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Precipitate evolution in the early stages of ageing in Inconel 718 investigated using small-angle x-ray scattering

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ABSTRACT

Microstructural evolution during the early stages of ageing (less than one hour) in a Ni–Cr–Fe based superalloy Inconel 718 (IN718) has been investigated using Small-Angle X-ray Scattering (SAXS). The effects of precipitate kinetics on the precipitate size distribution are compared indirectly with SAXS measurements by using Vickers microhardness data. The microhardness increased after 4 min of ageing at a temperature of 760 °C, although the recorded SAXS data did not reveal the precipitate size distribution. This indicates that the precipitates had not evolved enough to be detected, but still a small number of precipitates increased the yield strength. After ageing the alloy for the shortest period for which data were available, 8 min, clear evidence of precipitates could be found from the SAXS data, showing that the $\gamma''-$ precipitates are about 6 nm in width and 3 nm in height.

1. Introduction

Excellent mechanical strength and resistance to creep at elevated temperatures are important requirements in today's aircraft engines. Over the years, special kinds of alloys have been developed, often referred to as superalloys, which fulfil these requirements. The Ni–Cr–Fe based superalloy Inconel 718 (IN718) possesses excellent mechanical strength and resistance to creep at operating temperatures up to approximately 650 °C. These attributes make the alloy suitable for use in aero engines and power generation turbines [1]. IN718 is a precipitate hardening alloy. The two precipitates that contribute to increased hardness are the ordered face centre cubic (fcc) $\gamma'-$phase of composition Ni_3(Al,Ti), and the coherent ordered disc-shaped body-centred tetragonal (bct) $\gamma''-$phase, which comprises nickel, niobium, titanium and aluminium Ni_3(Nb, Ti, Al) [2]. When the IN718 alloy has been aged past its nucleation stage, approximately 13 vol% of γ'' and 4 vol% of γ' precipitates are present in the matrix [3]. The size of the precipitates depends on the ageing temperature and time of heat treatment, and has been measured by a number of researchers for ageing times longer than two hours, e.g. Camus and Engberg [4], Han et al. [5,6], Sundararaman et al. [7] and Slama et al. [8,9].

Transmission Electron Microscopy (TEM) dark field techniques have been the dominate method of studying nucleation and growth of γ' and γ'' precipitates. In the study by Alam et al. [10], WE91 (a variant of IN718) was investigated for very short ageing times. They utilised both TEM and atom probe tomography and compared Vickers microhardness data to the microstructural information. After ageing for 120 s, they found a small increase in microhardness, but no evidence of nucleation in the form of superlattice reflections. After 500 s evidence of superlattice reflection could be found. Prior to the observation of precipitates, clustering of aluminium/titanium and niobium has been shown to occur, which is believed to contribute to an increase in hardness. The ageing temperature was 706 °C. Nakai et al. [11] investigated the effects of solution treatment on microstructural evolution in the early stage of ageing. They used the Vickers microhardness technique to measure the hardness variation with ageing time, where the shortest ageing time was 10 s.

The morphology and distribution of stable precipitates in IN718 is well understood. However, less is known about the nucleation and growth stage of these precipitates. Measurements of the

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nucleation and growth processes in engineering alloys are valuable for calibrating and validating material models [12]. In this study, we consider measurements of alloy IN718 in the early stages of nucleation and growth using Small-Angle X-ray Scattering (SAXS) and Vickers microhardness data for comparison.

2. Experimental methods

Aged heat treated samples were investigated using the Vickers microhardness test method [13]. The samples were first hot rolled and solution treated at 954 °C for 1 h. Following this, ageing was carried out at eight temperatures (500, 550, 600, 650, 700, 760, 800 and 850 °C), each for eight different times (4, 8, 16, 32, 64, 120, 240 and 480 min), giving 64 samples. Each of the 64 aged samples was etched lightly in oxalic acid with a voltage of ~3 V to enable Vickers microhardness testing on a single grain. In order to obtain significant statistics, five measurements for each specimen were performed using a load of 200 g and a 15 s dwell time.

SAXS is a non-destructive technique for obtaining quantitative information such as shape and size distribution of clustering of elements that have high electron density contrast with respect to the matrix [14-17]. In IN718, γ' particles comprise nickel, aluminium and titanium, and γ'' nickel, niobium, titanium and aluminium, respectively. The chemical composition of alloy IN718 is given in Table 1. It is possible impurities segregate in the alloy, causing false results from the scattering of the x-ray beam. Since the alloying elements in superalloys are very controlled, only a small amount of impurities are expected. Because of this, the authors do not expect any large amount of impurities, which can segregate and change the SAXS-curves in the q-range used in this work.

