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Creep properties of a new Re free single crystal Ni-based superalloy, NKH71

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Abstract. The creep of the newly developed Ni-based, Re free, single crystal superalloy NKH71 was examined at 1273 K. The creep properties were compared to those of CMSX-4, a second-generation commercial superalloy containing 3%Re. When the (applied) stress was higher than 160 MPa, the rupture lives of the present alloy were shorter than those of CMSX-4. However, when the stress was below 160 MPa, the rupture lives of the present alloy became longer than those of CMSX-4. It was noted that this lower stress level was more important when Ni-based superalloys were used for engineering land-based gas turbines. TEM and SEM observations showed that NKH71 formed a rafted structure in the early stage of creep. This was because a large number of dislocations existed in the γ/γ' interfaces before creep. In contrast, CMSX-4 had very few dislocations before creep. This observation explained why NKH71 was inferior to CMSX-4 under high stress. However under lower stress, the γ/γ' rafted structure changed to a more complex morphology in CMSX-4, while that of NKH71 tended to remain unchanged. This conservation of the rafted structure lead to the good creep resistance of NKH71 under a lower stress. In brief, the new alloy was shown to have good creep resistance even without Re element.

1. Introduction

Recently, turbine inlet gas temperature of land-based gas turbines have increased to improve thermal efficiency and to reduce SO_x, NO_x and CO_2 emissions [1]. Ni-based superalloys can be used for this purpose. The service conditions for this application are less severe than for aero applications: e.g. lower stress. Re-containing Ni superalloys, have been used for aero turbine blades, since these alloys showed improved creep resistance. However, undesirable topologically close packed (TCP) phases are formed at higher temperatures in this kind of alloy [2–4]. Moreover, Re is an expensive element. Thus, we aimed to eliminate Re from Ni-based superalloys. The present alloy, NKH71, was produced for this purpose [5]. Indeed, this alloy shows good creep resistance, equal to or better than conventional second-generation Re-containing alloys such as CMSX-4. The present paper reports creep properties of NKH71 at 1273 K in comparison with CMSX-4.

2. Experimental procedure

Single crystals of a newly developed Ni-based superalloy, NKH71, were cast as cylindrical bars of 13 mm in diameter and having an orientation within 5 degrees from [001]. Chemical compositions of the alloy are given in Table 1 with those of CMSX-4 which contained Re and was used for comparison. The bars were solution treated at 1573 K for 2.88×10^4 s and double-aged at 1373 K for 1.44×10^4 s and at 1144 K for 7.2×10^4 s. Specimens for tensile creep having a gauge diameter of 6 mm and length of 30 mm were machined from the bars. Creep tests were conducted at 1273 K under the stress of 100–400 MPa. The creep strain was recorded using linear variable differential transformers attached to the gauge section. Some creep tests were interrupted in order to see structural changes, using 8 mm diameter and 50 mm length specimens. A field emission scanning microscope (FE-SEM) and a transmission electron microscope (TEM) were used to see these changes. The observed planes were (100) and (001). Samples for SEM observation were metallographically polished and electro-etched using a saturated oxalic acid solution. Discs of 3 mm diameter were spark-cut from creep interrupted specimens and twin-jet polished for TEM observation using a mixture of perchloric and ethanol. The dislocation density was measured on 200 μm^2 from TEM images.

Table 1. Chemical compositions of NKH71 and CMSX-4 (wt/%).

	Ti	Cr	Co	Mo	Ta	W	Re	Al	Hf	Ni
NKH71	1.2	12.2	—	0.5	5.8	7.8	—	5.0	—	Bal.
CMSX-4	0.9	6.4	9.3	0.6	6.2	6.2	2.8	5.5	0.1	Bal.

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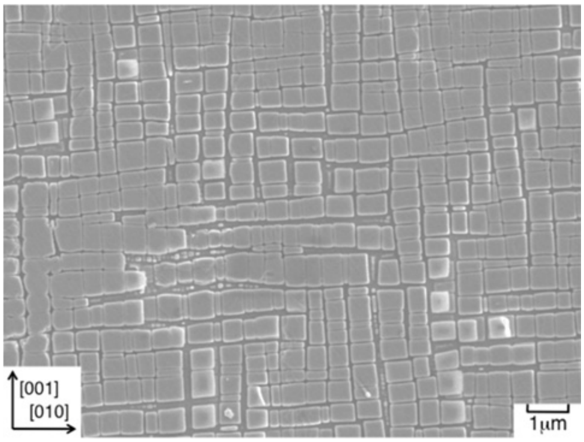

Figure 1. Scanning electron micrograph of the as-heat treated NKH71.

