PROCESSING EFFECTS ON MICROSTRUCTURE AND FATIGUE PROPERTIES OF NICKEL-BASE SUPERALLOYS.

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Processing Effects on Microstructure and Fatigue Properties of Nickel-Base Superalloys

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This report covers work performed on Contract N00019-79-C-0659 from December 1, 1979 to November 30, 1980. The purpose of the program is to develop a fundamental understanding of the influence of processing on the fatigue resistance of a nickel-base gas turbine disk alloy. Rene 95 has been selected as the model alloy for this investigation because it can be manufactured in a variety of product forms. The influence of processing will be studied in powder metallurgy (PM), cast and wrought (C+W), and
surface enhancement layers of René 95. The types of surface enhancement considered included high energy, low pressure plasma sprayed and high intensity shot peened layers. The plasma spray layers prepared for this study were not fully dense and therefore only their microstructure was studied.

A replication study of the crack initiation and growth process in PM René 95 with intentionally doped 10 mil diameter alumina particles showed that most of the fatigue process is crack growth. In the single specimen studied, crack initiation was detected at an alumina defect at less than one percent of the total fatigue life.

Cast and wrought René 95 with a necklace microstructure and eleven variations of PM René 95 were evaluated for tensile properties and cyclic defect tolerance. The defect tolerance was assessed by monitoring the crack growth from constant size EDM defects using a DC potential drop technique. The PM conditions included variations in powder size, HIP cycles, solution treatments, quench rates, and aging treatments. These processes resulted in two- fold variations in defect tolerance. These studies showed that those materials which were processed above the γ' solvus had a large grain size and possessed improved defect tolerance and retarded crack growth rates, particularly in the threshold regime. Evidence was presented which suggested that some of the improvements were caused by altered γ' distributions and composition and are not solely a result of the larger grain size. In PM René 95, increasing solution temperature increased the defect tolerance of material processed below the γ' solvus, while material processed above the γ' solvus had an opposite trend. The material with the best combination of strength and defect tolerance was -400 mesh (smaller than 37 microns) powder which was HIP'ed at 2200° F, approximately 65° F above the γ' solvus. This condition had 45 percent higher defect tolerance with less than five percent decrease in strength levels relative to the baseline condition, -140 mesh (smaller than 105 microns) powder which was HIP'ed at 2050° F.

Specimens with high intensity shot peening had defect tolerance four times greater than unpeened samples, most likely due to a compressive stress layer approximately 0.01 inch thick.
FOREWORD

This is the final report for Contract N00019-79-C-0659, Department of the Navy, Naval Air Systems Command. The work covered in this report was completed during the period from December 1, 1979 to November 30, 1980. The investigation was performed by the Materials and Process Technology Laboratories (M&PTL) of General Electric Aircraft Engine Group, Cincinnati, Ohio, 45216, and the Metallurgy Laboratory of General Electric Corporate Research and Development (CRD), Schenectady, New York, 12301.

The project engineer for the Department of the Navy is Mr. I. Maclin. The principle investigator for this program is Dr. R. H. VanStone (M&PTL). Technical assistance was provided by Dr. M. F. Henry (CRD), Ms. A. M. Ritter (CRD), Mr. D. M. Carlson (M&PTL), Mr. L. T. Duvelius (M&PTL), Mr. F. Brate (M&PTL), Mr. T. L. Richardson (M&PTL), Mr. W. R. Catlin (CRD), Mr. W. A. Seaman (CRD), Mr. C. P. Palmer (CRD), and Mr. R. A. White (CRD).
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INTRODUCTION AND TECHNICAL APPROACH

The continued demand for higher performance aircraft gas turbine engines with longer operating lives and improved reliability has encouraged the development of new turbine disk alloys which are capable of operating at higher stresses and higher temperatures.

Historically, disk alloys have been developed to improve tensile properties, creep strength, and rupture lives. Today's military and commercial customers are emphasizing life cycle management of their aircraft systems, thus shifting the disk alloy emphasis from tensile and creep properties to low cycle fatigue (LCF) capability. The intent of this investigation is to understand the influence of processing on the fatigue life of disk alloys. In a given high strength disk alloy, the size and location of the fatigue nucleation site has a significant effect on fatigue life. In addition to the initiation site location and size, fatigue lives of a given alloy are also dependent on its defect tolerance, that is, the resistance of a material to crack initiation and growth from the fatigue nucleation site. The principal goal of this investigation is to develop an understanding of the fatigue resistance through changes in processing. These modifications will result in different types of microstructures or residual stress patterns which can have a profound effect on defect tolerance.

This investigation will use two basic approaches. For selected conditions, PM Rene' 95 will be intentionally doped with large contaminants. The nucleation and growth of fatigue cracks from these defects will be studied by replicating surfaces of polished fatigue samples. The study of replicas taken at various fractions of fatigue life
will permit documentation of the crack nucleation and growth processes. Studies of this type have led to the understanding of classical low cycle fatigue of superalloys such as A286, IN718, and cast and wrought Rene' 95. One of the difficulties of these types of studies is the costly nature of performing these evaluations. These difficulties will be discussed in more detail in a subsequent section of the report.

The other approach taken in this investigation is to study the response of fatigue specimens to artificially introduced fatigue origins. The objective of such an approach is to have the life limiting fatigue crack nucleate and grow from an origin with a carefully controlled size and location. Wright has shown that electric discharge machined (EDM) defects have the same fatigue lives as naturally occurring surface defects of similar dimensions. Subsequently, Gangloff developed a direct current (DC) potential drop technique to monitor fatigue crack nucleation and growth from small EDM defects. This technique simultaneously provides a quantitative measure of defect tolerance and fatigue crack growth rates. This measurement of defect tolerance will be used as the major experimental technique in this investigation.

By combining the experimental observation of crack nucleation and growth from intentionally added defects with the monitoring of crack growth from EDM defects, those microstructural conditions which result in improved defect tolerance and thus higher fatigue capability can be identified. The microstructural conditions examined in this study include fourteen processing conditions. These were selected to be manufactured using the composition of Rene' 95 because it can be manufactured in powder metallurgy (PM) and cast and wrought (C+W) product forms. Most
of the conditions evaluated in this program will be in PM products because of the large variation in processing available for that product form. Necklace microstructure C+W will also be studied. Two surface enhancement processes, high energy, low pressure plasma spray layers and high intensity shot peened surfaces, will be evaluated.
PROCESS SELECTION

The processes selected for this investigation are to provide large differences in microstructure to significantly alter defect tolerance. The processes selected for PM, cast and wrought, and surface enhancement Rene' 95 will be discussed separately. The main emphasis in this program will be on PM Rene' 95 due to the greater flexibility in processing variables.

Powder Metallurgy

The powder processing variables that are thought to affect fatigue life in PM product form are listed below:

1. Powder size
2. Powder contaminants
3. HIP cycles
4. Solution treatment and quench
5. Aging treatment

It would be impossible to evaluate such a five factorial matrix, so several conditions have been selected on the basis of data generated in other programs and some degree of metallurgical judgement.

In a DARPA-sponsored program to develop improved disk alloy fatigue life, it was shown that defect tolerance of Rene' 95 increased with higher solution temperatures, higher HIP temperatures, and the use of fine powders. There appeared to be a continuing improvement in defect tolerance with alloy homogeneity resulting from reduced segregation distances (dendrite arm spacing) in a finer size powder and with more rapid homogenization associated with higher temperature HIP cycles. The most improved condition.
evaluated was -270 mesh (smaller than 53 microns) powder which was HIP'ed at 2200°F. This comparison had a fracture mechanics fatigue crack growth threshold which is the lower limit of experimental detectable crack growth of approximately 13 ksi \( \frac{\text{in}}{\text{in}} \). This is approximately 40 percent larger than the baseline condition (-140 mesh or powder smaller than 105 microns with a 2050°F HIP cycle). For this reason, a large part of this program will investigate the influence of homogeneity on the fatigue mechanisms and defect tolerance of Rene' 95.

In addition to the homogeneity, PM Rene' 95 will also be prepared to study the influence of HIP temperature and \( \gamma' \) distribution on defect tolerance. The \( \gamma' \) distributions will be altered through variations in solution treatments, salt quench bath temperatures, and aging cycles.

Several conditions will also be prepared from powder intentionally doped with alumina contaminants. The primary interest in this condition is to study the mechanism of fatigue crack initiation and early growth from naturally occurring defects. The conditions selected for these evaluations have been previously shown to have significant variations in defect tolerance.

Table I lists the conditions of PM Rene' 95 to be evaluated in this program. Examination of this table shows that the following variations have been included:

1. Three HIP cycles for a 2050°F solution treatment
2. Two HIP cycles for a 1925°F solution treatment
3. Two aging treatment variations on the baseline condition
4. One salt quench variation on the baseline condition
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<th>Dorset</th>
<th>HIP Cycle</th>
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<th>Salt (0.4 %)</th>
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* Baseline Condition
The undoped conditions will be evaluated using the potential drop/EDM defect technique while the four doped PM conditions (compacts N3-A, N10-A, N13-A, and N13-B) will be used in the replication studies.

**Cast and Wrought**

The standard "necklace" microstructure of cast and wrought (C+H) Rene' 95 will be studied in this program. This material consists of large warm-worked grains surrounded by small recrystallized grains. The condition was included in this program because this type of microstructure was not included in the PM processed material.

**Surface Enhancement Layers**

Two types of surface enhancement processes were studied in this investigation--high energy, low pressure plasma spray and shot peened layers. The intent of studying surface enhancement is to develop processing methods to suppress crack nucleation or retard crack growth. The fact that the environment can significantly affect the cyclic response of Rene' 95 to a defect has been demonstrated both in standard fatigue tests which show improved life for internal relative to surface nucleated LCF failures (1, 3) and by crack growth tests in vacuum which produce slower crack growth rates than companion tests in air. Floreen and Kane (13, 14) have shown a similar crack growth behavior in Inconel 718 at elevated temperatures. Thus if a surface enhancement layer can force all crack initiation to occur internally, then fatigue life can be improved. It is imperative in this approach that the surface defect tolerance not be impaired. It is that aspect of surface enhancement that will be studied in this investigation.

8.
Current state-of-the-art layers of high energy, low pressure plasma sprayed Rene' 95 will be manufactured and studied in this investigation. Jackson and Rairden[15] have shown that nickel-base superalloys can be plasma sprayed to produce a superfine grain structure. Their experiments resulted in tensile strengths and ductilities in plasma sprayed Rene' 80 which were superior to cast product of Rene' 80 at temperatures up to about 1300°F. Thermal fatigue wedges of plasma sprayed Rene' 80 showed no evidence of crack initiation at cyclic conditions which causes severe cracking in cast products. It was decided, therefore, to examine high energy, low pressure plasma sprayed Rene' 95 for its cyclic defect tolerance in this study. This material should have a microstructure much different than that of either PM or C+W product forms.

