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Intragranular precipitation in alloy 718

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Abstract

A combined energy-compensated 3-dimensional atom probe and transmission electron microscope characterization has been performed to determine the nature of the intragranular precipitates that form in Alloy 718 after various stages of a multistage heat treatment. Primary γ'' precipitates were found to form after the 870°C stage. The secondary precipitates formed at lower temperatures were not found to be uniform throughout their volume, but to be divided into two distinct regions enriched in either niobium, or in aluminum and titanium, that are characteristic of the γ'' and γ' phases, respectively. The experimentally determined partitioning data of the solute elements between the γ matrix and the γ'' and γ' phases was found to agree with the trends in the thermodynamic predictions. © 1999 Elsevier Science S.A. All rights reserved.

Keywords: Alloy 718; Precipitates; Superalloys

1. Introduction

Alloy 718 is a niobium-modified nickel-base superalloy that is used widely in gas turbine and related high temperature applications [1]. Alloy 718 derives its good mechanical properties at elevated temperature from a fine dispersion of $D0_{22}$-ordered γ'' and L_{12}-ordered γ' precipitates in a face-centered-cubic γ matrix. Both these precipitates have been described as Ni_3 (Nb, Al, Ti) with varying levels of Nb, Al, and Ti.

Previous atom probe field ion microscopy (APFIM) and transmission electron microscopy characterizations of these alloys have revealed the presence of primary γ'' precipitates and secondary γ'' and γ' precipitates [2–5]. These studies also revealed that there was a significant variation in the composition of the precipitates.

In this study, the variations in the compositions of individual precipitates are examined with an energy-compensated 3-dimensional atom probe.

2. Experimental

The nominal composition of the Alloy 718 used in this study was: Ni, 5.2 wt%, Nb, 0.43%, Al, 0.95%, Ti, 19.6%, Fe, 19.4%, Cr, 0.26%, Co, 3.0%, Mo, 0.16%, Mn, 0.21%, Si and 0.054% C (see Table 1 for composition in atomic percent). The material was characterized after the following five series of two-, three-, or four-stage heat treatments: 1 h at 1038°C + 8 h at 982°C + 8 h at 760°C + 8 h at 650°C (ABDE); 1 h at 1038°C + 8 h at 870°C (AC); 1 h at 1038°C + 8 h at 870°C + 8 h at 760°C (ACD); 1 h at 1038°C + 8 h at 870°C + 8 h at 760°C + 8 h at 650°C (ACDE); and 1 h at 1038°C + 8 h at 760°C (AD).

The material was characterized in the Oak Ridge National Laboratory's (ORNL) energy-compensated optical position-sensitive atom probe. A specimen temperature of 60 K, a pulse fraction of 20%, and a pulse repetition rate of 1500 Hz was used for all atom probe analyses.

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3. Thermodynamic predictions

The equilibrium compositions and the amounts of the possible phases were predicted from the Thermocalc program [6] and the results are presented in Table 1. At 1038°C (A), the γ matrix was predicted to be in equilibrium with a niobium-and titanium-enriched MC and a niobium- and molybdenum-enriched M_6C carbide. The $D0_{22}$-ordered γ'' precipitates were predicted not to be present at 1038°C (A) but formed at 982°C (B) and lower temperatures. These γ'' precipitates were predicted to be enriched in niobium and titanium and depleted in aluminum, chromium, iron, molybdenum, and cobalt. The $L1_2$-ordered γ' precipitates, the chromium-enriched σ and $M_{23}C_6$ phases were only predicted to be present at 760°C (D) and 650°C (E). The γ' precipitates were predicted to be enriched in aluminum, titanium, and niobium and depleted in chromium and iron, molybdenum, and cobalt. The volume fractions of both γ'' and γ' precipitates were predicted to increase with decreasing aging temperature. It is evident from these predictions that the γ'' precipitates may be characterized by their high niobium and low aluminum levels and the γ' precipitates by their high titanium, aluminum, and intermediate niobium levels. The complex trend of solute partitioning between these phases at the different aging temperatures may also be examined from these predictions.