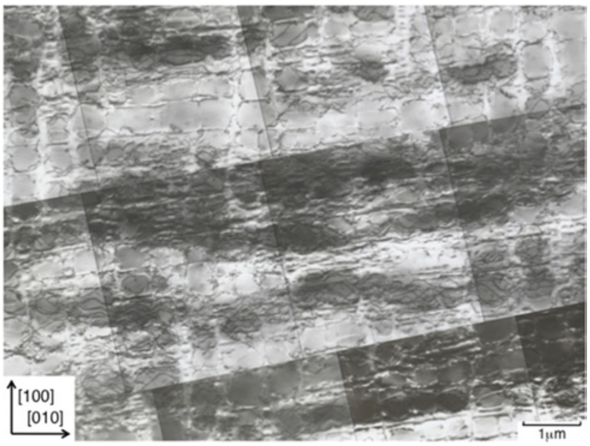

Figure 2. Transmission electron micrograph of the as-heat treated NKH71.

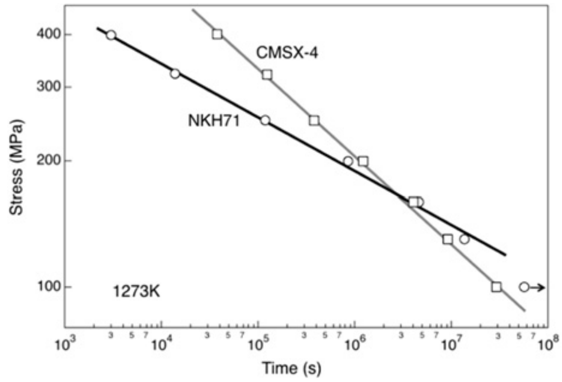

Figure 3. Stress-time to rupture curves of NKH71 and CMSX-4 at 1273 K.

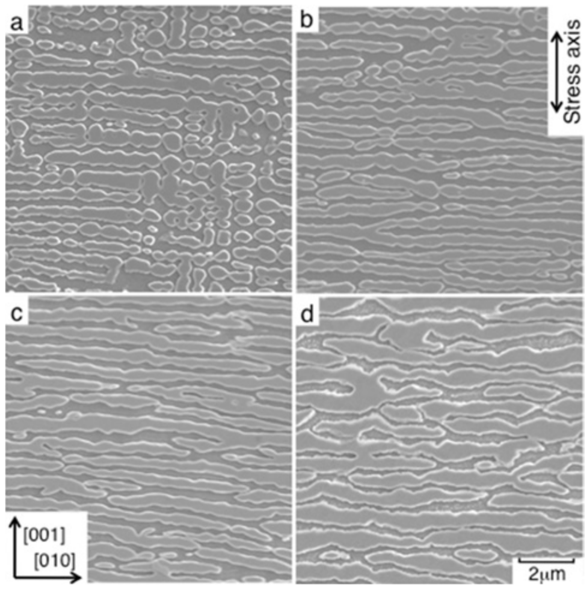

Figure 4. Scanning electron micrograph of the creep interrupted ((a) 3.60×10^4, (b) 3.60×10^5, (c) 3.24×10^6 s) and (d) ruptured specimens of NKH71 at 1273 K and 160 MPa.

3. Experimental result

3.1. The microstructure of as-heat treated NKH71

A scanning electron micrograph is shown in Fig. 1. Cuboidal γ' precipitates are regularly arrayed. The average edge length of the γ' precipitates is 0.5 μm and the width of average γ channels is 0.1 μm. The volume fraction of γ' is estimated as 78%. No eutectic γ' exists.

A transmission electron micrograph (TEM) is shown in Fig. 2. A large number of dislocations are observed in the γ/γ' interfaces even before creep deformation. Of course, no stacking faults exist in the cuboidal γ' precipitates.

3.2. Stress-time to rupture curves

The stress-time rupture curves at 1273 K are shown in Fig. 3, which also includes that of CMSX-4 for comparison. When the stress is over 200 MPa, the creep rupture lives of NKH71 are shorter than those of CMSX-4. However, when the stress is less than 160 MPa, NKH71 is stronger in creep rupture lives.

