MODELLING OF δ-PHASE DISSOLUTION DURING PREHEATING OF INCONEL718 TURBINE DISKS.

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Abstract

The relevance of δ -precipitates for controlling the grain size in Nickel-base alloys during the forging process is well known. However, a high volume fraction of δ -phase causes a lower Nb-content of the matrix, followed by a reduced fraction of γ "-precipitates and, thus, less favourable mechanical properties. Considering these facts, the possibility of calculating the dissolution kinetics of δ -precipitates as a function of time, temperature, chemical composition and size is of high importance for modelling the microstructure development during a forging process. In computer simulations of diffusional phase transformations (e.g. with software DICTRA) the high number of relevant alloying elements in INCONEL718 leads to massive problems with the numerical stability. Therefore, a multi-component precipitation model included in the thermo-kinetic computer program MatCalc was used to calculate the δ -phase dissolution at various temperatures. Interrupted annealing experiments were performed and the fraction, size distribution and morphology of the δ -precipitates were determined by means of quantitative optical microscopy. When comparing the experimental data with the calculations, very good agreement was found.

Introduction and Background

The interest in microstructure modelling has gained considerable momentum over the past two decades. The decisive reason for this development has been provided by the needs of industry to improve productivity and product quality. However, most of the published microstructure models [1-13] are more or less semi-empirical and, therefore, have no predictive capabilities with regard to second phase precipitation or dissolution kinetics. Especially in alloys, were the solubility temperature of precipitates is located in the range of the process window for hot deformation, the effect of varying phase fractions can significantly affect the microstructure and mechanical properties during and after the deformation process [11].

With the development of computers, two different concepts for calculating phase transformations in complex alloys have been pursued: On one hand, semi-empirical models based on the C-curve concept [14-25] have been developed to predict phase fractions transformed as a function of time and temperature. On the other hand, thermo-kinetic computer programs, such as DICTRA [29] or MatCalc [26, 27], provide the possibility for multi-component phase transformation computations based on the local-equilibrium-concept [26-29]. The major advantage of the second method is, that variations in chemical composition and their influences on transformation kinetics can be considered directly. The thermodynamic information (i.e. chemical potential gradients) that is needed to evaluate the diffusion coefficient matrix is calculated from the Thermotech nickel-base database [30]. Because reliable mobility data for niobium in nickel-base alloys was missing in the DICTRA mobility database, the corresponding parameter was determined from experimental data of Patil and Kale [31]. Thus, the dissolution calculations were performed without using any fitting parameter.

Experimental Investigations

The chemical composition of INCONEL718 used in this investigation is shown in table 1. The material was obtained as homogeneous preformed ingot with 203 mm (8") diameter. The specimens were cut and machined to 10 mm diameter and 4 mm height. The heat treatment was carried out in a conventional 1500 °C maximum temperature annealing furnace. Because of the good corrosion resistance of the alloy and the short annealing times at high temperatures, the influence of oxygen on the dissolution of δ -particles was neglected. After annealing, all specimens where water-quenched and prepared for optical and secondary electron microscopy.

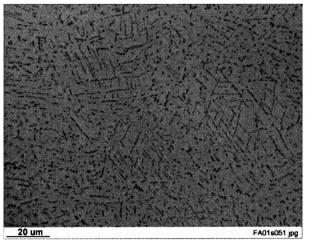
Table 1. Chemical Composition of INCONEL718 (wt%)

Ni	Fe	Cr	Со	Nb	Mo	Al	Ti	С	В
54,54	17,61	17,86	0,18	5,373	3,005	0,495	0,91	0,025	0,003

Two different reagents were used for δ -sensitive etching: Canada's solution (80 ml H₂O, 20 ml H₂SO₄, 20 ml HF, 10 ml HNO₃), 90 s wiping with cotton wool, and Schalk solution (50 ml H₂O, 10 mg K(SO₃)₂, 5 ml Keller solution – 190 ml H₂O, 3 ml HCl, 5 ml HNO₃, 1 ml HF), etching 30 min.

