DEVELOPMENT OF HOT ISOSTATICALLY PRESSED (AS-HIP)

POWDER METALLURGY RENE' 95 TURBINE HARDWARE

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ABSTRACT

This program was directed at producing premium quality aircraft turbine engine parts from as-compacted plus heat treated (As-HIP) Rene' 95 powder. The principal objective was development and demonstration of a reliable, low cost, reproducible production practice for making As-HIP turbine hardware.

The investigation was conducted by General Electric and two powder vendors, Crucible and CarTech, using argon atomized Rene' 95 powder. Experimental studies were performed to evaluate the effects of powder mesh size and particle size distribution, compaction temperature and pressure, and post compaction heat treatment on microstructure and mechanical properties. Shape definition studies were conducted to determine container fabrication and filling techniques required to produce the target turbine disk configuration.

The selected process consisted of consolidating -60 mesh powder in shaped metal or ceramic containers to full density by hot isostatic pressing at a temperature below the \vee ' solvus. This compaction procedure developed a desirable fine grain (< ASTM 8) microstructure. Heat treatment consisting of solutioning 30F below the \vee ' solvus followed by a 1000F salt quench and double aging treatment produced the required final microstructure and mechanical properties. Dimensional analysis of the final turbine disk shapes indicated both vendors developed shape-making processes capable of accurately and reproducibly fabricating the desired target configuration.

INTRODUCTION

Rene' 95 is a highly alloyed, precipitation strengthened nickel-base superalloy currently being specified for compressor and turbine disks and other hardware in advanced jet engines. Because of its high alloy content and strength characteristics, the alloy is costly and difficult to produce by conventional (cast plus wrought) techniques. Powder metallurgy (P/M)as a method for producing Rene' 95 parts offers potential advantages over conventional techniques. Most notable are lower costs resulting from improved metal utilization and fewer processing steps, and improved property uniformity and reliability due to the inherent homogeneity of powders.

Initial work on Rene' 95 P/M hardware involved development of hot isostatically pressed (HIP) preforms which were subsequently forged and heat treated to achieve properties equivalent to conventionally processed material. Refinement of this process yielded economically viable parts with improved microstructural and mechanical property homogeneity relative to cast plus wrought hardware

With the development of P/M technology, it became apparent that the economic advantages associated with HIP plus forge P/M processing could be enhanced substantially if material conversion ratios could be reduced and all metalworking operations eliminated. An example of the material, processing, and machining savings associated with application of As-HIP powder metallurgy techniques to disk components is illustrated in Figure 1. Elimination of the forging operation, substantial amounts of excess material, and the associated reduced machining operations all contribute to the significant cost reduction potential of As-HIP processing. The potential of As-HIP processing to provide exceptional economic and technical benefits unattainable by any other known practice was the catalyst which initiated this program.

PROGRAM SCOPE AND BACKGROUND

The objective of the program was to develop a production practice for producing flight quality As-HIP hardware from Rene' 95 powder. This task involved two principal areas of endeavor:

- 1. Process Parameter Definition
- 2. Shape Definition

Definition of As-HIP processing parameters encompassed all major variables having an effect on mechanical properties. The processing sequence and associated process variables considered in this study are summarized in Figure 2. In general, the process involves production of Rene' 95 powder, consolidation by HIP to obtain full density, and heat treatment to provide the desired microstructure and mechanical properties. Mechanical property goals for this program, shown in Table I, were based on current capabilities of HIP plus forge hardware.

A production practice for producing Rene' 95 powder was available at the inception of the program. Vacuum induction melting coupled with argon atomization had been proven as a reliable, cost-effective technique for producing powder with low oxygen and nitrogen contents. Thus, investigation of powder production process variables was not included in the program.

The Rene' 95 powder composition is the same as that used for cast plus wrought products with the exception of slightly reduced carbon and chromium contents. This chemistry is uniquely suited to P/M processing, since the balance between carbon content, $M_{C}C$ carbide formers (W and Mo), and MC carbide formers (Cb and Zr) suppresses carbide precipitation at prior powder particle boundaries. Suppression of this phenomenon is considered a prerequisite at

General Electric for all potentially useful P/M alloys. A recently issued General Electric patent (No. 3,890,816) describes the compositional limitations required to inhibit carbide segregation.

