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CREEP STRENGTH OF Ni-BASE SINGLE-CRYSTAL SUPERALLOYS ON THE γ/γ' TIE-LINE

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Abstract

The creep strength of Ni-base single-crystal superalloys with various γ' fractions was investigated at 900˚C, 392MPa and 1100˚C, 137MPa. Creep strength of two-phase alloys was superior to single phase alloys. It was shown that the optimum γ' volume fraction depends on the temperature. The creep rupture lives were the longest for the 70% γ' alloys at 900˚C, but they were obtained at about 55% practical γ' fraction at 1100˚C. Changes in the microstructure after creep rupture were studied. In ruptured specimens, a raft structure perpendicular to the stress axis was observed in the 40~60% γ' alloys. On the other hand, parallel rafts were observed in the 80% γ' alloy. This parallel raft structure could be explained by the change in the relative role between the matrix and the dispersed phase.

Introduction

It was first reported by two of the authors[1, 2] that the creep rupture life was the longest in the vicinity of 65% γ' under any creep condition in the polycrystalline Ni-base superalloy Inconel713C. This tendency was expected to be observed in single-crystal superalloys, and, in fact, in the last two decades, many studies had been carried out at around 60~70% γ' fraction to develop Ni-base superalloys. However, systematic studies have not been reported about the effect of γ' fraction for the strength of single-crystal superalloys. Generally, when the composition of each phase varies, it is difficult to evaluate the effect of the changes in the γ' fraction alone because the strength of each phase and the lattice misfit should also be influenced. Therefore, as tie-line single-crystal superalloys contain various γ' volume fractions, with each phase having the same composition, an investigation using these alloys was believed to produce results of great importance.

It is well known that the directional coarsening of γ', which is called rafting, takes place in the creep of Ni-base superalloys at high temperatures. Some observations imply that rafting reduces the creep resistance [3~5] at temperatures below 1000˚C and at higher stress. Therefore, modern Ni-base single-crystal superalloys were developed with the addition of Re, because it delays rafting despite the increased tendency to form topologically close-packed (TCP) phases. Currently, higher temperatures are required in practical applications of Ni-base superalloys, and it is difficult to prevent rafting above 1000˚C. However, it is possible to extend the creep rupture life with the formation of a stable raft structure[6]. From this point of view, the fourth generation superalloys[7, 8] were developed recently. The stable raft structure can be obtained by a large lattice misfit that produces a dense dislocation network on the γ/γ' interface. In this study, two creep conditions at 900˚C and 1100˚C were selected to investigate the effect of the γ' volume fraction in different creep behavior.

Experimental Procedures

Figure 1 is a schematic drawing of a pseudo-binary phase diagram of a (Ni, X)-(Al, Y) system. The points A and B are on the boundary line of γ-(γ+γ') and (γ+γ')-γ' at 900˚C, respectively. When the compositions of A and B are given as X_i and X_i', respectively, the composition (C_i) of a Ni-base superalloy for a given γ' fraction (f) is obtained by Eq. (1), which is the equation of a tie line representing the compositions of γ and γ' in an equilibrium state.

$$C_i = (1 - f)\, X_i + f\, X_i' \qquad (i : \text{Ni, Al, etc...}) \qquad (1)$$

The composition of each phase is maintained in the same way as in superalloys along the γ/γ' tie line. Since the strength of each phase and the lattice misfit between γ and γ' do not change, only the effect of a γ' volume fraction on the creep behavior can be discussed.

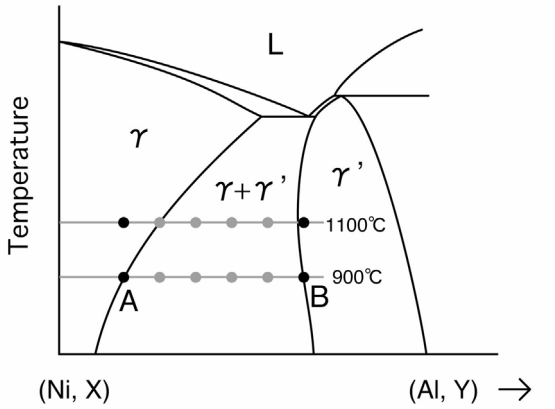

Figure 1. Schematic drawing of a pseudo-binary phase diagram for (Ni,X)-(Al,Y) system.

The equilibrium compositions of the γ and γ' phases in the single-crystal superalloy TMS-75[9] and TMS-82+[10] are

calculated by an alloy design program (ADP) developed at NIMS[11], as shown in Table 1. ADP can estimate not only the equilibrium composition of each phase and volume fraction but also the lattice misfit, specific gravity, solution index (S.I.) value, creep rupture life, and corrosion resistance. TMS-75 and 82+ are third- and second-generation superalloys, respectively. In comparisons with TMS-75, TMS-82+ contains less Re, but the creep rupture life of TMS-82+ at 1100˚C is improved by increasing the lattice misfit.