Shot peening has historically been used as a way to improve fatigue lives through use of residual stresses. This study will investigate high intensity shot peening as a means to improve fatigue life. High intensity shot peening results in a compressive residual stress layer approximately 0.01 inch thick.
MATERIALS

This section of the report will describe the processing, compositions, physical properties and microstructures of the materials studied in this program. Separate sections will describe powder metallurgy, cast and wrought, and surface enhancement layer materials.

Powder Metallurgy

Enough -140 mesh (smaller than 105 microns) powder was obtained for use in producing the PM Rene' 95 conditions listed in Table I and the high energy, low pressure plasma spray layers. This powder is from Special Metals lot number BN79018. The composition and mesh size analysis of this lot are listed in Tables II and III respectively. The composition of the powder is within the PM Rene' 95 specification which is also listed in Table II. The powder was loaded into two inch diameter cans which are 10 inches long and were processed according to the schedule in Table I. The density of each heat treated PM compact was determined by a water displacement technique. These data are shown in Table IV. All have densities typical of fully dense Rene' 95.

The microstructures of the conditions listed in Table I have been examined using conventional optical metallography and transmission electron microscope (TEM) replicas. The remainder of this section will use these results to describe the microstructures of the PM products.

The influence of HIP cycle temperature on the microstructures after heat treatment is shown in Figure 1. This figure shows optical micrographs -140 mesh Rene' 95 which was HIP'ed at 1925°F (Compact N2-B),
### TABLE II

**COMPOSITION OF RENE' 95 POWDER**

<table>
<thead>
<tr>
<th>ELEMENT</th>
<th>SPECIFICATION</th>
<th>ANALYSIS (WEIGHT PERCENT)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>0.04 - 0.09</td>
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<tr>
<td>Mn</td>
<td>0.15 MAX</td>
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</tr>
<tr>
<td>Si</td>
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<tr>
<td>S</td>
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<tr>
<td>P</td>
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<td>Mo</td>
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<td>Cb</td>
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</tr>
<tr>
<td>Al</td>
<td>3.30 - 3.70</td>
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<tr>
<td>B</td>
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<tr>
<td>W</td>
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<td>O</td>
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<tr>
<td>H₂</td>
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<td>Ni</td>
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<td>BALANCE</td>
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TABLE III

MESH SIZE ANALYSIS OF -140 MESH RENE' 95 POWDER

<table>
<thead>
<tr>
<th>MESH SIZE</th>
<th>POWDER SIZE (MICRONS)</th>
<th>FRACTION (%)</th>
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<td>-230 +270</td>
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<td>-270 +325</td>
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<td>-325 +400</td>
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<tr>
<td>-400</td>
<td>SMALLER THAN 37</td>
<td>35.5</td>
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## TABLE IV

**DENSITIES OF HEAT TREATED RENE’95 POWDER COMPACTS**

<table>
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<tr>
<th>COMPACT CODE</th>
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<th>g/cc</th>
<th>lb/in²</th>
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</tr>
<tr>
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<td>N1-C</td>
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<td>N2-B</td>
<td>8.29062</td>
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<td>N3-A</td>
<td>8.29251</td>
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<tr>
<td>N4-B</td>
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<td>N4-C</td>
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<tr>
<td>N12-A</td>
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<td>0.29956</td>
<td></td>
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<td>N12-B</td>
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<tr>
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<td>N13-B</td>
<td>8.28956</td>
<td>0.29950</td>
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Figure 1. Optical Micrographs of -1.0 Mesh Bend 95% Steel Aged at 1,025°F, (b) 2030°F, and (c) 2200°F and Subsequently Reheated to 2050°F Solution Treatment, 1000°F Salt Bench, and 1 Hour Oil Anneal.

15.
2050°F (Compact N1-B), and 2200°F (Compact N4-A). All of these compacts received a 2050°F solution treatment, a 1000°F salt quench, and an 8 hour, 1400°F age. The γ' solvus for Rene' 95 is typically about 2135°F (10) thus, the compacts HIP'ed at 1925°F and 2050°F (Figures la and lb respectively) were consolidated below the γ' solvus. The micrographs of these conditions show evidence of a remnant dendritic microstructure, most likely resulting from γ' which nucleates on interdendritic precipitates such as carbides. (10, 21) Figure 1c shows an example of Rene' 95 compacted above its γ' solvus. This processing eliminates the appearance of preferred γ' precipitation in interdendritic regions within individual powder particles and increases the grain size from approximately 8 to 30 microns. In this material, γ' is precipitated on prior particle boundaries, and the distribution of γ' within the grains is more uniform than the sub-solvus HIP cycles.

For the remainder of this report, the term finer γ' distribution means a reduction in the size of the γ' smaller than one micron as well as a more uniform distribution. The γ' on the prior particle boundaries probably results from preferential nucleation of γ' on the fine oxides and other phases which have been observed on prior particle boundaries of Rene' 95 using thin foil transmission electron microscopy. (27)

Figure 2 shows TEM replicas of the same materials which were shown in Figure 1. All three conditions show a triplex distribution of γ'. The largest γ' is on the order of 1 or 2 microns and is typically set up during sub-γ' solvus solution or HIP cycles. This γ' tends to form along prior particle or grain boundaries. The intermediate γ' precipitates have sizes of approximately 0.5 micron and form in grain interiors. In large powder particles, with their larger dendrite arm spacings, (16) γ' precipitates
Figure 2. TEM Replica Micrographs of -100 Mesh René 95 Which Was HIP'ed at (a) 1925°F, (b) 2050°F, and (c) 2200°F and Subsequently Received a 2050°F Solution Treatment 1000°F Salt Quench and a 1400°F/8 Hours Age.
form on interdendritic phases in conditions processed below the $\gamma'$ solvus. This leads to a large variation in $\gamma'$ distribution from grain to grain as shown in Figure 2b. This behavior is not observed in Rene' 95 processed above the $\gamma'$ solvus. In that case, each grain has a relatively uniform distribution of intermediate $\gamma'$ such as shown in Figures 1c and 2c. The fine $\gamma'$ shows up in the background of Figure 2 and cannot be easily resolved using TEM replicas.

Even though it cannot be observed metallographically, processing above the solvus may change the $\gamma'$ chemistry. When material is processed above the $\gamma'$ solvus, all the $\gamma'$ will go into solution and thus, can be homogenized. When such a compact is subsequently cooled below the $\gamma'$ solvus, the first $\gamma'$ which forms will have a composition which reflects the total alloy chemistry. This most likely is not the case when the HIP and solution temperatures do not exceed the $\gamma'$ solvus. In such a situation, the largest $\gamma'$ forms in the segregated interdendritic region, probably early in a HIP cycle, and will have a composition which is dependent on the local interdendritic chemistry. The phase transformations in these regions are very complex as evidenced by the presence of Laves phase in gas atomized Rene' 95(16) and its absence following HIP compaction either above and below the $\gamma'$ solvus.(10) Figure 3 shows higher magnification TEM replicas of intermediate $\gamma'$ which received (a) sub $\gamma'$ solvus, and (b) super $\gamma'$ solvus processing. Figure 3a shows a micrograph from compact N1-B, the baseline condition which contained -140 mesh powder which was HIP'ed at 2050°F, solution treated at 2050°F and aged at 1400°F for 8 hours. Figure 3b shows a micrograph from compact N12-A which contained -400 mesh powder which was HIP'ed at 2200°F followed by a solution and aging treatment identical to that for compact N1-B. The obvious difference between these conditions
Figure 3. TEM Replica Micrographs of (a) -140 Mesh René 95 HIP'ed at 2050° F and (b) -400 Mesh René 95 HIP'ed at 2200° F. Both Conditions Were Solution Treated at 2050° F and Aged for 8 Hours at 1400° F.
is the ellipsoidal morphology of the intermediate γ' in Figure 3a and the cuboidal shape of the γ' in Figure 3b. Although the shape shown in Figure 3b is the extreme case of cuboidal γ', in general, the intermediate-sized γ' in Rene' 95 processes above the γ' solvus had cubish shapes such as those shown in Figure 2c. This shape was never observed in materials totally processed below the γ' solvus. This change in shape strongly suggests a change in γ-γ' mismatch most likely brought about by a change in the alloy partitioning between γ and γ'.

Figure 4 shows optical micrographs of -140 mesh Rene' 95 which was HIP'ed at 1925°F (Compact N2-A) and 2200°F (Compact N5-B). Both materials were subsequently solution treated at 1925°F, quenched into 1000°F salt, and aged at 1400°F for 8 hours. The materials shown in Figures 1 and 4 are different in that the solution treatment for Figure 1 is 2050°F while that for Figure 4 is 1925°F. Comparing these figures shows that, as expected, the lower solution temperature increases the amount of large and intermediate-size γ' which forms during solution treatment. It is difficult to observe large differences in microstructure between Figures 4a and 4b due to the high volume fraction of large γ'. These differences can be seen more easily in TEM replicas of these microstructures which are shown in Figure 5. The major differences are larger grain size and more cuboidal γ' in material HIP'ed at 2200°F (Figure 5b) relative to that HIP'ed at 1925°F (Figure 5a).

In a HIP cycle, the cooling rate is slow, typically on the order of several hundred Fahrenheit degrees per hour. When cooling at this rate, the γ' which forms will coarsen at the high temperatures. If a structure is quenched from above the solvus the γ' distribution would be finer than those shown in Figures 1 through 4. Compact N4-C was solution treated at
Figure 4. Optical Micrographs of -120 Mesh Rod 93 which was HIP'ed at 1925°F Solution Treatment, 1000°F Salt Quench, and 1000°F 8 Hours Age.
Figure 5. TEM Replica Micrographs of -140 Mesh René 95 Which Was HIP'ed at (a) 1925°F and (b) 2200°F Prior to a 1925°F Solution Treatment, 1000°F Salt Quench, and a 1400°F/8 Hours Age.
2200°F and quenched through the γ' solvus into 1000°F salt and subsequently resolution treated at 2050°F, quenched again into 1000°F salt, and aged at 1400°F. Figure 6 shows optical and TEM replica micrograph of this compact. These micrographs show the large γ' grain size and the very fine γ' distribution. There is essentially no large or intermediate-size γ'. This suggests that the large and intermediate-sized γ' precipitates observed in the other materials forms during the solution treatment and/or the slow cool from the HIP temperature. This indicates that the large and intermediate γ' nucleates during solution treatment on sites which had formed previously during slow cooling from a super solvus treatment. A good indication of this phenomena is comparison of Compact N5-A (Figures 1c and 2c) which was HIP'ed at 2200°F and slow cooled with Compact N4-C (Figure 6) which was solution treated and quenched from 2200°F. Subsequent to this both compacts were solution treated at 2050°F for either 6 hours (Compact N5-A) or 1 hour (Compact N4-C). It is doubtful that this difference in time could result in the variations shown in Figures 2c and 6h.

It should be noted that heat treatments which involve a quench through the γ' solvus temperature would never be used for production Rene' 95 hardware due to the possibility of quench cracking. Even on the two inch diameter Compact N4-C evaluated in this program, some quench cracking was observed. This resulted in a loss of one of the fatigue specimens to be used for cyclic defect tolerance evaluation.

