EVOLUTION OF SIZE AND MORPHOLOGY OF γ' PRECIPITATES IN UDIMET 720 LI DURING CONTINUOUS COOLING

R. Radis¹, M. Schaffer², M. Albu², G. Kothleitner², P. Pölt² und E. Kozeschnik^{1,3,4}

¹Christian Doppler Laboratory for Early Stages of Precipitation, Graz University of Technology,
Kopernikusgasse 24, A-8010 Graz, Austria

²Research Institute for Electron Microscopy, Graz University of Technology,
Steyrergasse 17, A-8010 Graz, Austria

³Institute for Materials Science and Welding, Graz University of Technology,
Kopernikusgasse 24, A-8010 Graz, Austria

⁴Materials Center Leoben Forschung GmbH, Roseggerstraße 12, A-8700 Leoben, Austria

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Abstract

In this work, an analysis of the evolution of precipitates in the nickel-base superalloy UDIMET 720 Li during continuous cooling is carried out. Starting from the solution-annealed condition, the precipitation kinetics, the distribution and the morphology of particles during continuous cooling is studied experimentally by SEM, EFTEM and FIB. The results are compared to simulations using the software MatCalc. Depending on the cooling rate, discontinuous precipitation as well as mono-modal and multi-modal distributions are observed. The precipitate microstructure of various samples is investigated and evaluated quantitatively to determine the precipitate distribution parameters. The experimental results are discussed and compared with the results of the simulation. Finally, two parameter studies are carried out which indicate the sensitivity of the simulation results on the input data, i.e. the thermodynamic and diffusion databases.

Introduction

The term superalloy is generally used for a large amount of different alloys. The major motivation for development of these materials is the fabrication of gas turbines for aircrafts and power plants. For a high degree of efficiency, these machines require combustion at elevated temperatures. Usually, these alloys combine high strength and good corrosion resistance, which are essential for application in modern gas turbine aero-engines, e.g. for high-speed rotating components, such as high pressure turbine disks.

A good control of the technological properties of these alloys is inherently related to a proper control of the precipitation of a large amount of fine-dispersed coherent $\text{Ni}_3(\text{Al,Ti})$ particles (γ' precipitates) with ordered L1_2 structure in a face-centred cubic γ -matrix. The main strengthening mechanism in these materials is precipitation hardening. Depending on the cooling rate, the shape of these particles differ in a wide range, from simple spheres over cubes, octocubes to complex structures such as octodendrites and dendrites. This large variety of dissimilar morphologies, founded in dissolution and precipitation processes, is well known and documented in several articles, e.g. [1-11].

It is also well known that bimodal distributions of γ' precipitates in a γ matrix can lead to superior mechanical properties in these alloys [12-14]. Primary γ' precipitates arranged on grain boundaries prevent the growth of γ -grains during solution heat treatment. A second population of these precipitates appear intergranularly and is responsible for a high strength. Such microstructures are usually achieved by special multi-stage heat treatments, which are generally complex and costly. Some authors have already shown that such bimodal, or even multimodal, size distributions of γ' particles can also be achieved by a series of γ' nucleation bursts during continuous cooling from the solution annealed state [15-22]. Thereby, the individual particle populations precipitate at different temperatures and from different chemical compositions of the γ matrix.

Studying these nucleation and diffusion processes experimentally is expensive, especially in nickel based superalloys. With the development of specialized simulation software, e.g. the software MatCalc [23-25], it is possible, nowadays, to investigate these complex processes with computational methods on the researchers desktop.

J. Mao et.al. [20] carried out investigations of the evolution of multimodal γ' precipitate size distributions during continuous cooling in UDIMET 720 Li. However, his experimental methods have concentrated on SEM investigations, thus not capturing very fine γ' precipitates. These authors suggested to do further investigations with high resolution TEM microscopes.

In the present work both, experimental investigations using high-resolution techniques as well as theoretical simulations using the software MatCalc are applied to investigate the evolution of γ' precipitates in the Ni-base superalloy UDIMET 720 Li during continuous cooling. A series of experiments with varying cooling rates is performed and the results of the experimental investigation are compared to the predictions of the simulation. Furthermore, the theoretical background for the formation of multimodal size distributions of precipitates is presented and confirmed by experimental results where we identified mono-modal, bi-modal and tri-modal γ' size distributions.

