THE INFLUENCE OF HIGH THERMAL GRADIENT CASTING, HOT ISOSTATIC PRESSING AND ALTERNATE HEAT TREATMENT ON THE STRUCTURE AND PROPERTIES OF A SINGLE CRYSTAL NICKEL BASE SUPERALLOY

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SUMMARY

The results of a program to improve the cyclic properties of the single crystal superalloy PWA 1480 are reported. The program objective was to reduce or eliminate casting porosity as fatigue initiation sites by the application of improved commercial casting process parameters and hot isostatic pressing. An alternative to the standard PWA 1480 coating and aging heat treatment cycle was also evaluated for potential mechanical property improvement in high pressure hydrogen environment. Higher thermal gradient casting was found to provide a reduction in dendrite arm spacing, reduction in overall casting porosity density and, most importantly, a reduction in pore size. Tensile properties of PWA 1480 were not significantly affected by casting gradient while stress rupture lives were increased. Improvements in low cycle fatigue and high cycle fatigue lives were achieved as a result of the reduced pore size and improved homogeneity. The alternative heat treatment provides a slight increase in tensile ductility in hydrogen environment, but no apparent benefit to cyclic properties. The most dramatic improvements to material properties were provided by hot isostatic pressing. Increased high cycle fatigue life is directly attributed to elimination of casting pores as crack initiation sites. Increased stress rupture lives may be attributed to a delay in the onset of tertiary creep due to the absence of pre-existing voids and an increased volume fraction of fine gamma prime due to improved solution heat treatment. Hot isostatic pressing has now been instituted as a production requirement for the next generation of single crystal blades for the Space Shuttle Main Engine.

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Introduction

Single crystal (SC) superalloys are currently under development as a potential replacement for directionally solidified MAR-M246 + Hf as turbine blades in the Space Shuttle Main Engine (SSME) high pressure turbopumps. The operating conditions in the SSME, however, are significantly different from the conditions in airbreathing gas turbine engines for which SC alloys were developed.(1) Consequently, the primary deformation modes of SSME turbine blades are low cycle fatigue (LCF) and high mean stress high cycle fatigue (HCF) compounded by hydrogen environment embrittlement rather than creep/stress rupture assisted by oxidation and sulphidation, for which the alloys were developed. The stress rupture, low cycle fatigue, high cycle fatigue and hydrogen environment embrittlement resistance of the candidate SC alloys are superior to DS MAR-M246, however, significant improvements in fatigue properties have been projected due to the application of advanced processing methods.(1,2) In the absence of intentional carbides in most modern SC superalloys, interdendritic casting porosity has been found to be the primary initiation site for fatigue failures. Reduction in the density and, more importantly, size of the inherent porosity can be expected to substantially improve fatigue lives from the view point of increased crack initiation times.

Increased casting thermal gradient has been shown to provide reduced dendrite arm spacing (DAS),(1) improved chemical homogeneity(3) and reduction in interdendritic pore size.(4) The influence of increased thermal gradient on SC nickel base superalloys was first demonstrated in the early 1970's,(5) but the technology to apply significant improvements on a commercial scale has only recently been developed.(6) Hot isostatic pressing (HIP) has also been recognized as a valuable tool for improving the fatigue capability of cast superalloys.(7) Early attempts at applying the technology to SC superalloys met with only limited success due to problems unique to the SC structure. Typical HIP temperatures and pressures produced plastic flow around closing pores, leading to local recrystallization. Fatigue cracks then initiate at the isolated secondary grains, negating the benefits due to elimination of porosity.(8) Additionally, low carbon single crystal superalloys contain many alloying elements with high affinities for carbon. Extremely careful control of HIP autoclave environments must be exercised to avoid significant surface carburization.(9) Despite these early concerns, development of a SC HIP process has been spurred on for the SSME program due to the potential benefits in improved fatigue lives. Alternative heat treatments for several superalloys,(10) have been shown to provide improvements in hydrogen environment embrittlement resistance.(11) Since creep is not a primary mechanism in SSME turbine blade deformation, alternatives to the PWA 1480 heat treatment have been evaluated for improved hydrogen environment properties.

The program described herein was undertaken to evaluate the benefits in material properties due to high thermal gradient casting, hot isostatic pressing and alternate heat treatments for PWA 1480.