First, the γ and γ' single-phase alloys were cast as master alloys. These alloys were mixed to make a series of single-crystal superalloys, which had been designed to contain various volume fractions of the γ' phase at 900˚C. The γ' fraction in each alloy will decrease with increasing temperature above 900˚C, and, for consistency, each alloy is henceforth referred to in terms of the originally designed γ' volume fraction values, e.g., 20% γ'. The single crystals were produced in a directional solidification furnace at NIMS in the form of bars with a diameter of 10 mm. The longitudinal direction is near [001] in the 0~80% γ' alloys; however, it is near [111] in the 100% γ' alloys, according to the preferred growth direction of the primary crystal. Solution and two-step aging heat treatment were carried out on each alloy under optimum conditions for standard TMS-75, which contains a 63% γ' phase, as follows:

$$1300^{\circ}C/1h + 1320^{\circ}C/5h \rightarrow GFC(\text{Gas Fan Cool}) \rightarrow 1120^{\circ}C/5h \rightarrow GFC \rightarrow 870^{\circ}C/20h \rightarrow GFC$$

Constant-load tensile creep tests were performed at 900˚C, 392MPa and 1100˚C, 137MPa. After heat treatment and creep rupture, SEM studies were carried out.

Microstructure after Heat Treatment

Figure 2 shows the heat-treated microstructure of a series of TMS-82+. The 0~60% γ' alloys have a homogeneous microstructure, suggesting that the solution heat treatment was performed fully. The 0% γ' fraction alloy consists mostly of a single γ phase. In the 20% γ' alloy, fine spherical γ' precipitates were observed. The spherical shape of γ' should be derived from the relatively larger interfacial energy as compared with the elastic strain energy in the case of smaller precipitates. This microstructure suggests that the 20% γ' alloy consisted of only a γ phase during the first aging heat treatment at 1120˚C, and these fine γ' precipitates were considered to have appeared during the secondary aging treatment at 870˚C for 20 hours. In the 40% γ' alloy, the volume fraction of γ' was increased, and the shape of γ' appeared to be slightly cuboidal. The typical microstructure of a modern single-crystal Ni-base superalloy was observed in the 60% γ' alloy. The size of the cuboidal γ' precipitates was about 200~500nm.

With an increase in the volume fraction of γ', the γ-γ' eutectic should appear after the formation of the primary γ phase during solidification. However, in most of the Ni-base superalloys, the γ-γ' eutectic should transform to a γ' single phase during cooling, as drawn schematically in Figure 1. Therefore, this γ' single phase is called eutectic γ'. A very small number of eutectic γ' phases were observed in interdendritic parts of 70% γ' alloy, and eutectic γ' increased as the increase of γ' fraction.

Figure 3 represents the microstructure in an alloy of the TMS-75 series, which was designed to contain an 80% γ' phase after heat treatment. Three kinds of microstructures were observed in different brightness. One microstructure consists of cuboidal γ' and narrow γ channels, as in the 60% γ' alloy. On the other hand, the γ/γ' structure is coarser, resulting in a visually matrix-like γ' phase. The wavy γ/γ' interfaces suggest that the interfaces are in a semi-coherent state. In the interdendritic region, eutectic γ' was observed. The existence of a large amount of eutectic γ' indicates that this alloy cannot be completely solution-treated under the conditions applied in this study.

There was no γ phase in the 100% γ' alloy as shown in Figure 2; however, round and plate-like precipitates were observed in the γ' matrix. In particular, the interdendritic regions were densely populated with round precipitates. Generally, precipitates, called topologically close-packed (TCP) phases, are formed in long annealed Ni-base superalloys, particularly in the cases of Re-added alloys. The phases, σ, P, μ, and R are reported as TCP phases with different crystallographic structures[12, 13]. In this study, round-shaped and plate-like precipitates are expected to be TCP phases. The amount of these precipitates in 100% γ' alloy was only 2%; thus the creep strength of this alloy should not be significant influenced. In the heat-treated samples of the TMS-75 series, similar SEM photographs were obtained [14].

Creep Behavior

Figure 4 shows the resultant variation of the creep rupture life at 900˚C, 392MPa and 1100˚C, 137MPa against the designed amount of γ'. Some low γ' content alloys ruptured instantaneously when the load was applied. This instant rupture means that the yield stress and ultimate tensile stress of these alloys were same or smaller than the creep stress under each condition. In these cases, points were not plotted on the graph. Under both conditions, 0% γ' alloys were weaker than 100% γ' alloys. The longest rupture life was obtained with the 70% γ' and 63% γ' alloys at 900˚C and 1100˚C, respectively, but it should be noted that the γ' fraction at 1100˚C should have been smaller than the designed γ' fraction. When the γ' fraction of TMS-75 at 900˚C is 63%, it is calculated by ADP that the practical γ' fraction decreases to 47% at 1100˚C.