4. Microstructure

The previous investigation revealed that no intragranular precipitation occurred after the 1 h at 1038°C (A) or 8 h at 982°C (B) stages of the heat treatment [2–4]. Primary γ'' precipitates were observed in the material aged for 8 h at 870°C (C). The typical microstructures of the material after the ACDE and the ABDE heat treatments are shown in Figs. 1 and 2, respectively. The microstructure after the ACDE heat treatment is comprised of ≈ 0.3 μm long lenticular primary γ'' precipitates and a finer dispersion of 10–40 nm diameter secondary precipitates that form during the 8 h at 760°C (D) stage of the heat treatment. When the C stage is omitted, no primary γ'' precipitates are formed and the microstructure is significantly finer. No discernable contrast variations were evident in the field-ion micrographs of the secondary precipitates and the contrast difference with the matrix was small. Some contrast was evident within the precipitate in the transmission-electron micrographs. However, the high density of the precipitates in the microstructure made further interpretation difficult in this material. The details of other phases formed in this material are described elsewhere [4,5].

The partitioning of the alloying elements and the composition gradients across the primary γ'' precipitate-γ matrix interface were determined. An atom map of a volume containing a primary γ'' precipitate after the ACD heat treatment is shown in Fig. 3. The

Table 1
Equilibrium compositions of the phases at the different annealing temperatures as predicted from Thermocalc[a]

Phase		Cr	Fe	Nb	Mo	Ti	Al	Co	C	Volume %
Bulk	Temperature	21.80	20.30	3.20	1.81	1.15	0.96	0.26	0.26	
γ	1038°C	21.87	20.61	2.66	1.73	1.13	0.94	0.26	–	98.6
	982°C	22.82	21.43	2.09	1.89	0.93	0.96	0.26	–	94.3
	870°C	24.44	22.92	0.91	2.00	0.58	1.01	0.27	–	87.9
	760°C	25.15	24.85	0.36	1.82	0.22	0.61	0.28	–	79.5
	650°C	24.43	25.98	0.14	1.32	0.08	0.34	0 28	–	73.1
γ'	760°C	1.03	3.86	6.03	0.19	8.39	9.06	0.13	–	4.5
	650°C	0.67	3.01	5.98	0.12	8.13	9.40	0.12	–	6.9
γ''	982°C	1.38	1.95	20.85	0.45	3.42	0.27	0.18	–	5.1
	870°C	1.05	1.56	19.62	0.49	4.59	0.30	0.17	–	11.6
	760°C	0.66	1.09	19.86	0.47	4.41	0.26	0.15	–	13.5
	650°C	0.34	0.64	20.39	0.40	3.99	0.22	0 14	–	13.4
σ	760°C	50.68	18.40	–	13.43	–	–	0.35	–	1.3
	650°C	52.75	18.92	–	12.08	–	–	0.42	–	5.4
MC	1038°C	0.06	–	35.87	0.13	17.67	–	–	46.27	0.2
	982°C	0.05	–	36.18	0.13	17.29	–	–	46.33	0.6
	870°C	0 04	–	32.43	1.06	20.52	–	–	46.90	0 5
M_6C	1038°C	3.69	0.08	44.96	8.23	–	–	–	14.29	1.2
$M_{23}C_6$	760°C	66.90	1.73	–	9.94	–	–	–	20.70	1.3
	650°C	67.47	1.42	–	10.06	–	–	–	20 69	1.3

[a] The balance of each analysis is nickel and the data are in atomic percent.

