QUANTITATIVE MICROSTRUCTURE ANALYSIS TO DETERMINE OVERHEATING TEMPERATURES IN IN100 TURBINE BLADES

H. Huff and H. Pillhöfer

Department of Materials Technology
MTU Motoren- und Turbinen-Union München GmbH
Munich, Federal Republic of Germany

Abstract

The paper presents a method for the determination of overheating temperatures in nickel alloys by the evaluation of the gamma prime microstructure.

In the temperature range 1080 to 1200 °C, a stable fine structure with a close relationship between the gamma prime volume fraction and the temperature forms within 30 seconds.

Accordingly, overheating of turbine blades can be determined qualitatively above 1040 °C, and quantitatively above 1080 °C.

Furthermore, these relationships provide a better understanding of the formation of the fine structure of precipitation-hardening nickel alloys by showing how heat treatment in the partial solution range of the gamma prime phase and ageing influence the high-temperature strength-determining gamma prime structure.

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Introduction

In nickel-alloy turbine blades, overheating can produce certain, at times pronounced changes in the gamma prime phase. In the assessment of overheated parts for reusability, the criteria applied have been a combination of gamma prime particle size and shape (1). This approach, however, is relatively unsafe. It was thus appropriate to find a quantitative, practically exclusively temperature-dependent relationship between the formation of gamma prime particles and overheating conditions (T > 1050 °C). An attempt also had to be made to establish a component-related overheat ceiling.

Procedure

Principle of Measurement and Nomenclature

The volume fraction of gamma prime phase that does not go into solution in the gamma matrix under overheat conditions was selected and stereologically measured as a characteristic, temperature-dependent structural feature (2).

If cooling from a temperature condition in the partial solution range of the gamma prime phase (approximately 1000 to 1200 °C) is relatively fast, two different gamma prime particle dispersions occur:

1) Primary gamma prime particles; these include the usually larger, cubic to globular gamma prime particles stemming from the original condition

2) Secondary gamma prime particles; these include the fraction of small to very small, finely dispersed gamma prime particles that go into solution and reprecipitate at high temperature

Test Parameters and Microstructure Specimens

Airfoil sections in IN100 were heat treated and fan-cooled (cooling rate 120 °C/ min to 800 °C) in a lab-type vacuum furnace following long-term ageing (1000 °C/100 h + 900 °C/20 h + 800 °C/20 h)

Temp. °C	1040	1080	1100	1120	1140	1160	1180	1200	1210
Time	2 h	2 h	5 min 2 h 24 h	5 min 2 h	5 min 2 h 24 h	5 min 2 h	5 min 2 h	5 min 2 h	2 h

To simulate very short overheat periods, small, flat IN100 specimens (thickness < 2.5 mm) were fitted with thermocouples and immersed for short, specific holding times in a hot salt bath (Semper Neutral 950) at a preheat temperature of 900 °C. Preheating and overheating in the salt bath were both carried out in a furnace with high temperature gradient. The heating period lasted less than 20 seconds. The salt-bath treatment was carried out following the procedure of Böhm (3). Other heat-treatment parameters were additional temperature cycles following overheating, ageing isotherms at 800 to 950 °C, and different cooling rates after complete gamma prime dissolution.

Mechanical Properties

Separately-cast stress-rupture specimens (Ø 9.5 mm) in HIP + aged condition were overheated for 10 minutes at 1050 to 1200 °C on a hot gas test rig, and cooled at over 240 °C/min. One set of specimens was additionally overheated under a stress of 200 MPa (5 min at 1050 °C and 1075 °C, 30 sec at 1100 °C and 10 sec at 1125 °C). Other specimens were additionally aged for 8 h at 900 °C after overheating only.