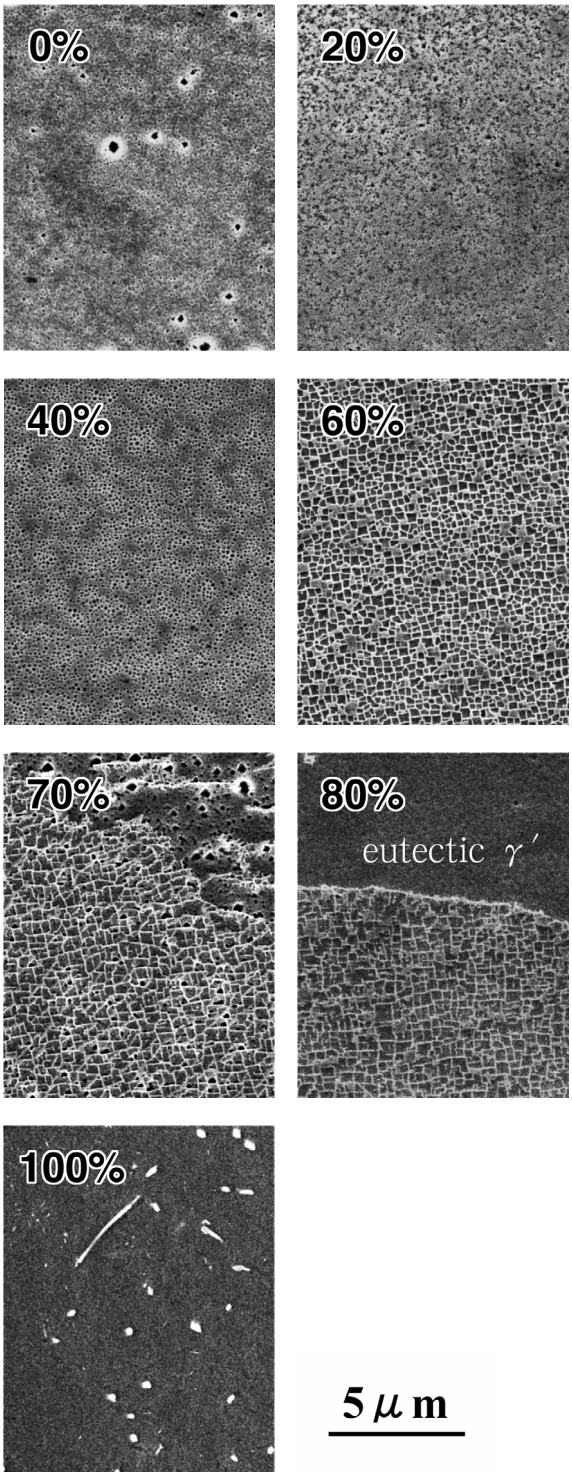

Figure 2. Microstructures of a heat-treated series of TMS-82+.

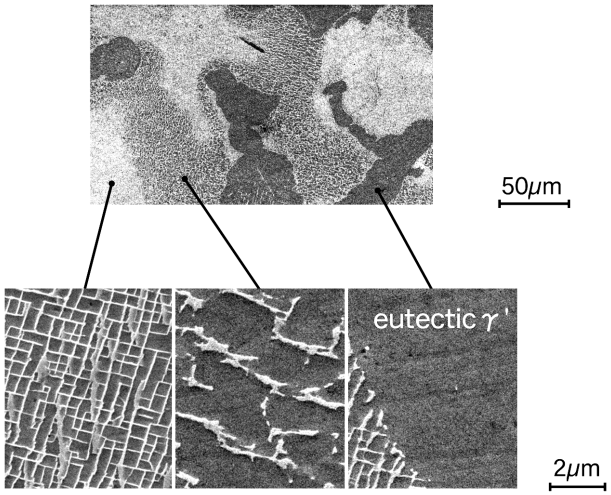

Figure 3. Microstructure of the heat-treated 80% γ' alloy in the series of TMS-75.