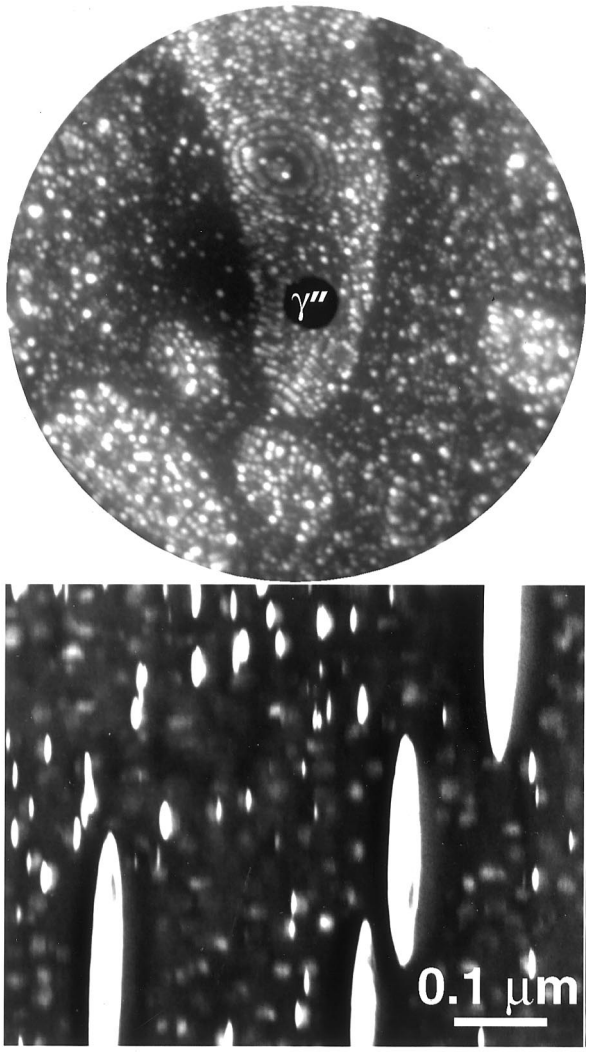

Fig. 1. Field ion and transmission electron micrographs of the microstructures after the ACDE heat treatment. The ACDE heat treatment results in ≈0.3 μm long lenticular primary γ″ precipitates and a finer dispersion of 10–40 nm diameter secondary precipitates.

corresponding composition profiles perpendicular to the γ–γ″ interface are shown in Fig. 4. The expected partitioning of iron, chromium, molybdenum, and cobalt to the γ matrix and niobium, and titanium to the γ″ phase is evident. The concentration profiles show enrichments and depletions close to the precipitate–matrix interface. Chromium and titanium exhibited slight depletions and iron a significant enrichment in the γ″ precipitate near the γ matrix. Manganese and aluminum exhibited local enrichments and niobium a depletion in the y matrix adjacent to the γ″ precipitate. Carbon exhibited a small local enrichment at the interface. The widths for these local solute enriched and depleted regions ranged from ≈1 nm for the aluminum and carbon to ≈5 nm for the other elements.

A representative atom map of a secondary precipitate in the γ matrix after the ABDE heat treatment is shown in Fig. 5. Since the extent of the precipitate is larger than the volume analyzed only a portion of the precipitate is evident. However, it is evident that the distribution of the different types of atoms is not uniform within this precipitate and there is a distinct interface dividing a niobium-enriched and an aluminum- and titanium-enriched portion. These two portions exhibited the solute partitioning behavior characteristic of the γ″ and γ′ phases, respectively, as indicated from the thermodynamic predictions. Most precipitates were found to exhibit this character. However, as a result of the limited extent of the volume analyzed in the 3-dimensional atom probe, it was not possible to unequivocally determine this dual character was present in all the precipitates intersected. It should be noted that no discontinuities at the γ″–γ′ interface were observed in the shape of the surfaces of these precipitates. Similar precipitates were observed in the AD, ACD, and ACDE heat treatments.