Three compacts were processed to alter γ' distributions through use of quench rates and aging temperatures. The optical micrographs showed only subtle differences, however, more significant changes in microstructure were observed using TEM replicas as shown in Figure 7. All three compacts contained -140 mesh Rene' 95 which was both HIP'ed and solution
Figure 6. Optical (a) and TEN Replica (b) Micrographs of -140 Mesh René 95 which was HIP'ed at 2050°F, Solution Treated at 2200°F, Quenched into 1000°F Salt, Re-Solution Treated at 2050°F, Quenched into 1000°F Salt and Aged at 1400°F for 8 Hours.
Figure 7. TEM Replica Micrographs of -140 Mesh Rene 95 which was HIP at 2050° F and (a) Quenched into 1000° F Salt but not Aged, (b) Quenched into 1500° F Salt and Aged at 1400° F for 8 Hours, and (c) Quenched into 1000° F Salt and Aged at 1800° F for 8 Hours.
treated at 2050°F. Figure 7a shows the microstructure of compact N4-B which was quenched into 1000°F salt but not aged. This processing is identical to the baseline compact (N1-B) except for the absence of an aging cycle. Comparison of Figures 7a and 3a show that the microstructures of these two conditions are very similar. Figure 7b shows the microstructure of compact N1-C which was quenched in 1000°F salt and aged at 1400°F for 8 hours. Increasing the salt quench bath temperature results in a slower quenching rate from the solution temperature. Comparing the baseline condition with a 1000°F salt quench (Figure 3a) to compact N1-C with a 1500°F salt quench (Figure 7b) shows that the slower quench rate increases the size of the fine γ' resulting in an almost bimodal γ' distribution. Figure 7c shows the microstructure of Rene' 95 compact N1-A which was quenched into 1000°F salt and aged at 1800°F for 8 hours. Increasing the aging temperature results in larger intermediate-sized γ'. This condition is almost analogous to a very low temperature solution treatment.

The microstructures shown in Figures 1 through 7 were from compacts containing -140 mesh (smaller than 105 microns) powder. Figure 8 shows optical micrographs of -400 mesh (smaller than 37 microns) powder which was HIP'ed at 2050°F (Compact N12-B) and 2200°F (Compact N12-A). Both of these compacts were solution treated at 2050°F, quenched into 1000°F salt and aged at 1400°F for 8 hours. As in the case of -140 mesh powder, HIP'ing -400 mesh powder below the γ' solvus results in preferential nucleation of γ' in the interdendritic regions (Figure 8a) while the 2200°F HIP results in a larger grain size and γ' decoration of prior particle boundaries. Comparison of equivalent processing conditions (Figures 1b to 8a and 1c to 8b) shows that the -400 mesh powder compacts have finer γ' sizes. This most likely results from the increased amount of prior
Figure 8. Optical Micrographs of -400 Mesh René 95 Which Was HIP Compacted at (a) 2050° F and (b) 2200° F Solution Treated at 2050° F Quenched into 1000° F Salt and Aged at 1000° F for 8 Hours.
particle boundary area and the reduced dendrite arm spacing in the -400 mesh powder. (16)

Figure 9 shows TEM replica micrographs of the two -400 mesh conditions. As was shown in the larger powder sizes, there is a more uniform distribution of γ' sizes in the super γ' solvus HIP'ed material. As noted previously, Figure 9 shows the 2200°F HIP material has a tendency to form more cubic-shaped γ'.

**Cast And Wrought**

Portions of a cast and wrought Rene' 95 high pressure turbine disk have been obtained. This disk was forged by Wyman-Gordon Company (SN 67586). The cast and wrought Rene' 95 chemistry limits and disk chemical analysis are shown in Table V. Note that the carbon and chromium content of PM (Table II) and cast and wrought (Table V) product forms are different. The composition of the forged Rene' 95 is within the specification shown in Table V. Following forging, this disk was solution treated at 2000°F for one hour, oil quenched, and aged at 1400°F for sixteen hours.

Optical micrographs of cast and wrought (C+W) Rene' 95 are shown in Figure 10. Figure 10a shows a low magnification micrograph showing large elongated warm-worked grains. Figure 10b shows the intersection of several of these grains. In the grain boundaries are MC carbides and a layer of fine recrystallized grains. The MC-type carbides in C+W Rene' 95, indicated with arrows in Figure 10b, are much larger than those in powder products due to the higher carbon content and the much slower solidification rate of a material starting from a cast ingot. The distribution of fine grains shown in Figure 10b has caused this microstructure to be termed "necklace" Rene' 95. (11) The micrographs in Figure 10 show metallographic sections parallel to the
### TABLE V

**COMPOSITION OF CAST AND WROUGHT REV. 95**

<table>
<thead>
<tr>
<th>ELEMENT</th>
<th>SPECIFICATION</th>
<th>ANALYSIS (WEIGHT PERCENT)</th>
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</thead>
<tbody>
<tr>
<td>C</td>
<td>0.13 - 0.17</td>
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</tr>
<tr>
<td>Mn</td>
<td>0.15 MAX</td>
<td>0.10</td>
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<td>Si</td>
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<td>P</td>
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<td>Cr</td>
<td>13.00 - 15.00</td>
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<td>Mo</td>
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<td>Fe</td>
<td>0.50 MAX</td>
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<td>W</td>
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<td>Ni</td>
<td>BALANCE</td>
<td>BALANCE</td>
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Figure 9. TEM Replica Micrographs of -400 Mesh René 95 Which Was HIP Compacted at (a) 2050°F and (b) 2200°F, Solution Treated at 2050°F, Quenched Into 1000°F Salt and Aged at 1400°F for 8 Hours.
Figure 10. Low (a) and High (b) Magnification Optical Micrographs of Cast and Wrought René 95. The Arrows in Figure 10B Indicate MC-Type Carbides.
radial plane of the disk. When the tangential plane of the disk is observed metallographically, the large warm-work grains have more circular cross-sections.

Figure 11 shows a TEM replica micrograph of necklace C+W Rene' 95. In this product form there is a duplex $\gamma'$ distribution—large primary $\gamma'$ several microns in size and the fine $\gamma'$ which is difficult to resolve in replicas. In necklace Rene' 95, the large $\gamma'$ appears to be larger than that in PM products, but they have similar fine $\gamma'$ distribution.

Surface Enhancement Layers

**High Energy, Low Pressure Plasma Spray.** During this investigation, 57.5 pounds of -140 mesh (smaller than 105 microns) Rene' 95 powder from Special Metals heat number BN79018, the same as was used in the powder metallurgy evaluation, was set aside for the plasma spray evaluation. In preparation for high energy, low pressure plasma spraying, the powder was screened to -400 mesh and -140/+400 mesh. The yield of -400 mesh powder from -140 mesh powder was 18.8 pounds or approximately 32.7 percent. This is consistent with the 35.6 percent -400 mesh yield reported in Table III.

Oxygen and nitrogen analyses were performed on the loose powder, compacted powder, and the plasma sprayed material. Those results are given in Table VI. The -140 mesh powder data show reasonable agreement with the oxygen analysis reported in Table II. Comparison of the oxygen contents of loose and consolidated powder show a significant increase in oxygen with reduced powder size. It should be noted that for these product forms, the current Rene' 95 powder metallurgy specification requires that oxygen content be less than the 150 ppm.
Figure 11. TEM Replica Micrograph of Cast and Wrought René 95.
<table>
<thead>
<tr>
<th>MATERIAL</th>
<th>NITROGEN* (ppm)</th>
<th>OXYGEN* (ppm)</th>
</tr>
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<tr>
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<td>36.27</td>
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<td>-400 Mesh</td>
<td>36.37</td>
<td>121.129</td>
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<td>CONSOLIDATED POWDER</td>
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<td>-140 Mesh (Compact Ni-B)</td>
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<td>-400 Mesh (Compact Ni2-B)</td>
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<td>126.138</td>
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<tr>
<td>PLASMA SPRAYED PLATE</td>
<td>89.79</td>
<td>268.261</td>
</tr>
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* DUPLICATE MEASUREMENT
Two plates have been plasma sprayed using state-of-the-art parameters for a high energy, low pressure plasma spray process. The Rene' 95 powder was sprayed on stainless steel plates to yield for each plate a useful plasma spray material region of approximately 1.5 x 0.25 inch. Portions of both plates were heat treated as follows in argon.

2050°F/1 Hr./retort cool
plus
1600°F/1 Hr./retort cool

Oxygen analysis of this plasma sprayed material, as shown in Table VI, indicates substantial oxygen pick-up occurred during this process. The density of the as-sprayed material was measured to be 95.7 percent of HIP compacted Rene' 95. Optical micrographs of as-plasma sprayed and sprayed plus heat treated Rene' 95 in the as-polished condition are shown in Figure 12. Both conditions show considerable porosity. One cannot distinguish original porosity from pullouts which occur during grinding and polishing. Micrographs of as-sprayed and heat treated material after heavy etching are shown in Figure 13. The as-sprayed conditions shows that many particles which are still essentially circular in cross-section and in general, maintained their dendritic microstructure. Many circular cross-section prior particle boundaries are also visible in light optical micrographs of this Rene' 95 after heat treatment. These particles show no evidence of dendritic structure after the same gamma prime etch that revealed a dendritic structure in the as-sprayed condition. This is in sharp contrast to the -400 mesh HIP compacted powder shown in Figure 8a, which received a similar heat treatment. In order to more fully understand the plasma spray process, the microstructures were
Figure 12. Optical Micrographs of (a) As-Plasma Sprayed and (b) Sprayed Plus Heat Treated René 95 In the As-Polished Condition.
Figure 13. Optical Micrographs of (a) As-Plasma-Sprayed and (b) Sprayed Plus Heat Treated René 95 After Heavy Etching.
studied using electron microscopy. Scanning electron microscopy (SEM) was used to further examine polished and deep etched surfaces. Examples of such samples are shown in Figure 14 for the as-sprayed and heat treated materials. This figure shows evidence of "splattered" as well as circular cross-section particles. Note that the dendritic structure shapes seen in circular cross-section particles in light microscopy are not evident in scanning electron microscopy. This suggests that the interdendritic boundaries are not decorated with intermediate or large size γ' particles. The material after heat treatment looks much more homogeneous in scanning electron microscopy than it did in light microscopy.

