size distribution of precipitates during tempering. Basics of the SANS technique can be found elsewhere [7]. The measurements were performed with the instrument SANS-2 at the Geesthacht Neutron Facility (GeNF). Selector-monochromated neutrons with a mean wavelength of $\lambda = 0.57$ nm and a wavelength spread of $\Delta\lambda/\lambda = 10\%$ were used. The neutron beam impinging on the 1 mm thick samples had a diameter of 8 mm. The samples were situated in a vacuum chamber without any windows influencing the beam, thus giving a low background. The samples were magnetized to saturation in a field of 2 T. Four different detector distances (1, 3, 9 and 21 m) were used together with appropriate collimations to cover scattering vectors q from 0.03 to 3 nm^{-1} ($q = 4\pi \sin(\theta)/\lambda$, where 2θ is the scattering angle). Scattered neutrons were recorded with a 50 cm × 50 cm area detector using 128 × 128 pixels. Measured intensities were corrected for the sample transmission, background intensity and detector response. Macroscopic differential scattering cross sections were obtained by calibration with a Vanadium standard. The error in the absolute scale of the obtained volume fractions introduced by this calibration is approximately 5%. Additional sources of error are mainly the measured sample transmission T_S (1%) and the sample thickness D (2%). The statistical error in the measured intensities is accounted for in the fitting procedure to calculate error bands for the obtained size distributions. The additional error in the absolute scale of the obtained volume fractions resulting from the errors in calibration, T_S, and D is approximately 6%. This additional error is added to the error in the total volume fractions obtained from the fit.

Table 1
Chemical composition of HS6-5-2 high-speed steel

	Nominal chemical composition									
	C	Cr	W	Mo	V	Si	Mn	Ni	Co	Fe
wt.%	0.9	4.1	6.4	5.0	1.8	0.27	0.27	0.18	0.84	Bal.
at.%	4.3	4.5	2.0	3.0	2.0	0.55	0.28	0.18	0.82	Bal.

2.3. EFTEM

EFTEM was used to acquire information on shape and size of the precipitates. An extensive description of this technique is given in [8]. The specimens were prepared employing standard methods [9]. For the investigations, a Philips CM20/STEM equipped with a Gatan imaging filter was used.

3. Results

3.1. Particle size distributions obtained from SANS

Different types of particles are expected to be the dominant source of SANS intensity in this type of steel: primary carbides that are stable at the tempering temperature, proeutectoid carbides that can dissolve or coarsen at tempering temperature and secondary hardening carbides that precipitate from the supersaturated matrix during tempering [1]. Consequently, changes in the SANS scattering curves during tempering are discussed in terms of changes in the size distribution of particles.

The SANS scattering cross sections were analysed by means of a so-called two-phase model according to [7],

$$\frac{\mathrm{d}\Sigma}{\mathrm{d}\Omega}(q) = (\Delta\eta)^2 \int_0^\infty n(R)V(R)^2 F(q,R)^2 \,\mathrm{d}R, \qquad (1)$$

where $\mathrm{d}\Sigma/\mathrm{d}\Omega$ represents the macroscopic differential scattering cross section. $\Delta\eta$ is the difference in the scattering length densities of precipitate and matrix, $n(R)\mathrm{d}R$ stands for the number density of precipitates with sizes between R and $R + \mathrm{d}R$, $V(R)$ the volume of the precipitates and $F(q,R)$ is the form factor of the precipitates. Previous investigations by Karagöz et al. [5] have shown that both prolate M_2C type and oblate MC type precipitates with an aspect ratio of $u = 5$ and $u = 0.12$, respectively, can be expected in the tempered condition. Since the size of both carbides is similar and SANS conducted on polycrystalline materials is not very sensitive with regard to the shape of precipitates, the scattering curves were analysed assuming the appearance of spherically shaped