3.3. Morphological change of γ' precipitates with creep

Scanning electron micrographs of NKH71 crept under the stress of 160 MPa are shown in Fig. 4. When crept for 3.60×10^4 s, some cuboidal γ' precipitates begin to connect with each other in the direction perpendicular to the stress axis (Fig. 4a). The rafted γ/γ' structures perpendicular to the stress axis are observed when crept for 3.60×10^5 s (Fig. 4b). When crept for 3.24×10^6 s, the fully rafted γ/γ' structure is observed and the linear γ/γ' interfaces are clearly seen (Fig. 4c). This corresponds to the occurrence of the minimum creep rate. When creep ruptured, the γ' precipitates have begun to merge even in the vertical direction parallel to the stress axis (Fig. 4d).

The morphological change is presented quantitatively in Fig. 5, where the aspect ratio, the ratio of the length over the width of γ' precipitates, is plotted against the creep time for NKH71 and CMSX-4 [6]. Here, the results for 160 and 250 MPa are shown. Before creep, the aspect

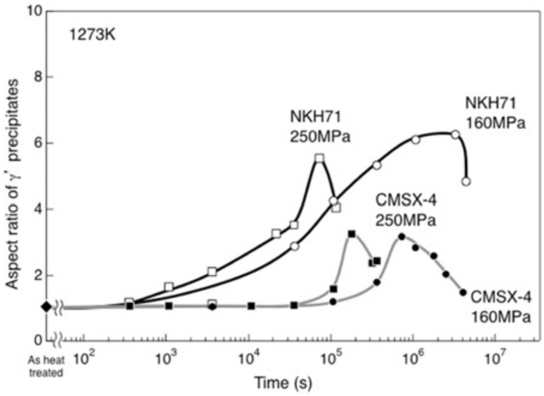

Figure 5. Changes in the aspect ratio of the γ' precipitates of NKH71 and CMSX-4 crept at 160 and 250 MPa with creep time.

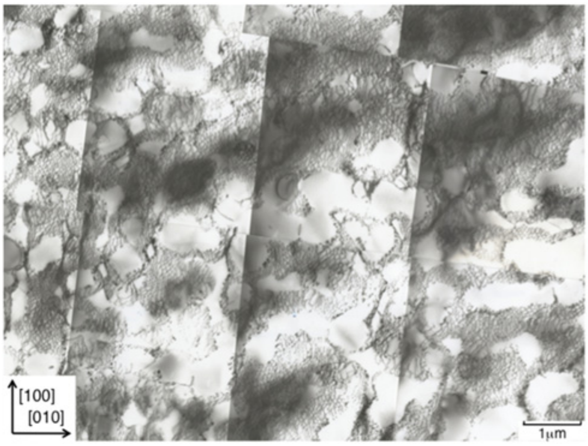

Figure 6. Transmission electron micrograph of interrupted creep specimen for 1.08×10^5 s at 1273 K and 160 MPa.

ratio is one. As the creep progressed, the aspect ratio increases with time, attains the maximum value and then decreases. The maximum aspect ratio is attained at a shorter time for 250 MPa creep than for 160 MPa creep. However, the maximum aspect ratio of 160 MPa creep is larger. On the other hand, the aspect ratio of CMSX-4 does not change until the creep time of 1.0×10^5 s. Then, the ratio increases and again decreases after attaining a maximum. The ratio of NKH71 is higher than that of CMSX-4 for all the creep times. In brief, the γ/γ' rafted structure is formed earlier in NKH71 than in CMSX-4. Also NKH71 keeps the γ/γ' rafted structure until rupture.

3.4. Dislocation structure at γ/γ' interfaces

Using TEM foils, parallel to (103), a transmission electron micrograph of the interrupted creep specimen of NKH71 for 1.08×10^5 s at 1273 K and 160 MPa is shown in Fig. 6, where the incident beam direction, B, is closed to [001]. A large number of dislocations are entangled with each other, existed at the γ/γ' interfaces in NKH71 even in the as-heat treated state. Few dislocations are observed in CMSX-4 before creep. No stacking faults are observed.