Experimental Procedure

The PWA 1480 test materials for this program were cast in commercial facilities, representing the range of available thermal gradients. Commercial facilities were specified to facilitate transfer of results to the SSME program without the need to scale up from laboratory experiments. Materials representing three casting thermal gradients were obtained in the form of 1.25 to 1.6 centimeter diameter cylindrical rods. Primary crystallographic orientation of the cast bars was maintained within 10 degrees of the <001> direction. All of the test material received standard PWA 1480 solution heat treatment. Low and intermediate thermal gradient cast materials were tested in the standard heat treat condition; 1080C/4 hours plus 871C/32 hours. The intermediate and high thermal gradient materials were tested with an alternative heat treatment of 1010C/2 hours plus 871C/48 hours to evaluate possible improvements in hydrogen environment embrittlement. The intermediate gradient material was also tested in the hot isostatic pressed (HIP) plus standard heat treated condition. A viable HIP schedule has been devised which avoids the microstructural pitfalls associated with the densification of SC superalloys. The process was developed by Rocketdyne for application to PWA 1480 and has been found to be generic to other single crystal superalloys with the adjustment of HIP temperature profiles. Details of the process are restricted by U. S. Patent Secrecy Order.

Mechanical test samples were low stress crush ground from the fully processed castings. Standard straight gage section samples of 0.625 cm diameter were employed for all tests. Tensile and low cycle fatigue tests were conducted according to ASTM standards with extensometry attached to the specimen gage section. Tensile tests were conducted at 20C in air and 34.5 MPa hydrogen gas to verify the viability of the various processes. Low cycle fatigue tests were conducted at 538C, 0.33 Hz and 2.0% total strain range. Stress rupture tests were conducted at 871C and 550 MPa initial stress. High cycle fatigue tests were conducted in load control at room temperature and 843C, at a stress ratio of R = 0.47 at 30 Hz. The cyclic test parameters were chosen to represent conditions of interest for SSME turbopump turbine blades.

Optical metallography was employed to document the density and size of casting porosity and DAS. Statistics of the pore distribution were calculated from quantitative metallography measurements. Gamma prime morphology and distribution were evaluated by scanning electron microscopy. Optical and SEM fractography were conducted on all failed samples, with special emphasis placed on characterization of fatigue initiation sites.

Results

Representative photomicrographs of the casting porosity and etched microstructures are shown for the respective materials in Figure 1-3, with average area fraction of pores, average pore sizes and maximum pore sizes also given. The observed density and size of the interdendritic porosity are reduced as the thermal gradient is increased. In addition, pore morphology changes qualitatively to a lower aspect ratio, more circular cross section

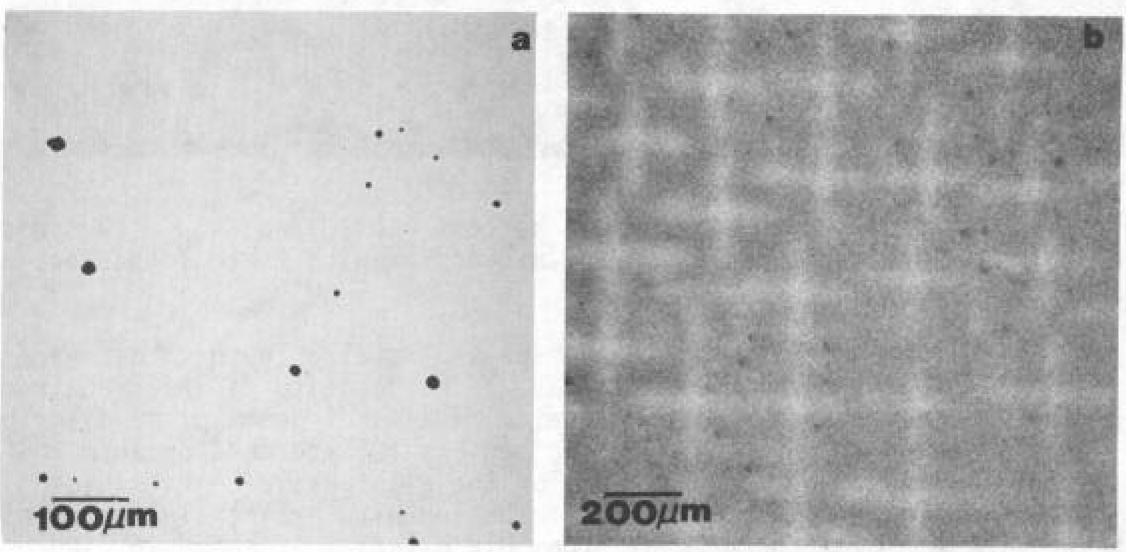

Figure 1. a)Typical porosity; area percent = 1.01 and d_{avg} = 32 micron; and b)typical microstructure; DAS = 446 micron; of low thermal gradient cast PWA 1480.

