The Effect of Volume Percent of Fine γ' on Creep in DS Mar-M200 + Hf

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A correlation was found to exist between creep life at 1800°F (1255 K) and the volume percent of uniformly dispersed fine (<0.5 μm) gamma prime (γ') precipitate in directionally solidified (DS) Mar-M200 + Hf turbine blade castings. A three-fold increase in creep-rupture life was realized when the amount of fine γ' increased from about the 30 pct level to approximately a 45 vol pct level. Heat treatment studies revealed that the amount of fine γ' developed in this alloy is strongly dependent on solution temperature, especially in the range from 2160 to 2210°F (1455 to 1483 K). Higher solution temperatures promote the development of greater amounts of fine γ' by more complete solutioning of the coarse γ' and eutectic γ-γ' constituents which subsequently reprecipitate as fine γ' upon cooling and aging.

DIRECTIONALLY solidified (DS) Mar-M200 + Hf* is a γ' [$Ni_3(Al, Ti)$] hardened nickel-base superalloy with excellent high temperature creep capability. The superior creep capability of DS Mar-M200 + Hf and other directionally solidified superalloys, compared to conventionally cast γ' hardened superalloys, is largely the result of the elimination of grain boundaries oriented transverse to the major stress axis.[3-5] In the case of DS Mar-M200, the ⟨100⟩ preferred cube orientation of the columnar grains may also be contributing to improved creep resistance, particularly in the 1400 to 1600°F (1033 to 1144 K) temperature range where Kear and Piearcey found a strong creep dependency on orientation in single crystal Mar-M200.[6] A third factor which affects high temperature creep resistance of DS Mar-M200 + Hf is the high volume percent of γ' in this alloy (~60 vol pct). The effectiveness of γ' strengthening is dependent not only on the volume percent but also on the size and distribution of the precipitate which is developed during solidification and heat treatment.[7] A fine uniform γ' dispersion can provide optimum creep resistance at a given volume fraction of precipitate. In a complex cast alloy such as Mar-M200, the character of the γ' precipitate which is developed can be extremely variable due to segregation and cooling rate effects.[1] Large amounts of γ-γ' eutectic phase and coarse γ' (>1.0 μm), which are developed during solidification, can account for up to 40 vol pct of the ~60 vol pct γ' in Mar-M200. Thus, only 20 vol pct of the total γ' may be available for precipitation as a fine (<0.5 μm) uniform dispersion.

Solution heat treatments at temperatures sufficiently high to homogenize the alloy and dissolve coarse γ' and eutectic γ-γ' constituents for reprecipitation in the form of a uniform fine γ' dispersion have been shown by Piearcey *et al*[8,9] to improve the longitudinal (parallel to growth direction) creep capability of DS Mar-M200. In this investigation, the effect of solution heat treatment temperature on the amount and distribution of each form of γ' phase (eutectic, coarse and fine) in DS Mar-M200 + Hf cast-to-size turbine blades was examined. Creep resistance of blade castings is correlated with the microstructure variations produced by the range of solution temperatures encountered in normal production heat treatments.

*Nominal composition (wt pct): 0.14C, 9Cr, 10Co, 12W, 5Al, 1Cb, 2Ti, 2Hf, 0.015B, 0.08Zr, balance Ni (Hafnium was added to the base DS Mar-M200 alloy for improved transverse ductility.[1,2])

EXPERIMENTAL PROCEDURES

Turbine blades of DS Mar-M200 + Hf were cast-to-size in various solid and hollow configurations using current production processes. Sections of the hollow blade castings were used for solution heat treatment studies described below. The solid blade castings were machined into longitudinal creep specimens for testing in air at 1800°F (1255 K) and 32 KSI (220 MN/m^2). Prior to machining, the solid blade castings were given a nominal standard heat treatment in production facilities as follows: a solution at 2200°F ± 20°F (1477 K ± 11 K)/2 h/AC, a standard blade coating diffusion cycle at 1975°F (1353 K)/4 h/AC and a precipitation/aging treatment at 1600°F (1144 K)/32 h/AC. The hollow blade sections were solution heat treated for a minimum of 2 h at temperatures from 2140 to 2250°F (1444 to 1505 K) in laboratory furnaces with the temperatures measured using Pt/Pt-10 Rh thermocouples and maintained within ±5°F (±3 K) throughout the experiments.