Selection of powder particle size can have a significant effect on process economics as well as mechanical property response in a P/M product. Powders are currently screened to -60 mesh in HIP plus forge processing, primarily because this practice has proven to be economically attractive and technically acceptable. However, the potential technical benefits associated with finer powder distributions in As-HIP hardware merited inclusion of a powder size variable in the program in spite of possible economic penalties.

The microstructural objectives for As-HIP Rene' 95 represented a radical departure from the "necklace" structure required in cast plus wrought and HIP plus forge components. The As-HIP process is incapable of producing a necklace microstructure, since development of a duplex structure is dependent on application of mechanical work to a coarse-grained preform. Therefore, microstructural variations during HIP are limited to: 1) development of a uniformly fine grained (ASTM 8-10) structure; or 2) development of a uniformly coarse (ASTM 5-7) grained structure. A fine grain size was desired in this study to permit greater microstructural flexibility, i.e., a coarse grain but the opposite is impossible. A fine grained structure is also more tolerant to subsequent heat treatments near the gamma prime (γ ') solvus temperature.

Initial studies to define fundamental HIP processing variables were completed prior to inception of the heat treatment and shape definition portions of the program. Major variables evaluated were the effects of HIP temperature and pressure on microstructure and resulting mechanical property levels.

Once the HIP structure was defined, the principal parameter affecting mechanical properties in As-HIP material was heat treatment. A two phase study was defined to screen a large number of solution temperature/quench media/aging cycle combinations followed by a more detailed mechanical property evaluation of the most promising treatments.

Shape definition objectives were based on development of a uniform, reproducible material envelope around a target turbine disk shape. Achievement of this goal permitted significant improvements in material utilization relative to current HIP plus forge technology.

Two powder vendors participated in the program. Each evaluated processing parameters and developed shape-making technology. Processing parameter and shape studies were initiated simultaneously by both vendors to maximize program efficiency.

RESULTS AND DISCUSSION

Powder Preparation

Crucible and CarTech each produced argon atomized Rene' 95 powder for the program in their own facilities. Crucible prepared master blends of -60 and -200 mesh powder while CarTech prepared -60 and -100 mesh blends. Individual vacuum-induction melted heats were argon atomized, screened to the desired mesh size, and blended to make the master blends. Chemical compositions of the master blends are shown in Table II. All blends met major and trace element requirements. Typical screen analyses of the -60 mesh products from both vendors are presented in Figure 3.

Process Parameter Definition

Initial Studies

The primary objective of the initial studies was to define the effect of HIP temperature and pressure conditions on microstructure and resultant mechanical properties. Both powder vendors participated in the studies, therefy providing a common base from which processing parameter and shape definition studies could be initiated.

Crucible consolidated -60 mesh and -200 mesh powder in 2½-inch diameter by 15-inch long compacts to study the effect of powder mesh size and HIP temperature on structure and properties. Compacts were consolidated at temperatures of 1950F, 2000F, and 2050F under 15,000 psi pressure for three hours. Density measurements after consolidation indicated all compacts achieved full density.

The effect of HIP temperature on microstructure was similar for both mesh size powders. Photomicrographs of the -60 mesh compacts, shown in Figure 4, indicated consolidation at 1950F and 2000F completely recrystallized all smaller powder particles but did not supply enough energy to recrystallize the larger, higher strength particles. The result was an inhomogeneous structure. Compaction at 2050F yielded an almost completely recrystallized structure. Although most particles recrystallized to a very fine, uniform grain structure (ASTM 8 to 10), a few retained the dendritic structure inherent in argon atomized powders. Some powder particles recrystallized into a relatively small number of grains (ASTM 8) making them more easily identifiable in the very fine grained matrix.

Tensile and stress-rupture properties were determined on all compacts after application of the standard* Rene' 95 heat treatment. Results indicated the 2050F HIP temperature produced the best combination of strength and ductility. The more extensive recrystallization produced at 2050F may have been responsible for this advantage in mechanical properties, since the 2000F solution treatment was probably not adequate to complete recrystallization of the larger particles in the 1950F and 2000F HIP compacts. However, application of a higher solution temperature capable of fully recrystallizing the 1950F and 2000F compacts might produce essentially equivalent microstructures and mechanical properties in materials consolidated over a wide temperature range. A thorough investigation of this effect was not pursued in this study.

