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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

A committee of the National Materials Advisory Board has surveyed the emerging technology of using powder metallurgy (P/M) processing to produce superalloys. Particular attention was directed to the fabrication of turbine disk materials. The key processing steps were identified as being powder atomization, powder handling and containerization, and consolidation and post-consolidation treatments. Consolidated powder properties, quality control,

20. continued

and process economies also were considered. The committee presents several conclusions and makes recommendations that reflect important future needs.

SUPERALLOYS FROM POWDER: PRODUCTION AND PROPERTIES

Report of

COMMITTEE ON SUPERALLOY POWDER ALLOYS

NATIONAL MATERIALS ADVISORY BOARD
Commission on Sociotechnical Systems
National Research Council

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The report has been reviewed by a group other than the authors according to procedures approved by a Report Review Committee consisting of members of the National Academy of Sciences, the National Academy of Engineering, and the Institute of Medicine.

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ABSTRACT

A committee of the National Materials Advisory Board has surveyed the emerging technology of using powder metallurgy (P/M) processing to produce superalloys. Particular attention was directed to the fabrication of turbine disk materials. The key processing steps were identified as being powder atomization, powder handling and containerization, and consolidation and post-consolidation treatments. Consolidated powder properties, quality control, and process economies also were considered. The committee presents several conclusions and makes recommendations that reflect important future needs.

PREFACE

Over the past decade, the production of superalloy parts using powder metallurgy (P/M) processing has moved from the research and development to the commercial stage. P/M technology offers several advantages over conventional ingot metallurgy practice--in particular, avoidance of macrosegregation, reduction in microsegregation of alloying elements, fine-scale microstructure, and increased materials utilization. The recent development of rapid solidification processing for the production of metal powders, with an attendant reduction in dendrite size, has further stimulated the fabrication of parts and materials using P/M processing.

At the urging of and with support from the Department of Defense (DoD), the National Materials Advisory Board (NMAB) instituted a committee study of P/M superalloy technology in the spring of 1979. The specific purposes were:

- To survey the technology of the new generation of superalloy products with emphasis on turbine disks made from powder.
- To assess the potential impact on advanced gas-turbine performance, reliability, fuel economy, and cost.
- To survey foreign developments in the powder field.
- To assess alloy development activities world-wide in light of material shortages, particularly cobalt and chromium.

To provide a framework for its study, the committee identified several key steps in the commercial P/M production route for gas-turbine disks. These included powder atomization, handling, containerization, and consolidation and post-consolidation treatments. Attention also was directed to properties after powder consolidation, quality control, alloying potential and flexibility, and economics. Oxide-dispersion-strengthened P/M superalloys were not considered since blade and vane materials were not of primary concern in this study.

This report constitutes the findings of the committee. Several conclusions are drawn from the study and specific recommendations for future work concerning P/M superalloys in general and turbine-disk technology in particular are made.

ACKNOWLEDGMENTS

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Chapter 1

CONCLUSIONS AND RECOMMENDATIONS

Despite the problems identified in the following conclusions, powder metallurgy (P/M) products are used currently in production parts, and the process has the potential for making even more-superior material. Resolution of the problems identified would lead to economies, broadened applications, and improved jet engine performance.

CONCLUSIONS

1. The predominant commercial atomization processes are the inert-gas and soluble-gas processes followed by the rotating-electrode process.

2. The centrifugal-atomization process, utilizing advanced design concepts, is in the pilot-plant stage, and promising techniques such as ultrasonic atomization are in the laboratory stage.

3. Present understanding of the mechanisms responsible for the break-up of liquid-metal streams via different atomization processes is limited. Correlations between process variables and particle size, particle shape, and distributions of size are empirical.

4. The use of high-thermal-conductivity gas during atomization results in fine structures and the formation of metastable phases, but the practical benefits of these phenomena in turbine-disk applications have not been established.

5. The lack of dependable quantitative methods for identifying the microchemistries and crystal structures of rapidly solidified superalloys has complicated analyses of the beneficial effects of rapid solidification on microstructure before and after consolidation.