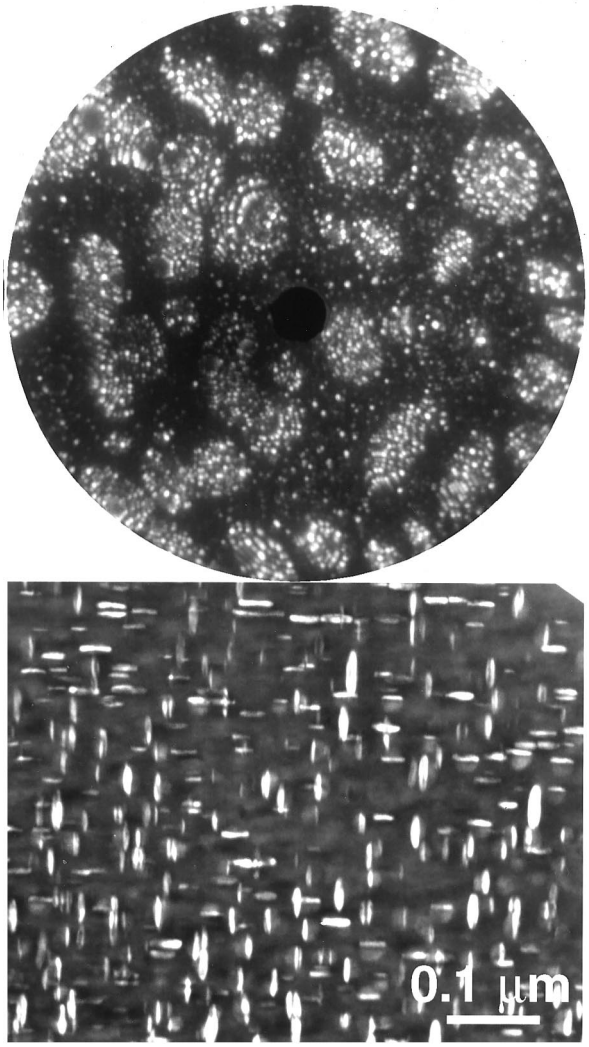

Fig. 2. Field ion and transmission electron micrographs of the microstructures after the ABDE heat treatments. No primary γ″ precipitates are formed in the ABDE heat treatments and the microstructure is significantly finer.