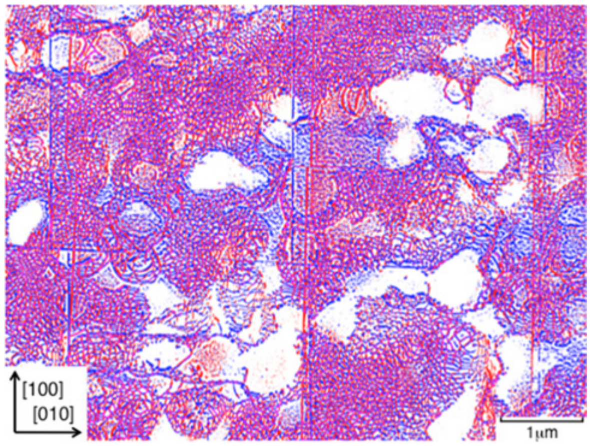

Figure 7. Superimposed image of transmission electron micrograph where B = [001], g = [020] and [200], on the same region at the γ/γ' interfaces, by using the interrupted creep specimen for 1.08×10^5 s at 1273 K, 1600 MPa. (g = [020] : red, [200] : blue, [020]+[200] : purple).

3.5. Quantitative examination of dislocations

The dislocation density at the γ/γ' interfaces is regarded as the quantitative parameter showing the change in the dislocation substructures at the γ/γ' interfaces with creep time. In order to make the quantitative measurement of the dislocation density, it must be understood that there are two invisibility conditions on the basis of the $g \cdot b = 0$ invisibility criterion. For example, when the TEM observations are performed with the incident beam directions, B = [001], and the g-vector, g = [020], dislocations with Burgers vectors of [101] and [10-1] are invisible, but are visible with B = [001], g = [200]. Hence the dislocation density cannot be accurately estimated by using only one g-vector. The TEM observations should be carried out with two different g-vectors on the same region and the images from each g-vector should be superimposed on the other to express the accurate dislocation substructures at the γ/γ' interfaces [7].

Using the invisibility condition of $g \cdot b = 0$ and two types of g-vector, the dislocations in the γ/γ' interfaces are characterized and their density measured. An example of a TEM image used for this analysis is shown in Fig. 7 for a specimen crept for 1.08×10^5 s under the stress of 160 MPa. The accurate expression of all dislocations at the γ/γ' interfaces can be attained by the TEM observations with the two different g-vectors on the same area.

The change in the dislocation density, thus determined, at the γ/γ' interfaces with creep time at 160 MPa is shown, in Fig. 8. Naturally, the density increases with creep time. Before creep, the density is $1.0 \times 10^{13}\ m^{-2}$ and it increases to $3.0 \times 10^{13}\ m^{-2}$ for the creep time of 3.6×10^4 s. This density stays the same until rupture.

The change in the dislocation density at the γ/γ' interfaces with creep time at 250 MPa is shown in Fig. 9. The dislocation density at the γ/γ' interfaces increases with increasing creep time until rupture. From these results, the change in the dislocation density at the γ/γ' interfaces with creep time at lower stress is different from that at higher stress.

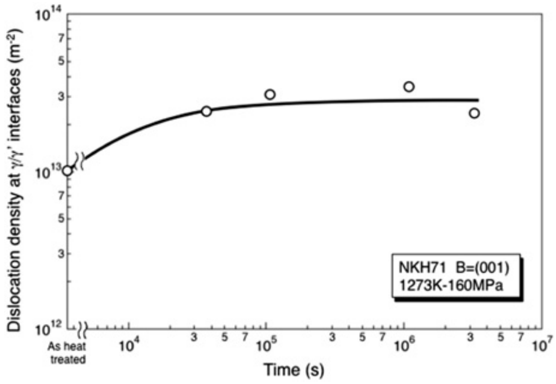

Figure 8. The change in the dislocation density at the γ/γ' interfaces with creep time at 1273 K and 160 MPa.

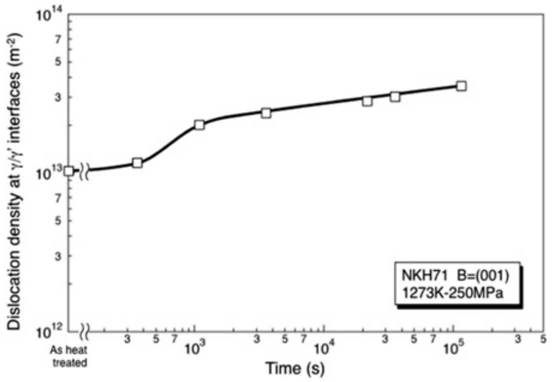

Figure 9. The change in the dislocation density at the γ/γ' interfaces with creep time at 1273 K and 250 MPa.

4. Discussion

As shown above, γ' precipitates show a rafted morphology earlier in NKH71 than in CMSX-4. This rafted γ/γ' structure is kept until rupture. The creep life of NKH71 is shorter than CMSX-4, when the stress is higher than 200 MPa. However, when the stress is below 160 MPa, the creep life of NKH71 is longer than that of CMSX-4. This result has been understood in terms of the dislocation structure in the γ/γ' interfaces, as described below.