CarTech consolidated -60 mesh powder in 3-inch diameter by 6-inch long compacts to study the effect of HIP temperature (1900F to 2050F) and pressure (15,000 and 30,000 psi) on structure and properties. Density measurements after consolidation indicated all compacts achieved at least 99.99% of theortical density.

Effects of HIP temperature and pressure on microstructure were similar to those observed in Crucible compacts, i.e., consolidation at 1900F and 2000F produced incomplete recrystallization, with large powder particles retaining the as-atomized structure. Increasing the HIP pressure to 30,000 psi had a \bullet negligible effect on the extent of recrystallization. As in the Crucible material, compacts consolidated at 2050F showed nearly complete recrystallization. Tensile and stress rupture properties of compacts heat treated using the standard Rene' 95 treatment again indicated the same trends noted in Crucible material.

As a result of these studies, a HIP temperature of 2050F and a pressure of 15,000 psi were selected by both vendors in the program. These conditions produced a uniform, almost fully recrystallized fine grained (finer than ASTM 8) microstructure. A brief mechanical property study also suggested

* Standard Rene' 95 heat treatment - 1650F/4 hrs -> 2000F/1 hr/0Q + 1400F/16 hrs/AC

these conditions produced the best combination of strength and ductility in As-HIP Rene' 95.

Heat Treatment Studies

Material for the heat treatment study was prepared by both Crucible and CarTech in the form of hollow cylindrical billets containing -60 mesh and the finer (-100 or -200) mesh powders. The powders were encapsulated in mild steel containers and HIP at 2050F and 15,000 psi pressure for three hours. Final dimensions of the cylinders were approximately 6.5 inches outside diameter by 2.75 inches inside diameter by 20 inches in length.

Density measurements at three locations in top and bottom billet slices indicated all compacts were fully dense. Microstructural examination of all billets indicated uniform, fine grain structures like those shown in Figure 5 were produced by both powder vendors.

Screening Studies

A number of two-inch thick slices from the -60 mesh powder billets were prepared for the heat treatment study. These slices were designed to simulate the turbine disk section size during heat treatment, thus generating mechanical property data typical of that expected from the full scale disk configuration.

The heat treatment study plan, presented in Table III, was designed to investigate the effects of solution temperature, quench media, and aging treatment on mechanical properties. Each two-inch thick slice was subjected to a solution treatment, then sectioned into quarter segments prior to application of the experimental aging treatments.

Each vendor solution treated two slices (1 and 2) using the standard (wrought) Rene' 95 solution temperature (2000F) followed by an oil quench, and applied eight different aging treatments to the quarter sections to establish a reference condition. The potential of higher solution temperatures was investigated in slices 3 and 4. Solutioning at a temperature just below the Y' solvus temperature (T) increased the quantity of Y' in solution prior to the quenching cycle while still maintaining a fine (ASTM 8 to 10) grain size. Crucible and CarTech used different solution temperatures, since the Y' solvus temperatures of their powders differed by approximately 20F due to minor chemistry variations. Aging treatments were identical to those evaluated with the 2000F solution.

The possibility of encountering quench cracking during the oil quench from T -30F was the primary reason for employing a slightly slower 1000F salt quench ^Son slices 5 and 6. The solution temperatures and aging treatments were identical to those used on slices 3 and 4.

Slices 7 and 8 were used to determine the effect of solution treating at a temperature above the Y' solvus. This procedure produced grain growth, along with dissolution of all large Y' formed during consolidation. Since oil quenching from these temperatures (2165 to 2185F) was anticipated to produce severe quench cracking, two slower cooling treatments were used.

Tensile and stress-rupture properties derived from Crucible and CarTech material in this study were virtually identical. Therefore, no attempt will be made to discuss the results separately. It is beyond the scope of this paper to present a detailed discussion o_2 the results of each heat treatment. The data have been previously published . A summary of the trends and observations follows.