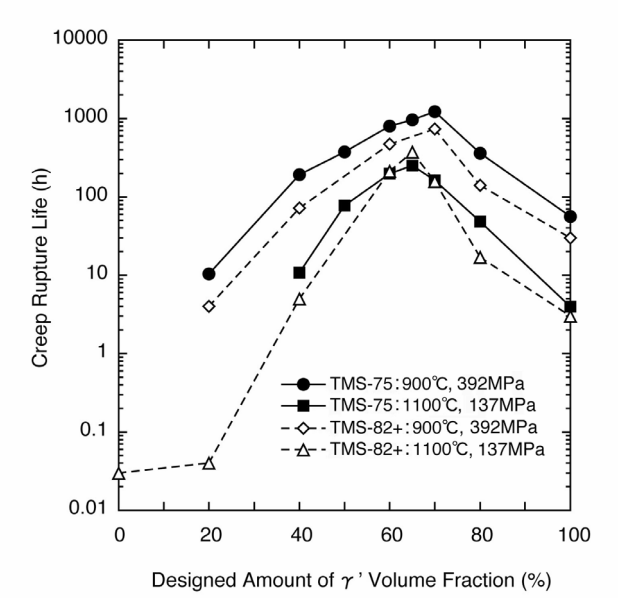

Figure 4. Relationship between the designed amount of γ' volume fraction and creep rupture life.

The volume fraction of γ' in each alloy was investigated by a point-count method after heat treatment at 900˚C and 1100˚C for 4 hours and a subsequent water quench, as shown in Figure 5. In both series of TMS-75 and 82+ quenched from 900˚C, the measured volume fraction shows good accordance with the designed amount of γ'. In cases of alloys quenched from 1100˚C, the volume fraction of γ' decreases. The 0% and 20% alloys contain almost no γ' phase at 1100˚C. However, the decrease in γ' was small in 60~80% alloys, and the microstructure in 100% alloys did not show a difference. These experimental facts mean that the γ single-phase area in the pseudo-binary phase diagram expands as the temperature increases, but the boundary line between the γ' single-phase area and the γ+γ' two-phase area does not change significantly, as shown in Figure 1.

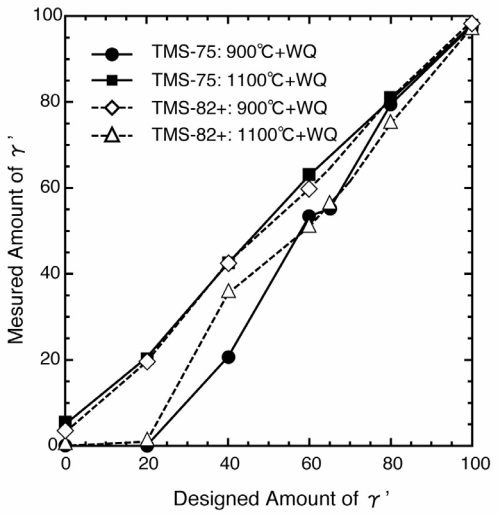

Figure 5. Relationship between designed amount of γ' and measured amount of γ' in each alloy quenched from 900˚C and 1100˚C.

Figure 6 represents the relationship of the creep rupture life and measured volume fraction of γ' in each series of a superalloy. The amounts of γ' at the longest creep rupture life are obviously different at 900˚C and 1100˚C. The longest rupture life was obtained with the 70% and 55% measured γ' volume fraction at 900˚C and 1100˚C, respectively. At 900˚C, the creep strength of the TMS-75 series exceeds that of the TMS-82+ series at all of the γ' volume fractions, but the longest rupture life of TMS-82+ is more than that of TMS-75 at 1100˚C.

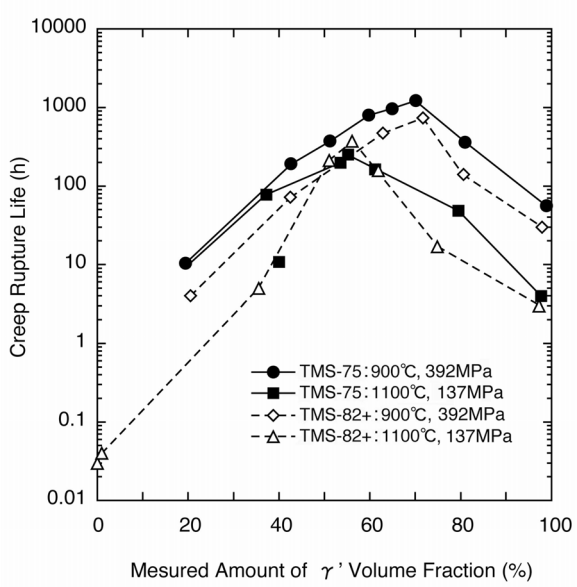

Figure 6. Relationship between the measured amount of γ' volume fraction and creep rupture life.

An instant rupture of the single γ phase alloy means that the yield stress and ultimate tensile stress of this alloy at each temperature were the same or smaller than the creep stress under each condition, namely, the viscous resistance for the dislocation motion in the γ phase was very low in spite of the large quantities of solution-hardening elements that were added. The single γ' phase alloy did not rupture instantly, but its creep rupture life was short. While the weak phases were combined, the longest life was obtained at the 50~70% γ' practical fraction under both creep conditions. In the light of these facts, it can be said that the Ni-base superalloys are strengthened by an interface rather than by dispersion strengthening.