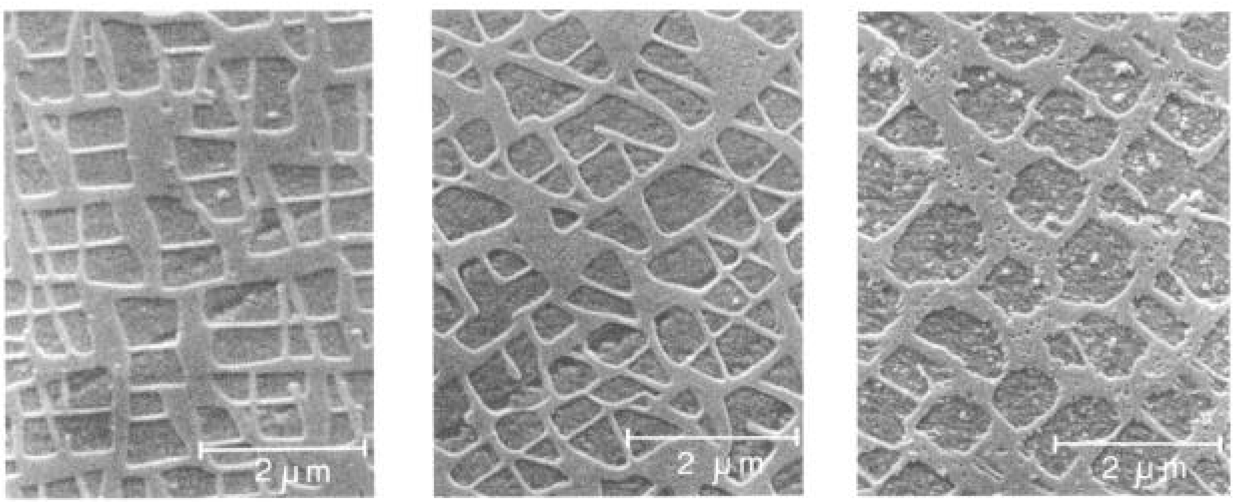
Microstructure

For quantitative evaluation of the microstructure, the gamma prime phase was etched selectively, using an etchant according to Ref. (4). SEM micrographs (5000X) were analysed with the aid of a Hewlett Packard HP 1000 process computer using the linear intercept method and special stereological programs (5, 6).

Results

Influence of Temperature on Gamma Prime Microstructure

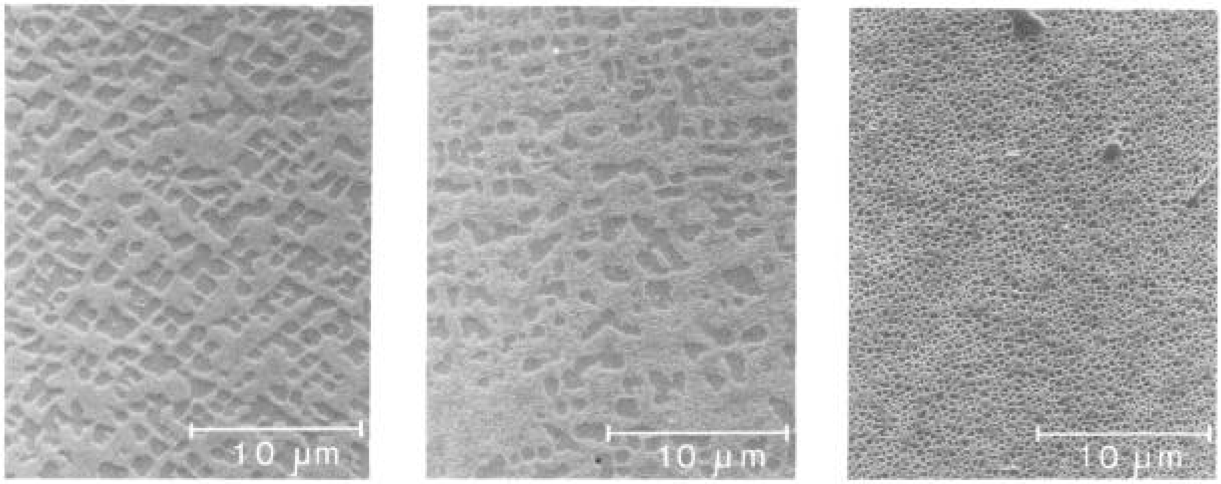
The SEM micrographs in figures 1 and 2 show the gamma prime microstructure of IN100 following exposure to several different temperature levels from 1040 to 1210 °C. The starting condition is also shown. The gamma prime particles appear as a dark phase.



a) b) c)

Figure 1 - Influence of temperature on the gamma prime phase in IN100; a) Starting condition, showing a uniform dispersion of the primary gamma prime particles b) 1040 °C c) 1080 °C

A second dispersion of small, secondary gamma prime particles appears in addition to the approx. 0.8 μm gamma prime particles of the starting condition as early as at 1040 °C, but even more clearly at 1080 °C. As can be seen in figure 2, the area fraction of the primary gamma prime particles reduces visibly from a temperature of 1100 °C on, whereas the gamma matrix fraction, appearing as a light area on the micrograph, increases. Secondary gamma phase particles are embedded in these light matrix areas. At 1200 °C, the primary gamma prime particles have been completely dissolved and re-precipitated.



a) b) c)

Figure 2 - Influence of temperature on the gamma prime phase
a) 1100 °C, b) 1140 °C, c) 1210 °C

Influence of Temperature and Holding Time on Volume Fraction

The results of the quantitative microstructure analysis of the primary gamma prime volume fraction are shown in figure 3. Regardless of the holding time (5 minutes, 2 hours, 24 hours), the volume fraction decreases continuously from an initial 55% with increasing temperature. The scatter in the values for each temperature is less than 5% by volume.