6. There is a limitation on achievable convective heat-transfer coefficient at the surface of atomized droplets, but the useful size range of superalloy powders cannot be reduced much below 10 μm without heavy penalties in terms of surface contamination, flowability, and handling. This combination limits the maximum achievable cooling rate in a liquid superalloy droplet to $\sim 10^7$ K/s.

7. A clear distinction must be made between cooling rates in a liquid droplet and during crystalline solidification. The latter is significantly lower (due to the heat of fusion) and is more important.

8. The complex heat-flow and solidification conditions in atomized droplets of engineering alloys are not well understood and indirect deduction of cooling rates during solidification from measured dendrite-arm spacings can be made only under special conditions.

9. Centrifugal atomization lends itself to close control of size distribution of the resulting powders.

10. Hot-isostatic pressing (HIP) of near-net- or sonic-disk-shape components is the most promising present consolidation technique for superalloy powders.

11. Current consolidation techniques primarily use shaped metallic containers. Ceramic containers, which are attractive for use in the production of complex shapes, are still in the development stage.

12. The production of metastable microstructures in powders that have been rapidly solidified offers considerable promise for the development of unique alloy properties. The value of any metastable microstructure will be determined by the extent to which its metastability persists during processing and subsequent use. The ability to retain the fine structure present in powders after consolidation has not been evaluated carefully. The changes in microstructure during the time, temperature, and pressure cycles of consolidation have not been evaluated thoroughly.

13. Existing data are insufficient to permit a direct comparison of the properties of P/M parts with those of conventionally processed components made from cast or forged material.

14. Existing data do not permit a detailed evaluation of the effect of powder-making processes on consolidated properties of superalloys.

15. Current models for evaluating the role of small flaws on the fatigue performance, particularly low-cycle fatigue (LCF), of superalloys are inadequate.

16. Metallic and nonmetallic contaminant inclusions, particularly when large ($\geq$5 μm), decrease low-cycle fatigue.

17. Gas entrapment results in the formation of excessive thermal-induced porosity (TIP) in gas-atomized P/M products, and this has a deleterious effect on properties. No nondestructive inspection techniques are presently available to permit determination of the size and number of inclusions or the amount of porosity in consolidated parts.

18. Near-net shapes currently cannot be inspected satisfactorily using sonic techniques.

19. The alloy composition constraints imposed by cast and wrought technology can be relaxed for P/M alloys. Alloy development programs for disk alloys similar to those under way for blades and vanes may be highly productive.

20. The P/M superalloy compositions presently used originally were developed for conventional casting and forging processes, but they have been modified to make them suitable for P/M processes.

21. The greater homogeneity and increased solubility of alloy elements obtained via rapid solidification of superalloy powders should permit increases in both the solvus temperatures and the amount of the solid solution strengtheners. High-cooling-rate solidification of powders, such as is achieved by various techniques, leads to fine dendrite-arm spacing and to compositional homogeneity, which maximize the contribution of each alloying element to the properties of the alloy. This also makes possible the development of metastable microstructures.

22. A high-gamma-prime solvus temperature is required for long-term alloy stability; however, as the solvus temperature approaches the alloy solidus, processing flexibility is reduced and a trade-off has to be made.

23. Analysis of available economic and cost data suggests that advantages exist for P/M processing of superalloy in disk applications. Existing data are too limited to permit a complete economic analysis. Missing is information concerning such factors as energy utilization and capital investment costs.

RECOMMENDATIONS

1. Ultrasonic and other innovative atomization processes for superalloys should be explored with emphasis on scale-up.

2. Commercial scale-up of the centrifugal atomization process should be encouraged in view of the powder particle-size control possible with this process. Consideration should be given to the comparative cost of the process with respect to existing commercial processes.

3. The development of processes that will reduce the volume fraction and size of nonmetallic inclusions and substantially reduce thermal porosity should be encouraged.

