E-9840

SHEAR RUPTURE OF A DIRECTIONALLY SOLIDIFIED EUTECTIC γ/γ' - α (Mo) ALLOY

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ABSTRACT

Directionally solidified γ/γ' - α (Mo) eutectic alloys are being evaluated for application as advanced aircraft engine turbine blades. Their excellent high temperature strength is partly due to their directionally aligned microstructure. However, alloys with such directional structures may display low shear strength at 760° C, the operating temperature of advanced blade roots. The objective of this investigation was to determine the shear rupture strength of the γ/γ' - α eutectic alloy and to possibly improve it by microstructural and heat treatment variations. Bars of γ/γ' - α alloy containing nominally 5.7% Al and 33.5% Mo by weight with balance Ni were directionally solidified at rates between 10 and 100 mm per hour. Materials were solidified in furnaces with thermal gradients at the liquid-solid interface of 250 or 100° C per cm. A limited number of longitudinal shear rupture tests were conducted at 760° C and 207 MPa in the as - solidified and in several heat treated conditions. It was found that the shear rupture failures are partly transgranular and that resistance to failure is promoted by good fiber alignment and a matrix structure consisting mainly of γ'. Well aligned as-solidified specimens sustained the shear stress for an average of 81 hours, while cellular material failed in one hour or less. A simulated coating heat treatment appeared to increase the transformation of γ to γ' and raised the average shear life of aligned specimens to 111 hours. However, heat treatments at 1245° C and especially at 1190° C appeared to be detrimental by causing partial solutioning of the γ', and reducing lives to 47 and 10 hours, respectively.

INTRODUCTION

Substantial improvements in the performance of aviation gas turbine engines could be achieved by the development of turbine blade alloys with resistance to long, repeated exposures at stresses and temperatures superior to that of existing superalloys. Certain directionally solidified eutectic alloys appear to have the properties needed to improve engine performance and among the most promising are the γ/γ' - α alloys.

A promising directionally solidified eutectic alloy has been developed under NASA contract by the General Electric Company (refs. 1 and 2). It is nickel base with 5.7% Al and 32.3% Mo by weight. This alloy upon solidification from the melt forms two phases, α, which is mainly Mo, and γ, a Ni solid solution. The α phase, under favorable directional solidification gradients and rates, forms fibers with parallel axial alignment which can extend the full length of elongated γ grains. From the γ, upon further cooling, γ', nominally Ni_3Al, precipitates. The alloy, as shown in table I, has excellent high temperature tensile and stress rupture properties in both longitudinal and transverse directions. Its high temperature ductility is superior to that of other directionally solidified eutectic alloys (ref. 1). These properties make it a prime candidate for advanced turbine blades.

The most advanced turbine blade alloy in current use is directionally solidified Mar-M200+Hf. This alloy at 960° C has a stress rupture life of 1000 hours at 150 MPa (ref. 3). For comparison, the $\gamma/\gamma' - \alpha$ alloy at this stress displays the same life at the higher temperature of 1010° C. At 760° C both alloys will withstand without rupture a stress of 600 MPa over 1000 hours.

Temperatures up to 760° C are anticipated in advanced blade roots. Shear stresses, mainly due to centrifugal loading, are a critical factor in this portion of the blade (refs. 4 and 5). The magnitude of these stresses depends on the root configuration, the design of which must take into consideration the material properties. In directionally solidified eutectic alloys, such designs are complicated by the anisotropy of properties, which are always better along the axis of solidification than transverse to it, and which do not favor shear loading. The work described in this paper was performed with a $\gamma/\gamma' - \alpha$ alloy based on the composition developed in reference 1. It was intended to determine the tensile shear rupture properties of the alloy and to investigate whether they could be modified and possibly improved by heat treatment.

EXPERIMENTAL PROCEDURE

Master Melts

All master melts were prepared from elemental constituents with a purity better than 99.8 percent. The charge composition contained 33 to 34% Mo, 5.7% Al, balance Ni. The molybdenum content was raised over that of the alloy in reference 1 to overcome melt losses. Charges of 1.5 kg were melted in 0.4 cubic decimeter zirconia crucibles. The melt chamber of the vacuum furnace was evacuated to about 1 Pa (10 μm Hg) and backfilled with argon. The initial charge was nickel to which molybdenum and aluminum were added. The melts were stirred by induction to effect thorough mixing and poured at about 1425° C. The zirconia shell molds, embedded in a fire-clay filler, had been preheated to about 875° C. These molds contained a cluster of 6 or 8 remelt bars, each 10 mm in diameter and about 130 mm long. The test specimens used in this program represent 3 master melts, all nominally of the same composition. Additional material, furnished by the General Electric Company was also used in this program; the procedure for the preparation of its master melt is reported in reference 1.