Table I: Chemical Composition of UDIMET 720 Li in wt% [26]

Cr	Ni	Co	Mo	Ti	Al	W	Fe	C	Mn	B
15.92	57.6	14.43	2.96	4.96	2.53	1.26	0.09	0.014	0.02	0.014

The chemical composition of UDIMET 720 Li was analysed using emission spectroscopy. The results are given in Table I in wt%.

Experiments and Simulation

Experimental

The precipitate microstructure of 13 samples is evaluated qualitatively and quantitatively to determine the precipitate distribution parameters and the morphology of the precipitates. The samples are cut into cylindrical pieces of $\phi 10 \times 10$ mm, except for two samples of size $\phi 5 \times 10$ mm to achieve the fastest cooling rates. Each sample is soaked at 1180 °C for 6 h and then quenched into water. In references [19,20], the super-solvus solution temperature is recommended with approximately $T_{sol}=1160$ °C. Scanning electron microscopic analyses have shown that this heat treatment is suitably for achieving solid solution condition and dissolving all precipitates.

Depending on the cooling rate, which is varied between 78 K/s and 0,00416 K/s, different equipment is used for different heat treatments. The samples are heated to a temperature of 1180 °C under argon atmosphere as fast as possible, kept on this level for 30 minutes to dissolve precipitates which formed temporarily before reheating, and then cooled continuously to 400 °C at different cooling rates (see Table II). Finally, they are quenched into water to suppress further precipitation. Afterwards, the distribution and the morphology of precipitates are studied by SEM, EFTEM and FIB. The relevant precipitation parameters are determined using the stereological correction procedure after Sonderegger [27]. In each case, more than 100 precipitates are measured to determine the distributions. For this correction, SEM penetration depth data were used from reference [28].

Table II: Continuous cooling rates of the present investigation

sample	cooling rate [°C/s]	cooling time [s]
1	78	10
2	19.5	40
3	6.5	120
4	3.25	240
5	1.625	480
6	0.867	900
7	0.433	1800
8	0.217	3600
9	0.03333	23400
10	0.01667	46800
11	0.00984	79200
12	0.00619	126000
13	0.00416	187200

Electron microscopy

The backscattered electron (BSE) images are recorded from polished cross sections by use of a 4-quadrant backscattered electron detector attached to a Zeiss DSM 982 Gemini scanning electron microscope (SEM). With decreasing energy of the primary electrons also the interaction volume of the electrons with the material decreases, thus enabling the detection of precipitates of decreasing size. But with decreasing electron energy also the detection sensitivity of the BSE detector decreases. Thus, as a compromise electron energies between 7 and 10 keV are used for the recording of the BSE images.

Energy filtered transmission electron microscopy (EFTEM) investigations are carried out on a Philips CM20/Scanning TEM (STEM) and a FEI Tecnai F20 both equipped with Gatan imaging filters (GIF). The microscopes are operated at 200 kV with a LaB₆ cathode and a Shottki field emitter, respectively. The pictures are recorded with the slow scan CCD camera integrated in the GIF with a local resolution of about 1nm. All images are processed with Gatan's Digital Micrograph, and corrected for drift, dark current and gain variations. Electron energy loss spectroscopy (EELS) and X-ray dispersive spectroscopy (EDX) are used for the precipitate analysis.

A dual beam-focused ion beam (DB-FIB) consisting of both, an ion and an electron source, offers a unique way for 3D analysis by sequentially milling the sample, and analyzing each freshly produced surface. From the series of data created in this way, a 3D volume model can be reconstructed. The fully automated 3D-EDXS method [36] is performed using the Nanolab Nova200 DB-FIB (FEI) and the Genesis EDXS detector (EDAX). The DB-FIB is operated with an ion beam current of 1 nA at 30 kV acceleration voltage during milling, and with an electron beam current of 2.2 nA at 15 kV acceleration voltage during SEM and EDXS analysis. The data is evaluated using customized routines such as image registration and adaptive threshold image segmentation before visualizing the models with the Amira software (Mercury Computer Systems SA). In the course of the experiment, a total sample volume of $4,45 \times 6,18 \times 2,55 \mu\text{m}^3$ is reconstructed with a voxel size of $35 \times 45 \times 150 \text{ nm}^3$.