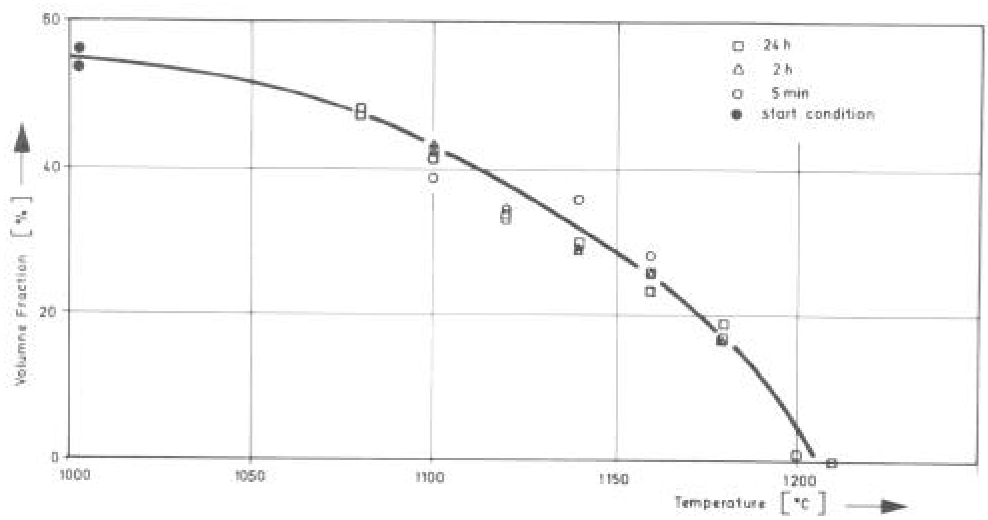


Figure 3 - Influence of temperature and holding time on the primary gamma prime particle volume fraction in IN100

Minimum Time Required for Change in Volume Fraction

The primary gamma prime volume fractions from the tests with short overheat times in the salt bath are shown in figure 4. The last two values shown in each curve are the results of overheat tests in a chamber furnace and represent the temperature-related equilibrium volume fraction for holding times of 5 minutes and 2 hours.

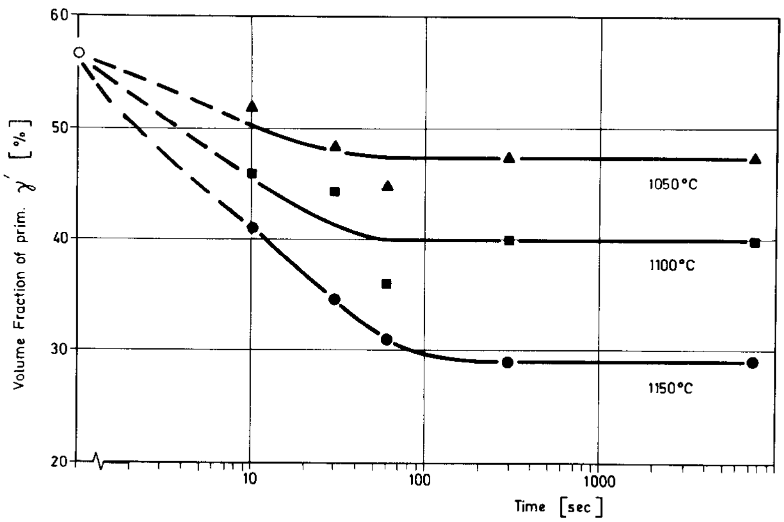


Figure 4 - Primary gamma prime particle volume fraction in relation to short holding times at different temperatures. In each case the last two values are equilibrium values (acc. to Böhm (3))

Influence of Cooling Rate

The influence of the cooling rate on the occurrence of secondary gamma prime particles was investigated optically, because it was also desirable to ascertain the secondary gamma prime volume fraction. After cooling from 1160 °C at a rate of 70 °C per minute, secondary gamma prime particles were still clearly present as a second dispersion of separate particles in addition to the primary gamma prime particles. At a cooling rate of 30 °C per minute most of the secondary particles have merged with the primary particles, and at the even slower rate of 8 °C per minute there is no secondary dispersion distinguishable in the SEM micrograph. Only the irregular shape of the primary particles still betrays partial redissolution.