Directional Solidification

The directional solidification was performed in a modified Bridgman furnace which was induction heated by a coil connected to a radio frequency generator (fig. 1). The remelt ingots were melted in high purity alumina crucibles of 12.5 mm inside diameter which were heated by radiation from a graphite susceptor. Argon flowed through the apparatus at 0.5 cubic meter per hour and protected the melt from oxidation. A thermal gradient, previously determined to be about 250° C per cm at the liquid-solid interface (ref. 6), was generated by a water spray at the base of the furnace setup. The solidified bars were withdrawn from the furnace by a controlled screw mechanism at rates of 10, 20, 30, 40 and 100 mm per hour. Optical pyrometry was used to monitor the temperature of the top of the melt and to assure melting of the entire remelt bar prior to starting the withdrawal. The material supplied by the General Electric Company had been directionally solidified at 20 mm per hour in a gradient of 100° C per cm as a 42 mm diameter bar (ref. 1). From this bar a half-round cylindrical section about 110 mm long was machined into three bars of about 12 mm in diameter.

Microstructural Evaluations

Each directionally solidified bar was surface ground along its length to provide a flat surface about 7 mm wide. This surface was then polished and the unetched surface examined by optical microscopy. The aligned structure was rated according to the standards developed by Henry and Jackson (ref. 2). Cross-sections and shorter longitudinal sections which were subjected to metallographic examinations for other purposes were mounted in bakelite and given a final polish with alumina slurries. Etching for optical microscopy was by swabbing with a solution of 1 g $CuCl_2$ in 80 ml ethanol, 20 ml concentrated HCl and 1.5 ml concentrated HNO_3. Specimens examined by electron microscopy were etched by swabbing with a solution of 3.5 g $FeCl_3$ and 3 g $CuCl_2$ in 45 ml concentrated HCl, 15 ml concentrated HNO_3 and 20 ml water. The fiber volume fracture was determined essentially according to ASTM standard E562-76 (ref. 7) by averaging 64 point counts of 100 intercepts each on replica electron micrographs. The orientation of fiber and matrix phases was determined by a X-ray diffractometer scan.

Heat Treatments

Specimens were tested in the as-directionally solidified (DS) or heat treated conditions. All heat treatments were performed prior to machining of the specimens. Four different heat treatments were used on selected specimens. Preliminary examination of shear rupture failures had indicated that the fracture path was at least partly transgranular. Therefore, solutioning treatments which would change the γ' distribution and morphology were evaluated to determine whether such changes would alter the fracture path and improve the shear strength. Two solutioning treatments of one hour at 1245° C (HT1) or at 1190° C (HT2) with water quenching were investigated. Since at its projected use temperature the alloy is not inherently oxidation resistant, it will be necessary to protectively coat blades made from it. Therefore, one heat treatment simulated the typical thermal exposure which blades would undergo during coating. This heat treatment consisted of 4 hours at 1095° C with air cooling plus 24 hours at 900° C with air cooling (CHT). Finally, the fourth heat treatment was a combination of the solutioning treatment at 1245° C, followed by the simulated coating heat treatment (COMB). The furnace atmosphere for all heat treatments was argon.

Mechanical Tests

The mechanical tests performed in this program were in longitudinal tensile shear rupture at 760° C. The specimen used for the tests is shown in figure 2(a). In most cases two specimens were machined from each directionally solidified bar. To assure a uniformly aligned structure and eliminate end effects, the button-head test sections of all specimens were located head-to-head in the middle of the bars (fig. 2(b)). The GE-furnished bar yielded 6 specimens in 3 adjacent head-to-head locations. This allowed for a direct comparison of the properties of adjacent specimens given different heat treatments, with a minimum of possible differences in solidification rates, composition, and as-solidified microstructure. The machined specimens were inspected for integrity and parallelism of the button-head test surfaces. Special care was taken to assure cleanliness of the test fixture holding the specimens, and proper alignment of the specimens in the apparatus. All shear-rupture tests were performed in air. The specimens were heated to 760° C in 3 to 5 hours and then held at temperature 1 to 2 hours before loading to 207 MPa. The applied load was calculated from the area formed by the shaft circumference and the axial length of the button-head test section. Typical failures for this test are shown in figure 2(c). The tests comprised the following numbers of specimens: 12 DS, 9 CHT, 8 HT1, 2 HT2, and 6 COMB. When early evidence indicated that a particular solidification rate (100 mm/h) or heat treatment (HT2) was definitely detrimental, no further specimens were so processed.

RESULTS AND DISCUSSION

The results of chemical analyses for the four master melts are given in table II. The analyses show that the compositions are all very similar. The metallographic examination of the as-solidified bars showed that the bars solidified at rates of 10, 20, 30, and 40 mm per hour had a longitudinal α fiber structure consisting of fibers with perfectly even distribution (A rating) or distributed in patterns of varying density (B rating). However, the one bar solidified at 100 mm per hour had a cellular microstructure (D rating) (ref. 2). Typical as-solidified microstructures are shown as transverse sections in figures 3(a) and (b), and as longitudinal sections in figures 3(c) and (d). The α phase (Mo) fibers, shown in figure 3(d), which occupy 24 percent of the volume in the γ/γ' matrix, are faceted (fig. 3(b)) and extend along the entire length of individual grains. At the grain boundaries, the fiber phase typically forms narrow lamellae (fig. 3(a)). The fibers have a <100> orientation along the axis of solidification. The γ and γ' phases of the matrix in the as-directionally solidified and all heat treated conditions similarly show a preferred <100> orientation.

The results of the shear rupture tests are presented in table III. The specimens are grouped by their nominal solidification rates. The effects of the various parameters on shear rupture are discussed below.

