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Numerical simulation of grain boundary carbides evolution in 316H stainless steel

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ABSTRACT

In the present work, a numerical model based on the coupling of Kampmann and Wagner Numerical (KWN) framework and thermodynamic software ThermoCalc has been developed to predict grain boundary precipitate evolution in 316H stainless steel during thermal aging. The model is calibrated and validated against precipitate size distributions obtained by accelerated isothermal heat treatment and analysed using scanning electron microscopy (SEM). Elemental distribution was also investigated using electron microprobe analysis (EPMA). The predicted average particle size, particle size distribution and precipitate number density predicted by the model were found to be in good agreement with the experimental results. The model was then applied to predict the particle size distribution after several years exposure at service temperature. It is demonstrated that these predictions are consistent with measurements from a service-exposed part. The sensitivity of the precipitate size distribution to temperature is emphasised, and it is demonstrated that the model has potential as a useful tool for predicting evolution of the precipitate size distribution during service, providing reliable thermal data are available for the whole service life.

1. Introduction

AISI 316 austenitic stainless steels are widely used reactor materials in conventional nuclear power plants because of their outstanding mechanical formability, good high temperature strength and corrosion resistance [1]. However, these materials are subjected to degradation due to thermal aging and other external factors (irradiation, stress, temperature, coolant media, etc.), which could affect the reliability of components [2,3]. Carbides are important precipitates in these austenitic stainless steels. However, these carbides are not always stable; they evolve under thermal aging or welding processes, which have an important influence on material performance. The dominant carbide phase in 316H is $M_{23}C_6$ and it precipitates on the grain boundaries, twin boundaries and dislocations [2,4–9]. The precipitation of this phase at grain boundaries depletes the local chromium content to an extent which is an important reason for the occurrence of intergranular stress corrosion cracking in stainless steels in high temperature water environment and also results in degradation of strength, toughness and creep ductility properties [10–13]. Many variables such as alloy compositions, exposure temperature and duration, and processing history affect the stability of $M_{23}C_6$ in multicomponent materials such as AISI 316 S S.

Several investigations have reported the precipitation behaviour of $M_{23}C_6$ during isothermal heat treatment in 316 S S. The evolution of $M_{23}C_6$ has been studied by a range of experimental techniques, but clearly there are practical limits to the duration of such experiments [2,13–15]. To be able to extrapolate these results to longer timeframes (40–60 years or beyond) requires a reliable method for predicting $M_{23}C_6$ evolution. A physical rather than empirical model potentially provides the most reliable extrapolation as well as helping to understand the mechanisms contributing to the microstructural change in this type of stainless steels. However, the model must have some specific features; it has to have ability to capture the complexity of the multicomponent system and account for the simultaneous occurrence of nucleation,

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growth and coarsening of the precipitates.

The Kampmann and Wagner Numerical (KWN) method is one of the most appealing approaches to provide a framework to predict precipitate evolution [16–18]. This method uses thermodynamic and kinetic (e.g. mobility) data to predict the essential processes that occur during precipitate evolution, namely nucleation, growth and coarsening. It has also been widely applied to a number of alloy systems (including stainless steel 316) with the advantage of naturally predicting the evolution of precipitates from nucleation to growth and coarsening without imposed constraints [16,17,19–22]. The KWN model requires knowledge of the thermodynamic properties of the precipitate and matrix phase (to calculate interfacial compositions) and the diffusion coefficient of the rate controlling element(s). These inputs can be derived by thermodynamic calculation and from literature. The precipitate/matrix interfacial energy is another critical parameter in the KWN model, and strongly influences both nucleation and growth behaviour. Interfacial energy is usually a calibration parameter in the KWN method, and is determined by fitting the model predictions to experimental results for a limited number of temperatures before validating the model across a wider temperature range.

The traditional KWN model was developed to model homogeneous precipitation in a supersaturated matrix. In this work, the KWN method is adapted to simulate the evolution of grain boundary precipitation, specifically the carbides, in 316H stainless steels. The interfacial energy in this alloy was calibrated and optimised using experimental data obtained from specimens exposed at different temperatures for different durations as described in detail later. ThermoCalc with the TCFE9 steels database predicting the phase stability and equilibrium compositions is used as input for the KWN precipitation kinetics model [23]. Once calibrated, the KWN model was applied to predict the likely precipitate evolution in the nuclear power plant materials over long-term service (many years). Such a microstructure model is a key step in producing a physical based ability to predict remnant service life.