The microstructures of the solution heat treated blade sections and the creep specimens after test were evaluated using conventional metallographic and replication techniques to determine the amount and distribution of eutectic γ-γ', coarse γ' (>1.0 μm) and fine γ' (<0.5 μm) in each sample. (The coating and aging heat treatments did not significantly alter the amount of coarse γ' and γ-γ' eutectic present compared with the as-solutioned condition.) The total vol pct γ' in the alloy was determined using standard phase extraction methods.[10] The volume percents of eutectic γ-γ' and coarse γ' were determined by area measurements and point counting techniques on representative photomicrographs taken at various magnifications from 250 to 7000 times. By a method of differences, the vol pct of fine γ' (hereafter V_f) was calculated based on the

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measured total γ' level in the alloy of approximately 60 vol pct. A minimum of three locations per sample were photographed and measured to calculate V_f. Results of those calculations indicated an approximate standard deviation of ±3 vol pct in the values of V_f for a given sample. Creep-rupture tests were performed using a standard constant-load creep frame.

RESULTS

1. Effect of Solution Temperature on Microstructure

The as-cast microstructure of a hollow blade casting of DS Mar-M200 + Hf shown in Fig. 1 consists of a γ nickel solid solution containing a dispersion of various sizes of γ' precipitate, discrete MC and $M_{23}C_6$ carbide particles, and a γ-γ' eutectic constituent. Elemental segregation during solidification results in the formation of areas of coarse γ' (*A*) near the interstices between secondary dendrite arms and relatively large areas of γ-γ' eutectic (*B*) as well as associated carbides (*C*) at grain boundaries and in the interdendritic regions between primary dendrite arms. Relatively coarse γ' particles (~1.0 μm) were also present in the dendrite core areas of the as-cast sample (*D*).

The microstructures of blade sections solution heat treated at temperatures from 2140 to 2250°F (1444 to 1505 K) for a minimum of two h show a gradual reduction in the amount of coarse γ' and the eutectic γ-γ' phase with increased solution temperature, Fig. 2. Figure 3 shows the γ' size and morphology in dendrite cores and near interstices for samples solution heat treated at various temperatures. A reduction in the amount of coarse γ' in both the dendrite core and interdendritic regions occurs with increased solution temperature. At approximately 2200°F (1477 K), the coarse cuboidal γ' is eliminated from the dendrite core regions. The remaining coarse γ' near the dendrite interstices is completely dissolved after a 2225°F (1491 K) solution treatment. The amount of γ-γ' eutectic constituent also decreased with increased solution temperature and only isolated regions remained after a 2250°F (1505 K) solution heat treatment for 4 h. Piearcey *et al*[6] reported that complete homogenization of the DS Mar-M200 alloy required a 2250°F (1505 K) solution treatment for at least 100 h, an observation which is consistent with the above findings. The observed increase in the fine γ' size from ~0.1 μm to ~0.3 μm (see Fig. 3) when the solution temperature increased from 2225 to 2250°F (1491 to 1505 K) is believed to be caused by a relatively slow cooling through the 2250 to 2200°F (1505 to 1477 K) range where diffusion rates are high and precipitate growth is enhanced.

Although sometimes a problem in large segregated castings, incipient melting was not observed in the hollow blade segments used in this study, even at the highest solution temperature of 2250°F (1505 K). Advances in DS casting technology over the past several years have resulted in reduced levels of segregation and hence a gradual increase in incipient melting temperatures.

