

FATIGUE PROPERTIES OF MA 6000E, A γ' STRENGTHENED ODS ALLOY

Y. G. Kim* and H. F. Merrick
Inco Research & Development Center
Sterling Forest, Suffern, NY 10901

*Korea Institute of Studies, Cheong-Ryang-Ri, Seoul Korea

MA 6000E is a corrosion resistant, γ' strengthened ODS alloy under development for advanced turbine blade applications. The high temperature, 1093°C, rupture strength is superior to conventional nickel-base alloys. This paper addresses the fatigue behavior of the alloy. Excellent properties are exhibited in low and high cycle fatigue and also thermal fatigue. This is attributed to a unique combination of microstructural features, i.e., a fine distribution of dispersed oxides and other non-metallics, and the highly elongated grain structure which advantageously modify the deformation characteristics and crack initiation and propagation modes from that characteristic of conventional γ' hardened superalloys.

INTRODUCTION

It has long been recognized that improved gas turbine engine performance is keyed to the development of new materials which allow higher turbine entry temperatures. One of the most demanding parts in the gas turbine is the first stage blade which is subjected to severe combinations of stress and temperature in a hostile environment. Present day turbine blade alloys are nickel-rich solid solution matrices hardened primarily by coherent ordered γ' precipitates(1). Dissolution and coarsening of γ' precipitates imposes an upper limit on the metal operating temperature of current blade alloys, despite advances in process technology, such as directional solidification.

Oxide dispersion strengthened (ODS) alloys attract great attention as advanced high temperature materials because they can retain useful strength up to a relatively

high fraction of their melting points(2). High temperature strength is due primarily to the coarse elongated grain morphology and the fine uniform dispersion of stable oxide particles. One such alloy under development as a turbine blade alloy is MA 6000E(3). The nominal composition of this alloy is .05C-15Cr-2Mo-4W-4.5Al-2.5Ti-2Ta-.01B-.15Zr-1.1Y₂O₃-Bal Ni. The alloy contains 2.5 volume percent oxide particles of about 350 Å volumetric average diameter. In addition, the alloy contains approximately 50-55 volume percent γ' precipitates. The 1093°C rupture strength capability of this alloy compared with DS MAR-M200 + Hf and several nickel-base eutectic alloys is illustrated in Figure 1.

Although creep and rupture strength are prime requirements for turbine blade alloys, fatigue resistance is also an important factor. The general behavior of the alloy is described in this paper.

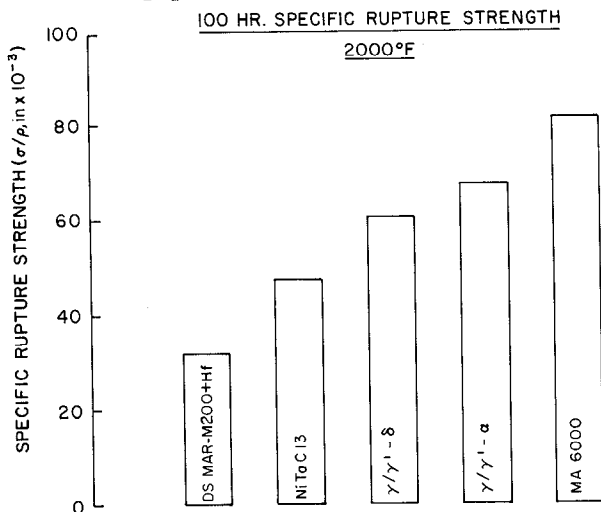


Figure 1. Comparison of the 1093°C/100-hour specific rupture strength of MA 6000E with DS MAR-M200 + Hf and several nickel-base eutectic alloys; NiTaC 13, γ/γ' - δ and γ/γ' - α .

EXPERIMENTAL PROCEDURE

MA 6000E powder was prepared by the mechanical alloying process(4). The raw materials used were as follows:

Nickel, Inco Type 123	- 4-7 microns (FSSS)
Elemental Chromium	- -200 mesh
Elemental Molybdenum	- -325 "

Elemental Tungsten	- -325	"
Elemental Tantalum	- -325	"
Titanium, Ni-17Al-28Ti	- -200	"
Aluminum, Ni-46Al	- -200	"
Boron, Ni-18B	- -200	"
Zirconium, Ni-28Zr	- -200	"
Yttria	- 250-400	Å

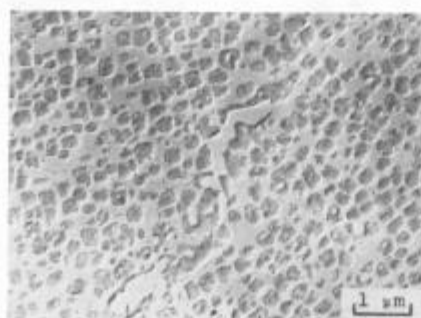
Mechanically alloyed powder was consolidated by extrusion. After screening to remove the coarse +12 mesh powder, the remainder was blended and sealed in 8.9 cm OD. mild steel cans. Extrusion to round bar was performed at 1038°C and 1010°C with ratios of 16:1 and 20:1. Metallographic examination of bar zone annealed at 1232°C showed that an essentially equivalent coarse, elongated grain morphology (GAR <10) was produced for all extrusions. The round bar was used for low cycle and high cycle fatigue tests.