For a fully aged IN718 alloy, the volume fraction ratio of γ''/γ' precipitates is about three [3]. Alam et al. [10] reported that the volume fraction ratio of the precipitates is higher for shorter ageing times (in their case 500 s for ageing at 706 °C) using Atom Probe Tomography (APT). Also, they reported that the γ' and γ'' precipitates are located in the same regions, and very often as co-precipitates forming a sandwich-like structure of the form $\gamma'/\gamma''/\gamma'$ or $\gamma''/\gamma'/\gamma''$.

From this knowledge, it is believed that SAXS can be used to predict the evolution of γ'' precipitates during ageing.

SAXS measurements were carried out at the synchrotron research facility MAX IV Laboratory located in Lund, Sweden, on the I911-4 SAXS beamline [18], at a fixed wavelength of 0.91 Å. The accessible scattering vector q-range was [0.01, 0.3] $Å^{-1}$, and the beam size was 200 × 300 µm. The q-range allows us to resolve particles between ~10 and 300 Å. SAXS patterns were acquired using a Pilatus 1 M two-dimensional detector with an exposure time of 150 s for each frame, normalised by the incoming photon flux and sample transmission. The hot rolled samples, with a thickness of ~40 µm, were first solution heat treated, and then subsequently aged at 760 °C for 4, 8, 16, 32 and 64 min. Details of how the precipitate size was extracted are given in Section 3.

Table 1
Chemical composition of alloy IN718 (wt%).

Ni	Cr	Fe	Nb	Mo	Ti	Al	Co
53.53	18.41	17.92	5.00	3.05	0.94	0.6	0.17
C	Mn	Si	P	S	B	Cu	
0.03	0.11	0.08	0.010	0.0004	0.002	0.13	

3. SAXS raw data

The inset graph in Fig. 1(a) shows the 2D SAXS pattern of a sample aged for 32 min, while the main graph shows the magnification of the pattern close to the beam stop. The colour scale represents the SAXS intensity for each pixel. The black horizontal and vertical lines are caused by the detector gaps. The larger black area in the middle is the beam stop. The structure of the 2D SAXS pattern shows a hexagonal shape, which indicate streaks in the projection in various directions. In the literature four different origins of streaks in SAXS patterns are reported: (I) presence of an oxide layer on the surface of the specimen [19,20], (II) presence of aligned anisotropically shaped precipitates [21,22], (III) possible double Bragg reflections within a single grain [23,24], or (IV) presence of ordered dislocations [25-27]. In further analysis, the main streaks are believed to arise from either (II) or (IV) as IN718 is generally resistant to oxidation, and the double Bragg reflections are assumed to be neglected.

To analyse the anisotropic behaviour, the intensity variation as a function of azimuthal angle φ was obtained for radius r_φ, which is illustrated in Fig. 1(b). The azimuthal intensity I(φ, t=32 min) shows symmetric behaviour, containing peaks and valleys (solid line). The large peak at $\varphi \approx 60^\circ$ disappears (dashed line) after subtracting the azimuthal intensity of the 4 min aged sample (without precipitates). Therefore, it can be assumed the streaks observed in the SAXS pattern (solid line) arise from structural anisotropy in the matrix, e.g. ordered dislocations, and not from the presence of aligned anisotropically shaped precipitates. The variation observed in the subtracted azimuthal intensity plot (dashed line) is therefore believed to arise from sample inhomogeneities. If aligned precipitates are present, distinct and symmetrical peaks should be observed in the subtracted spectra. The present peaks can be assumed to arise from the rolling direction during manufacturing and changes during heating.

From the analysis of the streaks above, the conclusion is that the precipitates are randomly oriented in the illuminated volume. With this knowledge, intensity plots are constructed by radial averaging of the data from the 2D SAXS plot and subtracting the constant value related to background scattering [28]. The curves contain information about the particle shape and size distribution.

Based on the particle shape (e.g. oblate spheroid) and the assumption of a given size distribution (e.g. log-normal), the scattering pattern can be fitted with a calculated intensity. Applying a size distribution $p(r)$ and neglecting the particle-to-particle scattering structure factor, the intensity I can be described as [29]:

$$I(q) = N|\Delta\rho|^2 \int_0^\infty |V_p(r)F_p(q,r)|^2 p(r)\, dr, \tag{1}$$

where $F_p(q,r)$ is the dimensionless form factor of a particle with radius r and volume $V_p(r)$ with a scattering vector $|\overline{q}| = q$. $|\Delta\rho|$ is the difference in scattering length density between the matrix and the precipitate, and N is the density of precipitates (number/m^3). Because the SAXS intensities are not absolute differential scattering cross sections, the density parameter N is a scaling factor only. The form factor of an oblate spheroid is given in [30].

For small values of q, Eq. (1) can be simplified using the Guinier approximation [31]:

$$I(q) \propto \exp\left(-\frac{q^2 R_g^2}{3}\right), \tag{2}$$

where R_g is the radius of gyration of the overall particle distribution. This is determined from the slope of the plot of ln(I) versus q^2 as q tends to zero (the Guinier plot). The relation between the radius of gyration and the size of the particle for an oblate