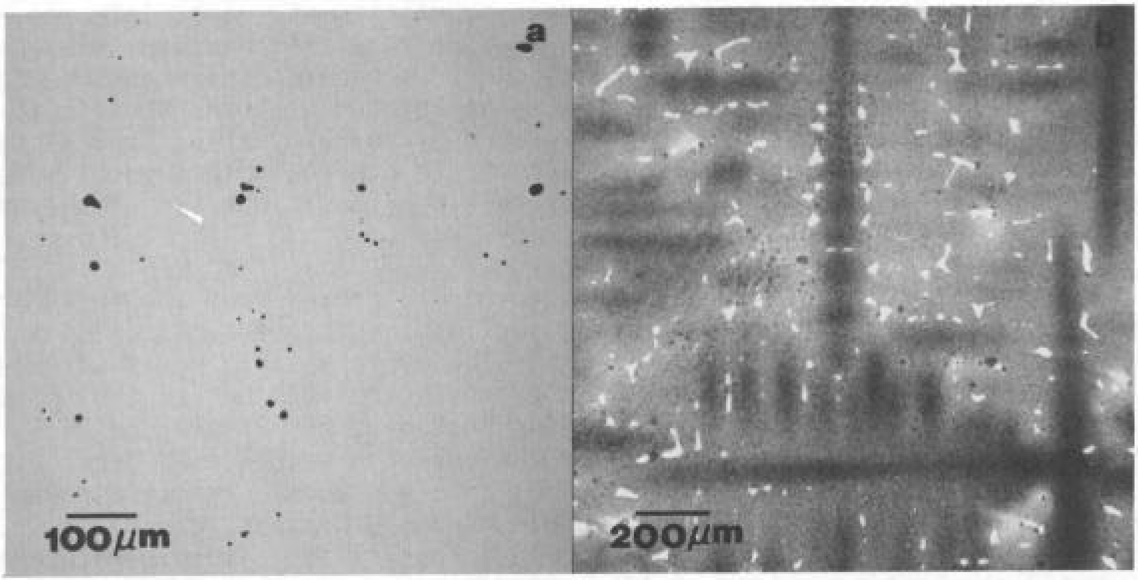

Figure 2. a)Typical porosity; area percent = 0.36 and d_{avg} = 20 micron; and b)typical microstructure; DAS = 324 micron; of intermediate thermal gradient cast PWA 1480.

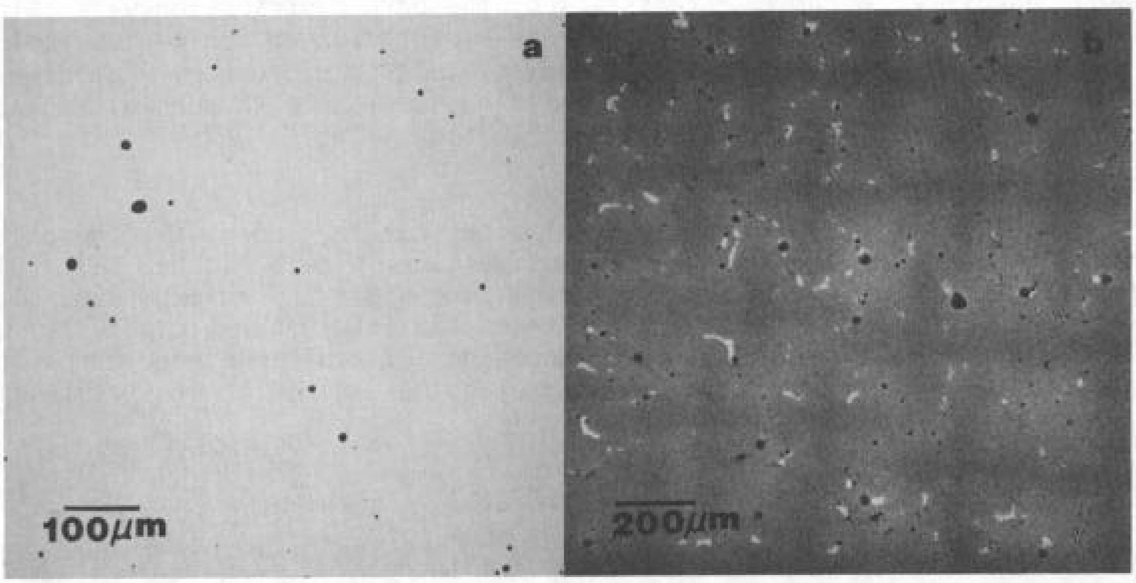

Figure 3. a)Typical porosity; area percent = 0.30 and d_{avg} = 14 micron; and b)typical microstructure; DAS = 222 micron; of high thermal gradient cast PWA 1480.

