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Solution Heat Treatment of Single Crystal Castings of CMSX-4 Nickel-base Superalloy

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Abstract

An investigation of the microstructure and mechanical properties for heat treated directionally cast rods, produced from the nickel-based superalloy, CMSX-4, is presented. The rods were cast using the Bridgman method for manufacturing single crystal structures. The microstructure of the cast rods consists of γ and γ' precipitates. This microsegregation has a negative effect on the microstructure and, hence, the mechanical properties of the castings. The solution heat treatment of the second generation, single crystal Ni-base superalloy, CMSX-4, is known to dissolve the eutectic γ and γ' region. This requires temperatures up to 1316°C and approximately 45 hours total time. These high temperatures and long processing times result in high costs. The aim of this study is to investigate the effect of the heat treatment protocol on the extent of improvement of quality of single crystal castings, as a basis for determining cost feasibility in practice.

Keywords

Nickel-base superalloys, CMSX-4, single crystal castings, solution heat treatment.

1 INTRODUCTION

During solidification through the mushy zone of the Nickel base superalloy, CMSX-4, some of the solute elements prefer to remain in the liquid phase while some elements preferentially diffuse to the solid phase forming a chemical heterogeneity in the solidified structure with a significant fraction of γ and γ' eutectic at the interdendritic region. It has been established that Co, Cr, W, Mo, and Re segregate preferentially to the dendrite cores, while Ti, Al, and Ta segregate preferentially to the interdendritic region. There are two important effects of this microsegregation in the solidified structure: chemical heterogeneity and microstructural heterogeneity. These effects have a direct impact on the mechanical properties and hence the performance of superalloys as high temperature materials.

The γ' precipitates in the interdendritic region of a solidified structure are coarse, irregular shaped and incoherent. Since the γ and γ' interface plays a major role in the development of strength and creep resistance, it is always desirable to have fine, uniform and coherent cuboidal shaped precipitates throughout the microstructure. The chemical heterogeneity in the solidified structure leads to chemical instabilities. Dendrite cores in the solidified structure, being rich in Cr and Re, are preferred locations for formation of the embrittling TCP phases that degrade both the creep and fatigue resistance of the alloy [1-3].

Solution heat treatment is a lengthy process; moreover it is also quite energy and capital intensive, and hence expensive. For this reason, a multistep cycle heat treatment process has been developed for single crystal castings, designed to completely solution the γ' and the most of the γ and γ' eutectic without incipient melting. Heat treatment is also used to reduce residual stresses in castings resulting from the casting process [3].

The all-important mechanical properties for turbine blades, as an example, (such as high-temperature creep resistance) depend largely on the alloy composition, given in Table 1, and on a proper heat treatment protocol. Microsegregation after casting can have either a beneficial or a deleterious effect on the property of the cast product. Most significantly, the chemical inhomogeneity associated with microsegregation usually leads to poor corrosion resistance. For these reasons, nickel-base superalloy turbine blades are subjected to a homogenisation heat treatment, so as to reduce or eliminate residual segregation patterns, and in order to re-dissolve non-equilibrium secondary phases produced by microsegregation [7-8].

Traditionally, two heat treatment stages are used for nickel-base superalloys. First is the solution heat treatment, designed to homogenise the microstructure and reduce the effects of segregation. Second is one or multi-step aging, designed to develop cuboidal γ′ precipitates.

This research focuses on the effects of solution heat treatment on directionally cast nickel-base CMSX-4 superalloy castings, through metallographic and mechanical property investigations [1-3].

2 MATERIALS AND EXPERIMENTAL PROCEDURE

Single crystal (SC) castings from the second generation Ni-base superalloy were produced in a VIMIC 2 E – DS/SX vacuum investment casting system manufactured by ALD Vacuum Technologies. The single crystal samples were cast in the [001] direction at average temperature gradient of 1.5 K/mm and withdrawal velocity of 3 mm/min (Bridgman Process for directional solidification). The chemical composition of the alloy is shown in Table 1.

Elements	Cr	Co	Mo	W	Ta
Weight %	6.5	9.0	0.6	6.0	6.5
Elements	Re	Al	Ti	Hf	Ni
Weight %	3.0	5.6	1.0	0.1	balance

Table 1 - Nominal compositions of CMSX-4

The heat treatment of the castings was performed in a laboratory vacuum furnace equipped with an induction heating system of 80kW total power, a working chamber of 600mm x 400 mm x 400mm, and a set of 9 sleeve protected thermocouples with a temperature measurement accuracy of 3°C. The heating process can be performed by convection to 950°C (in argon or helium atmosphere) or radiation to 1350°C (in vacuum). The furnace is equipped with a vacuum system consisting of a rotary and diffusion pumps capable of generating a vacuum of $5 \cdot 10^{-5}$ bar.