Optical microscopy was carried out on a Reichert MeF3. Three to five images per specimen were taken with a b/w digital camera having a resolution of 1024x768 pixels and 256 grey values. In figures 1 to 4, the dissolution sequence of the δ -particles at 1035 °C is shown. For shading correction and digital image recognition, a Zeiss KS400 system was used. Apart from determining the δ -phase fraction, the KS400-macro simultaneously allows the calculation of the amount, size and alignment of the particles. This feature gives the possibility to reliably separate carbides from the δ -precipitates. Varying the macro parameters for the particle detection between reasonable limits, a mean standard deviation for the accuracy of the method was calculated to be 4.78%. For comparison, two images were used for manual δ -phase fraction detection with line segment technique. The difference between the computer aided method and the manual evaluation was below 5%. The average variation of the δ -phase fraction along the cross-section of the specimens was 17.9%.

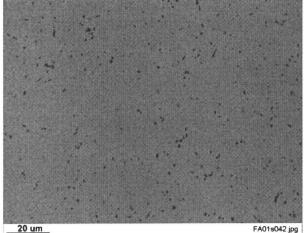
Secondary electron microscopy was carried out on a LEO 1450VP and it was used for additional verification of the optical methods.



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Fig. 1: Initial microstructure

Fig. 2: 1035 °C -3 min treated structure



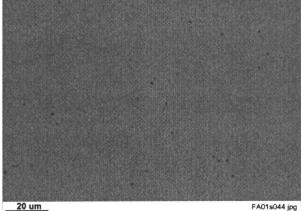


Fig. 3: 1035 °C -10 min treated structure

Fig. 4: 1035 °C -30 min treated structure

Numerical Simulations

The equilibrium solubility temperature of the δ -phase was calculated to be 1031,8 °C. The change of the stable phase fraction versus temperature together with experimental values obtained from the annealing experiments is shown in fig 5. At the solubility temperature of the δ -precipitate, a certain amount of MC-carbide and boride is present. In order to simplify the numerical simulation of the δ -phase dissolution kinetics, the equilibrium matrix composition at this temperature (table 2) was taken as the new overall system composition and the two rather stable carbide phases could be suspended. The simulations were thus carried out considering only the fcc-matrix phase and the δ -precipitate. The precipitate shape was assumed to be spherical at all simulation times.

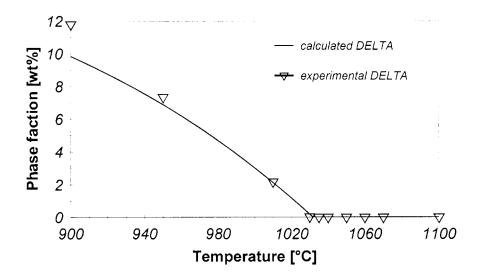


Fig. 5: Equilibrium δ -content vs temperature

Table 2. Calculated chemical composition of the FCC-matrix at 1031,8 °C in wt%

Ni	Fe	Cr	Со	Nb	Mo	Al	Ti	С	В
54,69	17,66	17,9	0,18	5,18	2,99	0,5	0,9	0,001	0,0003

The calculation of the δ -dissolution kinetics is based on the classical growth theory, where the variation of the particle radius R at time t can be expressed by the following equation [32]:

$$\frac{dR}{dt} = \frac{c_R - c(t)}{c_D - c_R} \cdot \frac{D}{R}$$
 [1]

Where c(t) is the time dependent concentration of the diffusion controlling element (here: Nb) in the Matrix, c_p the concentration in the particle, c_R at the interface and D is the volume diffusion coefficient of Nb in the matrix.

The influence of surface tension on the equilibrium amount of Nb at the precipitate / matrix interface c_M^e was taken into account by using the well-known Gibbs-Thomson equation [33]:

$$c_R(R) = c_M^e \exp\left(\frac{2\sigma_{MP}}{R_e T} \cdot \frac{V_P}{R}\right)$$
 [2]

Where σ_{MP} is the interfacial energy, V_P ist the molar volume of the precipitate and R_g ist the Universal Gas Constant. Due to the lack of experimental data for σ_{MP} in the literature, the interfacial energy of the δ -precipitate / matrix interface had to be estimated. A value of 0.1 J/m² was adopted in the calculations [34].