Heat Treatments

The overall average of the shear rupture lives for all as-solidified (DS) specimens was 81 hours. For heat treated specimens in the CHT condition the average life was 111 hours, and in the COMB condition 117 hours. Solution heat treatment averages were 47 hours for HT1, and 10 hours for HT2 conditions. These overall average include a large amount of scatter as is evident in table III, presumably due to undetected differences in the master melts, as will be discussed below.

A clearer comparison of the effects of the heat treatments is obtained by matching the results from the specimens cut from the same bars. As discussed above, specimens were cut head-to-head in the NASA-solidified bars or from adjacent head-to-head locations in the GE-furnished bar to minimize potential differences in variables, such as solidification rate, microstructure and composition. These same-bar comparisons are shown in figure 4. The data show that the simulated coating heat-treatment (CHT) generally results in longer shear rupture lives. In same-bar specimens the average life of CHT specimens is 118 hours against 71 hours for specimens in the as-solidified (DS) condition. CHT also appears superior to the HT1 solutioning heat treatment at 1245° C, averaging 95 against 75 hours. Compared to COMB (the combination of solution treatment at 1245° C followed by the simulated coating heat treatment) CHT specimens average 136 against 86 hours. The COMB heat treatment, however, displays an average life of 84 hours versus 56 hours for DS specimens. Compared against the DS condition, the HT1 heat treatment causes an average life decrease from 75 to 27 hours. The HT1 heat treatment is also detrimental when compared in same-bar specimens with the COMB condition, where it averages 9 hours against 133. The HT2 solutioning heat treatment was only given to two specimens and, due to the short lives obtained, no further tests were performed with specimens in this condition.

The resistance to shear rupture appears to be related to the amount and distribution of γ' present in the matrix, as is shown by the microstructures of tested specimens in figures 3(b) and 5(a) through (d). In the as-solidified structure (fig. 3(b)), the fibers are surrounded by γ' in a more or less continuous network of γ. By the simulated coating heat treatment the continuity of the network of γ has been broken up and pockmarked by transformation to γ' (fig. 5(a)). Heat treatment at 1190° C causes partial solutioning throughout the γ' (fig. 5(b)), and the fibers are now in contact with γ and γ' which shows evidence of solutioning. This structure resulted in the shortest shear rupture life. At the higher solutioning temperature at 1245° C (fig. 5(c)), most of the matrix has become γ phase, while much of the γ' is now agglomerated around the fibers. Subjecting this structure to the simulated coating heat treatment restores good shear resistance and the structure is now a network of γ islands in a matrix of γ' (fig. 5(d)). It therefore appears that the shear rupture resistance depends on the relative amounts and the morphology of the γ and γ' present, and may be affected by the quality of the fiber - matrix interface, which may possibly be strongest when it is purely $\alpha - \gamma'$.

The failure itself is a mixture of transgranular and intergranular modes. Detailed metallographic examinations of the transgranular portion of the failure did not conclusively reveal whether the fracture followed a preferred path through γ, γ' or the fiber-matrix interface.

Master Melts

A significant difference in the shear rupture lives was found between materials of different master melts. Overall the longest lives were obtained with specimens from master melt 12, while specimens from master melts 1 and the GE-furnished bar ranked lowest. No microstructural differences could be detected between the master melts, all structures being similar to those shown in figure 3. No conclusions as to the difference in lives could be drawn on the basis of the chemical analyses of the master melts (table II).

Solidification Rates

Significant differences in shear-rupture life were observed between materials with aligned and cellular structures. The two cellular specimens, solidified at the rate of 100 mm per hour failed very early (1 hr or less) during the tests, while the specimens with aligned structure, similarly heat treated, displayed lives greater by 2 to 3 orders of magnitude. On the other hand, the life of well aligned specimens solidified between 10 and 40 mm per hour could not be correlated with solidification rates. In the as-solidified condition, specimens of all master melts from nominal rates of 10, 20, 30, and 40 mm per hour had average lives of 91, 66, 108, and 133 hours respectively, while for master melt 12 the lives were 96, 177, 108, and 113 hours, respectively. For bars of all the master melts which were given the simulated coating heat treatment the lives were an average of 72, 84, 245, and 125 hours, respectively. Note that for the averages at 20 mm per hour a bias may have been introduced by the inclusion of two master melts (1 and GE-furnished), which are not represented at other rates. Microstructurally no consistent differences could be detected in fiber cross - section or spacing among specimens solidified at rates between 10 and 40 mm per hour.

Comparison with Other Alloys

A related alloy has been shear rupture tested by Pearson and Lemkey (ref. 8). This alloy, containing 29% Mo and 7.5% Al, has a matrix that is mostly γ', and the lives of specimens tested at 760° C and 207 MPa generally exceeded 150 hours in the as-solidified condition. The results of the present tests should also be compared with those reported by Sheffler, et al. (ref. 3) for $\gamma/\gamma' - \delta$ and B-1900 which were tested with Iosipescu type creep-shear specimens. While B-1900 had a life of over 10 000 hours under these conditions, $\gamma/\gamma' - \delta$ was not capable of sustaining a stress of 207 MPa. This is one of the principal reasons why $\gamma/\gamma' - \delta$ alloy, which otherwise displays outstanding high temperature tensile and rupture properties, appears to have limited usefulness for blade applications. The present tests show that $\gamma/\gamma' - \alpha$ which is stronger in stress rupture than $\gamma/\gamma' - \delta$ at airfoil operating temperatures, also has greater promise due to the increased shear rupture resistance in the blade root region.