The general effect of solution treatment on mechanical properties can be

described after examining data from samples receiving similar aging cycles. Table IV compares the tensile and stress-rupture properties produced by a solution/quench cycle and a 1600F/1 hr/AC + 1200F/24 hr/AC or similar aging treatment. The 2000F solution followed by oil quenching produced excellent ultimate strengths and ductilities, but yield strengths were near or just below the goals and stress rupture ductilities were marginal. Increasing the solution temperature to T_-30F and oil quenching improved yield strengths significantly while reducing tensile and rupture ductilities slightly. This trend was tempered substantially by using a slower 1000F salt bath quench rather than oil or 500F salt. Yield strengths were virtually unaffected while tensile and stress-rupture ductilities were enchanced significantly. Solutioning above the Y' solvus followed by relatively slow quenches to avoid cracking reduced yield strengths significantly, but improved tensile and rupture ductilities. These property changes were produced by the larger Y' particles precipitated during the slower quench and the coarser grain size (ASTM 5-7) resulting from solutioning above the Y' solvus temperature. An attempt to extract the best of both worlds by solutioning above the Y' solvus and slow cooling followed by re-solutioning below the γ ' solvus and rapid quenching did improve tensile and stress-rupture properties but also resulted in some quench cracking of the two-inch thick hollow cylinder slices.

Detailed Evaluation

Analysis of the results of over 50 solution and aging treatment combinations resulted in selection of eight powder mesh size/heat treatment combinations for detailed evaluation. The combinations, shown in Table V, were again applied to two-inch hollow cylindrical slices identical to those used in the screening evaluation. In addition to tensile and stress-rupture testing, low cycle fatigue (LCF), sustained peak low cycle fatigue (SPLCF) and crack propagation tests were conducted and results compared to data obtained from HIP plus forge material.

The mechanical properties summarized in Table VI indicate several mesh size/heat treatment combinations were essentially equivalent to the forged powder product. Tensile and stress-rupture properties were particularly outstanding, as evidenced by the fact that only three of the eighty data points failed to meet the program goal. Results of 900F strain-controlled LCF testing indicated nearly all combinations exceeded the average HIP plus forge data. At 1050F, load-controlled testing of notched (K_{\pm} =1.85) specimens suggested the two best As-HIP combinations were approximately equivalent to HIP plus forge results. Crack propagation testing, conducted using a precracked K_h specimen⁽²⁾, indicated all As-HIP results were slightly below the average HIP plus forge data'. SPLCF evaluation consisted of load cycling a notched (K_{\pm} =2.0) specimen to maximum stress in 10 seconds, holding at maximum stress for 90 seconds and unloading to a low prestress in lo seconds. These results comparison of disks 6H and 21H and A4C and C6D also suggested finer mesh size powders tended to exhibit lower 1200F SPLCF results.

Analysis of the data generated in the detailed evaluation indicated the best combination of mechanical properties was produced by the mesh size/heat treatment combination used on Crucible disk 6H. A description of the critical processing parameters for As-HIP Rene' 95 are presented in Table VII. Pertinent microstructural features produced by this processing sequence, including grain size and Y' size and distribution, are illustrated in Figure 6.

Shape Definition

The primary objective of this portion of the program was to define processing parameters required to produce the turbine disk target shape. The task was divided into several iterations by both vendors in order to incorporate experience gained in initial trials into refined container designs. Although Crucible and CarTech both used -60 mesh powder in the majority of their trials, some disks containing finer mesh powder were fabricated to determine the effect of mesh size on shape definition and handling procedures. Since each vendor employed different techniques, their results will be reported separately.

Crucible

Crucible chose to approach this task using two distinctly different container materials. The relative merits of metal containers shaped by shear spin forming and ceramic containers prepared using wax molds were examined.

A typical ceramic shell mold used to fabricate the turbine disk target shape is shown in Figure 7, along with examples of the prepared compacts after consolidation. Three shape iterations were completed before an acceptable target shape was achieved. The improvement in shape definition resulting from the iterative technique is illustrated in Figure 8. Dimensions of the second iteration shape before and after consolidation indicate the nature of the HIP compaction process. The concave shape of the bottom surface was corrected by adjusting initial container dimensions and applying more prudent handling techniques to avoid mold cracking prior to HIP. The result of these corrective actions was the third iteration disk, which successfully protected the target shape with a uniform ~ 0.125 -inch material envelope.