Solution heat treatment of DS Mar-M200 + Hf at temperatures above 2200°F (1477 K) has the advantages of a) improving alloy homogeneity, b) partially solutioning the γ-γ' eutectic, and c) dissolving coarse γ', thus making more Al + Ti available for subsequent reprecipitation in the form of fine γ' (<0.5 μm). The influence of the observed refinement and redistribution of γ' on creep resistance of DS Mar-M200 + Hf is described below. Figure 4 shows the effect of solution temperature on V_f. The sharp increase in V_f from 2160 to 2200°F (1455 to 1477 K) results from the rapid solutioning of coarse γ' with temperature over this temperature range as shown in Fig. 2. The more gradual increase in V_f from 2200 to 2250°F (1477 to 1505 K) represents the more sluggish solutioning of

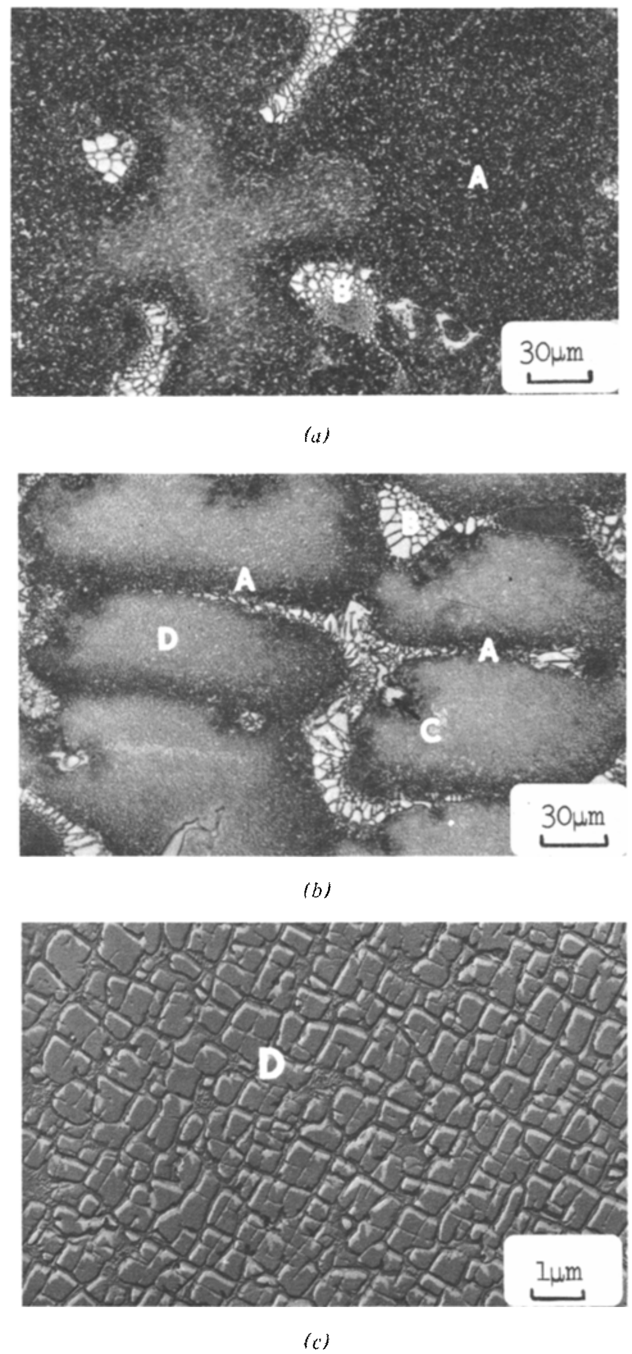


Fig. 1—As-cast microstructure of DS Mar-M200 + Hf turbine blade casting; (*a*) transverse section, (*b*) longitudinal section, (*c*) dendrite core of longitudinal section. Note coarse γ' in the interstices (A) and dendrite core (D) and the γ-γ' eutectic (B) and carbides (C) in the interdendritic regions.