CONCLUDING REMARKS

The directionally solidified $\gamma/\gamma' - \alpha$ eutectic alloy with 33 to 34% Mo and 5.7% Al by weight has good high temperature longitudinal and transverse tensile and rupture strength. In the present investigation it was found that the shear rupture properties of this alloy exceed those of the $\gamma/\gamma' - \delta$ alloy. It could be considered for use in present aircraft engines. However, in advanced engines, where higher design stresses are anticipated, the design of blade roots may have to accommodate certain shear rupture limitations. When the alloy in its directionally solidified form is used for turbine blades, a protective coating will be necessary to protect the airfoil portions from excessive oxidation. Indications are that a thermal treatment, such as is used to apply coatings will increase the shear rupture resistance of the alloy. A more detailed test program is necessary to statistically confirm the apparent heat treating effects on the shear rupture strength. Such a program should also determine how such a heat treatment will affect the tensile and stress rupture properties of the alloy.

SUMMARY OF RESULTS

A nickel base alloy containing by weight 33 to 34% Mo and 5.7% Al was directionally solidified at 10, 20, 30, 40 and 100 mm per hour in a high thermal gradient. Specimens of this alloy were shear rupture tested at 760° C and 207 MPa. Limited data show the following results:

1. Specimens with good fiber alignment had an average shear rupture life of 81 hours in the as-solidified condition. The microstructure of this alloy shows α fibers surrounded by γ' in a nearly continuous network of γ.

2. The shear rupture life was increased to an average of 111 hours by a simulated coating heat treatment of 4 hours at 1095° C with air cooling plus 24 hours at 900° C with air cooling. This heat treatment caused the network of γ to dissolve into islands pockmarked with γ'.

3. Solution treatments of one hour at 1245° C or 1190° C generally reduced the shear rupture life significantly to averages of 47 and 10 hours respectively. These heat treatments dissolved most of the γ' in the matrix.

4. Specimens given the 1245° C solution heat treatment followed by the simulated coating heat treatment displayed an average life of 117 hours. However, when specimens cut from the same bar were compared, the specimens with the combination heat treatment had shorter lives than those with the simulated coating heat treatment only. The matrix of the specimens with the combination heat treatment had a microstructure similar to that of specimens given the simulated coating heat treatment only.

5. Alignment of fibers is an important factor in shear rupture life. Good alignment was obtained at solidification rates up to 40 mm per hour and no consistent relationship between solidification rates and shear rupture lives was observed. However, at 100 mm per hour, the α phase formed a cellular structure, and failure occurred in 1 hour or less.

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TABLE I. - SELECTED PROPERTIES OF A DIRECTIONALLY SOLIDIFIED $\gamma/\gamma' - \alpha$ EUTECTIC ALLOY (REF. 1)

[Composition, wt %: 32.3 Mo, 5.7 Al, bal Ni. Density 8.65 g/cm^3.]

	Tensile properties			
	Temperature, °C	UTS MPa	0.2%YS MPa	Elongation, %
Longitudinal	750 900 1100	1100 890 450	975 880 440	17 26 40
Transverse	750 1100	925 280	890 265	17 80
	Longitudinal tensile shear properties			
	Temperature, °C	Ultimate shear strength, MPa		
	750 1100	618 193		
	Stress rupture properties			
	Temperature, °C	Stress, MPa	Avg. life, hr	Elongation, %
Longitudinal	750 750 900 1100 1100	700 800 375 110 125	510 84 88 154 50	24 16 47 17 18
Transverse	750 900 1000	600 250 100	26 14 20	2 6 2

TABLE II. - CHEMICAL ANALYSIS OF MASTER MELTS

Master melt	Weight percent		
	Mo	Al	Ni
1	34.39	5.57	Bal
7	33.03	5.53	Bal
12	34.32	5.78	Bal
GE	33.07	5.73	Bal

TABLE III. - SHEAR-RUPTURE LIFE AT 760° C UNDER 207 MPa STRESS

Identification		Solidification rate, mm/hr	Microstructural rating (ref. 2)	Rupture life, hr(1)				
Master melt	Bar			DS	CHT	HT1	HT2	Comb
7	G	11	A	86	70			
12	G	10	A		74	215		
12	J	9	A	96				106
1	R	20	A	8.5		29		
1	M	19	A	58	89			
1	S	20	A			0.8		163
1	T	20	A		51			23
7	E	20	A	42			(3)	
7	K (2)	20	A	97				
12	A	20	A	177		78		
GE	(4)	20	A	13.3	113	0.3		61
				67		9.8		
12	E	29	A		245			176
12	H	29	A	108		18.5		
7	H (2)	37	B				19.1	
7	L (2)	40	A-B		46			
12	C	40	A-B			25		174
12	F	39	A-B	133	203			
7	J	100	D	0.1	1.0			

(1) Condition
DS - as directionally solidified
CHT - 1095° C/4 h/AC+900° C/24 h/AC (simulated coating heat treatment)
HT1 - 1245° C/1 h/WQ
HT2 - 1190° C/1 h/WQ
Comb - 1245° C/1 h/WQ+1095° C/4 h/AC+900° C/24 h/AC

(2) One specimen only tested from this bar

(3) Failed on loading

(4) 6 specimens from 3 adjacent 12-mm diameter bars machined from a 42-mm diameter bar LDB259 directionally solidified by General Electric Company.