A second missing parameter was the mobility of Nb in the INCONEL718 matrix. However, this value could be assessed from experimental data of Patil and Kale [31]. The following expression was evaluated and added to the mobility database:

$$MQ(FCC A1\&NB,NI:VA) = -215000+R*T*LN(+2e-006)$$
 [3]

The original diffusivity values reported in this work [31] had to be slightly modified because, based on the current thermodynamic information, these values could not reproduce the given diffusivity values.

Results

A comparison of experimental and simulation data is shown in figure 6. The mean particle radius is plotted versus time for 6 different temperatures near and above the δ -solubility temperature. Additionally, a table of the experimental δ -phase fraction for the plotted dissolution simulations is shown in table 3. An initial particle radius $R_{t=0}=0.57~\mu m$ was evaluated with digital image analysis and utilized in the calculations for all temperatures.

Table 3.: Mean δ -fraction and standard deviation for experimental data plotted in figure 6

temperature °C	annealing time 3 min	annealing time 10 min	annealing time 30 min	annealing time 60 min	annealing time 120 min
1030	3.69±0.53	0.92±0,23	0.32±0.08	0.02±0.01	0.005±0.002
1035	2.90±0.39	1.33±0.12	0.21±0.04	0.02±0.003	0
1040	2.77±0.29	1.7±0.35		0.37±0.25	0
1060	2.64±0.73	0.36	0.01±0.005	0	0
1070	3.15±0.65	0.39		0	0
1100	1.51±0.07	0	0	0	0

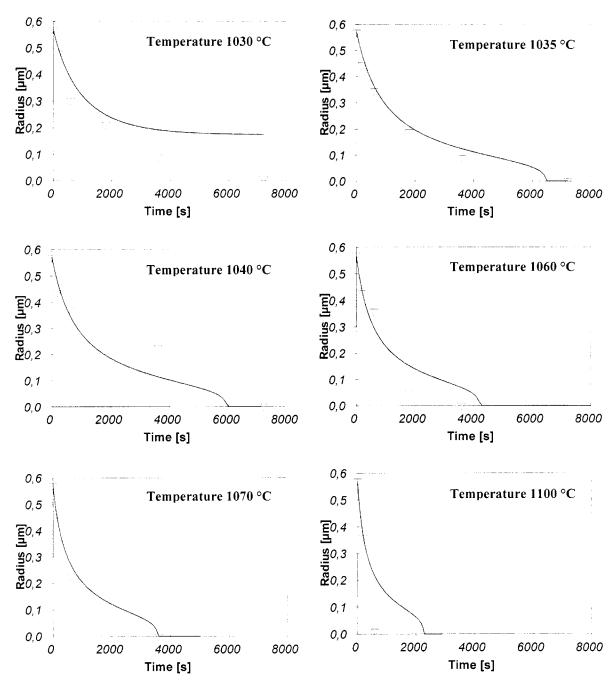


Fig. 6: Comparison of calculated and experimental mean particle size vs. time in the temperature range between 1030 and 1100 °C.

From figure 6 it is obvious that the kinetic simulations exhibit a very good correlation with the experimental data. For very small δ -fractions, corresponding to advanced δ -dissolution, the mean radius of the δ -particles is very hard to determine, due to the limitations in resolution of the used microscope (0.3 μ m). Calculated values above a threshold of 0.15 μ m are generally in good agreement with the experimental data points. The drop off in the curves at longer times is due to the influence of the surface tension.

Summary and Conclusions

The present investigations have shown that the thermo-kinetic simulations of the dissolution kinetics of the δ -phase in INCONEL718 based on classical growth theory and the thermodynamic information by the ThermoTech Nickel-based database give excellent agreement between calculation and experiment. A comprehensive program of annealing experiments above and at the solubility temperature of the δ -phase and within the range of the processing window for hot deformation was carried out to verify the predictions of the numerical simulation.

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