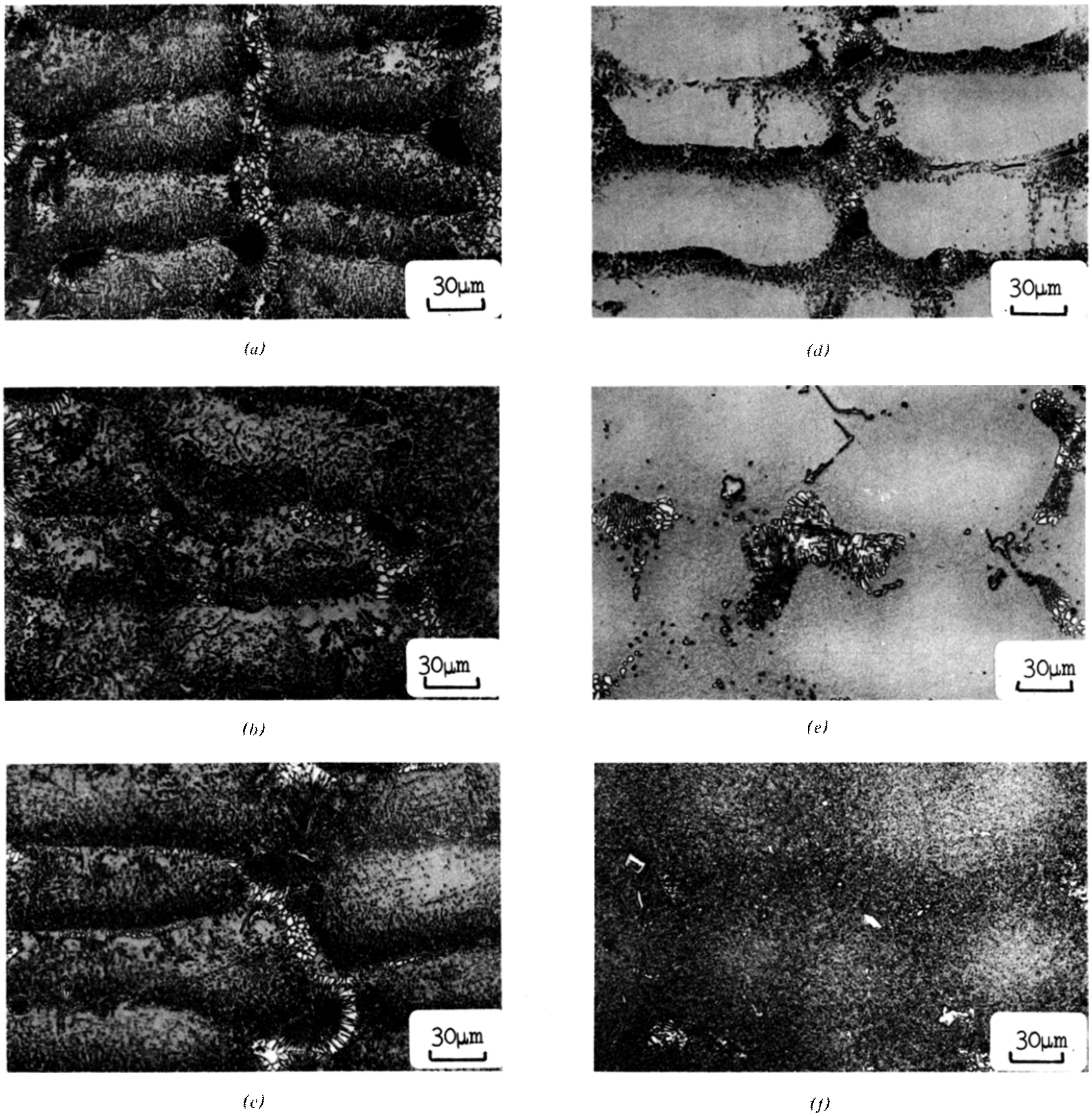


Fig. 2—Microstructure of DS Mar-M200 + Hf turbine blade castings solution heat treated at the temperatures indicated; (*a*) 2140°F (1444 K), (*b*) 2160°F (1455 K), (*c*) 2180°F (1466 K), (*d*) 2200°F (1477 K), (*e*) 2225°F (1491 K) and (*f*) 2250°F (1511 K).