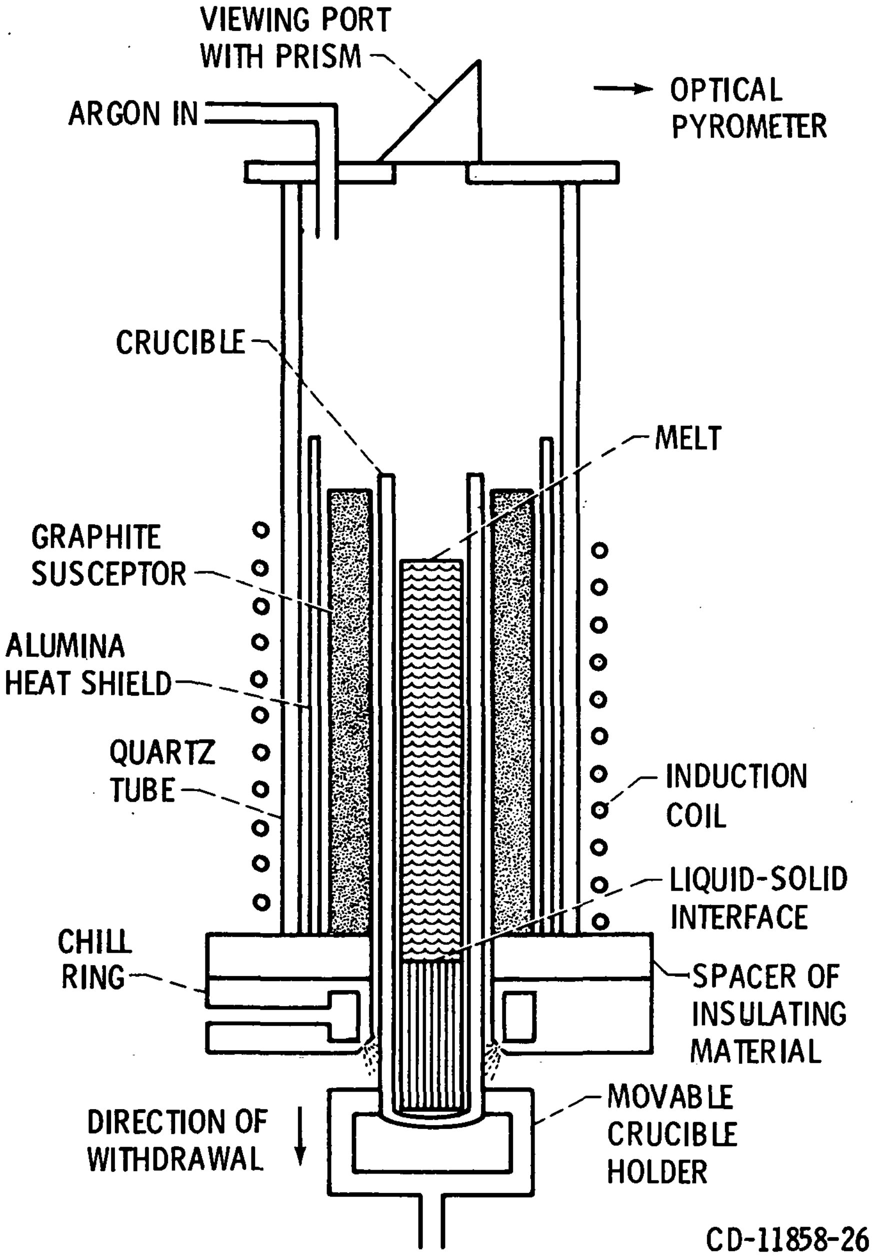
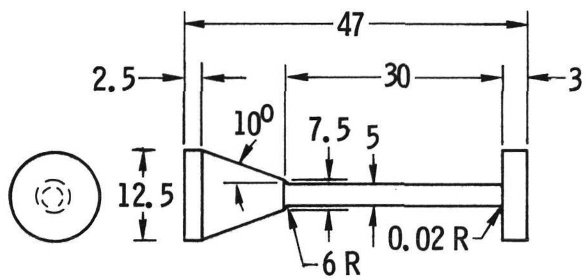
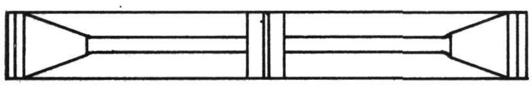


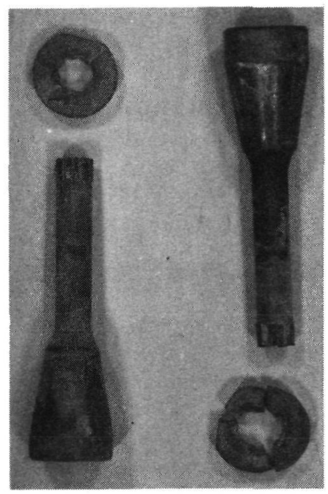
Figure 1. - Schematic representation of directional solidification apparatus.