The change in aspect ratios of the γ' precipitates of NKH71 and CMSX-4 with creep time under the stresses of 160 and 250 MPa are shown in Fig. 10. The ratio of CMSX-4 at 160 and 250 MPa increases with time and after reaching 3, the ratio decreases. The ratio of NKH71 at 160 MPa increases with time in the beginning and stays constant until rupture after attaining maximum of 6 until the 80% of its creep life. That is, the rafted structure of NKH71 under a stress of 160 MPa is stable for most of the creep life. On the other hand, the ratio of NKH71 under the stress of 250 MPa increases with time. After reaching 5 at 65% of creep life, the ratio decreases. That is, the rafted structure of NKH71 under 160 MPa is more stable than that under 250 MPa.

The dislocation density of NKH71 is plotted against the normalized creep time in Fig. 11. The density that increases rapidly up to the creep time of 3% of creep life is three times larger than the density before creep, but stays unchanged until rupture. When the stress is 250 MPa, the maximum value of the density is twice larger than before creep and increases further up to rupture. These observations indicate that the dislocation density unchanging causes the stable rafted structure under 160 MPa during creep.

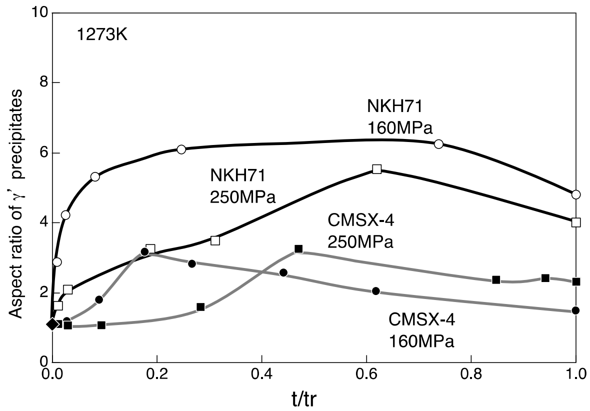

Figure 10. The change in the aspect ratios of the γ' precipitates of NKH71 and CMSX-4 crept at 160 and 250 MPa with normalized creep time (ratio of time to rupture).

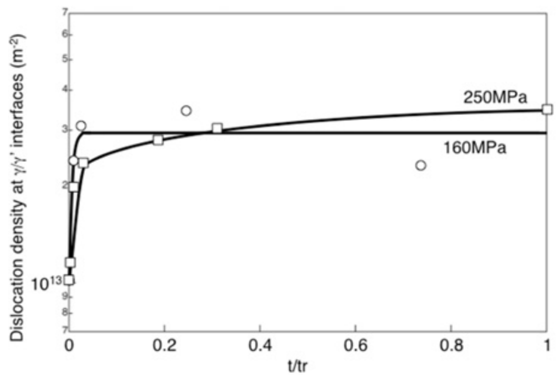

Figure 11. The change in the dislocation density at the γ/γ' interfaces of NKH71 crept at 160 and 250 MPa with normalized creep time.

It is well known that the creep resistance of Ni superalloys depends directly on the γ channel width [7,8]. When the stress is higher than 200 MPa, the γ/γ' rafted structure is formed earlier in NKH71 than in CMSX-4. This is due to a large number of dislocations existing before creep, the number larger than that in CMSX-4. This large number of dislocations makes the width of γ channels larger and, thus, the dislocations move more readily in NKH71. This accounts for the observation that NKH71 is weaker than CMSX-4 when the stress is large. However, when the stress is smaller, the rafted structure of NKH71 is more stable during creep. As a result, NKH71 is stronger than CMSX-4 when the stress is small.

5. Conclusion

The creep of a newly developed Re-free Ni-based superalloy, NKH71, is studied at 1273K under the stress of 100 to 400 MPa with SEM and TEM observations, in comparison with that of CMSX-4. The following conclusions are reached.

1. When the applied stress is smaller than 160 MPa, the creep life of NKH71 is longer than that of CMSX-4.
2. This result is correlated with a built-in dislocation density before creep. A large number of dislocations exist at the γ/γ' interfaces in NKH71 than in CMSX-4.
3. Further, the rafted structure, produced by creep deformation, is more stable in NKH71 than in CMSX-4 at lower stress.
4. The new alloy, NKH71, is shown to have good creep resistance even without the element Re.

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