PROCESSING EFFECTS ON THE PROPERTIES OF P/M RENE 95 NEAR NET SHAPES

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A series of powder metallurgy Rene 95 near net shapes were evaluated to determine the effect of varying structural conditions on properties. The material was produced by combining a unique consolidation process $\{CAP^R\}$ and thermomechanical hot working procedures. In addition to structure, the effects of powder mesh size, test temperature, and specimen size variations were evaluated. Principal emphasis was accorded to LCF testing. Results show little effect of the variables studied on tensile and stress rupture properties. The effect of structure on LCF life was very significant and much more critical than other factors studied. The results indicate that CAP^R , an alternate process to HIP, combined with thermomechanical rolling and/or isothermal forging can produce quality product with a high LCF defect tolerance level.

INTRODUCTION

Numerous programs are being conducted to increase the consistency and reliability of hot isostatically pressed Rene 95 parts by determining and eliminating factors affecting low cycle fatigue (LCF) life. Heavy emphasis has been given to improving powder cleanliness because of the association of LCF failures to inclusions. Some progress, at the sacrifice of cost, has been made by using finer mesh (-150 inclusive) powder. This approach has increased the consistency of as HIP LCF life, but is not compatible with the original purpose of applying P/M technology--cost reduction. The original intent of the P/M process to reduce costs by minimizing material input is defeated by improving LCF properties via increased costs due to lower powder yields, stringent cleanliness requirements and tighter quality control procedures.

The purpose of this effort was to study an alternative approach to the manufacture of Rene 95 near net shape parts

while maintaining the ultimate goal of performance at minimum cost. P/M technology, using a unique consolidation process, was combined with traditional hot working technology to produce structurally controlled parts that were evaluated for performance. An attempt was made to generate structures that would offer more tolerance to typical defects without employing costly production steps designed to minimize their presence. Evaluation placed emphasis of LCF life as affected by structure, powder mesh size, and LCF test temperature, strain ratio and specimen configuration. Included in the evaluation were an identification of defect location, defect size and SEM analysis of crack initiators. Structural effects on tensile and stress rupture properties were also evaluated.

PROCEDURE

Materials and Processing

Material from four different heats of Rene 95 were used in this study. Powder from each of these compositions was produced by the argon atomization process. The identification and chemical analysis of each Rene 95 heat is shown in Table 1. The specification for this nickel base alloy also requires an oxygen maximum of 150ppm. Oxygen levels in these four heats ranged from 96 to 126ppm. Since one of the factors to be studied in this investigation was mesh size, three of the four heats were screened to -60 mesh inclusive, while the fourth was screened to -150 mesh inclusive.

Powder preforms were prepared using the CAP^R (Consolidation by Atmospheric Pressure) process which involves the consolidation of powder via sintering in a glass container. Screened powder is chemically treated to activate powder particle surfaces utilizing a minor chemical additive common to the alloy composition. The treated powder is placed in a

Table 1.	Rene	95	Compositions
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	.04	<u>Cr</u> 12.00	3.30	3.30	Mo 3.30	7.00	<u>Ti</u> 2.30	3, 30	.006	<u>Zr</u>
Spec.	.09	14.00	3.70	3.70	3.70	9.00	2.70	3.70	.015	.07
KR443 KR464	.05	13.05 13.25	3.48	3.56	3.51	8.08	2.53	3.57	.008	.07
KR471	.06	12.83	3.50	3.55	3.55	7.82	2.53	3.52	.008	.05 .05
KR498	.06	13.48	3.42	3.70	3.48	7.78	2.55	3.60	.009	.05

shaped glass container, evacuated, sealed, and then sintered at a temperature and time determined by the alloy and the mass being consolidated. During the hold at temperature, the glass container softens and the resultant atmosphere to vacuum pressure differential causes the container to be formed inward. Consolidation occurs as a result of powder particle diffusion. Isostatic atmospheric pressure produces a preform which is shape consistent with the original glass mold to densities of 98 to 99+ percent.

Final consolidation of P/M CAP^R preforms can be achieved via a subsequent hot working operation (forging or rolling), or by hot isostatic pressing. Preforms in this study were consolidated to full dense near net shape parts via both rolling and forging. The structure of Rene 95 is normally controlled by a recrystallization heat treatment and controlled reductions during the hot working operation. Since one of the primary objectives of this study was to determine the effect of structure on properties, variations in both recrystallization temperature and reduction were employed on the preforms processed to a disc configuration. All material was given a common heat treatment involving solution treatment at 1093°C (2000°F) for one hour, quenching in oil and aging at 760°C (1400°F) for 16 hours.