In an early stage of creep at around 900˚C, dislocation loops expand into γ channels, leaving 60˚ dislocations on γ/γ' interfaces[15]. When the width of a γ channel decreases, the Orowan stress increases, producing a better creep strength. However, increases in the γ' volume fraction are limited for practical uses. Once the γ' fraction is beyond a limit, solution heat treatment cannot be performed fully, and eutectic γ' and coarsened γ' phases appear. In that case, the gross area of the γ/γ' interface decreases, therefore creep strength is expected to decrease.

In addition to a decrease in gross area of the interface, the coherency between the matrix and the precipitates has an influence on the mechanical properties. Generally, the creep strength of Ni-base superalloys decreases when the γ/γ' interfaces before creep are semi-coherent. The coarsened two-phase region observed in the 80% γ' alloy is considered to be in a semi-coherent state because the interfaces are rounded and wavy. This can be attributed to an increase in the creep rate because the semi-coherent interfaces act as dislocation sources. Consequently, the longest creep rupture life at 900˚C was obtained with 70% γ'.

At 1100˚C, the maximum creep strength was achieved at 55% of the measured γ' volume fraction. It is difficult to maintain the heat-treated microstructure during creep, and rafting takes place in the very early stage of creep above 1000˚C. When the creep stress is high enough, the shearing of rafts takes place easily, and creep rupture life should be very short. However, if the creep stress is not high enough, the formed raft structure is stable. Consequently, the steady creep region and creep rupture life should extend. A stable structure is considered to contain a long flat raft with a dense interfacial dislocation network, as concluded in previous studies[7, 8]. In addition to this characteristic, in this study, it was found that a raft structure has a high stability if it consists of nearly equal γ and γ' phases. It is reasonable to consider that the stability of the raft structure is affected by the volume faction of γ'. If the practical volume fraction of γ' phase is excessive, rafts of γ phase should become thinner than γ' rafts. Thinner rafts should be sheared to pieces more easily. Once the shear of rafts occurs, the γ' phase becomes similar to a matrix, and the raft structure must collapse with increasing creep rate because of decrease in gross area of the interface. In other words, the almost same amount of γ and γ' can causes stable raft structure and the longest rupture life.

The peak of the rupture lives at 1100˚C was more sharp in TMS-82+ rather than in TMS-75. It could be explained as follows. Due to larger negative misfit in TMS-82+ as compared with TMS-75[10], the driving force for rafting is larger in TMS-75. Since diffusion coefficient of Re is small, and Re is preferentially partitioned to γ[16], the mobility of interfaces in TMS-82+ should be higher than that in TMS-75. The larger driving force and the higher mobility of interface promote the formation of long flat rafts. Consequently, TMS-82+ shows a significant long rupture

life when the practical γ' volume fraction is 55%.

Microstructure after Creep Rupture

Figure 7 shows the microstructure of a series of TMS-82+ alloys after creep rupture at 900˚C, 392MPa. In comparison with the heat-treated specimens, the 0% γ' alloy did not change because of instant rupture. In a 20%γ' alloy, coarsened γ' precipitates were observed. In a 40% alloy after creep, round shaped γ' precipitate linked up to each other to form raft perpendicular to the stress axis. A wavy raft structure was observed perpendicularly to the stress axis in a 60% γ' alloy. Generally, rafting takes place gradually during creep in modern Ni-base single crystal superalloys at 900˚C. These rafts in the 60% γ' alloy must have been flat in the middle stage of creep, but they are considered to have been sheared to pieces in the late stage of creep. On the other hand, the direction of rafts was random in 70% γ' alloy, and the rafts in 80% γ' alloy was parallel to the stress axis. This anomalous raft structure was observed also in TMS-75 as described later. In a 100% γ' alloy, microstructure after creep was almost same as the heat-treated specimen.

Figure 8 shows the microstructure of TMS-82+ after creep rupture at 1100˚C, 137MPa. Coarsening occurred more significantly in the 20% γ' alloys at 1100˚C than at 900˚C. In the 40% γ' alloy, the flat rafts were formed after only 4 hours of creep. Raft structure collapsed after creep rupture in the 60~80% γ' alloys. At 1100˚C, raft structure is formed in the very early stage of creep, and long flat γ' phases are sheared gradually in the steady creep region, as reported before[10]. In the 70~80% γ' alloys, sheared rafts were parallel to the stress axis. The microstructures in 0% and 100% γ' alloys did not change significantly.