as pore size decreases. Dendrite arm spacing is also found to decrease as casting gradient is increased. An increase of homogeneity is inferred from a reduction in the amount of casting eutectic. The final gamma prime distribution between dendrite and interdendritic regions is also more uniform in the high gradient material. The influence of the alternative heat treatment is to force a more uniform gamma prime distribution with a slightly larger precipitate size. Microstructure of the HIP material is shown in Figure 4. Porosity has been completely eliminated and internal recrystallization has been avoided by the HIP process. The final microstructure is better homogenized due to increased time at the solution heat treatment temperature during the HIP process.

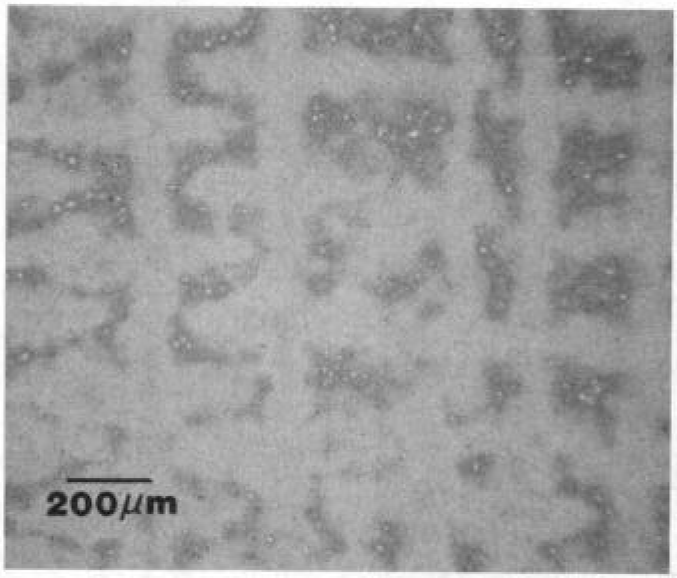

Figure 4. Microstructure of hot isostatically pressed PWA 1480 shows elimination of casting porosity and improved alloy homogeneity.

Tensile properties for the various materials are presented in Table I. Room temperature yield and ultimate strengths are unaffected by process parameters, except for increased ultimate strengths observed for the alternate heat treated material. The alternate heat treatment also provides a slight benefit in ductility in both air and high pressure hydrogen environments. Stress rupture results are given in Table II. Short time stress rupture life increased as thermal gradient increased and increased for HIP relative to non-HIP material. A SEM photomicrograph of a failed, low gradient cast, stress rupture sample is shown in Figure 5. Stress rupture failures initiate at interdendritic casting porosity and associated eutectic islands which link up by ductile overload in the intervening regions. Low cycle fatigue test results, presented in Figure 6, indicate slightly increased life as thermal gradient increased, for the strain range tested. LCF failures in the intermediate temperature regime initiate primarily at surface connected porosity and are Stage I, with the {111} fracture plane often encompassing the entire specimen. High cycle fatigue test results are presented in Figure 7. HCF life increases with increasing casting thermal gradient at

TABLE I. Typical Room Temperature Tensile Properties

Material Condition	Atm.	0.2% Yield (MPa)	Ultimate (MPa)	R. A. (%)	Elongation (%)
Low Grad/Std Heat Treat	Air	1024	1077	12.5	11.7
Int. Grad/Std Heat Treat	Air	1020	1179	5.6	4.0
Int. Grad/Alt Heat Treat	Air	992	1282	7.0	8.0
Int. Grad/HIP/Std Heat Treat	Air	1010	1122	9.2	12.5
Int. Grad/Std Heat Treat	H_2*	979	986	2.0	2.0
Int. Grad/Alt Heat Treat	H_2	1020	1089	4.0	3.0
Int. Grad/HIP/Std Heat Treat	H_2	970	1007	3.2	3.1
High Grad/Alt Heat Treat	Air	1082	1211	10.3	10.3