Computer simulations

For the theoretical analysis of the precipitation behaviour in UDIMET 720 Li, the simulation software MatCalc [23-25] is used. This programme uses a novel theoretical approach that has been developed for simulation of precipitation kinetics in general, multi-component alloys. The theoretical foundation of the precipitate growth formalism is based on the thermodynamic extremal principle of maximum entropy production [29]. The numerical time integration procedure for the evolution equations is similar to the classical numerical Kampmann – Wagner model [30]. Nucleation of precipitates is calculated from an extended Classical Nucleation Theory (CNT) [31,32] model for multi-component systems. The transient nucleation rate J ,

$$J = N_0 Z \beta^* \cdot P_{nuc} \cdot e^{\left(\frac{\tau}{t}\right)} \quad (1)$$

describes the rate at which nuclei are formed per one unit volume and time. The unity is $[(\text{new nuclei})/(\text{m}^3 \text{s})]$. In Equation (1), N_0 represents the total number of possible nucleation sites. The Zeldovich factor Z , takes into account that the nucleus is destabilized by thermal vibrations compared to the inactivated state and it is often of the order of 1/40 to 1/100. The atomic attachment rate β^* takes into account the long-range diffusive transport of atoms, which is necessary for nucleus formation if the chemical composition of the matrix is different from the chemical composition of the precipitate. Furthermore these factors are multiplied by a factor P_{nuc} , which is related to the probability of a single nucleation event

$$P_{nuc} = e^{\left(\frac{G^*}{kT}\right)} \quad (2)$$

and an expression which takes the incubation time τ into account.

$$\tau = \frac{1}{2\beta^* Z^2} \quad (3)$$

In Equation (2), k is the Boltzmann constant and T is the absolute temperature. The critical nucleation energy G^* is given by

$$G^* = \frac{16\pi}{3} \frac{\gamma^3}{D^2} \quad (4)$$

which is a function of the interfacial energy γ and the total driving force D .

Subsequently, a series of calculations for various cooling rates is presented and compared with the experiments. Finally, two parameter studies are shown with the aim of investigating the sensibility of the simulation results to the input data, i.e. to the accuracy of the independent thermodynamic and diffusion databases [33,34], which we used for our calculations.

Results

Precipitate morphology

The γ' precipitates observed in this study cover shapes from simple spheres over cubes, octocubes to very complex structures like octodendrites and dendrites. This transition is caused by the difference in lattice parameter of precipitate and matrix. This phenomenon is well known and it has been investigated several times (e.g. refs. [1,2,19]). Depending on the cooling rates between 78 K/s and 0.0042 K/s, different morphologies of γ' precipitates in UDIMET 720 Li develop. Corresponding micrographs are shown in Figure 1.

Figure 1(a) shows precipitates, which form during cooling from 1180 °C to 400 °C with a rate of 78 K/s. The shape of these particles is spherical or at least nearly spherical. Lower cooling rates cause an increase in the mean particle diameter as shown in **Figure 1(b)**. On further decreasing the cooling rate, the morphology of the microstructure gradually changes to more and more irregular precipitate shapes caused by the increasing misfit strain between particle and matrix with increasing precipitate size [2]. Figure 1(c) shows several non-spherical, faceted particles. In the lower right corner of the image, a precipitate with cubic cross section is observed. Another feature visible in this image is the observation of a second population of much smaller precipitates located in the corridors between the cubic particles. These multimodal particle distributions in UDIMET 720 Li on continuous cooling have been reported previously by, e.g., Mao et al. [20] and they are also predicted by the present numerical simulations with MatCalc. If the cooling rate is further decreased,

more and more cubes and octocubes are observed (Figures 1d,e). Finally, at the lowest cooling rates, the precipitate – matrix phase boundaries become incoherent and the precipitate shape resembles that of dendrites (Figure 1f).

The elongated precipitate shape at grain boundaries observed in sample 10 (Figure 1e) has stimulated further investigations. These shapes are produced by discontinuous precipitation, which occurs by the simultaneous precipitation at grain boundaries and grain boundary movement [35]. The right part of this image shows a very homogeneous distribution of precipitates with almost identical diameter, which is the result of a two-dimensional projection of a three-dimensional array of tubular precipitates in the normal to the growth direction of the precipitates. Figure 2 shows a corresponding microstructure reproduction performed with sequential sectioning using the FIB (focused ion beam) technique [36].