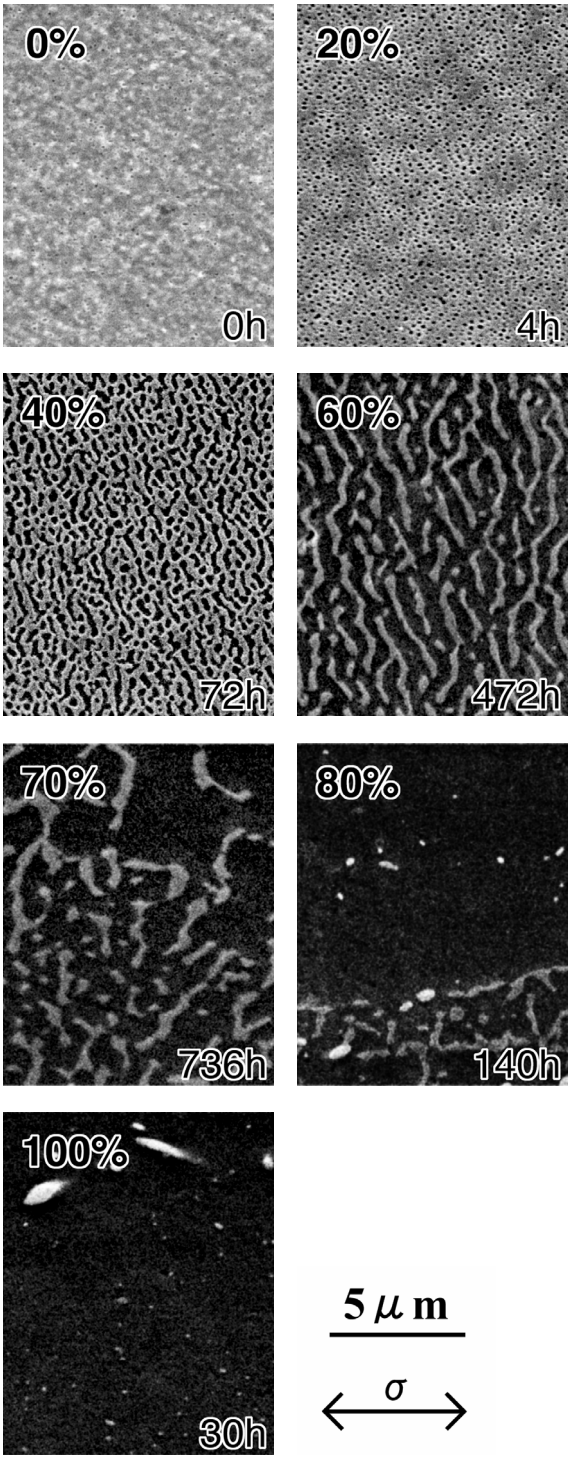

Figure 7. Microstructures of a series of TMS-82+ alloys after creep rupture at 900˚C and 392MPa. The volume fraction of γ' and rupture life are indicated in each micrograph.