*H_2 = 34.5 MPa Hydrogen Atmosphere

TABLE II. 871C, 550MPa Stress Rupture Lives

Material Condition	Time To Failure (Hours)	Elongation (%)
Low Gradient/Std Heat Treat	Not Tested	
Int. Gradient/Std Heat Treat	12.5	11.4
Int. Gradient/HIP/Std Heat Treat	61	10.6
High Gradient/Alt. Heat Treat	63.4	14.6

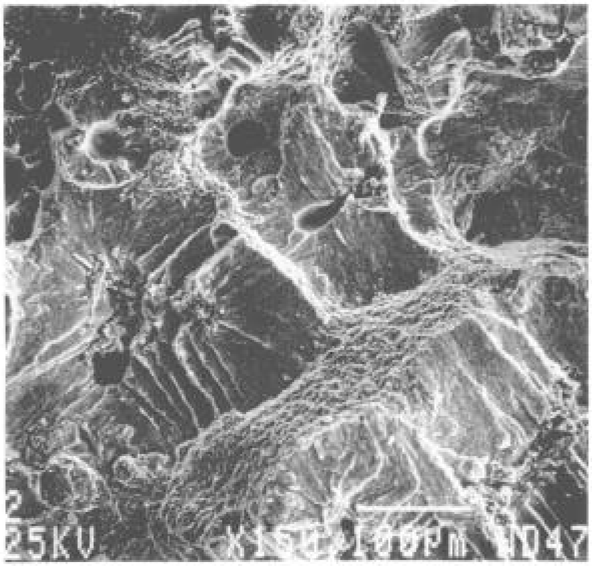

Figure 6. Low gradient cast stress rupture fracture surface.

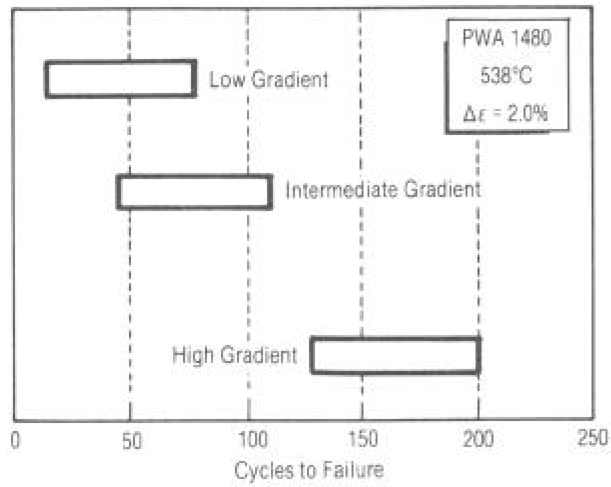

Figure 7. Low cycle fatigue life ranges at 538C and 2.0% strain range.

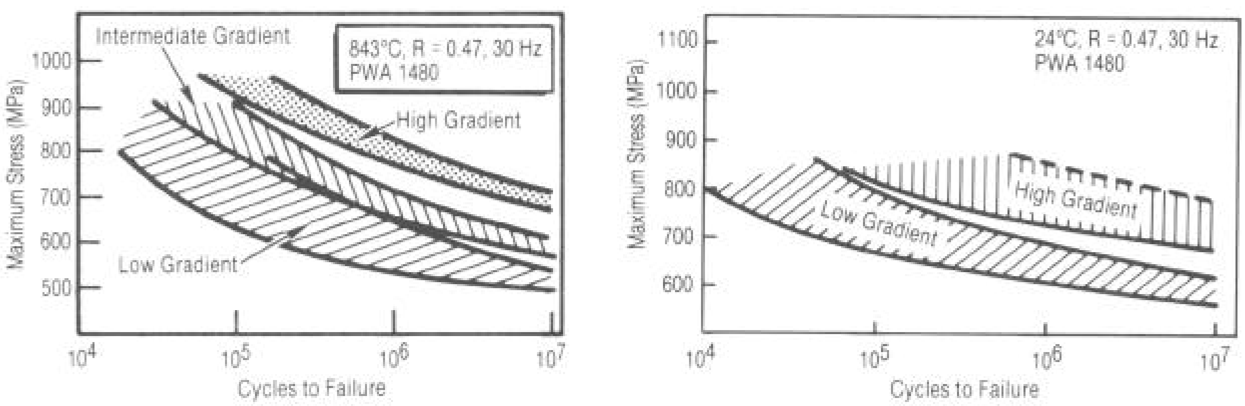

Figure 7. High cycle fatigue curves at R = 0.47, 30 Hz and a)843C and b)24C.