Precipitate size evolution

The samples 1–8 (spherical or nearly spherical precipitates), are also studied quantitatively with regard to the mean diameter. Since the determination of sizes and size distributions of precipitates with non-spherical morphologies is difficult, the quantitative analysis concentrates on samples with a mean precipitate diameter of less than 0.7 μm . This limit is well established in literature (see, e.g. ref. [37]). The measured and corrected mean diameters are summarized in Table III.

Table III: Measured and stereologically corrected [24] precipitate sizes

sample	cooling rate [K/s]	d_{mean} [nm]	
		measured	corrected
1	78	49.4	53.2
2	19.5	58.6	63.8
3	6.5	92.5	98.7
4	3.25	149.6	159.8
5	1.625	165.8	186.4
6	0.867	258.4	278.9
7	0.433	319	347.9
8	0.217	379.9	393.5

To complement the investigations, precipitation kinetics simulations are carried out with the software MatCalc. Depending on the applied cooling rate, the calculations predict the development of multi-modal particle distributions. The reason is found in the balance between continuously increasing supersaturation with decreasing temperature and the decreasing diffusivity of the precipitate-forming solute atoms. The interaction of these two counteracting mechanisms leads to the occurrence of ‘nucleation waves’.

Figure 1: Morphologies of γ' precipitates in UDIMET 720 Li depending on cooling rates between 78 K/s and 0.0042 K/s (a-c are EFTEM Cr-map images, d-f are SEM images)

Figure 2: 3D-reconstruction of precipitates at the grain boundary using the method FIB

Figure 3: Calculated nucleation rate (a) and number of precipitates (b) while continuous cooling from 1180 °C to 400 °C in 79200 s

Figure 3(a) shows a plot of the calculated nucleation rates demonstrating these oscillations. Figure 3(b) displays the predicted number of particles during cooling from 1180 °C to 400 °C. With each peak in the nucleation rate, the number of precipitates increases sharply. Due to these nucleation waves, various different precipitate size distributions are generated.

Discussion

Formation of multi-modal particle distributions during continuous cooling

Compared with isothermal heat treatments, the precipitation process during continuous cooling heat treatments is more complex. Generally, precipitation occurs in the three stages nucleation, growth and coarsening. In addition to the decrease of supersaturation in the matrix due to the formation of γ' precipitates, supersaturation of the matrix increases continuously due to the reduction of temperature. These two effects are counteracting mechanisms, which finally control the evolution of the multimodal precipitate distributions. In addition to this, the diffusivity of elements also decreases with decreasing temperature, thus making elements less and less mobile and the maximum diffusion field extension smaller and smaller. Mathematically, all these processes enter the expressions for the critical nucleation barrier G^* decreases (see Equation (4)) and the nucleation rate J (Equation 1). The competition between increasing driving force, decreasing diffusivity and narrowing of the diffusion fields finally leads to the observed oscillating nucleation characteristics.

Figure 4: Competition between increasing supersaturation due to continuous cooling and decreasing supersaturation due to the formation of γ' precipitates

Figure 4 schematically shows the different stages of the evolution of multi-modal distributions during continuous cooling. After cooling below solid solution temperature, a first nucleation burst of γ' precipitates occurs, characterized by low supersaturation and thus low nucleation rate. Figure 4(a) indicates that the particle spacing in the first nucleation wave is wide. At elevated temperature, the γ' precipitates are surrounded by large diffusion fields, causing extensive growth to large size.

On decreasing temperature, the maximum diffusion field extension reduces and, in the corridors between the existing precipitates of the first nucleation burst, the supersaturation in the matrix increases again (Figure 4(b)). This causes another nucleation burst at lower temperatures and a second γ' distribution of precipitates in between the precipitates of the first wave (Figure 4(c)). Because the diffusivity is much smaller at lower temperatures, the precipitates of the second wave grow slower and the mean precipitate size also remains much smaller. By further

continuous cooling another critical temperature can be achieved which eventually leads to a third nucleation burst and hence to a third population of γ' precipitates between the precipitates of the first and the second population. This process repeats until no further nucleation occurs. The simulation as well as the experimental observation clearly demonstrate this effect.

