PRACTICAL METHOD OF THERMAL HISTORY ANALYSIS BY \( \gamma' \) MORPHOLOGY

CHANGES IN NICKEL-BASE SUPERALLOY

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Summary

The morphological change of \( \gamma' \) in a directionally solidified nickel-base superalloy IN-713C has been quantitatively investigated by the application of a \(<100>\) oriented uniaxial tensile stress during annealing at elevated temperature. In agreement with previous work, it has been found that the coarsening of \( \gamma' \) to platelets, or rafts, was developed in an orientation perpendicular to the applied stress. Furthermore, the degree of rafted \( \gamma' \) has been determined as function of stress, temperature, and time by measurement of the aspect ratio, \( c/a \), of \( \gamma' \). That is, a map of \( c/a \) for stress-temperature for 100 hours was made from experimental data. Conversely, using the above map, it was suggested that it was possible to estimate the stress distribution for actual components such as turbine airfoils after elevated temperature performance by microstructural observation of significant parts. Also, the technique developed in this study is considered to be generally applicable to other single crystal and directionally solidified nickel-base superalloys.
Introduction

Morphological changes of γ' were observed in IN-713C turbo-charger turbine wheels which were exposed to an endurance test for about 100 hours (1). The coarsening of γ' to rafted structures in the airfoils of the wheels was observed in an orientation perpendicular to the applied stress imposed on these parts due to the centrifugal force. The extent to which γ' morphology transformed to the configuration consisting of parallel stringers was observed to vary with the location in the airfoil where the temperature and stress were also different. This suggested that the degree of rafted γ' may depend on stress and temperature as a function of time.

Some experimental studies for morphological changes in γ' were made for single crystal U-700 (2), Ni-Al (3), Ni-13Al-9Mo-2Ta (4), and several polycrystals of nickel-base superalloys. In these investigations, γ' shape change and its dependence on stress orientation and sense of stress have been studied. However, the dependence of the degree of morphological change in γ' on stress levels, temperature, and time has not been experimentally examined except for theoretical discussions in the Ni-Al single crystal (3). Conversely, if these relations were known, there may be the possibility that the stress and/or temperature of actual parts such as turbine airfoils could be estimated by observation of microstructures in significant locations.

Therefore, the present study was carried out to obtain the quantitative relation between the degree of rafted γ' and stress-temperature-time using directionally solidified IN-713C exposed to tensile stress annealing tests.

Procedure

Directionally solidified IN-713C was produced in the form of 60 mm diameter bars using the withdrawal technique. The withdrawal rate was 20,000 cm/hr. X-ray diffraction measurements indicated that the dispersion of <100> oriented columnar grains was confined to within 10 degrees of the axis of the bar ingot. The alloy composition of IN-713C used in this study was as follows: Ni-13.5Cr-6.0Al-0.75Ti-2.0Nb-4.3Mo-0.12C in weight percent. Stress annealing test specimens were machined to the shape as shown in Figure 1. The specimens were designed to have three diameter gauge sections in order to obtain three stress levels at the same time in a single test specimen. The macrostructure of the test specimen shows that columnar grains are almost parallel to the axis of the specimen.

Fig. 1 Macrostructure of stress annealing test specimen of directionally solidified IN-713C.

All specimens were given a solution treatment at 11770°C at 2 hours in vacuum atmosphere in accordance with standard conditions. Stress annealing tests were conducted in air using conventional creep rupture
testing techniques at temperatures of 800, 850, 900, 950, and 1000°C, at a stress range from 2.9 to 39.3 MPa, and for annealing times of 24, 70, 100, and 192 hours. After stress annealing, specimens were cross sectioned in three areas having different gauge diameters to observe the microstructures of planes parallel to the specimen axis by scanning electron microscope.

**Experimental Results**

Typical microstructures of specimens stress annealed at 800°C for 200 hours and at 900°C at 18 MPa are shown in Figure 2 and Figure 3, respectively. In both figures, the tensile stress axes are vertical in the photograph views. As can be seen, morphological changes of γ' are observed in the manner in which rafted γ' grows in an orientation perpendicular to the applied stress. These results are equivalent to previous results for single crystals such as U-700 (2) and Ni-13Al-9Mo-2Ta (4) but are contrary to the results for Ni-Al (3).

![Fig. 2 γ' morphological changes in IN-713C at 800°C for 200 hours at three stress levels of a) 220, b) 300 and c) 390 MPa. The tensile stress axes are vertical in the photograph views.](image)
The plate-like coarsening of γ' is more marked at higher applied stress (Figure 2) and for more prolonged exposure (Figure 3). Also, in comparing the micrographs in Figure 2 with Figure 3, it appears that the degrees of flatness of rafted γ' are more pronounced at higher temperatures.