Thermal fatigue tests were performed on bar extruded 7:1 at 1010°C and hot rolled a total of 50% at this same temperature. Zone annealing again provided a coarse, elongated grain morphology.

Prior to machining test specimens, bar and plate were heat treated: 1/2 hr/1232°C/AC + 2 hrs/954°C/AC + 24 hrs/843°C/AC. The microstructure of the alloy after zone recrystallization and heat treatment is illustrated in Figure 2.



(a)



(b)

Figure 2. Micrographs illustrating (a) the grain morphology and (b) the uniform oxide and Y dispersion in MA 6000E.

RESULTS

A. Low Cycle Fatigue

Fully reversed axial low cycle fatigue tests were performed in air at a frequency of 6 cpm using hour-glass shaped specimens and constant total diametral strain control. Test data showing the cycles to specimen fracture as a function of the computed longitudinal strain range applied for ambient temperature and 760°C are plotted in Figure 3. Although some scatter in data is apparent, the plot nevertheless indicates that the low cycle fatigue resistance of MA 6000E at room temperature and 760°C is similar for lives less than about 10^3 cycles. For lives greater than this, the room temperature fatigue resistance appears to be somewhat greater than that for 760°C. Fatigue fracture occurred in a transgranular mode at both test temperatures. The test data would indicate that the fatigue resistance of MA 6000E at 760°C is significantly better than that of the cast superalloy MAR-M200 in conventionally cast or directionally solidified form(5).

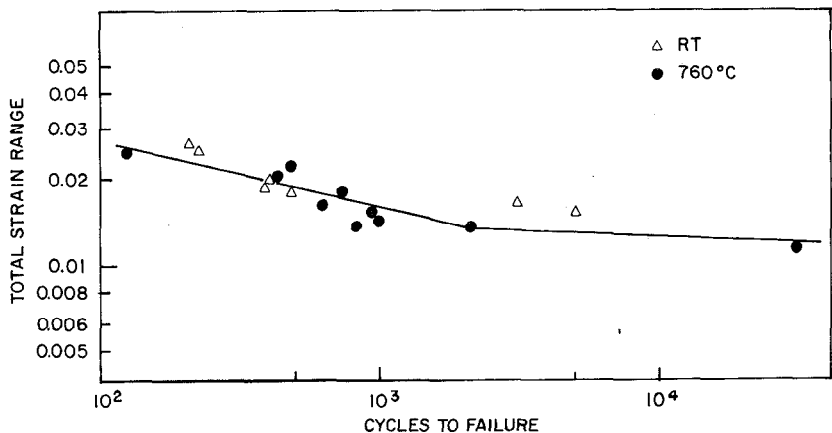


Figure 3. Low cycle fatigue behavior of MA 6000E.

By monitoring the changes in specimen load during the course of the fatigue tests, it was noted that at room temperature MA 6000E exhibits an initial period of cycle strain hardening, followed by strain softening. No cyclic hardening was observed at 760°C; only softening. The absence of hardening at 760°C is similar to that observed

for alloy 718(6,7). Other conventional γ' superalloys such as Waspaloy(6) and Udimet* 700(8) exhibit both cyclic hardening and softening at ambient and elevated temperature. Cyclic softening in γ' hardened superalloys has been attributed to shear of γ' and resultant disordering of the precipitates during the course of fatigue. However, evidence for shear of γ' precipitates in MA 6000E was not obtained. An examination of specimens tested at room temperature and 760°C showed that, in contrast to conventional superalloys(6, 9) at similar temperatures, the dislocations are homogeneously distributed (Figure 4).