both room temperature and 870C. Alternative heat treatment was found to have no discernable influence on fatigue life. Those test results are, therefore, included in the representative fatigue curves. Representative fractographs are presented in Figure 8. HCF failures initiate predominantly at internal interdendritic casting porosity at 870C. Propagation is Stage II at 870C until overload is reached or until the crack meets the specimen surface, when crack growth becomes Stage I. Room temperature propagation is predominantly Stage I from near surface pore initiation. HCF life increases as initiating pore size decreases. HCF curves for HIP material are compared to the non-HIP material in Figure 9. The greatest benefit in fatigue life is obtained at room temperature. Failures of the HIP material initiate at the specimen surface, eutectic islands or at isolated small carbides, as shown in Figure 8d.

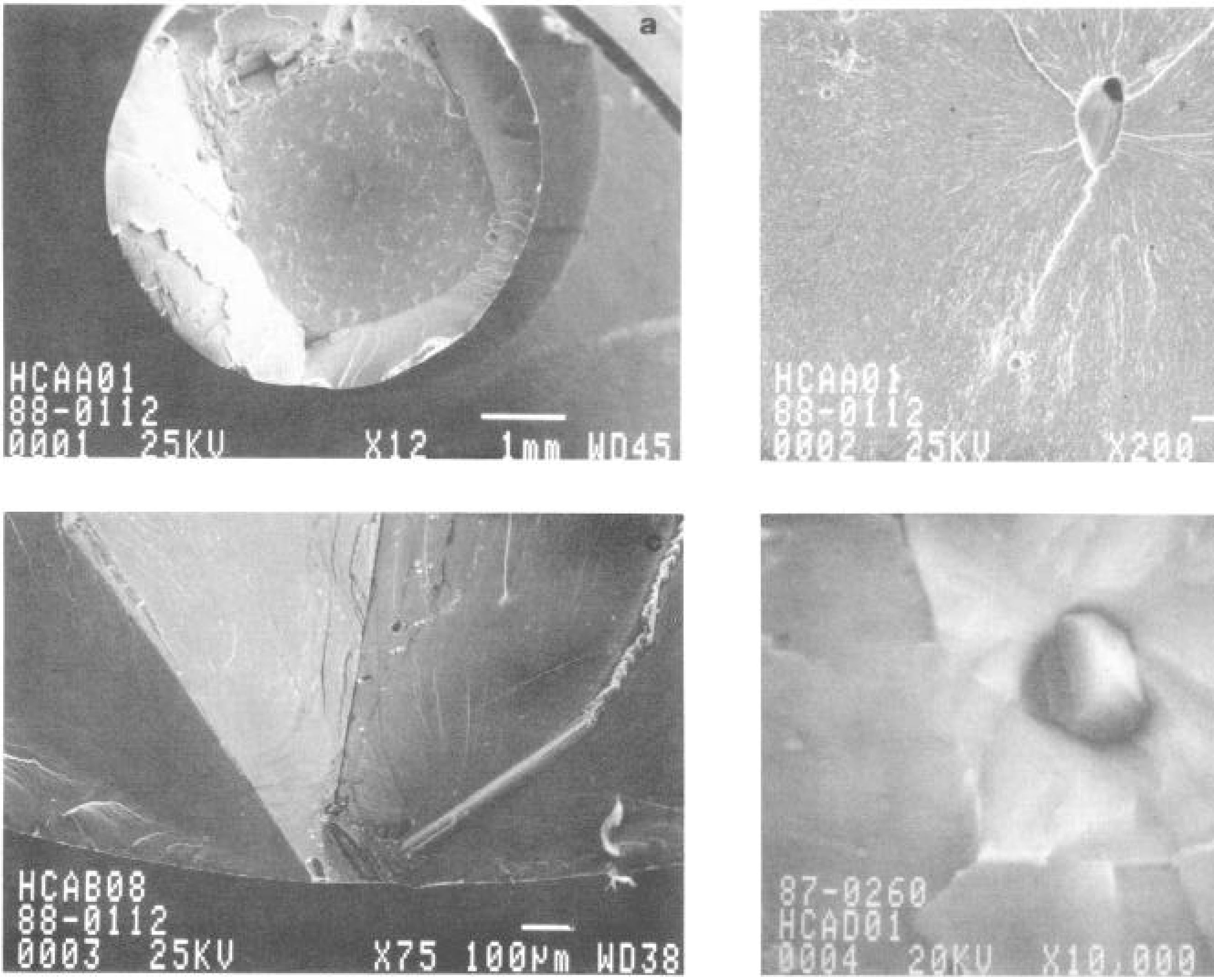

Figure 8. Representative high cycle fatigue fractographs. a) and b)high
dient cast, 843C, R = 0.47, maximum stress = 793 MPa, N_f=924,869; c)low
dient cast, 24C, R = 0.47, maximum stress = 793 MPa, N_f=111,789 and
intermediate gradient cast, 24C, R = 0.47, maximum stress = 896 MPa and
N_f=6.95 X 10^6