It is well known that single crystal alloys cannot be used for the intended high temperature applications through solution heat treatment only. Since the solidus of polycrystalline alloys is below the γ' solvus temperature, this prevents the complete dissolution of γ' using solution heat treatment. The increased melting temperature of single crystal alloys frequently allows refinement of the γ' microstructure with a solution annealing followed by two aging steps. In the cast alloy with a high volume fraction of γ and γ' eutectic, the complete dissolution of γ' by the appropriate heat treatment is of extreme importance. In addition to dissolving the γ and γ' eutectic and solutioning the γ' for subsequent re-precipitation, the solution heat treatment also reduces the chemical segregation of the elements.

Heat Treatment Stage	Temperature-time settings
Solution heat treatment (annealing)	1277°C / 4h → 1287°C / 2h → 1296°C / 3h → 1304°C / 3h → 1313°C / 2h → 1316°C / 5h → GFC*
Aging 1	1140°C / 6h → AC**
Aging 2	871°C / 20h → AC**

* Gas furnace quench ** Air cooled

Table 2 - Heat treatment protocol

The experimental observation of solution heat-treated samples were obtained through metallographic examination and analysed using both Scanning Electron Microscopy (SEM) and Optical Microscopy (OP) at all stages of the heat treatment process. These stages were chosen as follows:

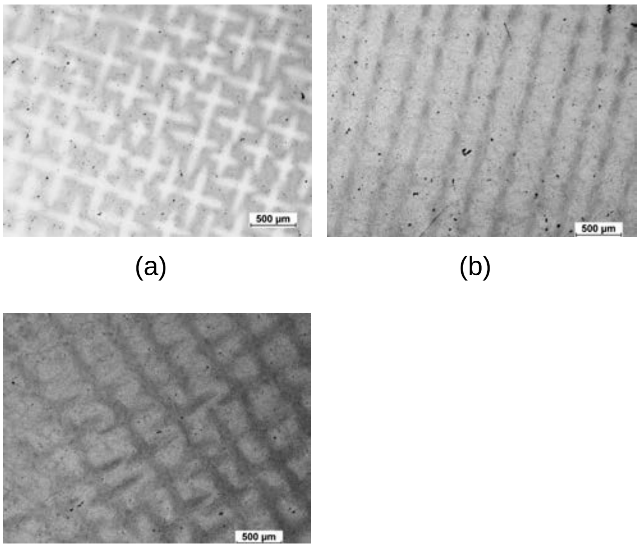
1. Samples after solution heat treatment;
2. Samples after aging 1;
3. Samples after aging 2.

For determining the crystal orientation, the Laue method was used, while hardness tests were undertaken to determine the mechanical strength of the samples.

3 RESULTS AND DISCUSSION

3.1 Investigation of microstructure using OP

Figure 1 presents optical photomicrographs of the interdendritic region of solution heat treated CMSX-4 samples showing single crystal microstructure of the castings after different heat treatment stages. It can be seen that the γ and γ' eutectic has been dissolved.



(a) (b)

(c)

Figure 1 - Microstructure of heat treated sample after the following stages: (a) solution heat treatment (annealing), (b) aging 1, and (c) aging 2

3.2 Investigation of microstructure using SEM

For the SEM investigation, a 10% H_3PO_4 solution etchant was used. This etchant was prepared for electrolytic etching with the following parameters:

voltage 3 Volts, amperage 0.2 Amps, time duration 3-5 seconds.

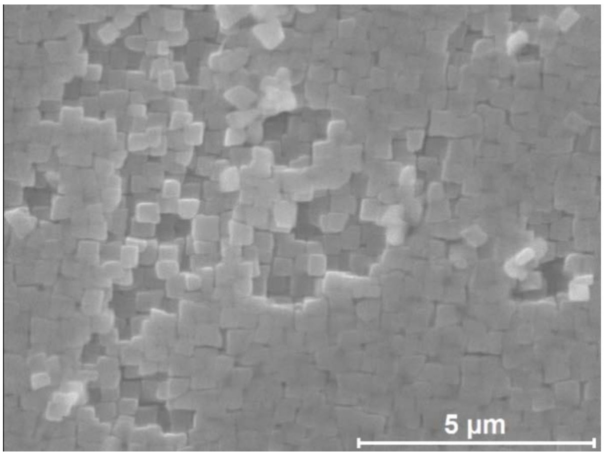


Figure 2 - Sample after annealing.