(a) SKETCH SHOWING DIMENSIONS (IN mm).

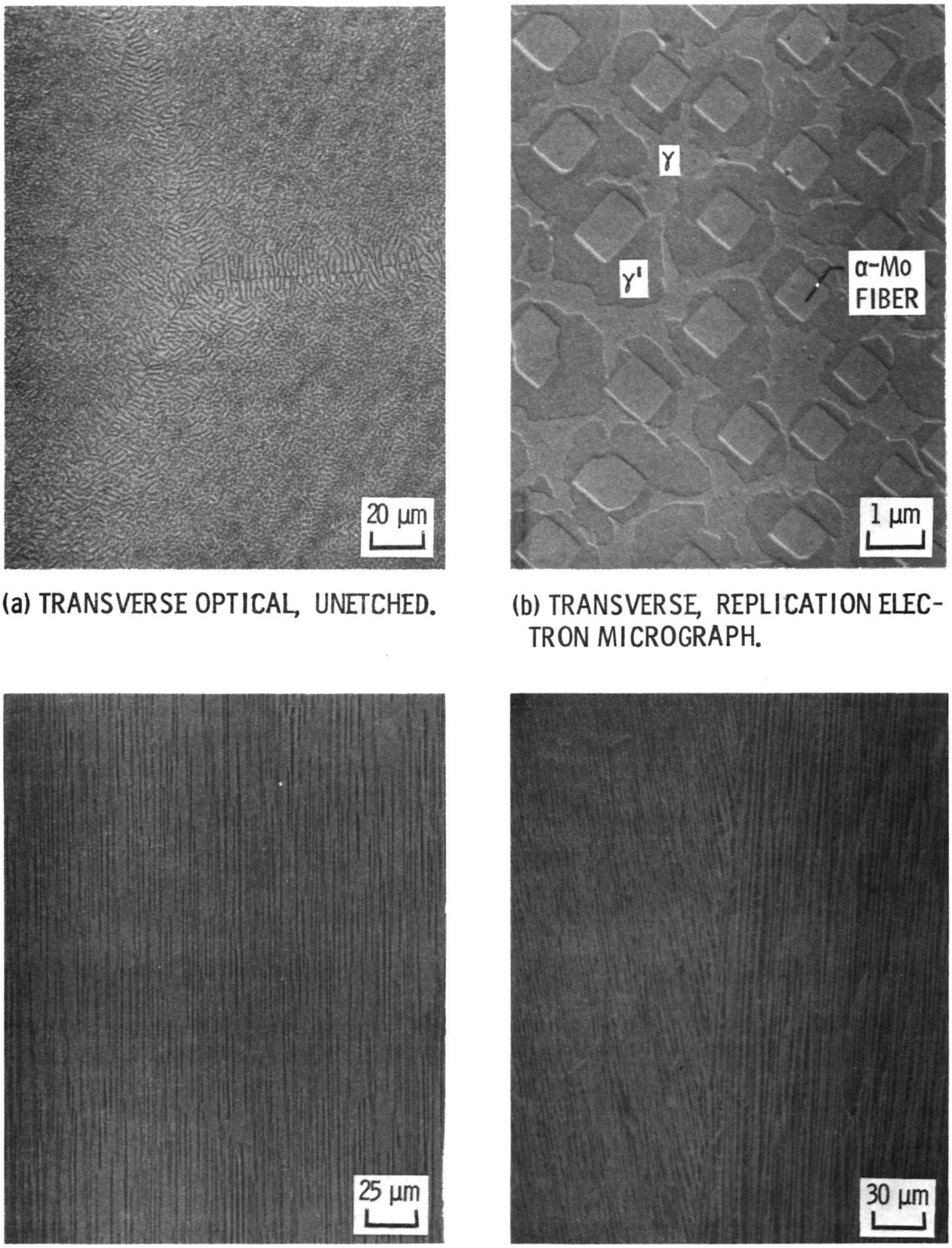


(b) LAYOUT FOR MACHINING TWO HEAD-TO-HEAD SPECIMENS FROM INDIVIDUAL DIRECTIONALLY SOLIDIFIED BAR.



(c) TYPICAL FAILURES.

Figure 2. - Specimens for shear rupture tests.



(a) TRANSVERSE OPTICAL, UNETCHED.

(b) TRANSVERSE, REPLICATION ELECTRON MICROGRAPH.

(c) LONGITUDINAL OPTICAL, UNETCHED.

(d) LONGITUDINAL, SCANNING ELECTRON MICROGRAPH.

Figure 3. - Typical microstructures of as-directionally solidified γ/γ'-α eutectic alloy.