The degree in flatness of rafted γ' can be expressed by the aspect ratio, c/a, where c and a are the average length of γ' in a parallel and perpendicular direction to the applied stress, respectively. The c/a ratios were measured for all tested specimens to establish quantitatively the morphological changes of γ' under the various stress annealing conditions. The measurements were made on photographs of secondary electron micrographs using the point counting method. This method is described as follows. The micrographs, whose magnification are about 7,000 times, were further enlarged about 2 times by copy duplication. On a square of 50 mm x 50 mm in the enlarged photograph, two directional straight lines parallel and vertical to the applied stress were drawn in space of 4 mm respectively. Then, the numbers of points of intersections between the lines and perimeters of γ' were counted for each direction. In this way, c/a could be simply obtained from the ratio of two sums of
counting numbers for each direction. While it is recognized that accuracy will increase with a decrease in line spacing, the above line spacing was considered sufficient to achieve the accuracy required in this study. The aspect ratio measurement results are shown in Figure 4 and Figure 5. The changes in c/a with annealing time at 900°C at three stress levels are shown in Figure 4. As can be seen, c/a values decrease with annealing time under constant applied stress, but attain approximate equilibrium in about 100 hours. Both equilibrium and transient values of c/a are smaller for higher stress levels. The results at 800°C and 1000°C are shown in Figure 5. At 800°C, although c/a values decrease with annealing time, equilibrium was not attained even after 200 hours. On the other hand, at 1000°C, ten hours was required to attain equilibrium for all stress levels. At both temperatures, c/a values are also smaller for higher stress levels at every annealing time.

Fig. 4 Changes in aspect ratio of 𝛽 in IN-713C with annealing time at 900°C.
Discussion

The experimental results obtained in this study indicate that the degree of the shape changes of γ' could be determined as a function of stress, temperature, and time under the conditions in which the present investigation was carried out. In order to clarify this, a map was constructed relating aspect ratio, c/a, of γ' to applied stress and temperature at constant exposure time, using the results obtained above. As a typical case, the map for 100 hours of exposure time is shown in Figure 6. As can be seen, contour lines for c/a can be approximately drawn through the plotted points of actual measurements. Conversely, using this map, it is suggested that stress or temperature in actual high temperature performance parts such as turbine airfoils can be estimated by the measurement of γ' aspect ratio in a certain performance time, if either stress or temperature are already known.
Fig. 6 The map of aspect ratio, $c/a$, of $\gamma'$ for stress and temperature in IN-713C in the case of 100 hours exposure.
There is another case regarding application of γ' aspect ratio to estimate stress in alloys. This example is shown in Figure 7. The photograph on the left exhibits γ' structure in IN-713C under the applied stress of 20.5 MPa at 850°C for 100 hours. As can be seen, the degree of γ' rafting is different around the MC carbide. This indicates that stress around a carbide can vary relative to location. Calculations were made of the local stress distribution around the carbide using the map displayed in Figure 6. The results are shown on the right in Figure 7 by dividing the regions around the carbide into eight portions. These estimated local stresses in each section vary from about 25 MPa to about 15 MPa. It was observed that local stress maxima were generated on either side of the carbide, while stress minima were generated above and below the carbide. On the other hand, the diagonal sections around the carbide exhibited stresses approximately equivalent to the applied stress (20.5 MPa) and are considered to be an average of maximum and minimum values in local stress. This suggests that these regions are not affected by the existence of the carbide.

In this way, if the map of γ' aspect ratio is known, it is considered possible to estimate not only the homogeneous stress distribution, but also local stress concentration around inclusions (e.g., carbides) by observations of the microstructures in alloys.

The directionally solidified specimens of IN-713C used in the present investigation consisted of columnar grains solidified to an approximate <100> orientation. This suggests that the morphological behavior of γ' in this study may not be essentially different from single crystal alloys. Accordingly, the technique developed in this study can be generally applied to both single crystals and directionally solidified alloys in the case where uniaxial stress is imposed on alloys in the <100> orientation during elevated temperature performance.

Conclusion

We have shown that the morphological changes of γ' in directionally solidified IN-713C were experimentally determined as function of stress, temperature, and time by the application of a <100> oriented uniaxial tensile stress annealing test, where the morphological changes were quantitatively evaluated by the measurement of γ' aspect ratio. These results suggest that it may be possible to estimate the stress distribution in actual parts such as turbine airfoils during elevated temperature performance.

It is suggested that the technique developed in this study can be generally applied to other single crystal and directionally solidified nickel-base superalloys.
Scanning electron micrograph
The tensile stress axis is vertical in the photograph view.

Estimated local stress values divided around a MC carbide.

Fig. 7 Estimation of local stress distribution around MC carbide in IN-713C under stress of 205 MPa at 850°C for 100 hours using the map of aspect ratio
References

1. J. Ohe and S. Wakita: To be published.