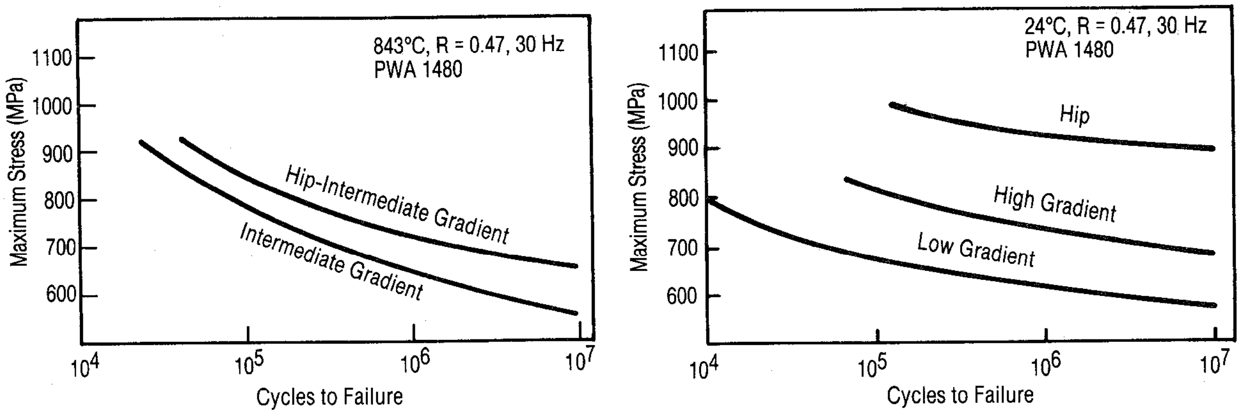

Figure 9. Comparison of fatigue life of HIP versus non-HIP PWA 1480 at a)843C and b)24C.

Discussion

Higher thermal gradient single crystal casting has been found to provide benefits in terms of microstructure and improved cyclic loading properties. Increased casting thermal gradient reduces the DAS, provides improved chemical homogeneity and reduces the size and density of the interdendritic porosity. The improved homogeneity and reduced pore size are both direct consequences of the reduced DAS. Shorter diffusion distances within the solidification front reduce the amount of solute rejected from each dendrite core and reduce the local magnitude of interdendritic eutectic formed. These shorter diffusion distances also improve the homogenization provided during a conventional heat treatment cycle. Casting porosity is formed by the inability of the liquid metal to flow into the dendritic interstices and subsequent shrinkage as the remaining liquid freezes. Finer DAS reduces the volume of liquid in the interdendritic region, resulting in a finer pore size. Pore morphology is also changed to a more spherical shape as surface tension reduces the surface area to volume ratio.

Tensile properties at room temperature are virtually unaffected by the microstructural improvement due to high gradient casting. The primary benefit is manifested as an increase in stress rupture capability at high stresses. There are several factors which contribute to this improvement. The reduction of the amount of casting eutectic increases the free concentration of the alloying elements responsible for the formation of fine gamma prime. A slightly increased volume fraction of the optimum size precipitates is then produced. In addition, the gamma prime size in both dendritic and interdendritic regions is more uniform and closer to the optimum size for creep resistance. The onset of tertiary creep is also delayed due to the reduction of porosity size and density in the initial microstructure. Crack nucleation must then take place on a broader scale, prior to linkup and net section loss leading to failure.

Low cycle fatigue lives were found to increase with increased casting thermal gradient. The relatively high strain range employed produces a significant amount of macroscopic strain. Under these conditions, crack initiation can occur in slip bands, reducing the importance of internal defects as initiation sites. The improved LCF capability in this test regime is primarily due to the improvement in strength and/or ductility afforded by the improved alloy homogeneity. High cycle fatigue lives are very clearly controlled by crack initiation at internal defects; especially at low temperature. Increased HCF lives with increased casting thermal gradient are a direct consequence of reduced interdendritic pore size and lower aspect ratio morphology. This benefit has recently been analytically predicted through linear elastic fracture mechanics calculations.(12) It is apparent, though, that return on investment for increasing thermal gradient will diminish due to practical limitations on production casting gradients.