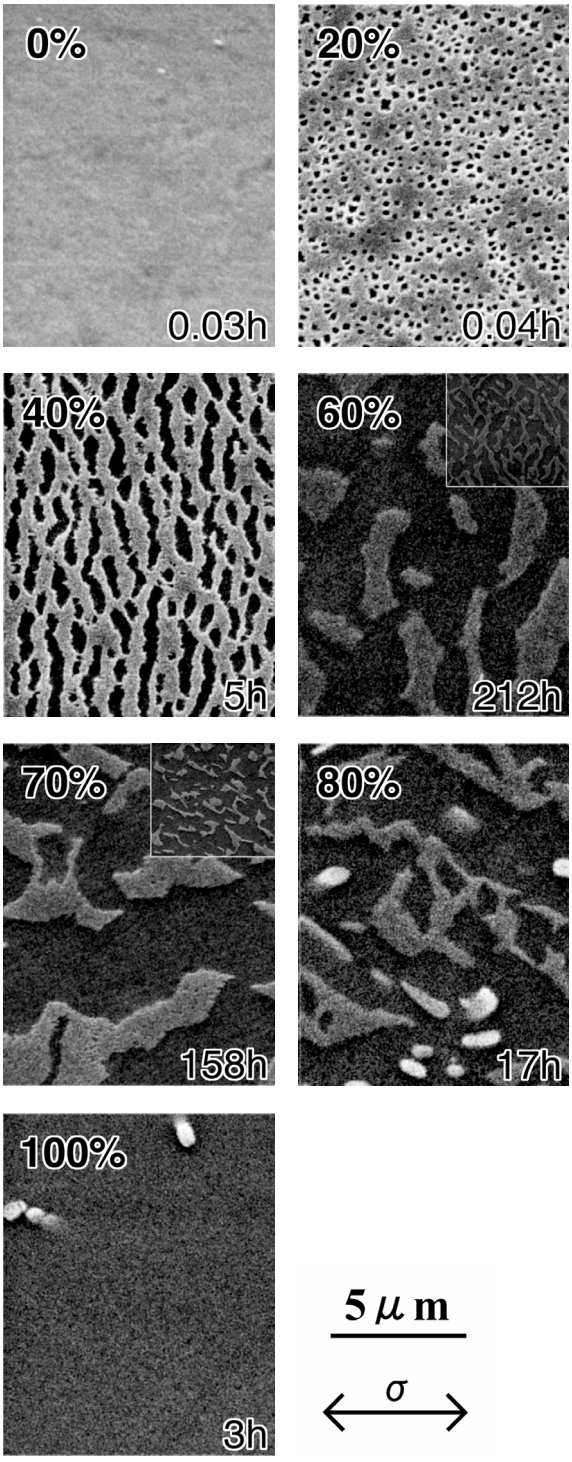

Figure 8. Microstructures of a series of TMS-82+ alloys after creep rupture at 1100˚C and 137MPa. The volume fraction of γ' and the rupture life are indicated in each micrograph.

Anomalous rafting was also found in 80% γ' alloys in a series of TMS-75. Figure 9 shows the microstructure in 80% γ' alloy of TMS-75 after creep rupture at 900˚C, 392MPa and 1100˚C, 137MPa. The γ' phases tended to align perpendicular to the stress axis in primary dendrite arms at 900˚C, 392MPa. However, fine rafts were formed parallel to the stress axis in secondary dendrite arms, and the dispersed γ phases were also arranged along the same direction. At 1100˚C, 137MPa, parallel rafts were observed in all regions except in the eutectic γ'. For the case of compression creep with a negative lattice misfit[17~19] or tensile creep with a positive lattice misfit[15, 20], the parallel raft structure had already been reported; however, these parallel rafts were observed in Ni-base superalloys that had a negative lattice misfit after a tensile test.

This anomalous raft structure can be explained by the change in the relative role between the matrix and the dispersed phase, as shown schematically in Figure 9. As is well known, if an alloy composed with a γ matrix and γ' precipitates has a negative lattice misfit, perpendicular rafts are formed. However, in an alloy containing dispersed γ phases in the γ' phase, parallel rafts can be formed because this situation is similar to the lattice misfit changing from negative to positive. In fact, dispersed γ phases in the γ' phases were observed in some parts of the heat-treated 80% γ' alloy. These parts composed with the dispersed γ in γ' phase are considered to expand into the dendrite cores during creep. When the relative role between the matrix and the dispersed phase changes before the formation of rafts, the direction of rafts should also changes. Since this change is slow in the dendrite cores of the 80% γ' alloy crept at 900˚C, both type of rafts were observed. However, at 1100˚C, this change should be fast enough, so that parallel rafts were formed.

When plastic flow can be neglected, the direction of rafting is affected also by the elastic misfit, m. A reversal in the sign of $m\delta\sigma$ changes the type of rafting (δ: lattice misfit, σ: applied stress) [6]. According to this criterion, the perpendicular rafts should be formed in the tensile creep of an alloy containing dispersed γ phases in a γ' matrix. Calculated maps for stable shapes of coherent precipitates [21, 22] also predicted that a perpendicular raft is stable under such a condition. However, it should be noted that these arguments are limited to an elastic regime. On the other hand, the direction of the raft is controlled by the sign of σδ in a plastic regime[6], so a parallel raft becomes stable, as actually observed in an 80% γ' alloy.

Summary

• The γ' volume fractions of tie-line alloys at 900˚C were in good accordance with the original designed values. The 0~60% γ' alloys had an almost homogeneous microstructure; however, there were differences in the dendritic and interdendritic regions in the 70~100% γ' alloys.

• Creep tests at 900˚C, 392MPa and 1100˚C, 137MPa were carried out. Some low γ' content alloys ruptured instantaneously. This means that the viscous resistance for the dislocation motion in the γ phase was very low.

• Creep strength of two phase alloys were superior to single phase alloys. It was shown that the optimum γ' volume fraction depends on the temperature. The creep rupture lives were the longest for the 70% γ' alloys at 900˚C, but they were obtained at about the 55% γ' fraction at 1100˚C. At 900˚C, narrower γ channels causes longer rupture life. However, the almost same amount of γ and γ'

can produce stable raft structure and the longest rupture life. It will be possible to develop new single-crystal superalloys that are tailored for different applications by controlling the γ' volume fraction accurately.

• The second-generation Ni-base single-crystal superalloy TMS-82+ was designed to form a stable raft structure at above 1000˚C by having a larger negative lattice misfit. The creep rupture life of TMS-82+ is longer than that of the third-generation superalloy TMS-75 with only 60~70% γ' at 1100˚C region, where the raft formation is effective for strengthening, while a series of TMS-82+ were weaker than TMS-75 at 900˚C.

• In a ruptured specimen, a raft structure perpendicular to the stress axis was observed in the 40~60% γ' alloys. On the other hand, parallel rafts were observed in the 80% γ' alloy. This parallel raft structure can be explained by the change in the relative role between the matrix and the dispersed phase.