Spun metal containers were prepared from Type 321 stainless steel, Inconel 601, and mild steel. A typical stainless container prior to assembly, along with examples of the compacts after consolidation and after container removal are presented in Figure 9. Again, three shape iterations were required to achieve the desired target configuration. Figure 10 indicates the marginal material envelope of the second iteration disk produced with a mild steel container. This deficiency was corrected by changing container dimensions to produce the third iteration disk. The difference in compactibility of -60 mesh and -200 mesh powder is shown by comparing third iteration disks shown in Figures 10b and 10c. The coarser -60 mesh powder due to its superior flow characteristics. Thus Crucible, in three shape iterations, developed the technology required to produce the target turbine disk configuration using two different container materials.

CarTech

CarTech also elected to develop the required shape technology using spun metal containers. Mild steel and stainless steel were both investigated, although most shape trials were made using mild steel containers.

The spun metal containers prior to fabrication were very similar to those shown from the Crucible study in Figure 9. Three shape trials were completed by CarTech to achieve the target configuration. Extensive dimensional analysis of the second shape trial was used to modify the third iteration container design. Results of this dimensional analysis, presented in Figure 11, indicate relatively uniform shrinkage in all areas and excellent shape reproducibility. The improvement in material envelope achieved in the third shape. trial is illustrated in Figure 12. These results indicate CarTech also developed the shape technology required to produce accurate, reproducible turbine disk shapes conforming to the target configuration.

SUMMARY

A new PM technology, designated As-HIP, was developed to produce high quality Rene' 95 jet engine hardware. This technology consists of three primary operations: powder production by argon atomization, compaction to a target shape by hot-isostatic pressing (HIP), and heat treatment to develop

required mechanical properties.

HIP parameters were studied by two powder vendors, Crucible and CarTech, to determine conditions required to produce the desired microstructure and mechanical properties. Heat treatment studies were designed to define the solution temperature, quench media, and aging treatment necessary to yield mechanical properties equivalent to those achieved in HIP plus forge hardware. Shape definition trials were also successfully completed by both powder vendors to permit accurate, reproducible manufacture of the desired target turbine disk shape.

Evaluation of this development study indicates that the new As-HIP P/M technology has the capability to revolutionize the aircraft engine rotating parts industry. The potential to produce premium quality nickel-base superalloy flight hardware at significantly reduced costs relative to conventionally forged or P/M HIP plus forged components makes the As-HIP process one of the most exciting and technologically important metallurgical developments of recent years.

ACKNOWLEDGEMENTS

The authors would like to acknowledge the financial support of the U.S. Army Aviation and Systems Command and guidance of Mr. Gene Easterling and Mr. Jan Lane which helped make this program possible.

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CRUCIBLE	-60 Mesh -200 Mesh	.059 .058	12.84 13.00	3.51 3.53	8.17 8.25	2.60 2.59	3.60 3.53	3.55 3.49	3.35 3.27	.04 .05	.009 .009
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CRUCIBLE	-60 Mesh -200 Mesh	۲.01 01	.15	.003		.004		.08 .18	50* 89*	27* 34*	3* 3*
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	TABLE IV SOREDNING TEST RESULTS ON -60 MESH MATERIAL	I TEST R	ESULT	B	8	ILW HS	RIA.				
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	TABLE Y	1 1	PONDER MESH STZE/HEAT TREATHENT PARAMETERS Included in detailed evaluation	ent parameters Ion
Perfect Variation	Net	Meth Sim	Solution fresteent	Aging Treatment
Crucible	3	8	21007/1 br/10007 Salt Q	16008/1 hr/AC+12008/24 hrs/AC
Creefble	NIS.	8	2100F/1 hr/1000F Selt Q	16009/1 hr/AC+12009/34 hrs/AC
Creekhie	634	8	80757/1 hr/500F SAIt Q	16009/1 hr/AC+12008/84 hrs/AC
Crucible	NCR.	8	20757/1. hr/00	1600P/1 hr/AC+1200P/26 hrs/AC
Carfied	3	3	00/24 V/45208	15008/% hrs/AC412008/24 hrs/AC
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Carthele	Q	100	30/34 1/45LOB	16007/1 hr/AC+12007/24 hrs/AC
Carthoch	-	9 9	2060/1 hr/00	16007/1 hr/AC+38007/24 hrs/AC