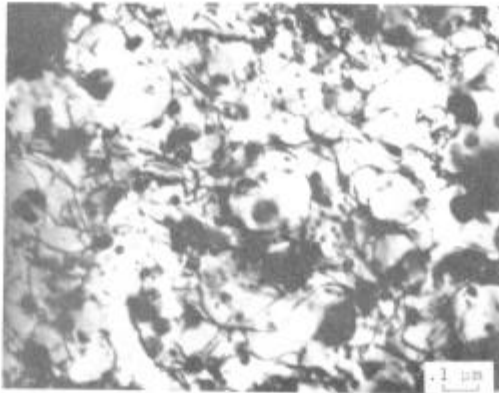


Figure 4. Micrograph illustrating the dislocation distribution after fatigue testing at 760°C. $E_T = 2\%$, $N_f = 423$ cycles.

B. High Cycle Fatigue

Rotating bending fatigue tests (R.R. Moore type) were performed at room temperature, 760°C and 982°C. A frequency of 8000 cpm was employed for tests at room temperature, while a lower frequency of 6000 cpm was used for tests at elevated temperature. Figure 5 showed plots of the fatigue data for the three test temperatures. The 10^7 fatigue strengths of MA 6000E are 676 MPa, 483 MPa and 287 MPa at RT, 760°C and 982°C, respectively. From a comparison with available data, it is noted that the fatigue strength of MA 6000E is substantially higher at all test temperatures than conventional wrought or cast superalloys.

A further feature is the high fatigue endurance ratio exhibited by MA 6000E. A comparison of the endurance ratio

*Trademark of Special Metals Corp.

at 10^7 cycles of MA 6000E and other superalloys is given in Table 1. Typically the values of endurance ratio at ambient temperature for conventional superalloys lie between .2 and .4, whereas that for MA 6000E is .52, a number usually associated with body centered cubic materials. Higher endurance ratios have been observed also for TD-Nickel (10) and MA 753(11) and would appear to be a characteristic of oxide dispersion strengthened alloys. It has been reported however, that high endurance ratios can be realized in conventional superalloys through thermomechanical processing which promotes ultra fine grain sizes(12).

Table 1. Comparison of Room Temperature Fatigue Endurance Ratio of MA 6000E with Other Superalloys.
 10^7 Cycle

Alloy	Fatigue Strength (MPa)	UTS (MPa)	Endurance	Ref.
MA 6000E	676.6	1290.3	.52	
Udimet 700	276.0	1407.6	.20	A
Waspaloy	303.6	1276.5	.24	A
INCONEL* Alloy 718	558.9	1390.4	.40	B
INCONEL* Alloy 706	499.5	1274.7	.39	B
MA 753	558.9	1161.1	.48	C

REF.:

- A Y. Nishiyama, et al, Jnl. Jap. Met. Soc., Vol. 38, 1974. p. 779.
- B Inco Brochures, No. 20M 2-68 T-39 and No. 20M 9-70 T-45.
- C J. H. Weber and M. J. Bomford, ASTM STP-520, 1972, p. 427

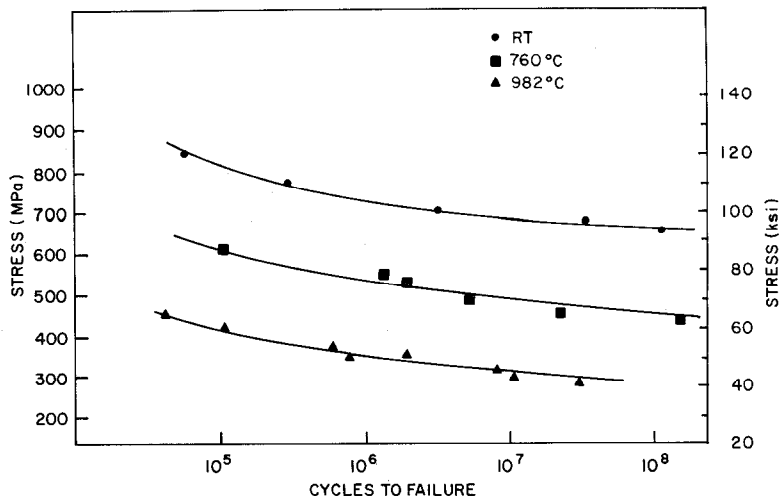


Figure 5. High cycle fatigue behavior of MA 6000E.

Metallographic observations of MA 6000E broken specimens revealed that, as in the case of low cycle fatigue, fatigue failure occurred through a transgranular crack propagation mode for all test temperatures. An optical micrograph of the fracture profile of a specimen tested at 982°C is shown in Figure 6. Scanning electron metallography of fracture surfaces revealed three different morphological regions. A general view of the surface of a specimen tested at room temperature, shown in Figure 7, indicates the crack initiation area, A, the stage II fracture zone, B, and the tensile rupture area, C. Higher magnification examination of the crack initiation area revealed a faceted appearance indicating that initial crack propagation occurs along well defined slip planes(6,9). In the stage II fracture zone, fine, regularly spaced striations are present. These are illustrated in Figure 8. The tensile rupture area showed a very fine "dimpled" appearance indicating that fracture occurs by a void coalescence mechanism. The absence of intergranular crack initiation and propagation at elevated temperatures is in contrast to that observed in most conventional superalloys.