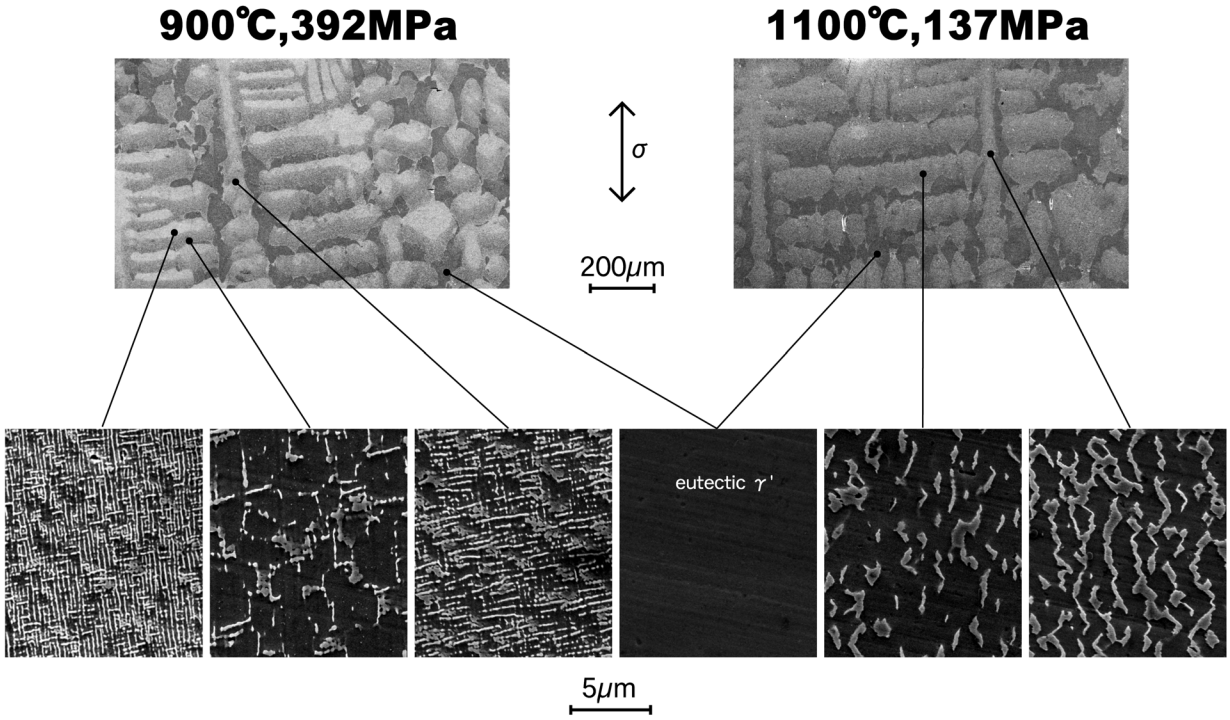

Figure 9. Raft structures perpendicular to the stress axis in 80% γ' alloy of TMS-75 after creep rupture at 900˚C and 1100˚C.

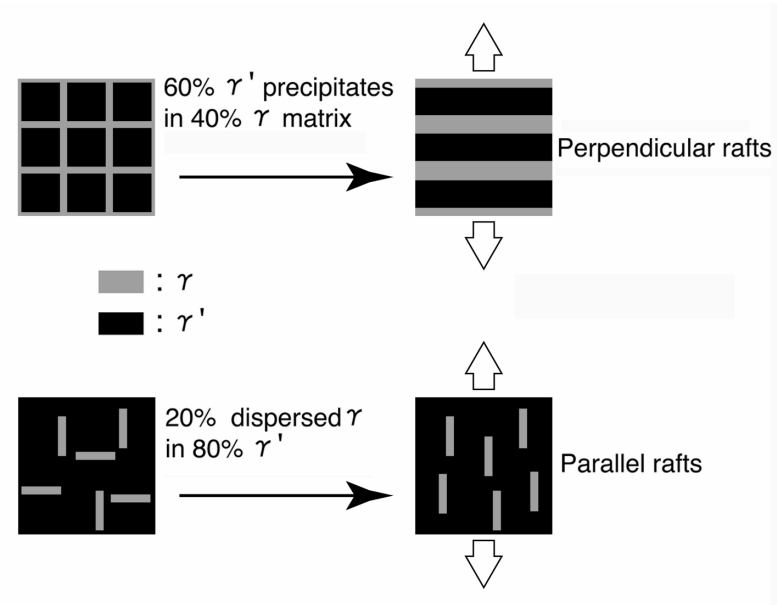

Figure 10. Schematic drawing of the formation of perpendicular and parallel raft in superalloys having negative lattice misfit.

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