2. Material details and experimental method

2.1. Materials

Type 316H stainless steel obtained from the body of a 64 mm wall thickness steam header of an advanced gas-cooled reactor (AGR) component provided by EDF Energy is used in this work. The as-received material block from EDF had been in service for 91,595 h at 460–540 °C. The chemical composition of this material is shown in Table 1. The chemical composition of the 316H stainless steel from the NIMS Material Database is listed in Table 1 as well [24], hereafter it is referred to as 316NIMS. The 316NIMS experimental data is used in later sections for comparison and validation of our results and models.

2.2. Experimental method

The as-received material was separated into small pieces (10 mm × 10 mm × 5 mm) and these small pieces were solution-treated at 1100 °C for 1 h to dissolve the carbides precipitated during service ageing, and were then water quenched to avoid precipitation during cooling. These as-quenched specimens were then isothermally aged at 700 °C and 800 °C for 1 h, 10 h and 100 h, respectively. The thermally aged specimens were mounted in conductive resin, and prepared using standard metallographic methods, finishing with a polish using Oxide Polishing Suspension (OPS).

Table 1
Compositions (wt%) of selected 316 alloys.

Alloy	C	Mn	Si	P	S	Cr	Ni	Mo	Cu	Fe
316H	0.07	0.98	0.42	0.021	0.014	17.17	11.83	2.19	0.15	Bal.
316NIMS	0.07	1.65	0.61	0.025	0.007	16.6	13.6	2.33	0.26	Bal.

Note: chemical compositions of 316H and 316NIMS [24].

A Quanta 650 scanning electron microscope (SEM) was used for precipitate observation. Compositional contrast backscattered electron (BSE) imaging was used to image the precipitates, and the accelerating voltage used during imaging was 15 kV. It should be noted that, to determine the measured precipitate size distributions, ImageJ software [25] was applied to calculate the areas of the particles in BSE images and then the particle areas were converted to equivalent circle radii to compare with the model predictions. In addition, due to the limitation of resolution, only particles with equivalent radii larger than 25 nm were measured.

A challenge in using SEM to quantify particle size distributions is to determine the maximum depth from which the back-scattered signal is emitted, and thus the effective volume of material being sampled. In the present work, the Monte-Carlo electron-sample interaction simulation software Casino [26] was used to determine the equal interaction volumes of the material with the beam for the imaging conditions used (10,000 electrons at 15 kV accelerating voltage with an assumed beam diameter of 10 nm, the density of the steel with 7.79 g/cm^3). This simulation suggested a maximum penetration depth of approximately 350 nm, and it may be assumed that back-scattered electrons that penetrate up to half this depth will be emitted and thus contribute to the detected signal. Therefore, an approximate conversion between number/area measured from the SEM image and volume fraction can be made, assuming the sampling depth to be half the maximum penetration depth:

$$N_v = \frac{N_A L}{2} \tag{1}$$

where N_v is the volumetric number density, N_A is the measured number density from the SEM image (per unit area of the image) and L is the total interaction depth (350 nm in this case). Since (as shown later) the sampling depth is typically much greater than the measured particle size (radius), it is reasonable to assume that in most cases the whole particle is captured inside the sampled volume, and thus truncation effects were ignored.

The chemical composition of the precipitated carbides and other intermetallic phases (η and σ) were investigated using a JEOL JXA-8530 F FEG-EPMA. The analyses were run at 9 kV, 43 nA at a magnification of 6500 × and 50 ms dwell time on each point.

3. Precipitation kinetics simulation

The carbide of interest, $M_{23}C_6$, can precipitate both on grain boundaries and in the grain interior. Grain boundaries are known to be potent nucleation sites for this phase, and it is grain boundary nucleated $M_{23}C_6$ that forms first during the early ageing stages [2,27]. It is the grain boundary $M_{23}C_6$ that has the most important effect on the long term performance of the alloy, particularly resistance to stress-corrosion cracking, due to its effect on local chromium depletion as already discussed. Therefore, in the present work, the model is focussed on predicting grain boundary $M_{23}C_6$ evolution.

The KWN method is applied to predict precipitation kinetics. A full description of the KWN method is given elsewhere [22]. The model describes the nucleation, growth, and coarsening of particles