the γ-γ' eutectic constituent. The most notable feature of the V_f *vs* solution temperature correlation is the wide range of V_f values which can result within a relatively narrow range of heat treatment temperatures. This is particularly evident near the steep portion of the curve in Fig. 4. For example, the solution temperature tolerance of ±20°F (±11 K) for a standard 2200°F (1477 K) solution heat treatment of blade castings can result in a range of V_f values as large as 31 vol pct [V_f = 22 vol pct at 2180°F (1466 K) to 53 vol pct at 2220°F (1488 K)]. The effect of such a wide variation in V_f on creep resistance can be significant as discussed below.

2. Effect of Volume Percent Fine γ' on Creep Resistance

The examination of creep specimens machined from solid turbine blades which were solution heat treated at 2200°F ± 20°F (1477 K ± 11 K) revealed varied microstructures as shown in Fig. 5. Although part geometry and specific casting process parameters can influence resultant microstructures, it is more likely that, for the configurations examined, the variation in the amount of coarse γ' and eutectic γ-γ' shown in Fig. 5 are due to solution temperature variations within the standard 2200°F ± 20°F (1477 K ± 11 K) solution treat-

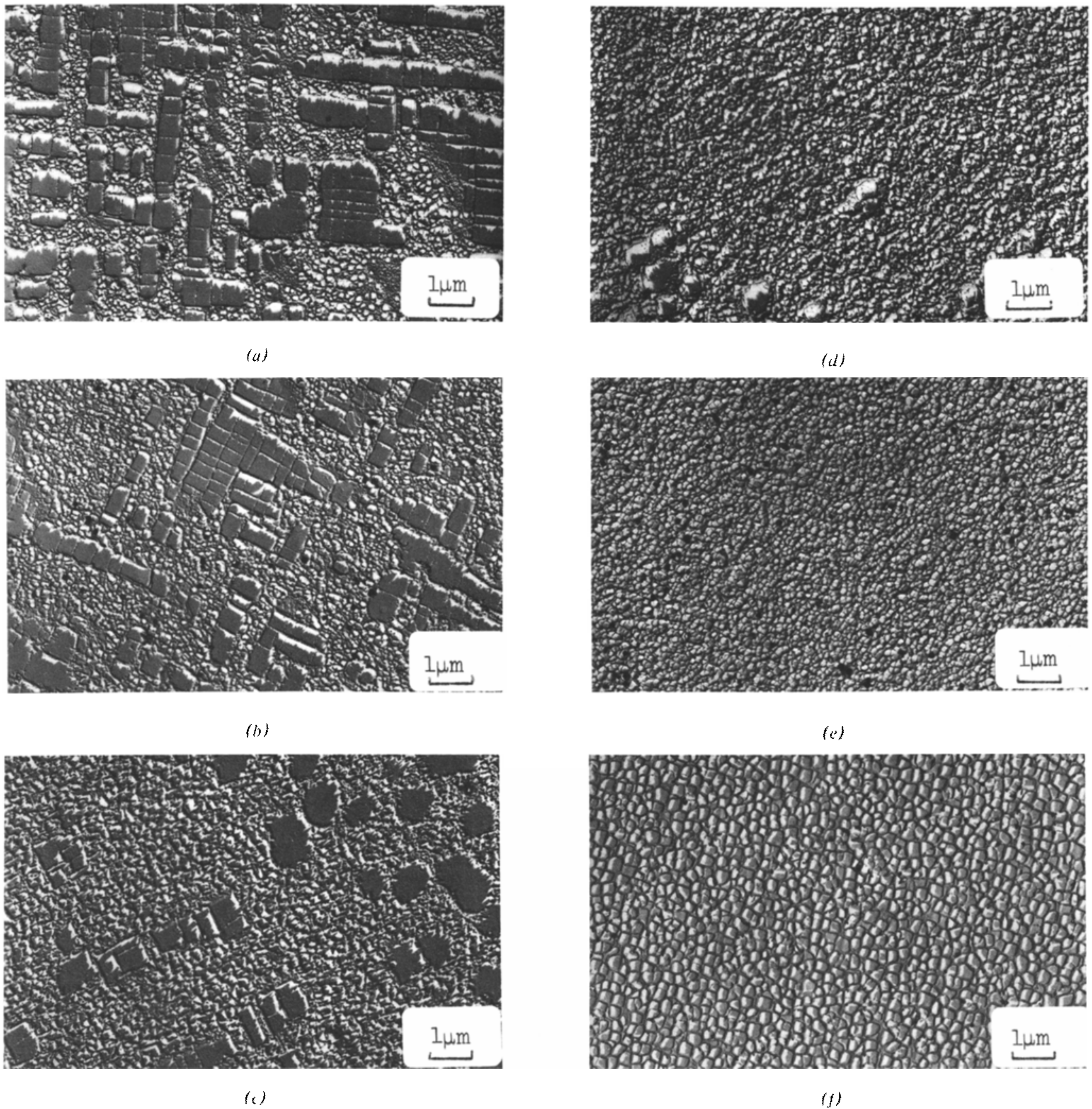


Fig. 3—Electron photomicrographs of DS Mar-M200 + Hf turbine blade castings solution heat treated at the temperatures indicated: (a) 2140°F (1444 K), dendrite core; (b) 2160°F (1455 K), dendrite core; (c) 2180°F (1466 K) near dendrite interstice; (d) 2200°F (1477 K) near dendrite interstice; (e) 2225°F (1491 K), dendrite core; (f) 2250°F (1505 K), dendrite core.