The material was examined further using transmission electron microscopy (TEM) on thin foils and extraction replicas from polished and etched metallographic cross-sections. These types of examinations on the as-sprayed material reveals a very inhomogeneous microstructure. There are numerous, apparently intact, possibly unmelted particles exhibiting dense interdendritic precipitation. A general low magnification micrograph is shown in Figure 15. A higher magnification micrograph of a particle with dendritic structure is shown in Figure 16. The sample consists mainly of a mixture of grains, several microns in size, and what appear to be dendritic regions from unmelted particles as shown in Figure 17. There is extensive precipitation throughout the non-dendritic portion of the matrix. These precipitates outline a macropattern of ripples or layers as shown in Figures 17 and 18. Some of the interdendritic and matrix precipitates have been identified by electron diffraction as MC type carbides. Most of the interdendritic precipitates in the circular cross-section particles are several times larger than those in the fine grained regions.
Figure 14. Scanning Electron Micrographs of Deep Etched Metallographic Sections of (a) As-Plasma Sprayed and (b) Sprayed and Heat Treated René 95.
Figure 15. TEM Micrograph of an Extraction Replica From a Metallographic Section of As-Plasma-Sprayed René 95.
Figure 16. TEM Micrograph of an Extraction Replica From a Metallographic Section of As-Plasma-Sprayed René 95 Showing a Region Having A Dendritic Structure.
Figure 17. TEM Micrograph of an Extraction Replica Showing the Typical Structure of a Metallographic Section of As-Plasma-Sprayed René 95.
Figure 18. TEM Micrograph of an Extraction Replica Showing a Rippled or Layered Precipitation Pattern in As-Plasma-Sprayed René 95.
Thin foil TEM micrographs of what seems to correspond to the ripples show sequence of somewhat columnar grains separated by what appears to be a fault line, the nature of which is still undetermined. Examples of these are shown in Figures 19 and 20. Some of the fine grains throughout the matrix have a higher dislocation density than in the dendrites and larger grains. This makes imaging of precipitates difficult, but some matrix and prior particle boundary precipitation can be seen. It is also difficult to distinguish intermediate size $\gamma'$. The fine $\gamma'$, as seen in Figure 21, has sizes ranging from 200 to 600Å. In the as-plasma sprayed condition, the grain size is on the order of 1 micron.

Transmission electron microscopy also revealed large increases in homogeneity after heat treatment. Intragranular, intergranular and prior particle boundary precipitates are seen, but no selected area diffraction has yet been performed to identify them. Although most of the grains are considerably larger (several microns) than in the as-plasma sprayed material, there are regions like that shown in Figure 22 where bands of small grains have been retained. The fine $\gamma'$ observed after heat treatment is slightly larger (400 to 1000Å) than that in the as-sprayed material as seen in Figure 23. Some intermediate size $\gamma'$ occurs, primarily at the grain boundaries. One unhomogenized particle was seen in thin foil examination with remnant dendrites outlined by small precipitates, probably MC type carbides. This area is shown in Figure 24 and should be considered atypical.

Due to the high porosity in plasma sprayed Rene' 95 layers, no mechanical property evaluation of high energy, low pressure plasma spray layers of Rene' 95 will be reported.
Figure 19. Thin Foil TEM Micrograph of a Rippled Area Showing Fault-Like Lines in As-Plasma-Sprayed Rene 95.
Figure 20. Thin Foil TEM Micrograph of a Rippled Area Showing a Fault-Like Line in as-Plasma-Sprayed Rene 95.
Figure 21. Thin foil TEM Micrograph Showing Fine in As-Plasma-Sprayed René 95.
Figure 22. Thin Foil TEM Micrograph Showing a Region of Retained Fine Grains in Plasma Sprayed René 95 After Heat Treatment.
Figure 11. Thin Foil TEM Micrograph Showing Fine γ in Plasma Sprayed René 95 After Heat Treatment.
Figure 24. Thin Foil TEM Micrograph of a Particle with Dendritic Structure Found in Plasma Sprayed René 41 After Heat Treatment.
High Intensity Shot Peening. Powder metallurgy compact N6-A was processed identical to the baseline compact (N1-B) listed in Table I. The test specimens were then machined from this material and shot peened using 5550 hard shot to a C Almen strip value in the range of 4 to 6 C. Each specimen received 125 percent coverage. The range of C almen strip values determined during processing ranged from 4.4 to 5.0. These shot peening parameters typically result in compressive stress layers 0.01 inch thick. Experimental determination of the residual stresses has not been performed at this time.
The emphasis in this program is on fatigue crack propagation rates as a measure of cyclic defect tolerance in high strength superalloys. This approach is one which considers the fatigue process to be one of essentially propagation. The assumption is best checked by direct observations of the initiation and early growth stages of fatigue.

Such direct observations should be aimed at learning how real defects behave and how cracks grow. To accomplish this, four of the material conditions selected for powder metallurgy studies were produced with 1000 particles of nominally 0.010 inch diameter alumina (Al₂O₃) per pound of Rene' 95. Alumina was selected because of its good phase stability in a nickel-base superalloy, and because alumina defects have been found in gas atomized superalloys. This ceramic doping is similar to that reported by Pfouts, et al.(1) The four conditions selected can be identified in Table I. The four compacts are -140 and -400 mesh powder which was HIP'ed above and below the γ' solvus temperature.

The direct observations of fatigue cracks emanating from ceramic defects were made by studying the free surface of a specimen gauge section with a high surface to volume ratio. In this technique,(7) the fatigue test is interrupted frequently, cooled to room temperature, and plastic replicas are made of the free surface. After completion of the test, the crack nucleating defect is easily located. The replicas can then be examined to observe the fatigue process from zero cycles onward at the location of the initiation site. In this particular study, replicas were metal vapor shadowed and examined using scanning electron microscopy. Replicas were taken at mean load rather than at minimum or zero load in order to improve the ability to find and distinguish

53.
cracks. Prior to testing, specimen gauge sections were metallographically polished and chemically etched with an etchant that preferentially attacks \( \gamma' \). Note that this one stage plastic replica is the topological mirror image of the real specimens surface. Hills and valleys on the specimen are seen as valleys and hills respectively on the replicas. Therefore, porosity and etched out \( \gamma' \) on the specimen appear as bumps on the replica, and the replica has tongues of replica material that were down inside cracks on the specimen surface.

The specimen selected had a uniform sheet gauge section of 0.050 in by 0.400 in by 1.0 in for a surface to volume ratio of 45 in\(^{-1}\) and is shown in Figure 25. All tests were run at 1000°F and 20 cpm in air with an \( P \)-ratio (minimum/maximum load) of 0.05. Four specimens of each doped material condition were machined and are available for testing.

Several preliminary tests were performed without surface replication to find a stress level that would yield reasonable lives for this type of study. Those results are summarized in Table VII. Based on previous experience with higher dopant densities, the first test was conducted on doped -140 mesh material with a 2200°F HIP and a 2050°F solution treatment and was tested with maximum cyclic stress of 120 ksi. The test was terminated after 125,000 cycles without failure. A specimen of -400 mesh material with a 2050°F HIP and a 2050°F solution treatment was then run with a maximum stress of 150 ksi. This resulted in 6899 cycles to failure.

A second specimen of -400 mesh powder with a sub-solvus HIP and solution treatment was then run with a maximum stress of 150 ksi with surface replication at several points in life including zero cycles. The specimen failed in 8668 cycles with a surface intersected dopant particle as the initiation site. Three scanning electron micrographs
Figure 25. Drawing of Axial Sheet Fatigue Specimen Used in Replication Studies.
### TABLE VII

**AXIAL SHEET FATIGUE SPECIMEN RESULTS**

1000F Air, 20 cpm, \( R = 0.05 \)

\( \text{Al}_2\text{O}_3 \) Doped Specimens

1400F/8 Hours Age

<table>
<thead>
<tr>
<th>MESH</th>
<th>HIP (°F)</th>
<th>SOLUTION (°F)</th>
<th>MAX (ksi)</th>
<th>( N_e )</th>
<th>REPLICAS</th>
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<td>150</td>
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<td>No</td>
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<tr>
<td>-400</td>
<td>2050</td>
<td>2050</td>
<td>150</td>
<td>8668</td>
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</table>

(1) Terminated prior to failure
of the zero cycle replica at the initiation site for this specimen (K191) are shown in Figure 26. Figure 26a is low magnification showing the entire surface intersection of the dopant particle, while Figure 26b shows the right hand side at higher magnification, and Figure 26c shows the left side at high magnification. The numerous "bumps" on the replica at the inclusion, represent porosity in the Al$_2$O$_3$. The principal stress direction is vertical in the micrographs.

Figure 27 is a high magnification micrograph of the right hand side of this dopant particle as shown after 50 cycles (0.6 percent of life). A comparison of Figures 27 and 26b reveal that after these 50 cycles there is a fatigue crack of approximately 0.00045" (11.4 microns) at the upper, right quadrant of the dopant particle. Micrographs of the dopant particle on the 1100 cycle (13 percent of life) replica are shown in Figure 28. The fatigue crack on the right hand side is now 0.00115 inch long. There is still no crack visible on the left hand side. The sharp line in the inclusion in Figure 28b represents a crack in the ceramic. Note also the visible prior particle boundary (PPB) approximately 0.001" to the left of the dopant particle in Figure 28b. Note the average surface crack growth rate was $9 \times 10^{-5}$ in/cycle for the first 50 cycles and $7 \times 10^{-7}$ in/cycle between 50 and 1100 cycles.

Figure 29 shows the dopant particle after 2400 cycles (28 percent of life). The fatigue crack on the right hand side is now 0.00225 inch long, without any visible crack on the left side. Figure 30 is lower magnification micrograph of the replica at 5400 cycles (62 percent of life). The fatigue crack is 0.0033 inch long on the surface on the right side. There is still no crack visible on the left hand side.
Figure 2b. SEM Micrographs of (a) a Replica of the Entire, (b) Right Side and (c) Left Side of Fatigue Nucleating Alumina Defect in Specimen K191 Prior to Fatigue Cycling.
Figure 27. SEM Micrograph of a Replica of the Right Side of the Fatigue Nucleating Alumina Defect in Specimen K191 After 50 Fatigue Cycles at 1000° F. The Arrow Indicates the Extent of Crack Growth.
Figure 28. SEM Micrographs of a Replica of the (a) Right and (b) Left Sides of the Fatigue Nucleating Alumina Defect in Specimen K191 After 1100 Fatigue Cycles. At 1000 °F, the Arrow Indicates the Extent of Crack Growth.
Figure 29. SEM Micrographs of a Replica of the (a) Right and (b) Left Sides of the Fatigue Nucleating Alumina Defect in Specimen K141 After 2400 Fatigue Cycles at 1900°F. The arrow indicates the Extent of Crack Growth.
Figure 29. SEM Micrographs of a Replica of the (a) Right and (b) Left Sides of the Fatigue Nucleating Alumina Defect in Specimen K101 After 2400 Fatigue Cycles at 1000°F. The arrow indicates the Extent of Crack Growth.
Figure 30. SEM Micrograph of a Replica of the Fatigue Nucleating Alumina
Defect in Specimen K191 After 5,000 Fatigue Cycles at 2000°F.
The Arrow Indicates the Extent of Crack Growth.
Figure 31 shows the dopant particle on the 8000 cycle replica. The surface fatigue crack lengths are now 0.0185 inch on the right hand side and 0.0085 inch on the left side.

Table VIII gives surface crack lengths for all replicas taken, not just those shown in Figures 26 through 31. Values for the right hand side are plotted in Figure 32. Examination of these surface lengths shows that the alumina particle resulted in a 0.45 mil long surface crack during the first 50 cycles. This crack did not grow appreciably until sometime between 700 and 1100 cycles. This initial crack growth thus most likely represents a process of local accommodation of strain concentration in the vicinity of the inclusion. This study shows that the fatigue process with cycles nucleation at inclusions is primarily a crack growth process.