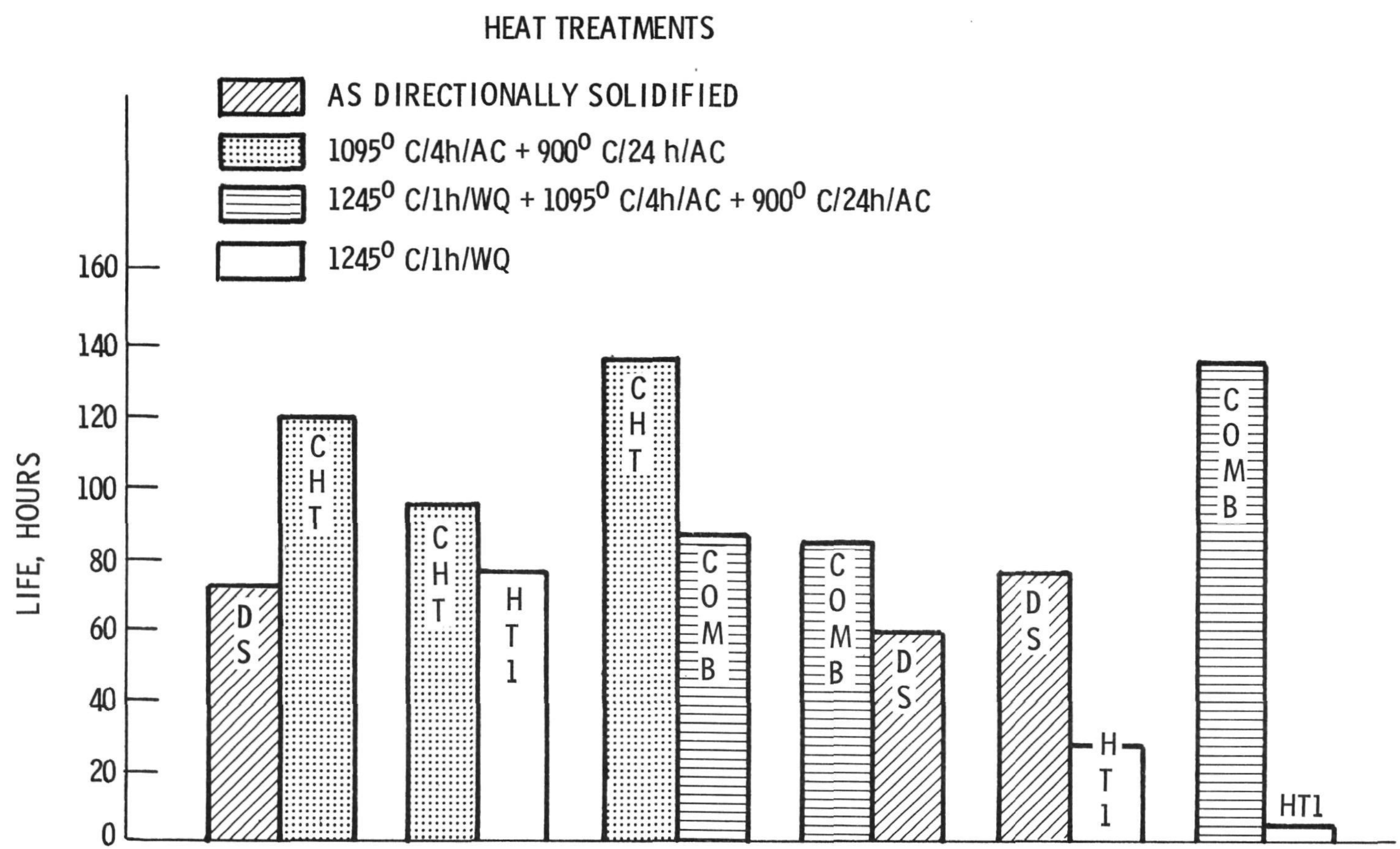
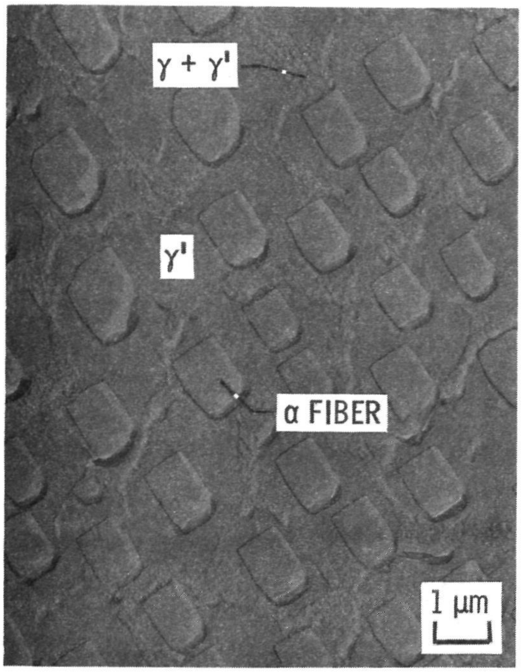
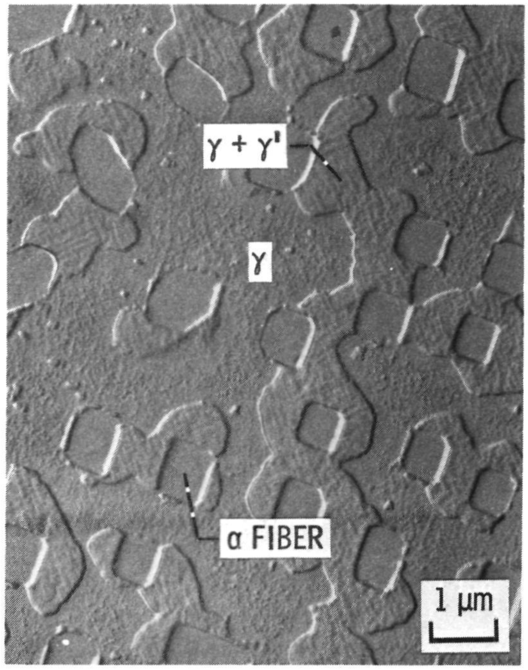