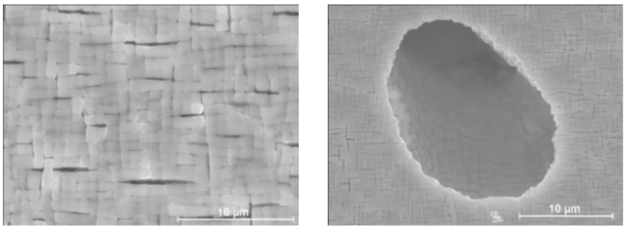


Figure 3 - Sample after aging 1, with evidence of gas porosity (right).

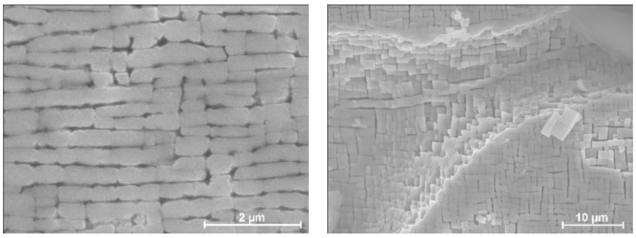


Figure 4 - Sample after aging 2 - dendrite core.

The microstructure of the heat treated samples following each step of the heat treatment indicates that the γ' and the γ/γ' eutectic completely soluted after the end of the solution heat treatment (annealing) stage.

3.3 Mechanical Properties: Hardness

Mechanical hardness tests were performed for the single crystal castings after each of the three different heat treatment stages, in addition to the as-cast samples. The results obtained show an improvement in the mechanical hardness property of the Nickel-base superalloy after the heat treatment process.

Test Sample	Hv (kg/mm^2)
As-Cast sample	436
Solution heat treatment	483
Aging 1	438
Aging 2	472

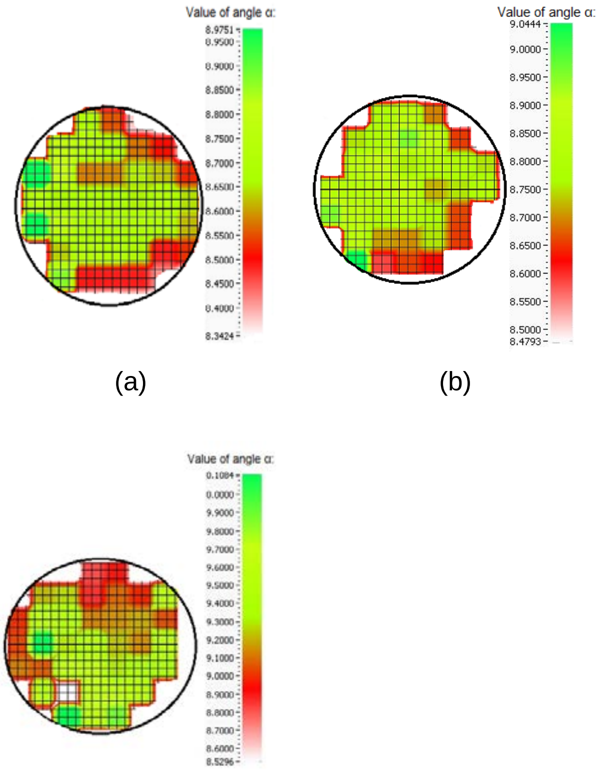
Table 3 - Vickers Hardness Hv

3.4 Diffraction Analysis for Single Crystal structures after heat treatment

A specialised X-ray EFG diffractometer, equipped with a goniometer allowing evaluation on three-dimensional surfaces, was used to determine the crystal orientation distribution on the surface of the single crystal castings. Only samples without defects (such as slivers, freckles, low angle boundaries or high angle boundaries) were used in this investigation. The crystallographic orientation of the samples was determined by Laue back-reflection techniques.

The strength of metals decreases with increasing temperature. Since mobility of atoms increases rapidly with temperature, it can be appreciated that diffusion–controlled processes can have a very significant effect on high-temperature mechanical properties. The assessment of crystalline quality, including the crystallographic orientation, is a very important element of manufacture of single crystal castings made of superalloys.

Superalloy castings display anisotropic mechanical behaviour, in which the material properties (and hence mechanical properties) depend on the crystallographic direction. Hence, the assessment of the crystallographic orientation of single crystal castings forms the basis for determining the mechanical properties of the castings.