Hot isostatic pressing, when properly applied, provides a significant improvement in high cycle fatigue life, especially at lower temperatures and long lives. At elevated temperatures stress rupture becomes a contributing factor in the final failure and reduction in initiating defect size is not as significant. Again, the increased cyclic life can be partially attributed to the removal of porosity as fatigue crack initiation sites since no appreciable affect of HIP was found on tensile properties. The high temperature, high mean stress fatigue capability in the creep/fatigue regime may also be improved slightly by the increased stress rupture life obtained due to improved solution heat treatment and pore removal. Alternative heat treatment had no significant impact on the cyclic properties of PWA 1480 since defect tolerance, rather than bulk mechanical properties, is the overriding factor in HCF life. This conclusion is consistent with recent results on the fatigue capability of rafted microstructures.(2)

Conclusions

High thermal gradient casting is an effective method for improving the homogeneity and reducing the size, amount and aspect ratio of interdendritic porosity in single crystal nickel base superalloys. Primary benefits due to improved thermal gradients are manifested as increased stress rupture and increased fatigue lives. A diminishing rate of return on improved properties attributed to high thermal gradient casting places practical limits on its use for production hardware.

Alternative heat treatment does not significantly affect the cyclic properties of PWA 1480 since fatigue life is predominantly controlled by crack initiation at microstructural defects. Hot isostatic pressing is an effective method of improving the cyclic properties of single crystal superalloys especially in the long life regime. Increased time at the solution heat treat temperature during HIP improves the alloy homogeneity and provides a significant increase in stress rupture life. Elimination of casting porosity shifts fatigue crack initiation sites to less severe defects such as carbides and, therefore, significantly improves fatigue life. Improved casting homogeneity and reduction of the initial pore size due to high gradient casting improves the homogenization due to solution heat treatment and increases process parameter windows for the HIP process, reducing the possibility of recrystallization due to pore closure.

Acknowledgements

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REFERENCES

1. W. T. Chandler, "Materials for Advanced Rocket Engine Turbopump Turbine Blades," (NASA CR-174729, Lewis Research Center, 1983.

2. T. Khan et. al., "Single Crystal Superalloys for Turbine Blades: Characterization and Optimization of CMSX-2 Alloy" (Paper presented at the 11th Symposium on Steels and Special Alloys for Aerospace, Paris Air Show, Le Bourget, 6 June 1985).

3. M. McLean, Directionally Solidified Materials for High-Temperature Service (London, The Metals Society, 1983).

4. D. D. Pearson, D. L. Anton and A. F. Giamei, "High Thermal Gradient Superalloy Crystal Growth" (Presented at NASA Conference on Structural Integrity and Durability of Reusable Space Propulsion Systems, Cleveland Ohio, June 4-5,1985).

5. J. K. Tien and R. P. Gamble, "The Suppression of Dendritic Growth in Nickel-Base Superalloys during Unidirectional Solidification," Materials Science and Engineering, 8(1971),152-160.

6. M. J. Goulette, P. D. Spilling and R. P. Arthey, Proceedings of the Conference on In-situ Composites III (Lexington MA., Ginn and Co. 1979).

7. D. J. Kenton, "HIP - Nemesis of Shrinkage in Investment Castings," Diesel and Gas Turbine Progress, March 1976.

8. T. E. Strangman, M. Fujii and X. Nguyen-Dink, "Development of Coated Single-Crystal Superalloy Systems for Gas Turbine Applications," Superalloys 1984, ed. M. Gell et. al. (Warrendale, PA: The Metallurgical Society, 1984), 795-804.

9. R. L. Dreshfield, "Application of Single Crystal Superalloys for Earth-to-Orbit Propulsion Systems", (Presented at the 23rd Joint Propulsion Conference, San Diego, CA, June 29-July 2,1987).

10. T. Khan and P. Caron "Effect of Heat Treatments on the Creep Behaviour of a Ni-Base Single Crystal Superalloy", (Paper presented at the 4th RISO International Symposium on Metallurgy and Materials Science, Denmark, 1986).

11. Rocketdyne Division unpublished research.

12. S. Majumdar and R. Kwasny, "Effects of a High Mean Stress on the High-Cycle Fatigue Life of PWA 1480 and Correlation of Data by Linear Elastic Fracture Mechanics" (NASA CR-175057, Argonne National Laboratory, November 1985.)