It is obvious, the surface replicas give no information on crack growth rates, but some insight can be gained by fractographic examination. Figure 33 shows scanning electron micrographs of the fracture surface at the initiation site. The right hand side of the fractographs of Figure 33 correspond to the right hand side of the replicas of Figures 26 through 31. One can see that the relatively flat portion of the fatigue surface is broader on the right hand side than on the left. This relatively flat zone is approximately 0.0085 inch on the right side and 0.001 inch on the left side at their intersection with the free surface. Examination of Table VIII shows that both of these lengths correspond to between 5400 and 8000 cycles for their respective sides on the replicas. It appears then, that the fatigue crack initiated at one corner of the alumina particle at its intersection with the free surface, spread around the perimeter of the inclusion in Region I type growth, and then grew.
Figure 31. SEM Micrograph of a Replica of the Fatigue Nucleating Alumina Defect in Specimen K191 After 8000 Fatigue Cycles at 1000°F. The Arrows Indicate the Extent of Crack Growth.
TABLE VIII

REPLICATION STUDY RESULTS OF SPECIMEN K191 FROM COMPACT ML3B
-400 MESH, 2050F HIP, 2050F SOLUTION, 1400F AGE
TEST AT 1000F/AIR/20 CPM
MAXIMUM CYCLIC STRESS=150 KSI; R=0.05

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<th>RIGHT SIDE*</th>
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</tr>
<tr>
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</tr>
<tr>
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<tr>
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<td>FAILURE</td>
<td></td>
</tr>
</tbody>
</table>

* SEE FIGURES 26 THROUGH 31
Figure 32. Variation of the Surface Fatigue Crack Length on the Right Side of the Fatigue Nucleating Alumina Defect in Specimen K191 with Fatigue Cycles.
Figure 11. SEM Micrographs of the Fracture Surface in the Vicinity of the Fatigue Nucleating Alumina Defect in Specimen K191.
symmetrical to final failure. It is postulated that local dopant morphology influenced the initial asymmetric growth.

Figure 34 shows a fractograph of the other half of the fatigue specimen tilted to show both the fracture surface and the free surface. The "left-right" relationship is reversed from micrographs of the replicas. It can be observed that the shape, cracks, and pores in this Figure 34 correspond to those observed in the replicas.
Figure 34. SEM Micrograph Showing Both the Fracture Surface and Prepolished Surface of Specimen K191 in the Vicinity of the Fatigue Nucleating Alumina Defect.
MECHANICAL PROPERTIES

The mechanical properties determined in this investigation include both tensile and fatigue properties. The tensile properties of each condition were determined at both room temperature and an elevated temperature (either 1000°F or 1200°F) using a specimen with a reduced gage section having a circular cross-section diameter of 0.25 inch and a uniform gage length of one inch.

The main emphasis of this investigation is the evaluation of the fatigue properties. High strength disk alloys most frequently fail by fatigue which initiates at discrete fatigue nucleation sites.\(^{(1-6)}\) The previous section described how the fatigue process in the presence of such a nucleation event is primarily one of crack growth. The replication technique has several major disadvantages. One is that the crack depth cannot be monitored. In addition, by intentionally adding dopants, the dopant location and shape is not precisely controlled. The replication technique is also very costly. An alternate approach would be to artificially introduce a defect having a controlled size, shape, and location and monitor the crack growth from this defect.

Wright\(^{(8)}\) has shown that commercially processed Rene' 95 fatigue specimens containing electric discharge machined (EDM) defects have lives equivalent to specimens containing naturally occurring surface defects of the same size. The artificial defect approach was further improved by the development of a direct current (DC) potential drop technique to monitor crack growth out of a small EDM defect. The details of this experimental technique have been described by Gangloff\(^{(9)}\) and will be summarized here. A small EDM defect is introduced into a specimen surface, and direct current is passed through the sample. The
potential drop between two points on either side of the defect is measured. As the crack emanating from the defect increases in area, the potential drop increases. Thus, this approach can be used to monitor the initiation and growth of cracks from EDM defects. Figure 35 shows a scanning electron microscope (SEM) micrographs of a fractured EDM defected specimen monitored with DC potential drop. This specimen was 0.20 inch in diameter, had a chord shaped defect 0.004 inch deep with a surface length of 0.056 inch and had potential monitoring probes located 0.016 inch on both sides of the EDM defect.

An analytical model has been developed to monitor the crack depth as a function of EDM defect dimensions and potential lead position. Experimental verification of this model was established for high stress cracking by growing fatigue cracks in several alloys over the temperature range from room temperature to 1000°F and subsequently fracturing them to reveal the extent of crack growth. The sensitivity and stability of this potential drop method is less than 0.001 inch. Gangloff (9) showed that the predicted and experimentally observed crack lengths varied by less than 15 percent. The use of the potential drop system allows a quantitative determination of the crack growth rate rather than just a single parameter such as cycles to failure. These data can then be analyzed using the concepts of linear elastic fracture mechanics to obtain the crack growth rate (da/dN) as a function of the stress intensity factor range (ΔK). This will permit comparison of the defect tolerance of materials without the contribution of secondary factors such as yield strength. The K-solution used to analyze these data has been described elsewhere. (9) This test method has been used to evaluate the crack growth properties of a 10 Ni steel where it was shown that the fatigue crack growth rate results (da/dN vs. ΔK) are in good agreement with data obtained in an interlaboratory round-robin test evaluation using the more
Figure 35. SEM Micrograph of Fractured EDM Defect Tolerance Specimen Monitored with DC Potential Drop Technique.
standard compact specimens. (19,20) An example of fatigue crack growth curve for Compact Ni-B of PM Rene' 95 is shown in Figure 36. This is the baseline condition for PM Rene' 95 in this investigation. The square symbols represent data from 0.66 percent longitudinal strain control tests with a semicircular 0.004 inch radius defect. For the remainder of this report, this defect will be called the semi-circle defect. The (X) symbols in Figure 36 represent data from load control tests using the chord defect shown in Figure 35. Duplicate load and strain control data are shown in Figure 36. The points with growth rates close to $4 \times 10^{-8}$ in/cycle indicate $\Delta K$ levels where no growth was detected in 28,800 fatigue cycles (triangle). This curve has two regions— the nearly vertical threshold regime (Region I) and the lower sloped linear region (Region II) which in Figure 36 starts at crack growth rates close to $10^{-6}$ in/cycle. It should be noted that the strain control tests start at $\Delta K$ levels of 10 or 11 ksi $\sqrt{in}$. This sometimes results in artificial thresholds such as shown in Figure 36. These type of results will not be interpreted as real crack growth rates. The baseline curve is the current best estimate of the crack growth rates in Compact Ni-B. The line shown in Figure 36 will be shown in all subsequent crack growth plots to be used on a comparison basis. The overlap in data between load and strain control specimens with their various defect shapes and 60 percent difference in load suggests that the fracture mechanics approach properly describes crack growth in superalloy disk materials. The $\Delta K$ values were calculated using the load range for the load control tests and the maximum load for the strain control tests, thus ignoring the compressive portion of the loading cycles. It should be noted that the DC potential drop method was used as a screening test in this investigation.
Figure 36. Fatigue Crack Growth Curve of -140 Mesh René 95 which was HIP'ED at 2050°F, Solution Treated at 2050°F, Quenched into 1000°F Salt, and Aged at 1400°F.
to evaluate metallurgical variables. More extensive testing using this or more standard test methods is required to unequivocally determine the cyclic crack growth rates.

This test was used to evaluate the defect tolerance of the various Renz' 95 processes studied in this program. On almost each condition, four defect tolerance fatigue tests were performed at 1000°F with a frequency of 0.33 Hz (20 cycles per minute). Two specimens had an hourglass geometry with a 0.2 inch minimum diameter and contained the chord EDM defect geometry shown in Figure 35. These specimens with chord defects were tested in load control (sinusoidal waveform with an R-ratio of minimum to maximum load of 0.05) in order to estimate the fatigue crack growth threshold and the crack growth rates in Region I. The values of threshold and Region I crack growth rate measured in this program should be viewed as relative values rather than absolute ones. The threshold values (ΔK_{th}) reported here are determined from the minimum load where crack growth was detected and the dimensions of the EDM chord defect, thus assuming the EDM defect is a crack. Previous studies(18) have shown that reductions in the notch root radius can decrease ΔK_{th} measured in this fashion. The defects used in this study were introduced using a razor blade as the EDM defect which has a notch root radius less than 1 mil. The defect geometry was kept constant during this study, so there should be no notch root radius effects. There is still the question as to whether the ΔK_{th} reported here represents an estimate of the true ΔK_{th} or the stress required to nucleate a crack at the sharp notch. In addition, cracks growing from the chord defect at low stresses, such as those used in this program, have been observed to tunnel preferentially at the center of the notch. This is not considered using the uniform growth algorithm described by Gangloff(9) and used in this study. This may result in errors in the ΔK calculation in the region of low crack growth.
The other two fatigue samples had a uniform cross-section of 0.25 inch and were tested in a strain control mode, using a total longitudinal strain range of 0.66 percent \( (A_e = 1.0) \) and a triangular wave shape. These strain control samples had the 0.004 inch radius EDM defect. The strain control tests were terminated after approximately 0.035 inch of crack growth. The cycles required to grow the crack to this length is defined as cycles to termination \( (N_t) \). This value is in excess of 90 percent of the cycles to failure for these experimental conditions in Rene' 95. Under most conditions a test of this type performed on a material with the strength and modulus of Rene' 95 has a starting \( \Delta K \) of 10 or 11 ksi \( \sqrt{\text{in}} \). The strain control test provides both Region II crack growth rates and \( N_t \) which is an experimental measure of residual life. Previous investigations \(^{(10,18)} \) have shown that \( N_t \) is a single parameter which reflects changes in either and/or both Regions I and II of the fatigue crack growth curves. In these strain controlled tests the specimen yields to approximately 0.1 percent plastic strain during the first fatigue cycle. For the remainder of the test, the specimen is cycled with no detectable plasticity at the maximum tensile stress and minimum compressive stress set by the first cycle. It should be noted that in these tests, \( N_t \) is probably influenced by both the maximum applied stress set first loading cycle and the crack growth rate curve. The measure of maximum \( (\sigma_{\text{max}}) \) and minimum stress \( (\sigma_{\text{min}}) \) used during this report will be that value measured at half of \( N_t \).
The tensile properties at room temperature and 1000°F have been determined for the PM conditions listed in Table I. Strain and load control cyclic defect tolerance tests were also performed on these compacts. Table IX lists the tensile properties and $N_t$ values for the PM materials. Also shown in this is $\sigma_{\max}$ and $\sigma_{\min}$ at $N_t/2$. The values are listed in the same order, i.e. the first $N_t$ value listed corresponds to the first $\sigma_{\max}$ and first $\sigma_{\min}$ value.