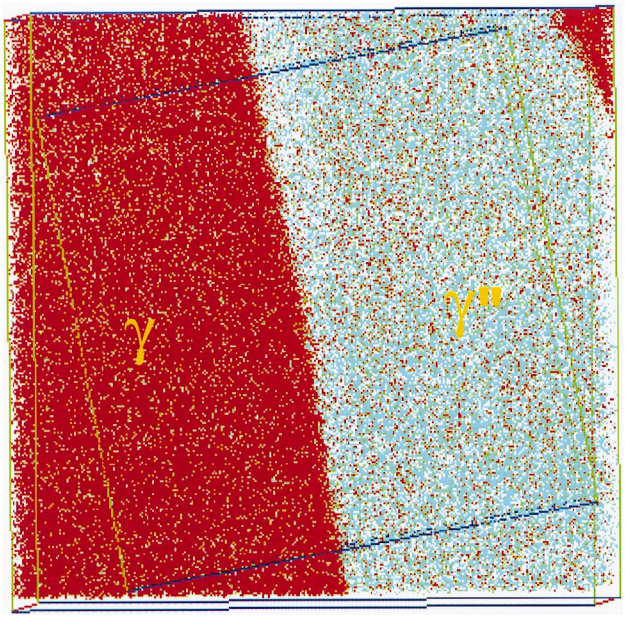

Fig. 3

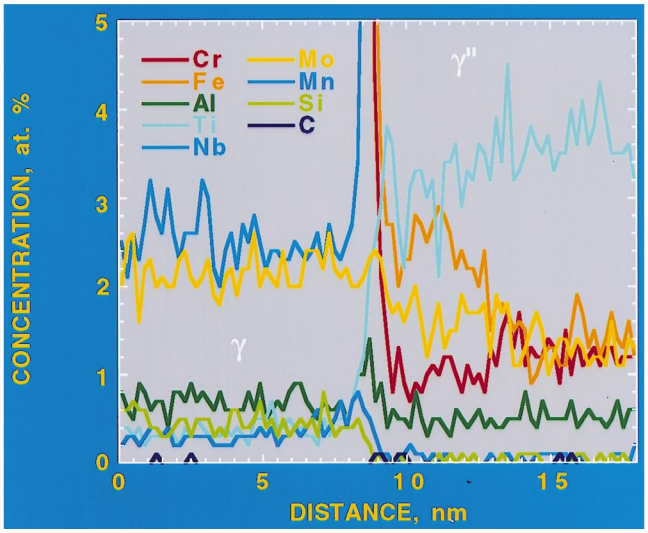

Fig. 4

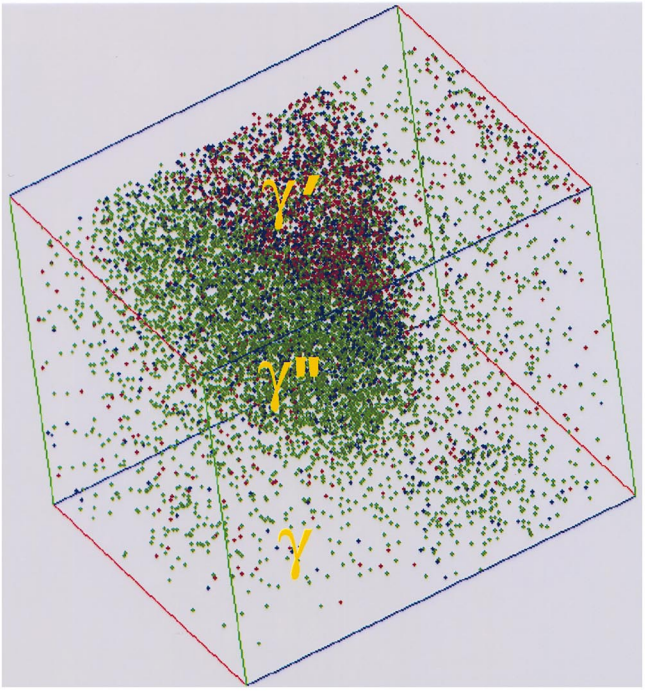

Fig. 5

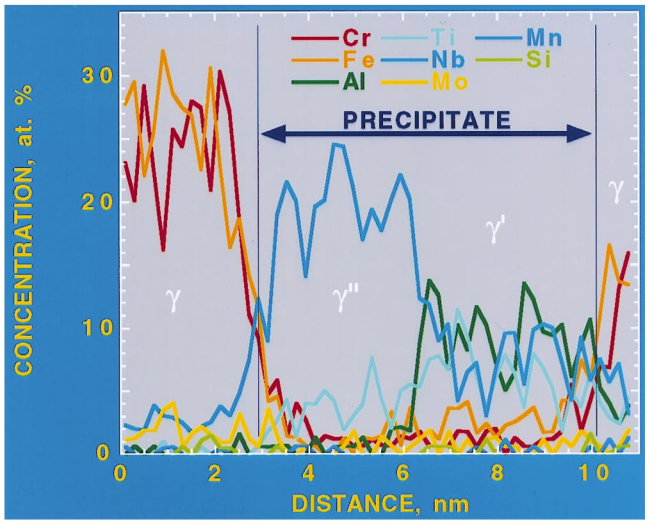

Fig. 6

Fig. 3. Atom map of the γ matrix and a primary γ″ precipitate in the ACD material. The inner box (18.1 × 18.1 × 18.2 nm) is the volume selected for the composition profile shown in Fig. 4. Atom colours are as shown in Fig. 4.

Fig. 4. Composition profile from the γ matrix into a primary γ″ precipitate in the ACD material. Note the solute enrichments and depletions at the precipitate–matrix interface.

Fig. 5. Atom map of a secondary precipitate and the γ matrix in the ABDE material. The box is 11.2 × 12.3 × 14.1 nm in extent. Al atoms are red, Ti atoms are blue, and Nb atoms are green.

Fig. 6. Composition profile from the γ matrix through a secondary precipitate into the γ matrix in the ABDE material. Note the precipitate has both γ′ and γ″ components.

The corresponding composition profile through this secondary precipitate is shown in Fig. 6. In this composition profile, a 4-nm diameter cylinder of analysis with its major axis oriented normal to the internal interface in the precipitate was used. Consequently the normals to both curved precipitate–matrix interfaces are inclined slightly to the axis of the cylinder which results in small concentration gradients at these interfaces. The interface between the niobium-enriched γ'' and aluminum- and titanium-enriched γ' portions of this precipitate is clearly evident. Chromium was found to partition to the γ matrix and was approximately the same in both precipitate regions. Iron was found to partition to the γ matrix and slightly higher levels were observed in the γ' regions than in the γ'' regions. Niobium partitioned preferentially to the γ'' regions and was significantly enriched in the γ' regions. Molybdenum partitioned to the γ matrix and was slightly higher in the γ'' than in the γ' regions. Titanium partitioned preferentially to the γ' regions and then the γ'' regions.

Aluminum partitioned to the γ' regions and was depleted in the γ'' precipitates. These trends are all consistent with the thermodynamic predictions.

5. Summary

A combined energy-compensated 3-dimensional atom probe and transmission electron microscope characterization has determined the nature of the intragranular precipitates that form in Alloy 718 after various stages of a multistage heat treatment. The secondary precipitates were not found to be uniform throughout their volume but to be divided into two distinct regions enriched in either niobium, or in aluminum and titanium, that are characteristic of the γ'' and γ' phases, respectively. The experimentally determined partitioning data of the solute elements between the γ matrix and the γ'' and γ' phases was found to agree with the trends in the thermodynamic predictions.

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[6] Computed with the use of the Ni-data database, a thermodynamic database for calculation of phase equilibria in multicomponent alloys, ThermoTech Ltd., UK.