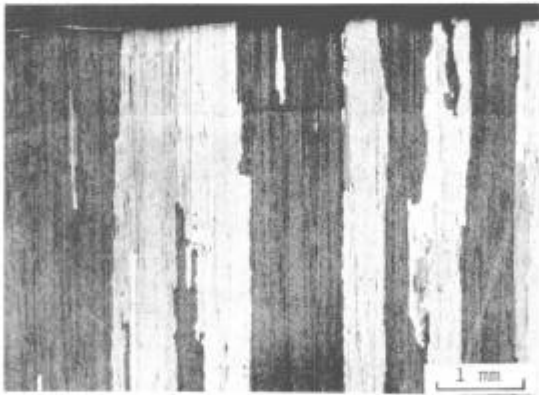


Figure 6: Fracture profile of specimen tested in high cycle fatigue at 982°C. $N_F = 1.98 \times 10^4$ cycles.

C. Thermal Fatigue

Fluidized bed thermal fatigue tests were performed in air using bed temperatures of 1088°C and 316°C and with an immersion time in each bed of three minutes. A double wedge specimen having edge radii of 0.1 cm and 0.07 cm was employed. Other details of specimen geometry have been described elsewhere(13). The tests were run at Illinois Institute of Technology Research Institute under contract to the NASA-Lewis Research Center. The comparative thermal fatigue resistance of uncoated and NiCrAlY coated MA 6000E

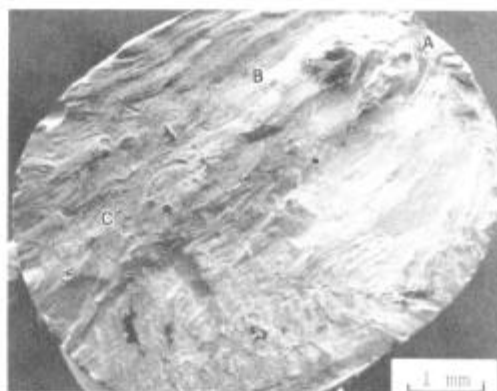


Figure 7. Scanning electron micrograph showing fatigue fracture surface of specimen tested at room temperature, $N_f = 3.35 \times 10^7$ cycles. A = crack initiation area; B = fatigue crack propagation zone; C = tensile rupture area.

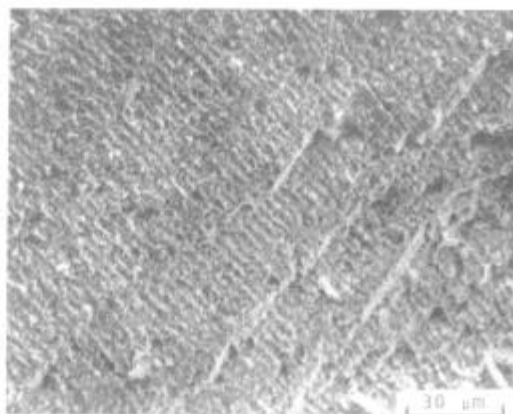


Figure 8. Scanning electron micrograph detailing fatigue striations observed in the crack propagation zone, B, of Figure 7.

and other nickel- and cobalt-base alloys was determined by monitoring the number of cycles required to initiate the first crack. For the specific test conditions, cracks initiated in uncoated MA 6000E after 10,250 cycles, whereas 12,750 cycles elapsed before cracks developed in the coated material. Typically, conventionally cast superalloys crack within 500 cycles, whereas several thousand cycles are required before cracks initiate in directionally solidified alloys. A comparison of MA 6000E with some other alloys is given in Table 2.

Table 2. Comparison of Thermal Fatigue of MA 6000E with Other Alloys

<u>Alloy</u>	<u>Cycles to 1st Crack</u>
MA 6000E	10,250
MA 6000E + NiCrAlY Coating	12,750
IN-738	100
MAR-M509	238
B-1900	400
DS IN-100	2,400
DS MAR-M200	2,450
DS MarM200 + NiCrAlY Coating	6,500
NASA TAZ-8A + RT-XP Coating	12,500
Single Crystal MAR-M200	15,000 Uncracked

DISCUSSION

The present study of MA 6000E has established that the alloy possesses excellent fatigue resistance, both mechanically and thermally imposed. The relatively poor fatigue properties of conventional γ hardened nickel-base superalloys have been attributed to a number of factors. These include the coarse planar deformation mode (at ambient and moderately elevated temperatures), the presence of defects such as coarse MC-type carbides, and porosity in cast alloys, which lead to easy crack initiation(9), and to intergranular crack propagation at elevated temperatures.