Figures 37 shows the variation of the 1000°F yield and ultimate tensile strengths of -140 mesh Rene' 95 as a function of HIP and heat treatment temperatures. All the data shown in this figure represent materials which were quenched into 1000°F salt and subsequently aged at 1400°F for 8 hours.

Several trends are apparent from Figure 37. When this alloy is solution treated below the $\gamma'$ solvus temperature (approximately 2135°F), increasing solution temperature generally increases both yield and ultimate tensile strength. By solution treating at a lower temperature, a larger fraction of the $\gamma'$ is formed during solution treatment and thus, a smaller amount of $\gamma'$ is available for precipitation hardening and the strength is thereby diminished. This trend is identical to those observed in previous investigations. The exception is the Rene' 95 which was HIP'ed at 1925°F. For this temperature, solution treatments below the $\gamma'$ solvus decreased the ultimate tensile strength but had no effect on the yield strength.

Figure 37 also shows that HIP'ing or solution treating above the solvus temperature diminishes both yield and ultimate tensile strength, most likely due to the larger grain size. It appears that the ultimate tensile strength is affected more than the yield strength. A previous investigation suggested that this results from a higher work hardening rate in the finer grain material.
Figure 37. Variation of the 1000°F Yield and Ultimate Tensile Strengths of -140 Mesh Rene 95 as a Function of HIP and Solution Treatment Temperature.
The other major variation examined for -140 mesh powder were changes in quench rates and aging temperatures. The influence of these variations on yield and ultimate tensile strength are shown in Figure 38. All the conditions shown in this figure were HIP'ed and solution treated at 1550°F. The aging times were 8 hours. Figure 38 shows that Rene' 95 which was quenched into 1000°F salt (circles) increases in strength with a 1400°F age but is overaged at 1800°F. A slower quench rate, 1500°F versus 1000°F salt quench, decreases both yield and ultimate tensile strengths for Rene' 95 aged at 1400°F. Examination of micrographs in Figure 7 shows that the 1800°F age and the 1500°F salt quench conditions result in coarser γ' distributions, most probably the reason for the loss in strength.

Examination of Table IX shows that -400 mesh powder has a higher strength than -140 mesh compacts for both 2050°F (Compacts N12-B and N1-B) and 2200°F (Compacts N12-A and N5-A) HIP cycles. Most of the strength probably results from reduced grain size and finer γ' distributions.

The defect tolerance results will be presented using two types of data--Nt values and da/dn - ΔK curves. In the crack growth rate figures, the baseline curve from Figure 36 will be shown for comparison. The Nt value is an experimental value of residual life for a controlled initiation site. Figure 39 shows the variation of Nt with HIP and solution temperature for -140 mesh Rene' 95. Several trends are apparent in these data. First, in materials compacted below the γ' solvus (circles and triangles), increasing the solution temperature increases defect tolerance. Second, HIP compaction at 2200°F results in higher Nt values. When compared to results of a previous study, there are two surprising results. The excellent results obtained from the compact HIP'ed at 2050°F and solution treated at 2200°F differ from that of VanStone and Gangloff.
Figure 38. Variation of 1000°F Yield and Ultimate Tensile Strengths of -140 Mesh Rene 95 as a Function of Quench and Aging Conditions.
Figure 39. Variation of Cycles to Termination ($N_t$) at 1000°F with Solution and HIP Temperatures for -140 Mesh Rene 95.
which suggested that HIP'ing above the γ' solvus is more beneficial than super-solvus solution treatments. The major differences between these two studies is the quench rate from the super γ' solvus temperature.

In this investigation the alloy was quenched into 1000°F salt. In the previous study, a Rene' 95 compact was quenched into 2090°F salt followed by a 1000°F salt quench. Based on the quench bath temperatures, it would be expected that the material evaluated in this program has a higher quench rate through the γ' solvus. Comparing the micrographs in Figure 6 to those from the previous study shows that the material from Compact N4-C had a finer γ' size distribution. This confirms that it has a higher quench rate which suppressed nucleation of large and intermediate γ'. These differences in distribution of γ' may alter deformation modes and, in turn, defect tolerance.

The second surprising result is from the most defect tolerant metallurgical condition identified to date in this investigation: 2200°F HIP cycle with a 1925°F solution treatment. The low solution temperature for this condition contradicts a previous study.\(^{(10)}\) That investigation showed that decreasing the solution temperature for Rene' 95 HIP'ed at 2050°F decreased defect tolerance in a fashion similar to the 2050°F HIP portion of this study; however, no evaluation of the influence solution temperature on Nₜ with material HIP'ed above the γ' solvus was performed. At this time it is difficult to explain this behavior.

It is also interesting to consider the position of Compact N1-A, the condition aged at 1800°F on Figure 39. Table IX shows the average Nₜ value for the Compact is 3255 cycles. This would continue the trend shown for sub-solvus processing shown in Figure 39. This point is not shown in Figure 39 because compact N1-A was not quenched or aged after the 1800°F treatment.
It should be noted that Compact Ni-B, which is the baseline condition, was solution treated at 2050°F and has an \( N_t \) value of approximately 4500 cycles. In the previous study \(^{(10)}\) when Rene' 95 powder was processed identically, except that it was solution treated at 2090°F, the \( N_t \) value was 6000 cycles. The crack growth rates for compact Ni-B, which are shown in Figure 36, are higher than those reported previously by VanStone and Gangloff \(^{(10)}\). The major difference between these two investigations are the powder source and solution temperature. Based on the strong influence of solution treatment on defect tolerance, it is believed that this is the major difference between the \( N_t \) values in the two studies.

The crack growth rate curves for the conditions represented in Figure 39 are shown in Figures 40 through 44 along with the baseline curve from Figure 36. Figures 40 and 41 show the crack growth rate curves of -140 mesh Rene' 95 which was HIP'ed at 1925°F and solution treated at 1925°F and 2050°F respectively. These conditions are represented by circles in Figure 39. The curves in Figures 40 and 41 show that Rene' 95 HIP'ed at 1925°F seems to have a lower fatigue crack growth threshold \( (\Delta K_{th}) \) than the baseline condition. Both these conditions have Region II crack growth rates similar to or slightly lower than the baseline conditions. These results suggest that HIP'ing well below the \( \gamma' \) solvus temperature may decrease defect tolerance due to higher crack growth rates in the threshold regime. The similarity in \( N_t \) values between these compacts and the baseline condition is not surprising in that they all have similar Region II crack growth rates and that the initial \( \Delta K \) in the strain controlled test is approximately 10 ksi \( \text{in.} \), therefore nearly into the Region II crack growth regime.
Figure 40. Fatigue Crack Growth of -140 Mesh Rene 95 which was HIP at 1925° F, Solution Treated at 1925° F, Quenched into 1000° F Salt and Aged at 1400° F.
Figure 41. Fatigue Crack Growth Curve of -140 Mesh Rene' 95 Which was HIP at 1925° F, Solution Treated at 2050° F, Quenched into 1000° F Salt, and Aged at 1400° F.
Figure 42 shows the fatigue crack growth curve of Compact N4-C. This material was HIP'ed at 2050°F and subsequently solution treated at both 2200°F and 2050°F. Each solution treatment was followed by a 1000°F salt quench. This processing cycle resulted in the very fine $\gamma'$ distribution shown in Figure 6. There is only one set of load control test data shown in Figure 42 due to the quench cracking which occurred in this compact. This frequently occurs when quenching large grained structure through the $\gamma'$ solvus. Figure 42 shows that this compact has a Region I behavior similar to that of the baseline material but has much reduced Region II crack growth rates relative to the baseline. In the vicinity of 15 ksi $\sigma_{\text{in}}$, there is an arch in the crack growth rate curve from one strain controlled test. This resulted from an excursion in the potential-cycle data and is considered to be an artifact. The improved defect tolerance for this condition (triangles in Figure 39 at 2200°F) results mainly from the change in the Region II crack growth rates.

Figures 43 and 44 show the crack growth curves of -140 mesh Rene' 95 which were HIP'ed at 2200°F. These conditions are represented by squares in Figure 39 and are among the most defect tolerant conditions studied in this program. Compact N5-A was solution treated at 2050°F and is shown in Figure 43 while compact N5-B was solution treated at 1925°F and shown in Figure 44. Both conditions appear to have a slightly higher $\Delta K_{\text{th}}$ than the baseline condition and the Region II crack growth rates appear to be approximately 30 percent lower than the baseline condition. The Region II crack growth rates for compact N5-B (Figure 44), the compact solution treated at 1925°F, appears to be lower than those in compact N5-A (Figure 43) which was solution treated at 2050°F. The high
Figure 42. Fatigue Crack Growth Curve of -140 Mesh Rene 95 which was HIP at 2050° F Solution Treated at 2200° F, Quenched into 1000° F Salt, Re-Solution Treated at 2050° F, Quenched into 1000° F Salt, and Aged at 1400° F.
Figure 43. Fatigue Crack Growth Curve of -140 Mesh Rene 95 which was HIP at 2200°F, Solution Treated at 2050°F, Quenched into 1000°F Salt, and Aged at 1400°F.
Figure 44. Fatigue Crack Growth Curve of -140 Mesh Rene 95 which was HIP at 2200° F, Solution Treated at 1925° F, Quenched into 1000° F Salt and Aged at 1400° F.
$N_t$ values in compact N5-B probably result from both the high $\Delta K_{th}$ and lower Region II crack growth rates. It is interesting to note that the strain-controlled tests shown in Figure 44 have extremely long artificial tails. It is felt that this indicates a resistance to crack growth at low crack growth rates or improved $\Delta K_{th}$.

Several general trends can be made from the data presented in Figure 40 through 44. The crack growth behavior in the Region I (threshold) and Region II portions of the curve appear to result from different processing effects. The crack growth threshold seems to increase with increasing HIP temperature. On the other hand, the Region II crack growth rates seem to be related to the HIP'ing above the $\gamma'$ solvus which increases the grain size, refines the $\gamma'$ size, and apparently alters the $\gamma'$ chemistry. Figures 40 through 44 show that processing Rene' 95 above the $\gamma'$ solvus retards crack growth rates. In addition, the material which was solution treated above and quenched through the $\gamma'$ solvus had an even lower Region II crack growth rate (Figure 42). This compact had a very fine and uniform distribution of $\gamma'$.

Figure 45 shows the variation of $N_t$ with aging and quenching temperatures. All the data in this figure represent -140 mesh Rene' 95 which was both HIP compacted and solution treated at 2050°F. Figure 45 shows that the defect tolerance of material aged at 1400°F is similar to that which received no aging; however, aging at 1800°F results in a lower $N_t$ value.

Figure 46 compares the fatigue crack growth curve of compact N4-B to the baseline condition. These two materials were processed identically except that compact N4-B did not receive the 8 hour, 1400°F age of the
Figure 45. Variation of Cycles to Termination ($N_t$) at 1000°F with Quench Rate and Aging Conditions for -140 Mesh René 95.
Figure 46. Fatigue Crack Growth Curve of -140 Mesh Rene 95 which was HIP at 2050° F, Solution Treated at 2050° F, Quenched into 1000° F Salt and was not Aged.
baseline condition. The absence of the age appears to reduce \( \Delta K_{th} \) but does not alter the Region II crack growth rates.