Testing

Standard tensile and stress rupture test bars were machined and low stress ground. Tensile tests were conducted at room temperature and 648°C (1200°F). Stress rupture tests were conducted at 648°C (1200°F) with an applied stress of 1034 MPa (150,000 psi). A series of low cycle fatigue bars were also machined and low stress ground. Test variations included temperatures of 538°C (1000°F) and 399°C (750°F), large diameter (10mm) and small diameter (5mm) specimens and total strain ranges of 0.66, 0.78 and 1.0 percent. Fatigue testing was done in air using a closed loop universal test machine. All testing was done in the longitudinally strain controlled mode (A=1, K=1, 20cpm) at a frequency of 0.33Hz.

Structure and Phase Analysis

Standard optical metallographic techniques were used to evaluate the initial structures of all parts tested. Fracture surfaces of LCF specimens were examined by scanning electron microscopy. The purpose of this effort was to identify failure location, measure defect size, and to determine defect morphology and principal elements associated with the defect.

To clarify an anomaly with powder mesh size effects, selected samples were subjected to phase analysis via x-ray diffraction. Material from heats KR464-3 (-60 mesh) and KR498-6 (-150 mesh) were subjected to extraction procedures that allowed identification of carbide, intermetallic, and non-metallic phases. A standard hydrochloric acid/methanol solution was used for the extraction.

RESULTS AND DISCUSSION

Microstructure

Processing of the CAPR preforms was varied to yield three structural conditions in parts produced from the -60 mesh powder and two structural conditions in parts produced from -150 mesh powder. Thermomechanical processing was designed to yield two structural extremes: 1) a fine grained highly recrystallized structure, and 2) a thin necklace structure with large warm worked grains surrounded by a small amount of fine recrystallized grains. These two structures are illustrated in Figure 1. These two structural extremes were obtained in parts produced from both -60 and -150 mesh powder. In addition, processing on several parts produced from -60 mesh powder was varied to yield an intermediate structure of coarse warm worked grains, but with a greater degree of recrystallization than observed in the previously shown thin necklace structure.

Tensile and Stress Rupture Properties

The average tensile properties of the material studied in this investigation are listed in Table 2. Room temperature ultimate and 0.2% yield strengths of 1720 MPa (250 ksi) and 1350 MPa (195 ksi) exceed specification requirements of 1565 MPa and 1227 MPa respectively by a wide margin. Euctility values also exceed requirements of 10 and 12 percent for elongation and reduction of area. Elevated temperature strength and

Table 2. Average Mechanical Properties

					Rupture		
	Ultir	nate	0.2%	/ield	Life	Elong.	R.A.
Property	(MPa)	(ksi)	(MPa)	(ksi)	(hours)	(%)	(%)
Tensile	1720	250	1350	195		14.0	18.0
Room Temp.							
Tensile	1600	230	1275	185	_	12.0	16.0
640°C(1200°F)							

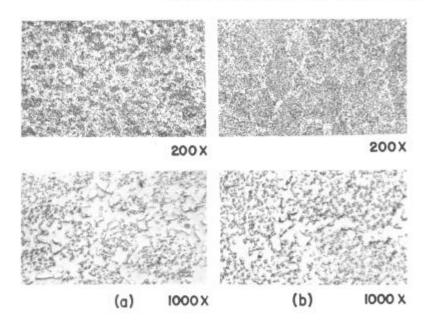


FIGURE 1: RECRYSTALLIZED (a) and THIN NECKLACE (b)
RENE 95 MICROSTRUCTURES

ductility at 640°C (1200°F) also exceed specification requirements. Stress rupture tests at 640°C (1200°F) with an applied stress of 1034 MPa (150 ksi) showed an average life of 230 hours with an average elongation of 6%. Rene 95 specification requirements are 25 hours and 2% elongation for the same test conditions. There appeared to be no significant trend in either tensile or stress rupture properties as a function of the structural variations studied.

Low Cycle Fatique Properties

The results of low cycle fatigue testing are illustrated in Figure 2. The illustration includes the powder metallurgy HIP low cycle fatigue goal (solid line) for -150 mesh Rene 95 and individual data points obtained in this investigation that illustrate effects of structure, mesh size, specimen size and test temperature. Internal and external nucleation of observed defects are also noted. The results show all materials tested, regardless of structure, meet or exceed the as-HIP Rene 95 LCF goal, and the specification requirement of 10,000 and 5,000 cycles for tests at 538°C (1000°F) for total strain ranges (et)