Cooling rate to 800 °C (°C /min):	2	8	72	240	800
Gamma prime volume fraction (%):	54	52	50	43	21

The maximum fraction of precipitated gamma prime phase after long-term ageing is 55 to 57% by volume. The precipitation of the gamma prime phase grows less and less with increasing cooling rate. For comparison, the cooling rate of the airfoil area of a blade in still air is roughly 200 °C per minute in the upper temperature range.

Volume Fraction after Ageing

IN100 specimens with a starting volume fraction of 46% uniform gamma prime phase were aged at temperatures ranging between 800 and 950 °C for periods of between 30 minutes and 125 hours. The results can be seen in figure 5.

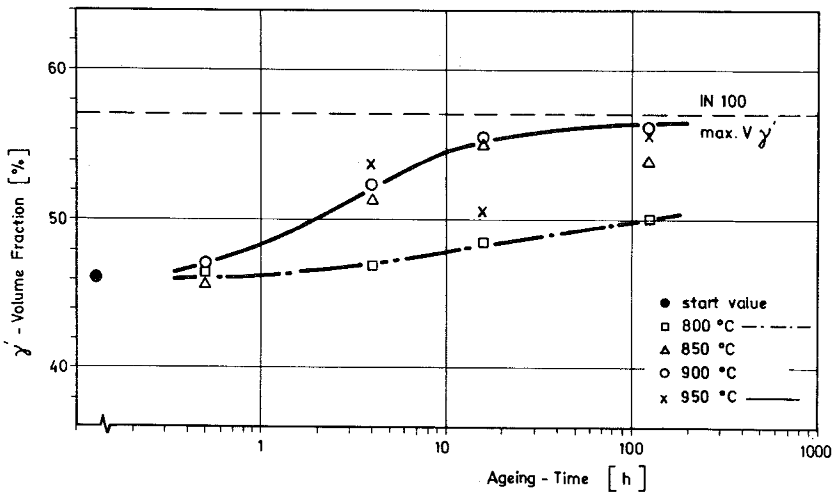


Figure 5 - Influence of ageing on the volume fraction of precipitated gamma prime phase in IN100

The maximum possible volume fraction is not reached at 800 °C. But, at the two higher ageing temperatures the volume fraction approaches its maximum within a few hours.

Stress-rupture Strength

To ascertain the length of time a blade will withstand overheat temperatures when subjected to centrifugal force, and to establish reasonable periods of stress for simulation of overheat conditions, stress-rupture tests were carried out on IN 100 specimens at elevated temperatures at a stress of 200 MPa. The results are summarised below:

Test temperature (°C):	1050	1075	1100	1125	1150
Time to rupture (minutes):	18	9.5	2.7	1.0	0.5
(2 specimens)	163	7.2	3.2	1.5	0.8

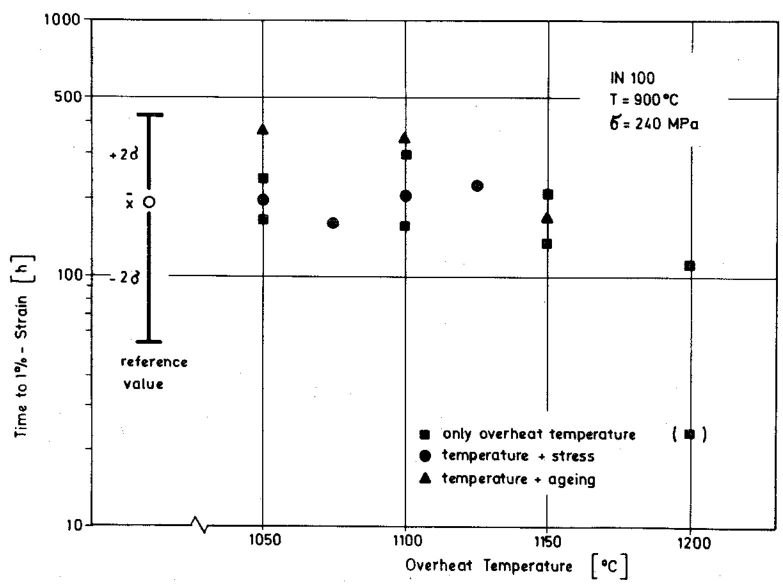


Figure 6 - Influence of overheating at various temperatures on the stress-rupture strength of IN100 at a test temperature of 900 °C and 240 MPa stress

Figure 6 shows the stress-rupture strength of IN100 specimens overheated deliberately at various temperatures. One set of specimens was overheated only, whilst a second set was both overheated and subjected to a load, and a third was aged at 900 °C for 8 hours after overheating. For comparison, a typical mean value with standard deviation for non-overheated material is also plotted for the test conditions 900 °C/240 MPa. The value at 1200 °C is shown in brackets because a fault was suspected in the simulation conditions.