(a) (b)

(c)

Figure 3 - Crystallographic maps indicating the angle of deviation α at the base (a), in the middle (b) and at the top (c) of the single crystal as-cast samples.

For acceptable mechanical performance of the castings, it is generally required that the value of the angle of deviation α from the [001] direction (the single crystal growth axis) should not exceed 15°. The greater this angle of deviation, the lower the creep resistance of the castings.

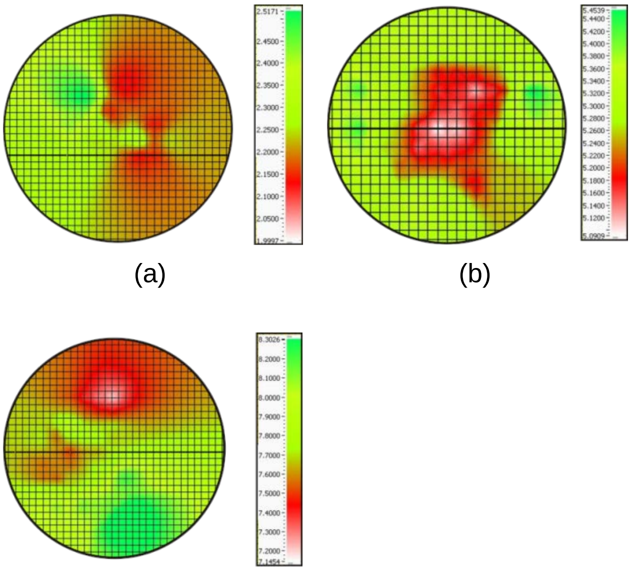
Firstly, the cross-sections of the as-cast castings were investigated at three points along the blade axis.

The map (distribution) of the crystallographic orientation on the studied surfaces is given in Figure 5. The angles of deviation α between are found to range between 8.75° and 9.40° as shown in Table 4. This proves a relatively high degree of structural homogeneity.

As Cast Sample	Angle of deviation α ± 1.0
1 (base)	8.75
2 (middle)	8.80
3 (top)	9.40

Table 4 - Values of deviation angle α from the single-crystal growth axis for the as-cast samples.

Secondly, the cross-sections of the heat treated samples were investigated at the same surfaces as for the as-cast samples. These resulting crystallographic maps are shown in Figure 6.



(a) (b)

(c)

Figure 6 - Crystallographic maps indicating the angle of deviation α for the (a) solution heat treated sample, (b) after aging 1, and (c) after aging 2.

The maps (distribution) of the crystallographic orientation on the studied surfaces are given in Table 5.

Heat Treated Sample	Angle of deviation α±1.0
1 – Solution heat treated	2.20
2 – aging 1	5.09
3 – aging 2	7.70

Table 5 - Values of angle α describing the deviation of the [001] γ' direction from the single-crystal growth axis for the heat treated samples

All values of the angle α are smaller than 15^0 and comply with global manufacturing standards in the aircraft industry. The lowest value of the angle α is found to occur after the solution heat treatment process, which indicates that the heat treated castings will have the highest creep resistance, a critical requirement for turbine blades which operate under high temperature conditions.

4 CONCLUSIONS

The solution heat treatment of the nickel-base superalloy, CMSX-4, results in the elimination or reduction of microsegregation. This allows the manufacture of castings of a more uniform, homogeneous microstructure.

The evaluation of the microstructure using both OP and SEM shows transformation of the dendritic structure to cuboidal γ/γ′ microstructure.

The solution heat treatment (annealing) process dissolves the γ/γ′ eutectic regions early in the heat treatment cycle at temperatures up to about 1316°C.

The mechanical properties investigation by hardness measurement has shown improved properties for the solution heat treated samples as compared to the as-cast samples.

This research has successfully shown that the solution heat treatment process dissolves both the γ′ precipitates formed during cooling from solidification and the γ/γ′ eutectic, and reduces the degree of chemical segregation due to the partitioning of some of the elements to the dendrite core and interdendritic regions. The resulting crystallographic orientation shows significant improvement. All these phenomena improve the material quality and mechanical properties of the single crystal castings.

The research further shows that the aging processes do not improve either the crystallographic orientation or the material hardness of the casting. In fact, there is a reduction in the degree of crystallographic orientation. In addition, there is a significant reduction in hardness after the first aging stage to the as-cast level, improving after the second aging stage to close to the level achieved after the solution heat treatment stage.

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6 BIOGRAPHY

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