Figure 47 shows the fatigue crack growth rate curve of compact N1-A, which is identical to the baseline compact except that it was aged at 1800°F rather than 1400°F. The threshold crack growth rates are similar for both conditions but the higher aging temperature in compact N1-A results in higher Region II crack growth rates. This behavior most likely caused the large decrease in \( N_t \) values with increasing aging temperatures shown in Figure 45. Previous studies\(^{18,27}\) have shown in several superalloys that a 1600°F aging treatment results in a lower defect tolerance than a 1400°F age condition primarily due to increased fatigue crack growth rates in the Region II portion of the fatigue crack growth.

Figure 45 also shows that decreasing the severity of the quench slightly improves defect tolerance. The fatigue crack growth data for compact N1-C is shown in Figure 48. Compact N1-C was quenched from the solution treatment into 1500°F salt while baseline compact N1-B was quenched into 1000°F salt. There is a larger variation in crack growth rate between the two strain controlled tests in Figure 48 (squares) than is ordinarily observed. Based on the load controlled tests, it is believed that the crack growth rates for the slower quench rate compact are similar to those of the baseline Rene' 95. This seems to be reasonable in that the approximate 10 percent improvement in defect tolerance shown in Figure 45 would be difficult to observe in the log-log plot in Figure 48.

The variations in \( N_t \) in Figure 45 are interesting when considering the microstructure of these conditions shown in Figure 7. Increasing
Figure 47. Fatigue Crack Growth Curve of -140 Mesh René 95 which was HIP at 2050° F, Solution Treated at 2050° F, Quenched into 1000° F Salt, and Aged at 1800° F.
Figure 48. Fatigue Crack Growth Curve of -140 Mesh Rene 95 which was HIP at 2050°F, Solution Treated at 2050°F, Quenched into 1500°F Salt, and Aged at 1400°F.
the aging temperature, which reduces defect tolerance, increases the size of intermediate-sized \( \gamma' \) but a fine distribution of \( \gamma' \) is still observed (Figure 7c). On the other hand, Rene' 95 quenched at a slower rate from the solution temperature seems to have a more uniform distribution of these finer \( \gamma' \) precipitates (Figure 7b). This data suggests that a finer and more uniform distribution of \( \gamma' \) will improve defect tolerance.

The influence of reduced powder size was investigated for two conditions. The finer powder has a more rapid solidification rate as evidenced by reduced dendrite arm spacing\(^{(10)}\) and thus is expected to have more uniform initial composition variations. Both -140 mesh (smaller than 105 micron) and -400 mesh (smaller than 37 micron) powder was HIP compacted at both 2050°F and 2200°F. The processing, strengths, and \( N_t \) values are repeated in Table X for easy comparison. All the compacts were solution treated at 2050°F, quenched into 1000°F salt, and aged at 1400°F for 8 hours. It should be noted that the time at the solution temperature was one hour for all four materials except compact N5-A which was solution treated for 6 hours. Table X shows that the improvement in \( N_t \) with either super \( \gamma' \) solvus HIP'ing (compact N5-A) or reducing powder size (compact N12-B) is on the order of 10 percent. When both of these variations are combined there is an improvement of approximately 45 percent in defect tolerance. The improvement reported here is similar to those reported for -140 and -270 mesh Rene' 95.\(^{(10)}\) The crack growth rate determination of these two conditions were not completed during this investigation so it is difficult to fully understand this improvement. The previous investigation\(^{(10)}\) strongly suggested that the use of fine powder and super \( \gamma' \) solvus HIP'ing improved \( \Delta K_{th} \).

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TABLE X

COMPARISON OF -140 AND -400 MESH RENE' 95

PROCESSING AND PROPERTIES

<table>
<thead>
<tr>
<th>COMPACT CODE</th>
<th>MESH SIZE</th>
<th>HIP* TEMPERATURE (°F)</th>
<th>0.2% YIELD STRENGTH (ksi)</th>
<th>ULTIMATE TENSILE STRENGTH (ksi)</th>
<th>Nt CYCLES</th>
</tr>
</thead>
<tbody>
<tr>
<td>N1-B</td>
<td>-140</td>
<td>2050</td>
<td>167.7</td>
<td>229.0</td>
<td>4250, 4575</td>
</tr>
<tr>
<td>N5-A</td>
<td>-140</td>
<td>2200</td>
<td>161.9</td>
<td>219.7</td>
<td>4625, 5800</td>
</tr>
<tr>
<td>N12-B</td>
<td>-400</td>
<td>2050</td>
<td>173.0</td>
<td>231.8</td>
<td>4625, 5375</td>
</tr>
<tr>
<td>N12-A</td>
<td>-400</td>
<td>2200</td>
<td>165.9</td>
<td>223.3</td>
<td>6350, 6375</td>
</tr>
</tbody>
</table>

* Compacts were solution treated at 2050°F, quenched into 1000°F salt, and aged at 1400°F.
As noted previously, HIP'ing above the \(\gamma'\) solvus alters the microstructure in three basic ways—increases the grain size, refines the \(\gamma'\) distribution, and alters the \(\gamma'\) shape presumably due to the influence of \(\gamma'\) composition on \(\gamma-\gamma'\) mismatch. Comparison of the optical micrographs of -140 and -400 mesh Rene' 95 HIP'ed at 2200°F in Figures 1c and 1b, respectively, show that the -400 mesh material has a smaller grain size. The fact that -400 mesh compact HIP'ed at 2200°F had higher \(N_t\) values than its -140 mesh counterpart may suggest that \(\gamma'\) composition and distribution may have a larger influence on defect tolerance than grain size.

The \(N_t\) values reported in this study were determined in a strain control fatigue test with a total strain range of 0.66 percent. For an alloy with the strength and elastic modulus of Rene' 95, the material deforms during the first loading cycle to a plastic strain of approximately 0.1 percent. For the remainder of the test, the alloy is strained elastically with very little change in the maximum tensile or minimum compressive stresses. If the fatigue crack growth rates do not change, one would expect that reducing yield strength would improve \(N_t\) because the maximum stress during strain cycling (\(\Delta \sigma_{\text{max}}\)) and, in turn, the initial range in stress intensity factor would decrease. As a result, \(N_t\) would increase with decreasing yield strength. Figure 49 shows the variation of 1000°F \(N_t\) with 1000°F yield strength for -140 and -400 mesh Rene' 95. In general, there seems to be no such trend in this figure, in that the lowest strength condition evaluation (Compact N1-A) with its 1800°F age also had the \(N_t\) value. It is interesting to compare the \(N_t\) values of this 1800°F age (Compact N1-A) with its average \(N_t\) value of 3255 with Compact N12-A which contains -400 mesh powder which was HIP'ed at 2200°F and had an average \(N_t\) value.
Figure 49. Variation of Cycles to Termination ($N_t$) at 1000°F with 1000°F 0.2% Offset Yield Strength of -140 and -400 Mesh Rene 95.
value of 6330. As shown in Table IX, Compact N12-A had an 18 percent higher 1000°F yield strength and an 8 percent higher $T_{\text{max}}$, than Compact N1-A. If the defect tolerance of these two conditions were compared at the same stress, the differences would probably be larger than those revealed by $N_t$. Conversely, those improvements which are accompanied by decreases in yield strength and thus $T_{\text{max}}$, would be diminished. The two conditions which fall in this category are Compact N1-C with its 1500°F salt quench and Compact N5-B which was HIP'ed at 2200°F but solution treated at 1925°F, well below the $\gamma'$ solvus.

For those materials which were both HIP'ed and heat treated at temperatures below that of the $\gamma'$ solvus (open points), defect tolerance seems to increase with yield strength. The closed points in Figure 49 represent data on materials which were processed at 2200°F and show a significant improvement with respect to materials processed below the $\gamma'$ solvus. This condition also seems to show the expected trend of decreasing $N_t$ with increasing strength.

Figure 49 shows that -400 mesh powder which was HIP'ed at 2200°F (closed squares) seems to have the best combination of strength and defect tolerance. One of the more defect tolerant conditions evaluated was -140 mesh powder, HIP'ed at 2200°F, and solution treated at 1925°F (closed circles) with $N_t$ values of approximately 7000 cycles. As previously mentioned this result must be tempered by its lower $T_{\text{max}}$ and yield strength. Comparison of Figures 43 and 44 show that the Region II crack growth rates of compacts N5-A and N5-B (2200°F HIP, 2050°F and 1925°F solution respectively) are similar. Based on the results to date, one might expect even further improvement in both strength and defect tolerance if -400 mesh powder was processed in this fashion.
The room temperature and 1200°F tensile properties and 1000°F defect tolerance of cast and wrought (C+W) Rene'95 have been determined in a fashion similar to that for the powder metallurgy (PM) Rene'95. Table XI lists the tensile properties and $N_t$ values for this material. The tensile properties are typical for C+W Rene'95. These strengths for this material are slightly higher than those for the PM materials evaluated in this program. This most likely results from the residual work in the necklace microstructure.

The $N_t$ values for C+W are higher than the PM baseline. The position of C+W on the PM $N_t$ yield strength comparison in Figure 49 shows that C+W appears to be an extension of the sub-solvus processed PM data. Figure 50 shows the fatigue crack growth curve of C+W Rene'95. The three load control test results shown in this figure are those reported by Kelly, Gangloff and Henry. Also shown in this figure is the PM baseline curve from Figure 36. The crack growth curve of C+W Rene'95 appears to have a slightly higher $\Delta K_{th}$ and a lower Region II crack growth rate. The $\Delta K_{th}$ and Region I crack growth rates were determined using a chord defect introduced with a different type of electrode which results in larger notch root radius. For this reason, this comparison of $\Delta K_{th}$ may not be valid. The lower Region II crack growth rates are consistent with the higher $N_t$ values in the C+W Rene'95. The specimens from the forged disk have $N_t$ values close to that of PM Rene'95 Compact NS-A which contained -140 mesh powder which was HIP'ed at 2200°F. Comparison of the fatigue crack growth curves for these two conditions (Figures 43 and 50) show that crack propagation rates are similar.
TABLE XI

PROPERTIES OF CAST AND WROUGHT RENÉ 95

<table>
<thead>
<tr>
<th>TEMPERATURE (°F)</th>
<th>0.2% OFFSET YIELD STRENGTH (ksi)</th>
<th>ULTIMATE TENSILE STRENGTH (ksi)</th>
<th>ELONGATION (%)</th>
<th>REDUCTION IN AREA (%)</th>
<th>N* (CYCLES)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Room Temp.</td>
<td>199.0</td>
<td>243.8</td>
<td>13</td>
<td>13</td>
<td>--</td>
</tr>
<tr>
<td>1000</td>
<td>--</td>
<td>--</td>
<td>--</td>
<td>--</td>
<td>5200, 5550</td>
</tr>
<tr>
<td>1200</td>
<td>182.3</td>
<td>222.3</td>
<td>10</td>
<td>15</td>
<td>--</td>
</tr>
</tbody>
</table>

*The max. Values For These Tests Were 160 And 161 KSI; The min. Values Were -12 And -18 KSI Respectively.
Figure 50. Fatigue Crack Growth Curve of Cast and Wrought Rene 95.
Surface Enhancement Layers

The only surface enhancement treating which was evaluated for mechanical properties was the high intensity shot peen layers. The mechanical property evaluations on the shot peened specimens were the same as those reported in the powder metallurgy section, except that no load control tests have been performed on the shot peened samples at this time.