The 1% strain values of the specimens up to an overheat temperature of 1150 °C are in the middle of the range for normal, non-overheated material. A decrease in the values sets in only after this temperature has been reached. Remarkable are the clearly higher strain values at 1050 and 1100 °C of the material aged after overheating and before the stress-rupture test.

Discussion

The noted progressive solutioning of the primary gamma prime phase at temperatures in excess of 1000 °C, as verified stereologically at 1080 °C and above, is caused by the temperature-dependent solubility of the gamma prime phase or of the gamma prime producers Al and Ti in the solid nickel solution. This relationship is illustrated in the Ni-Al and Ni-Ti binary alloy diagrams of Ref. (7). The equilibrium of the primary gamma prime phase and solid solution fractions is frozen in by rapid cooling and is preserved as an optically assessable change in the original gamma prime distribution.

A similar relationship between the solution or precipitation temperature and the primary gamma prime fraction has been found in other nickel-based alloys with high gamma prime-content, such as U700 (4, 8), SRR99 (3) and DS MAR-M 200 (9). Holmes (10) also observed differing gamma prime dispersion after heat treatment below the gamma prime solvus in IN100.

Minimum Time Required to Dissolution of the Gamma Prime Phase

It was demonstrated experimentally that approximately 60 seconds are sufficient for the temperature-related phase equilibrium to set in, and that just 30 seconds are enough for 90% of the equilibrium value to be approached (Figure 4).

Estimation of the diffusion rates from an investigation into the high-temperature brazing of IN100 (1) and experience with grinding of blade roots (12) confirm that the gamma prime phase goes rapidly into solution.

Evaluation of Other Factors Influencing the Gamma Prime Fraction

Other factors having a critical influence on the gamma prime distribution and its evaluation are:

a) The rate of cooling after overheating, which must be high enough to preserve the gamma prime fraction typical for the overheat temperature in question. However, secondary gamma prime particles can no longer form if the rate is too high, but they will form following brief ageing.

b) Treatment after overheating; long holding times after overheating cancel out overheat-related differences in the gamma prime structure.

c) The accuracy of the quantitative evaluation is determined essentially by the preparation of the microsection and the stereological evaluation, where care must be taken to ensure that stereologically correct preconditions for area or volume analysis are created by avoiding excessive selective etching of the gamma prime particles.

Other factors affecting the gamma prime structure, such as local and absolute fluctuations in the alloy composition, caused for example by dendritic segregation of the gamma prime producers, effect of stress and life-related changes to the gamma prime particles, do not seem to be of great significance with regard to the structure - temperature evaluation.

Conclusions

A method has been developed that uses microstructural analysis to clearly identify and quantify past overheat temperatures on IN100 rotor blades.

The relationship noted to exist between the volume fraction of primary gamma prime phase and temperature appears to permit

1) Safe and quantitative (± 20 °C scatter) identification of past overheating in the 1080 to 1200 °C range

2) At least qualitative indication of overheating in the lower temperature range of 1040 to 1080 °C

The method operates on and is thus also limited by the premise that a metallographically apparent structural differentiation of the gamma prime phase is allowed to form and survive. This premise again involves the following constraints:

a) The material needs at least 30 seconds or so at overtemperature

b) The rate of cooling from overtemperature should be faster than about 100 °C per minute

c) Overheating must not be followed by an extended holding time (of an hour or longer) at a lower temperture level (above 1000 °C)

The method of measurement can generally be transferred to all blade materials with high gamma prime fraction.

From data obtained in stress-rupture tests with simulated overheating, an overheat ceiling has been derived for IN100.

Taking degradation in stress-rupture performance as a criterion and allowing a safety margin of 20 °C, the overheat ceiling is 1130 °C for IN 100.

Quantitative stereological analysis of the gamma prime microstructure appears to provide a key to a better understanding of the effects of heat treatment on nickel-based superalloys. New insights are gained into the effects, for example, of homogenizing and ageing.

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