ment range. The level of fine γ' observed among the samples examined ranged from 21 to 46 vol pct, which is within the scatter band of data shown in Fig. 4 for this solution heat treatment range.

Results of 1800°F/32 KSI (1255 K/220 MN/m^2) creep-rupture tests for the machined-from-blade specimens are plotted in Fig. 6 as a function of V_f. The increase in rupture life with V_f shown in this plot indicates that the amount of fine γ' plays a significant role in the 1800°F (1255 K) creep resistance of DS Mar-M200 + Hf. Based upon the correlation in Fig. 6, the average 1800°F/32 KSI (1255 K/220 MN/m^2) rupture life of DS Mar-M200 + Hf blades increased by approximately a factor of three (40 h *vs* 120 h) as V_f increased from 30 to 45 vol pct. Even greater increases in rupture life may be possible as the maximum level of 60 vol pct fine γ' is approached. Creep-rupture tests on turbine blade specimens solution heat treated at 2250 to 2260°F (1505 to 1511 K) have resulted in rupture lives of up to 200 h at 1800°F/32 KSI (1255 K/220 MN/m^2).[12] Post-test examination of these specimens revealed that V_f levels above 55 vol pct had been achieved.

DISCUSSION

The concept of maximizing the creep resistance of γ' strengthened nickel-base superalloys by heat treating to develop a uniform dispersion of fine γ' (*i.e.*, maximize γ-γ' interfacial area) is well established. However, quantitative data which demonstrate