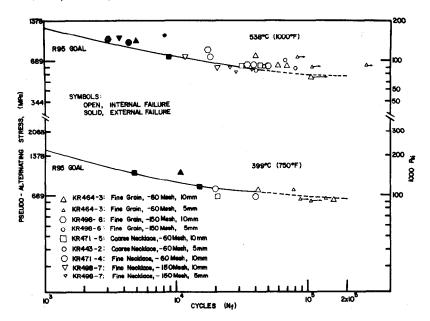


FIGURE 2: INFLUENCE OF PROCESSING AND TESTING ON LCF LIFE OF CAP RENE 95

of 0.66 and 0.78%, respectively. Specific effects on Rene 95 LCF life observed in this study are as follows:

Effect of Structure: Data showing the effect of structure on LCF life as a function of test variations are listed in Table 3. Tests conducted at 539°C (1000°F) for ϵ_t 's of 0.66 and 0.78% on large bar specimens (10mm) indicate that structure is the most significant factor affecting the LCF life of Rene 95. Fine grained (highly recrystallized) Rene 95 exhibits superior LCF life over both coarse and thin necklace structures. For the same test conditions, the coarse necklace structure (moderate recrystallization) shows slightly better LCF properties than the thin necklace structure (least recrystallization). Similar results were obtained on tests conducted at 399°C (750°F) on small bar specimens (5mm) for an ϵ_t of 1.0%.

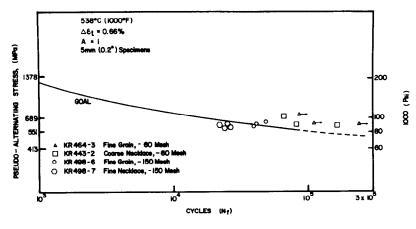
The most significant factor attributable to improved LCF properties of HIP Rene 95 is generally accepted to be cleanliness. Cleanliness and hence, defect size, is currently being controlled in HIP materials by limiting powder size to -150 mesh, thereby screening out large inclusions and their potentially damaging effects. The effect of powder mesh size on

Table 3.	LCF	Structure	Effects	at	538°C	(1000°F))
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Heat A) Large	Structure Bar Specime	LCF Life (cycles) n, et=0.669	Failure Location	Defect Size (mils)	Mesh <u>Size</u>
KR443-1 KR464-3 KR498-6	Fine Grain	131,718 71,291 33,564	Internal Internal Internal	28 16 5	-60 -60 -150
KR443-2 KR471-5	Coarse Necklace	31,664 44,750	Internal Internal	9 3	-60 -60
KR471-4 KR498-7	Thin Necklace	51,236 21,059	Internal Internal	<1 3	-60 -150
B) Large	Bar Specime	n, εt=0.789	%		
KR464-3 KR498 - 6	Fine Grain	40,876 15,248	Internal Internal	7.8 ~3	-60 -150
KR471-5	Coarse Necklace	48,947	External	N.D.	-60
KR471-4 KR498-7	Thin Necklace	18,210 10,827	Internal Internal	<1 N.D.	-60 -150
N D = Not	Determinahl	Δ			

N.D.= Not Determinable.

LCF properties of material evaluated in this study presented somewhat of an anomaly. Material produced from -60 mesh powder consistently showed higher LCF life when compared to material produced from -150 mesh powder, regardless of structure or test variations. This effect is illustrated in Figure 3.



EFFECT OF POWDER MESH SIZE ON LCF LIFE OF RENE 95 FIGURE 3:

obtained from examining fracture surfaces revealed a maximum defect size of 6 mils2 in -150 mesh material as contrasted with defects as large as 28 mils2 in the -60 mesh material. These results suggest that optimum processing can yield controlled microstructures, which can overcome substantial variations in material cleanliness. For example, LCF data obtained in this study on fine grained -60 mesh material is equal to or better than corresponding data for as-HIP -150 mesh product. Also, thermomechanically processed Rene 95 produced using -60 mesh powder appears to yield more consistent LCF data as evidenced by the high percentage of internal failures observed for Et's of 0.66 and 0.78%, regardless of variations in specimen size and test temperature. Overall results on structurally controlled -60 mesh CAPR plus hot worked Rene 95 show a defect tolerance level approximately four times that of HIP Rene 95. The data identify material structure control as a significant factor in achieving improved LCF life in this allov.