In the case of MA 6000E, the presence of a uniform dispersion of fine oxide particles promotes homogeneous fatigue deformation, thus preventing the concentration of slip and an early incidence of crack formation. Slip dispersal due to the oxide particles is probably the main reason why good fatigue properties are a feature of ODS type alloys(11,14). The ease of fatigue crack initiation is probably also reduced by the relatively smaller size of M(C,N) type carbonitrides in MA 6000E, compared with conventional superalloys. Another factor of importance is the elongated grain shape which prevents the intrusion of intergranular crack initiation and propagation as the test temperature is increased(5,9).

The excellent thermal fatigue properties of MA 6000E are somewhat surprising in view of the fact that it is considered that a (100) texture is needed for good thermal fatigue resistance. MA 6000E was tested with a (110) texture. It is reasoned, therefore, that, although a (100)

texture may provide enhanced resistance to thermal fatigue, the inherent alloy strength and advantageous microstructure, e.g., the highly elongated grains, are dominant factors.

CONCLUSION

MA 6000E was designed as a high strength, creep resistant alloy. It is evident from the results presented here, however, that the alloy also possesses excellent all round fatigue resistance. This is attributed to the unique combination of microstructural features which are generic to mechanically alloyed materials.

ACKNOWLEDGEMENTS

The authors acknowledge the technical assistance of T. W. Fuller during the course of this work. Discussions with T. K. Glasgow (NASA) and Inco personnel were appreciated. The experimental work was conducted under a NASA contract (NAS 3-20093).

REFERENCES

- (1) Decker, R. F. and Sims, C. T., "The Metallurgy of Nickel-Base Alloys", The Superalloys, (Wiley, New York, 1972, ed. by C. T. Sims and W. C. Hapell), p. 23.
- (2) Wilcox, B. A. and Clauer, A. H., "Dispersion Strengthening", Oxide Dispersion Strengthening, (Gordon and Breach, N.Y., 1968), p. 61.
- (3) Merrick, H. F., Curwick, L. R. and Kim, Y. G., "Development of Oxide Dispersion Strengthened Turbine Blade Alloy by Mechanical Alloying", NASA CR-135150, January 1977.
- (4) Benjamin, J. S., "Dispersion Strengthened Superalloys by Mechanical Alloying", Met. Trans., Vol. 1, 1970, p. 2943.
- (5) Leverant, G. R. and Gell, M., "The Elevated Temperature Fatigue of a Nickel-Base Alloy, MAR-M200, in Conventionally-Cast and Directionally-Solidified Forms", Trans. ASM, Vol. 245, 1969, p. 1167.
- (6) Merrick, H. F., "The Low Cycle Fatigue of Three Wrought Nickel-Base Alloys", Met. Trans., Vol. 5, 1974, p. 891.
- (7) Fournier, D. and Pineau, A., "Low Cycle Fatigue of INCONEL 718 at 298K and 823K", Met. Trans., Vol. 8A, 1977, p. 1095.
- (8) Wells, C. M. and Sullivan, C. P., "The Low Cycle Fatigue Characteristics of a Nickel-Base Superalloy at Room Temperature", Trans. ASM, Vol. 57, 1964, p. 841.

- (9) Gell, M., Leverant, G. R. and Wells, C. M., "The Fatigue Strength of Nickel-Base Superalloys", ASTM STP 467, 1969, p. 113.
- (10) Hane, R. K. and Wayman, M. L., "The Fatigue and Tensile Fracture of TD-Nickel", Trans. AIME, Vol. 239, 1967, p. 721.
- (11) Weber, J. H. and Bomford, M. J., "High Cycle Fatigue Properties of a Dispersion Strengthened Ni-Base Superalloy", ASTM STP 520, 1972, p. 427.
- (12) Brown, E. C., Boettner, R. C. and Runkle, D. C., "MINIGRAIN Processing of Nickel-Base Alloys", Super-alloys-Processing, Proceedings of the 2nd International Conference, 1972, MCIC-72-10, p. L1.
- (13) Bizon, P. T. And Spera, D. A., "Comparative Thermal Fatigue Resistances of Twenty-Six Nickel- and Cobalt-Base Alloys", NASA TN D-8071, October 1975.
- (14) Glasgow, T. K., "An Oxide Dispersion Strengthened Alloy for Gas Turbine Blades", NASA TM 79088, April 1979.