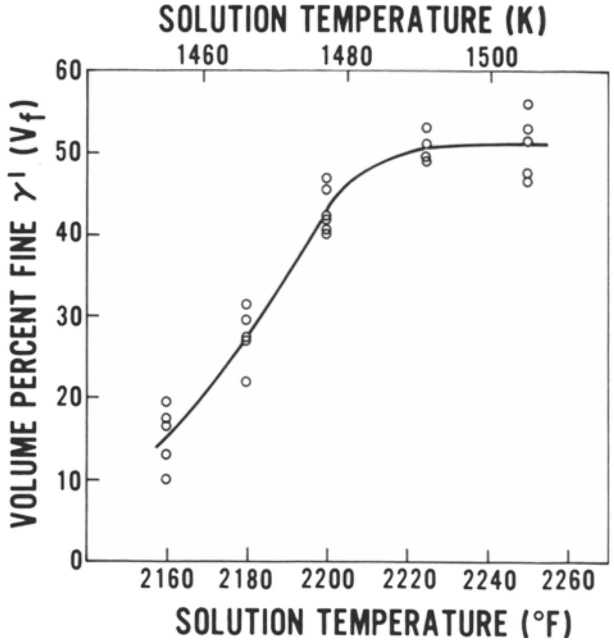
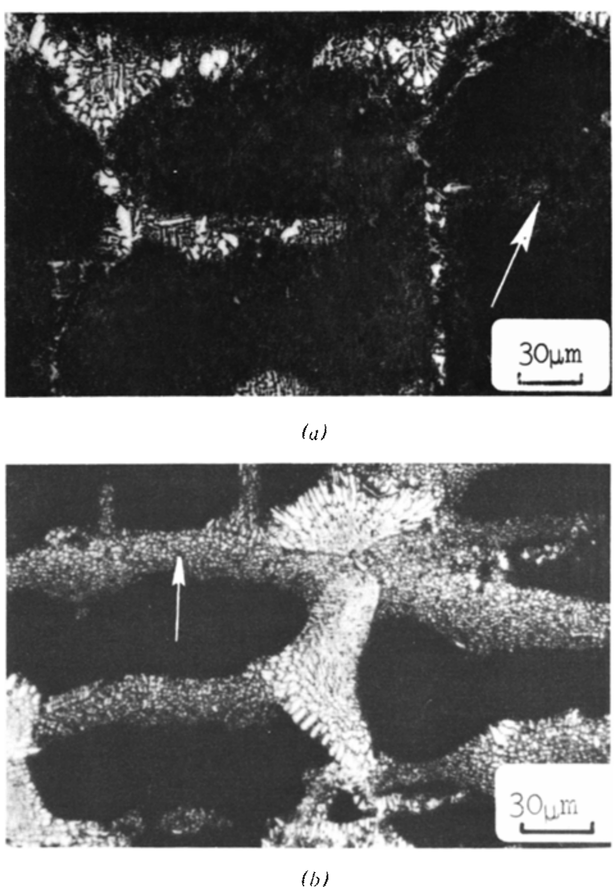


Fig. 4—Effect of solution temperature on the volume percent fine γ' in DS Mar-M200 + Hf.



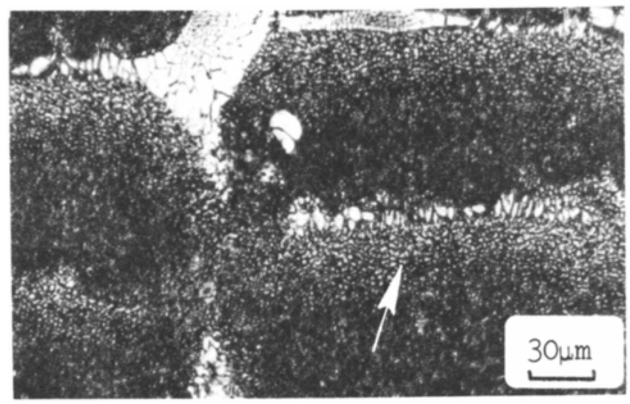


Fig. 5—Microstructure of variation observed in DS Mar-M200 + Hf turbine blade castings heat treated in production facilities at 2200° F ± 20° F (2) AC + 1975° F (4) AC + 1600° F (32) AC [1477 K ± 11 K (2) AC + 1353 K (4) AC + 1144 K (32) AC]. Note the varying amount of coarse γ' in the interstices (arrows); (*a*) light, (*b*) moderate, and (*c*) heavy.

