The Temporal Evolution of the Nanostructure of a Model Ni-Al-Cr Superalloy

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Abstract

The early to the later stages of precipitation of ordered $\gamma'$-precipitates ($L1_2$) in Ni-5.2 Al-14.2 Cr (at.%) are studied at 873 K. Precipitates with radii as small as 0.45 nm are characterized fully by three-dimensional atom-probe (3DAP) microscopy. Contrary to what is often assumed by theory or in models, the average precipitate composition is shown to evolve with time, such that solute concentrations decrease toward an equilibrium value given by the solvus lines. Power-law time dependencies of the number density, mean radius, and supersaturations of Al and Cr are discussed in light of theoretical predictions for Ostwald ripening.

Introduction

The precipitation of $\gamma'$-phase from a supersaturated solution ($\gamma$) in a temperature range where nucleation and growth is observable has been studied in Ni-Al alloys by both direct [1,2] and indirect [3] imaging techniques. In the present investigation, 3DAP microscopy is employed to characterize the identical, ternary alloy, Ni-5.2 Al-14.2 Cr (at.%), aged at 873 K, as studied by references [4,5]. This method allows the direct, spatial characterization of the chemical composition on a sub-nano- to nanometer scale via the reconstruction of a volume of material, typically $10^5$ nm$^3$ and $10^7$ nm$^3$ for the conventional 3DAP and the local electrode atom-probe (LEAP) [6] microscopes, respectively. The experimental procedures are described in [7,8].

Results and Discussion

After homogenization, Ni-5.2 Al-14.2 Cr (at.%) decomposes at 873 K into a high number density, $N_v$, of nanometer-sized, spheroidal $\gamma'$-precipitates. Misfit between the $\gamma$ and $\gamma'$ phases is near zero [9], and the precipitates are coherent and spheroidal up to 1024 h [7,8]. The $\gamma'$-precipitation is first observed after 0.17 h of aging and the precipitates' average radius, $<R>$, and volume fraction, $V_f'$, are determined to be 0.74 nm and 0.11 %, as shown in Fig. 1. A sharp rise in $N_v$ at a constant $<R>$ is observed between 0.17 h and 0.25 h aging times, indicating that nucleation predominates. 3DAP microscopy detected precipitates as small as $R = 0.45$ nm (20 atoms). After 0.25 h and until 256...
h, precipitate coalescence is observed, as seen for 4 h in Fig. 2. Given the precipitate radii and small misfit, this is not believed to be a result of elastically driven particle migration, and marks the finest scale, as well as smallest \( V'_f \), where this phenomenon has been observed in the solid state. Peak \( N_v, (3.2 \pm 0.6) \times 10^{24} \text{ m}^{-3} \), is achieved after 4 h of aging, after which the transformation enters a quasi steady-state regime with a constant power-law dependence of \( t^{-0.64 \pm 0.06} \). In this regime, \( V'_f \) steadily increases (upper panel in Fig. 1), indicating that the transformation is not complete, yet \( <R> \) has a temporal dependence of \( 0.30 \pm 0.04 \), which is approximately consistent with the \( t^{1/3} \) prediction for Ostwald ripening. This effect has also been observed in Ni-14 Al (at.%) aged at 823 K [1].

The average compositions of the \( \gamma \) (Fig. 3) and \( \gamma' \)-precipitates (Fig. 4) continually evolve. The matrix becomes more enriched in Ni and Cr and depleted in Al with time. Between 4 and 16 h, the solute far-field concentrations change slowly (\( dc/dt \rightarrow 0 \)), and the quasi-stationary approximation can be applied after 16 h. Assuming this approximation, Marquis and Seidman [10] recently determined the solid solubilities, \( c_{i \text{eq}} \), in a ternary alloy. They applied \( c_{i}(t)= \kappa t^{-1/3} + c_{i \text{eq}} \) to fit the experimental data. Employing this approach \( c_{i \text{eq}} \) are 16.69 ± 0.22 at.% for Al and 6.77 ± 0.15 at.% for Cr. Straightforwardly, the matrix supersaturations, \( \Delta c = c_i(t) - c_{i \text{eq}} \), are determined (Fig. 3). Their temporal behavior is in approximate agreement with the prediction of \( \Delta c \sim t^{-1/3} \) for Ostwald ripening.

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References