Regardless of the aforementioned structural effects, the data does not explain why CAPR plus hot worked -150 mesh material does not exhibit as good or better LCF life than -60 mesh material. Additional studies, utilizing SEM and X-ray diffraction analysis, revealed the apparent cause for this discrepancy. SEM analysis of fracture surfaces showed failures initiating primarily at inclusions (occasional void). The initial crack was intergranular in nature, becoming transgranular as crack growth progressed. Typical fractures showing variations in inclusion type and size are illustrated in Figure 4. While crack initiation sources in -60 mesh material

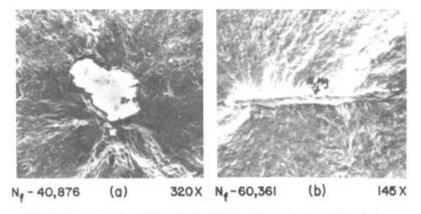


FIGURE 4: LCF CRACK INITIATION SITE INCLUSIONS (a) ZR, 7.8 MILS2 AND (b) A1, Mg, 20 MILS2

were inclusions traceable to melting refractories, sources in the -150 mesh material were found to contain substantial amounts of columbium, molybdenum and titanium, rather than the expected aluminum, magnesium and zirconium. X-Ray diffraction studies revealed an M₃B₂ boride phase in the -150 mesh material which was not present in the -60 mesh material. A review of the material process history showed that the -150 mesh material was consolidated at a higher temperature than the -60 mesh material, apparently resulting in boride formation during sintering. The presence of the boride phase could account for the LCF differences between structurally similar -60 and -150 mesh materials. This data suggests that intermetallic phases, especially borides, can be potentially more harmful to Rene 95 LCF life than non-metallic inclusions.

Effect of Test Variables. The effect of test temperature on LCF life was limited to tests on material produced from -60 mesh powder. These data show no significant effect of test temperature on LCF life of Rene 95 large bar specimens (10mm) for an ϵ_t of 0.78%. Tests on small bar specimens (5mm) and an ϵ_t of 0.66% similarly show no significant effect of test temperature. All failures were internal with crack initiation beginning in most instances at inclusions. Test data did show slightly better LCF life for tests conducted at 399°C (750°F) on fine grained material tested at an ϵ_t of 1.0%. However, the validity of this slight difference is questionable because all failures were external and had no readily evident source of crack initiation.

The effect of increasing total strain range (ϵ_t) variations shows reduced LCF life for all structures tested at 578°C (1000°F). Similar results were observed when testing at 399°C (750°F). Failure locations for ϵ_t of 0.66% were internal, and generally originated at an inclusion. A total strain range of 0.78% usually resulted in internal failures, although several external failures were noted. At a total strain range of 1.0%, all failures were external with crack initiation sources undetectable. The aforementioned effects were more pronounced on material produced from -150 mesh powder, again suggesting that cracks initiate preferentially at borides as opposed to inclusions.

Data showing the effect of specimen size on LCF life are listed in Table 4. Large bar specimens were 10mm (0.4") in diameter and small bar specimens were 5mm (0.2") in diameter. At 538°C (1000°F), a reduction in specimen size increases LCF life for both fine grain and necklace structure Rene 95 material produced from both -60 and -150 mesh powder. The effect is most pronounced for -60 mesh material. In fact, tests on fine grain -60 mesh material using small bar specimens did not fail

		Temperature	Specimen	LCF Life*	Mesh
Heat	Structure	(°C)	Size	(cycles)	Size
KR464-3	Fine Grain	538	Large	71,291	-60
KR464-3	Fine Grain	538	Small	237,772+	-60
KR498-6	Fine Grain	538	Large	33,564	-150
KR498-6	Fine Grain	538	Sma 11	42,784	-150
KR498-7	Fine Necklace	538	Large	21,059	-150
KR498-7	Fine Necklace	538	Small	25,061	-150

during testing and had to be removed from test. Limited data at 399°C (750°F) shows similar results. All failures were internal and initiated at sites containing inclusions or voids. The overall results tend to support the theory that large bar test specimens better reflect the actual performance of a material in an operating environment.

CONCLUSIONS

The following conclusions are presented relative to P/M Rene 95 near net shape parts produced by ${\sf CAP^R}$ consolidation plus hot working:

1. Structural variations effected by controlled hot deformation of Rene 95 preforms have a pronounced effect on the LCF properties of this alloy.

2. A fine grained, highly recrystallized structure in Rene 95 results in superior LCF life, when compared with necklace structures and with the goal LCF curve for as-HIP Rene 95.

3. Results confirm the initiation of LCF failures at inclusions, voids, or intermetallic compounds. However, the defect tolerance level of CAPR plus hot worked Rene 95 appears to be superior to as-HIP Rene 95 regardless of powder mesh size or LCF test conditions.

4. Strain ratio and specimen size LCF test variations have significant effects on LCF life, while test temperature has no significant effect on life.

5. CAPR plus hot worked Rene 95 preforms provide an alternative process to HIP for the manufacture of Rene 95 near net shapes. The process offers lower cost combined with higher and more consistent mechanical properties, and a greater LCF defect tolerance level.

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