the influence of a given amount of the fine γ' dispersion on creep resistance, particularly for a complex alloy such as DS Mar-M200 + Hf, have not previously been published. The rupture life *vs* V_f studies described above have shown that the 1800°F (1255 K) creep resistance of DS Mar-M200 + Hf steadily increased as the volume fraction of uniform fine γ' increased and the amount of coarse γ' and γ-γ' eutectic constituents decreased. The increase in creep resistance was especially apparent at V_f values above 35 vol pct. The quantitative mechanisms responsible for the improved creep resistance with increased V_f are not known at this time but increased γ-γ' interfacial area and decreased interparticle spacing associated with a uniform distribution of fine γ' are the most likely contributing factors. Examination of the creep curves for low and high V_f specimens from another investigation[12] revealed that minimum creep rate (MCR) was reduced as V_f was increased. (Specimen size limitations did not permit accurate creep rate measurements on the machined-from-blade specimens examined in this investigation.) This decrease in MCR with increased V_f is consistent with the increased dislocation/particle interaction which might be expected as the volume fraction of fine uniformly distributed γ' particles is increased and interparticle spacing decreased.

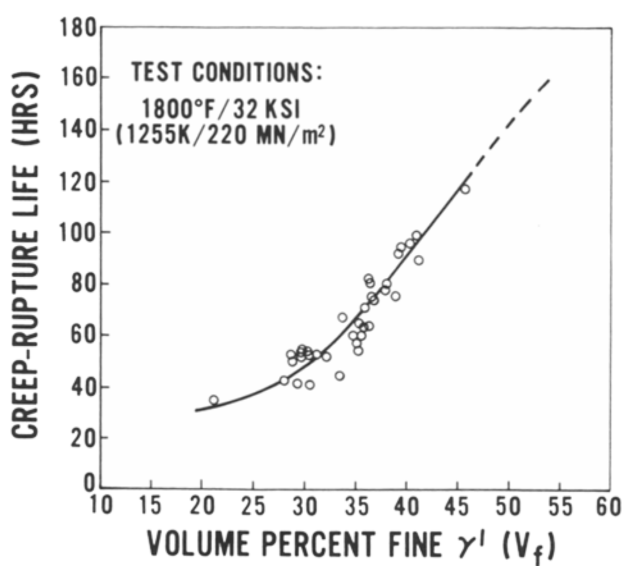


Fig. 6—Correlation between 1800° F/32 KSI [1255 K/220 MN/m²] creep-rupture life and vol pct fine γ' in DS Mar-M200 + Hf turbine blade castings.

As shown by the V_f *vs* solution heat treatment temperature studies, a critical temperature range (2160 to ~2210°F) [1455 to ~1483 K] exists for DS Mar-M200 + Hf over which V_f can vary significantly. Above that range, V_f is relatively constant and therefore, in order to assure that the highest level of fine γ' is attained, solution temperatures above that critical range should be used. The solution temperature/V_f/rupture life relationships found for DS Mar-M200 + Hf can probably be extended to other γ' strengthened DS alloys. However, the practical application of higher solution temperature heat treatments must take into consideration the potential incipient melting problems associated with heavily segregated parts.

CONCLUSIONS

1) The vol pct of fine γ' developed in DS Mar-M200 + Hf increases significantly with solution temperature in the range of 2160°F (1455 K) to approximately 2210°F (1483 K). Above 2210°F (1483 K), V_f is relatively constant. The increase in V_f with increased solution temperature is due to the gradual solutioning of the coarse γ' and eutectic γ-γ' constituents and subsequent reprecipitation of a uniform dispersion of fine γ' on cooling.

2) The 1800°F (1255 K) creep-rupture life of DS Mar-M200 + Hf is dependent on the vol pct of fine γ' developed during heat treatment. Rupture life increases steadily as V_f approaches the maximum attainable level of 60 vol pct for this alloy.

3) Maximum 1800°F (1455 K) creep life for DS Mar-M200 + Hf blade castings is achieved when the highest possible solution temperatures are used. The solution temperature limit, however, must be determined by incipient melting considerations where heavy segregation is present.

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