Table XII lists the tensile properties of the shot peened samples. This table also contains the results of Compact NL-B which was reported in Table IX for comparison purposes. Both compacts had identical processing except for the shot peening. The shot peened material has slightly lower strengths and significantly lower tensile ductility. The decrease in tensile properties probably results from the tensile residual stress at the interior of the specimen.

Figure 51 shows the variation of crack depth, as measured using the DC potential drop tests for the strain control tests on peened and unpeened specimens containing the 4 mil radius semicircular defect. At short crack depths, the crack growth rates in the shot peened samples are much slower than those without surface enhancement. Deep in the sample, the crack growth rates are very similar. The lines shown between 30 and 40 mils of crack depth for each test have the same slope. Thus, it appears that the crack growth rates are similar in this portion of the sample, a region presumably beyond the high residual compressive layer. The maximum tensile stress was 146 and 147 ksi in the tests on shot peened samples while those stresses for samples from Compact NL-B as shown in Table IV are approximately 6.5 percent higher. The $\sigma_{\text{min}}$ values for the shot peened samples are -26 and -25 ksi, slightly lower than those for the baseline tests.
<table>
<thead>
<tr>
<th>PROPERTY</th>
<th>COMPACT N6-A (SHOT PEENED)</th>
<th>COMPACT N1-B (NOT SHOT PEENED)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.2 Percent Offset Yield Strength (ksi)</td>
<td>171.5 165.3</td>
<td>180.1 167.7</td>
</tr>
<tr>
<td>Ultimate Tensile Strength (ksi)</td>
<td>220.0 201.8</td>
<td>240.8 229.0</td>
</tr>
<tr>
<td>Tensile Elongation (%)</td>
<td>6.6 3.8</td>
<td>16.6 14.0</td>
</tr>
<tr>
<td>Reduction in Area (%)</td>
<td>10.9 7.8</td>
<td>17.2 17.8</td>
</tr>
</tbody>
</table>
Figure 51. Variation in Crack Depth with Fatigue Cycles for Shot Peened and Non-Shot Peened Rene 95.
These results suggest that the residual stresses act to alter the effective stress so that crack growth is retarded in the presence of compressive residual stress. The $N_e$ values for the shot peened samples were 16050 and 19525 cycles, roughly four times greater than the unpeened condition. This should not be interpreted as an improvement in inherent defect tolerance for several reasons. First, this test only compares life of surface defects. In the shot peened condition, samples with crack initiation sites in regions of residual tensile stresses may have lower lives than an unpeened sample with an identical nucleation site. Second, the degree of improvement for surface nucleation sites is highly dependent on the defect size because the test is being conducted in a residual stress gradient. Presumably, samples with smaller defects would show a greater improvement while the improvement for a larger defect would be smaller.
DISCUSSION AND CONCLUSIONS

This investigation has studied the fatigue process in powder metalurgy (PM) and cast and wrought (C+W) Rene' 95. In aircraft turbine disk materials, the lowest fatigue lives result from conditions where fatigue cracks nucleate and grow from non-intentionally added inclusions such as ceramics.(1-6) This type of fatigue process was studied using two experimental techniques--replication metallographic study of intentionally doped PM products and defect tolerance evaluation of PM and C+W Rene' 95 with carefully controlled size, shape, and orientation EDM defects.

The replication study showed that in this type of fatigue process, crack nucleation occurs during the first one percent of fatigue life with the remainder being crack growth. Thus, the development of a defect tolerant disk alloy depends upon identification of a material with slower fatigue crack growth rates.

Most of the remainder of this investigation was spent studying the microstructure, defect tolerance, and crack growth characteristics of C+W Rene' 95 with a necklace microstructure, eleven variations of PM Rene' 95, and shot peened Rene' 95.

The critical question is what metallurgical processing results in a defect tolerant microstructure. Examination of Figure 49 shows that the materials which have the high defect tolerance have large grain size due to either HIP or solution temperatures above the \(\gamma'\) solvus (closed points). In addition, the necklace C+W disk, with its large warm worked grains, also had high \(N_t\) values for its strength level. Processing a \(\gamma-\gamma'\) super-alloy above its \(\gamma'\) solvus can result in three simultaneous microstructural
changes--increase in grain size, refinement in \( \gamma' \) distributions, and alteration of \( \gamma' \) chemistry. The first two have been observed directly while the change in \( \gamma' \) composition has been inferred based on the variation in \( \gamma' \) shape such as shown in Figure 3. The change in shape most likely results from a change in the \( \gamma - \gamma' \) mismatch.

The influence of increasing grain size on the retardation of crack growth rates is well documented in nickel-base alloys,\(^{(10, 22, 23)}\) titanium alloys\(^{(24, 25)}\) and steels.\(^{(27)}\) One has to be extremely careful in attributing improved defect tolerance in superalloys solely to increase grain size due to the alteration in \( \gamma' \) distribution and shape. For all the PM conditions evaluated in this program, the apparent fatigue crack growth threshold \( (\Delta K_{th}) \) as measured by the stress intensity required to break free from a sharp EDM notch seemed to increase with HIP temperature. Those conditions HIP'ed at 2200°F as shown in Figures 43 and 44 appeared to have \( \Delta K_{th} \) values above that of the 2050°F HIP baseline. It appears that the threshold of Compact N4-C which was HIP'ed at 2050°F and solution treated at 2200°F has a \( \Delta K_{th} \) similar to the baseline condition. Thus, it appears that HIP'ing above the \( \gamma' \) solvus and not just a thermal cycles is necessary to improve \( \Delta K_{th} \). These results are similar to the results reported by VanStone and Gangloff\(^{(10)}\) in an earlier study.

The Region II crack growth rates appear to be influenced by both super \( \gamma' \) solvus processing and alterations in \( \gamma' \) distribution resulting from sub \( \gamma' \) solvus heat treatments. As noted previously, studies of aging and quenching variations showed that Region II crack growth rates were accelerated when there were sharply divided \( \gamma' \) distributions of intermediate and small sized \( \gamma' \), such as in Rene' 95 aged at 1800°F (Figure 7).
Thus, it appears that the best microstructure would be a totally uniform $\gamma'$ distribution. The condition with that type of structure was compact N4-C which was solution treated at 2200°F and quenched through the $\gamma'$ solvus. This was the only condition with a rapid cool through the $\gamma'$ solvus. The microstructure and crack growth curve of this material is shown in Figures 6 and 42 respectively. This compact had the lowest Region II crack growth rates of any condition. In addition, this was the only condition where a major change in the slope of the Region II crack growth curve was observed. It should be noted that this type of heat treatment is very impractical for high $\gamma'$ content superalloys like Rene' 95 due to the high probability of quench cracking.

The next best condition for Region II crack growth appears to be those where there is a distinctly bimodal distribution of $\gamma'$ with only large and small $\gamma'$. This situation only occurs when the material is processed above the $\gamma'$ solvus. During these high temperature cycles the intermediate sized $\gamma'$ which forms in prior interdendritic regions are put into solution. All the large grained Rene' 95 had bimodal $\gamma'$ distribution and had retarded stage II crack growth rates. Again, this is consistent with results of previous studies on Rene' 95.\(^{(10, 22)}\)

Although several observations have been made relating to $\gamma'$ distribution and Region II $da/dN$ values, the grain size may still play an important role.

The processing conditions in this program were designed, in part, to attempt to determine if compositional homogeneity was necessary to improve defect tolerance. In a previous study,\(^{(10)}\) a correlation between $N_e$ values and homogeneity was noted using data similar in nature to that shown in Table X. As in that study,\(^{(10)}\) this investigation showed that
the combination of fine powder with its smaller dendrite arm spacing with super $\gamma'$ solvus HIP cycles leads to improved defect tolerance. Several comparisons help to shed light on the role of homogeneity on defect tolerance. The first comparison is between compacts N4-C and N5-A where the HIP and solution exposures were switched. Compact N4-C which was solution treated at 2200°F was resolution treated at 2050°F in an attempt to set similar $\gamma'$ populations. This material was also quenched from the 2200°F solution cycle. The defect tolerance of this material was slightly higher than the Rene' 95 HIP'ed at 2200°F. This suggests that maybe the fine $\gamma'$ distribution in the material quenched from 2200°F has a larger role than homogeneity. This concept is reinforced when compacts N5-A and N5-B are compared. Both of these compacts were HIP'ed at 2200°F, but N5-A was solution treated at 2050°F while N5-B was solution treated at 1925°F. Both materials received an identical quench and age. Based on the concept of homogeneity, the material with high solution temperature should have the greater $N_e$ value, but the opposite behavior occurred. Again, this suggests that homogeneity alone does not control defect tolerance.

This investigation has shown that the defect tolerance of Rene' 95 can be varied by a factor of two. No clear cut correlation between microstructure and fatigue properties could be made. The most obvious missing link is the study of the cyclic deformation modes. The study of the deformation modes might help to explain a very interesting behavior. Figure 39 shows that when materials are HIP'ed below the $\gamma'$ solvus, defect tolerance increases with increasing solution temperature. Those conditions HIP'ed above the solvus have an opposite trend. A
similar type of behavior is shown in Figure 49 in that the defect
tolerance of material processed below the \( \gamma' \) solvus (open points) in-
creases with increasing strength while Rene' 95 processed above the \( \gamma' \)
solvus has the opposite trend.

This study also helped to identify a processing sequence for Rene'
95 with a 45 percent improvement in defect tolerance with less than 5
percent loss in strength properties. This condition is -400 mesh powder
which was HIP'ed at 2200°F. Of all the metallurgical conditions evaluated in this
program, this condition shows the most promise to be able to manufac-
ture production hardware with a minimum of process development. It is
not clear that powder as fine as -400 mesh is necessary for this improve-
ment in that VanStone and Gangloff\(^{10}\) noted similar improvements using
-270 mesh powder. The use of fine powder HIP'ed above the \( \gamma' \) solvus
provides an opportunity to significantly improve the fatigue properties
of aircraft engine disks, however, considerable work would be needed to
determine the effect of these processing changes on other properties
and on overall production capability and cost.

Work on high intensity shot peening has suggested that residual stresses
act to modify the applied stress so that retard crack growth is retarded in
regions of residual compressive stresses and accelerated in regions having
residual tensile stresses. For the 4 mil radius semicircular surface de-
fect used in this study, fatigue life was significantly improved. However, it
may be possible to alter or even reverse this improvement with changes in in-
itiation site size and location.

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REFERENCES


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