Comparison between theoretical predictions and experimental results

Figure 5 summarizes and compares the experimental and computational results where the three solid lines represent the three predicted precipitate populations. The dashed lines indicate regions where overlapping of distributions is predicted. From these results it is easily seen that the mean diameter of precipitates increase linearly with cooling time if plotted in double logarithmic scale. This confirms the observations of Mao et.al. [20], who derived a phenomenological relation (Equation 7) for the size of precipitates of the first population as a function of cooling rate.

$$\log D_{\gamma'} = 0.0165 - 0.354 \cdot \log \frac{dT}{dt} \quad (7)$$

Very good agreement between the experimental data of the present work and of the data of Mao et.al. [20] is observed. It is important to emphasize that the present simulations are predictive in character and no fitting parameters are employed. In this sense, prediction and experiment can be considered to be in satisfying agreement over a wide range of different cooling rates, at least for the first population.

Despite the correct prediction of the size of the primary precipitate population, deviations in the second and third population are observed. The measured sizes for the second and third population are generally too small compared to the simulations. The differences between simulation and experimental observation can be attributed to a number of simplifying assumptions in the model as well as difficulties in the measurement procedures:

- The precipitation kinetics model implemented in MatCalc is a mean-field type model and it is therefore

strictly valid only for dilute solution, where the size of the precipitates are assumed to be sufficiently small compared to the diffusion fields surrounding the precipitates. This is certainly not valid in the present UDIMET 720 Li alloy due to the high amount of alloying elements. Often, the precipitates are located very close to each other and the corridors between the precipitates for other populations are too narrow.

- Both, the simulation with “MatCalc” and also the correction method after Sonderegger [27] require spherical shape of precipitates. This prerequisite is not entirely fulfilled for larger precipitates.

Furthermore, it should be noted that theoretical predictions can only be as exact as the accuracy of the input data, i.e. the thermodynamic and diffusion databases [33,34]. To investigate the sensitivity of the simulation results on variations of input data, two parameter studies are carried out for the most sensitive and, probably, also most undetermined parameters interfacial energy and diffusivity of alloying elements. Both parameters are varied by constant factors.

Figure 6(a) shows a distinct maximum of the predicted mean precipitate size when varying the value of the predicted interfacial energy. The maximum is observed when multiplying the predicted interfacial energy by a factor of 0.8. A linear dependence of the mean precipitate size is observed on the diffusion coefficient (Figure 6(b)), if the curves are plotted in double logarithmic scale. This means that the precipitate size scales directly with the diffusion coefficient. A change in the diffusion coefficient by a factor of two causes a variation in the mean precipitate size by a factor of 1.5.

This parameter studies show in a realistic manner the strong dependence of the simulation on the accuracy of the input data. Furthermore, it should be noted that these parameters are experimentally determined and uncertainties in the diffusion coefficient by a factor of two are realistic. Concerning these facts, the accuracy in predicting the precipitate sizes must be considered as excellent. It is again emphasized that no fitting parameters have been employed in the simulations and all values have been taken from the independent thermodynamic and diffusion databases [33,34].

Figure 5: Average precipitate radius over the cooling period from 1180°C to 400 °C

Figure 6: Parameter studies concerning the interfacial energy (a) and the diffusion coefficient (b)

Summary and Conclusions

- On continuously cooling samples from the alloy UDIMET 720 Li, depending on cooling rate, discontinuous precipitation as well as mono-modal and multi-modal distributions are observed.
- With decreasing cooling rate, the morphology of γ' precipitates in UDIMET 720 Li change from spheres over cubes and octocubes to incoherent dendritic structures (see also ref. [2] for other nickel based superalloys).
- The theoretically predicted average precipitate size and the experimental diameter of the γ' precipitates of the present work and of the work [20] are in excellent agreement over a wide range of cooling rates, at least for the first population.
- On a logarithmic scale, the average precipitate radius versus cooling time shows an approximately linear relation. This is in agreement with the observations in ref. [20].
- The theoretically predicted multi-modal precipitate distributions are experimentally confirmed.
- The parameter studies on the influence of the thermodynamic and diffusion data on the simulation results clearly indicate a strong dependence of the results from the input data. Particularly important seems to be accurate data on the diffusion coefficients of the alloying elements over the entire range of temperatures.

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