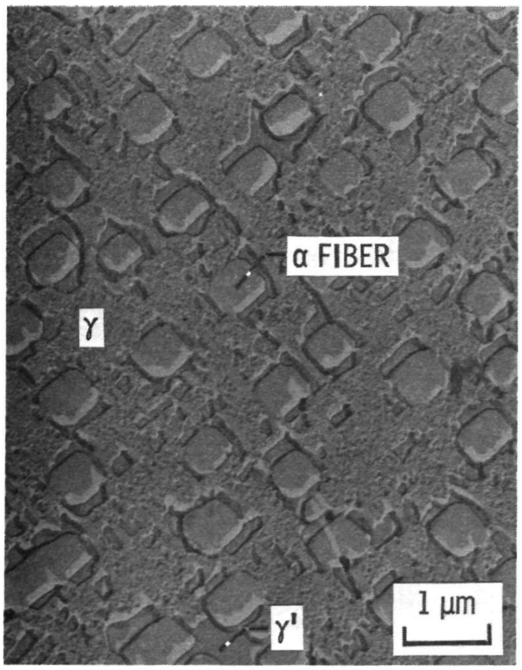
Figure 4. - Comparisons of average shear rupture lives of directionally solidified γ/γ' - α alloy at 760^0 C and 207 MPa. (Average based on 2 to 5 tests for each condition.)



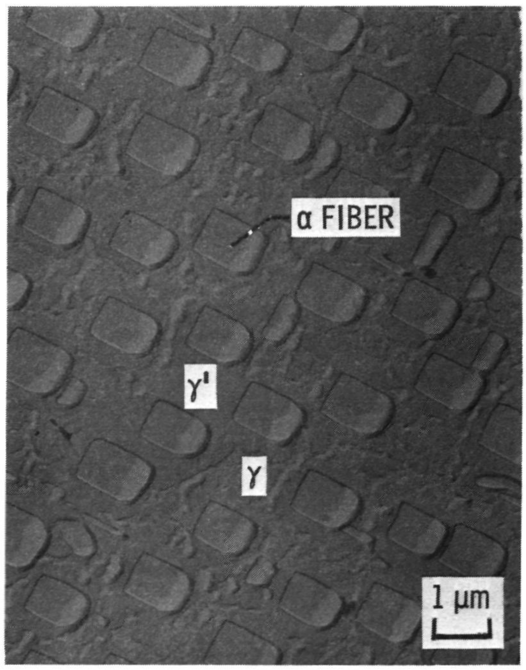
(a) 1095^{0} C/4 h/AC + 900^{0} C/24 h/AC.



(b) 1190^{0} C/1 h/WQ.



(c) 1245^{0} C/1 H/WQ.



(d) 1245^{0} C/1 h/WQ + 1095^{0} Cα h/AC + 900^{0} C/24 n/AC.

Figure 5. - Electron micrograpns by replication of directionally solidified γ/γ'-α eutectic alloy, heat treated.

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15. Supplementary Notes

16. Abstract

Directionally solidified γ/γ' - α (Mo) eutectic alloys are being evaluated for application as advanced aircraft engine turbine blades. Their excellent high temperature strength is partly due to their directionally aligned microstructure. However, alloys with such directional structures may display low shear strength at 760° C, the operating temperature of advanced blade roots. The objective of this investigation was to determine the shear rupture strength of the γ/γ' - α eutectic alloy and to possibly improve it by microstructural and heat treatment variations. Bars of γ/γ' - α alloy containing nominally 5.7% Al and 33.5% Mo by weight with balance Ni were directionally solidified at rates between 10 and 100 mm per hour. Materials were solidified in furnaces with thermal gradients at the liquid-solid interface of 250 or 100° C per cm. A limited number of longitudinal shear rupture tests were conducted at 760° C and 207 MPa in the as-solidified and in several heat treated conditions. It was found that the shear rupture failures are partly transgranular and that resistance to failure is promoted by good fiber alignment and a matrix structure consisting mainly of γ'. Well aligned as-solidified specimens sustained the shear stress for an average of 81 hours, while cellular material failed in one hour or less. A simulated coating heat treatment appeared to increase the transformation of γ to γ' and raised the average shear life of aligned specimens to 111 hours. However, heat treatments at 1245° C and especially at 1190° C appeared to be detrimental by causing partial solutioning of the γ', and reducing lives to 47 and 10 hours, respectively.

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