4. Fundamental aspects of the break-up of liquid-metal streams should be studied to provide the background necessary to achieve better control of existing processes and to develop new processes.

5. More studies using high-conductivity gas in conventional gas atomization should be conducted to identify beneficial effects on microstructure and to provide a data base for comparison with the RSR process.

6. Advanced techniques of microstructural analysis, such as scanning transmission electron microscopy (STEM) and scanning auger microscopy (SAM), should be applied more extensively in the characterization of the complex structures obtained during the rapid solidification of superalloy powders.

7. Theoretical studies should be conducted to address nucleation and growth and the sequence of formation of metastable phases and their stability in rapid solidification.

8. New techniques for detecting and separating nonmetallic and other foreign inclusions from superalloy powders should be developed.

9. A data base and predictive modeling capability for producing near-net shapes of complex parts are needed.

10. New container materials and containerization methodology for predictably obtaining near-net shapes with closer tolerances should be explored.

11. The ability to retain metastable phases during hot consolidation of superalloy powders should be evaluated.

12. Novel, low-temperature consolidation techniques that do not destroy the beneficial effects on microstructure of prior rapid solidification should be developed.

13. A data base that will permit direct comparison of the properties of P/M parts with those of conventionally processed components made by forging or casting should be developed.

14. A better LCF data base that will permit comparison of the performance of wrought and various P/M products should be developed.

15. Advanced nondestructive testing (NDT) techniques for the detection and characterization of small defects pertaining to P/M superalloy products should be explored.

16. Reliable and economical techniques for inspecting near-net shapes should be developed.

17. A methodology for determining the effect of small defects on fracture-related properties of P/M products should be developed.

18. A simpler (less time-consuming) and cheaper test than LCF should be developed for the evaluation of lot samples of P/M production parts.

19. The role of carbon, boron, and zirconium in P/M alloys should be investigated.

20. Rapid solidification techniques should be studied further to quantify the benefits of the cooling-rate variable in existing alloy compositions and for new compositions.

21. The development of alloy powder compositions that retain a high degree of metastability after fabrication (into engine components) should be pursued actively.

Chapter 2

INTRODUCTION

The development of superalloy powder metallurgy largely grew from the demand for gas-turbine hot-section components with increased high-temperature strength and fatigue resistance. Within the past decade, superalloy parts production by the P/M process has moved from the research and development stage to substantial commercial production for almost all of the newer military jet engines. State-of-the-art problems exist and most are covered in this report, but they are not great enough to overshadow the significant current and future potential advances offered by the P/M process. Technological advances such as hot-isostatic pressing (originally developed as a process for bonding nuclear-fuel elements) played a major role in the new P/M process for making high-performance materials. This report summarizes the current status of the P/M superalloy field with particular reference to gas-turbine-disk technology.

The key factors of P/M superalloy commercial production and use are treated in some detail in separate sections of this report. They are: powder atomization, handling, containerization, consolidation, post-consolidation treatment, properties and quality control, alloying, and economics.

A common commercial process for making P/M superalloy parts involves: vacuum induction melting of the superalloy composition, inert-gas atomization, powder characterization and handling, container production and loading, vacuum outgassing, hot-isostatic pressing, and decanning and final processing. The basic processing steps are illustrated in Figure 1.

In addition to the predominant inert-gas atomization process, soluble-gas (vacuum) atomization and the rotating-electrode process (REP) also are used in commercial production. Besides the hot-isostatic-pressing method, a forge-and-extrude process of powder-filled metal containers is used in conjunction with a subsequent superplastic forming step to make shaped parts.

The superalloy powder presently being used in production is spherical in shape and varies in size distribution depending on the atomization process used and the operating conditions of atomization. A typical gas-atomized powder is shown in Figure 2. Microstructurally, the powder particles exhibit a fine dendritic pattern (Figure 3). Because of the basic characteristics of powder, or microingots, versus slow-cooled conventional ingots, segregation is minimized and it may be possible to make alloys that heretofore were unattainable.

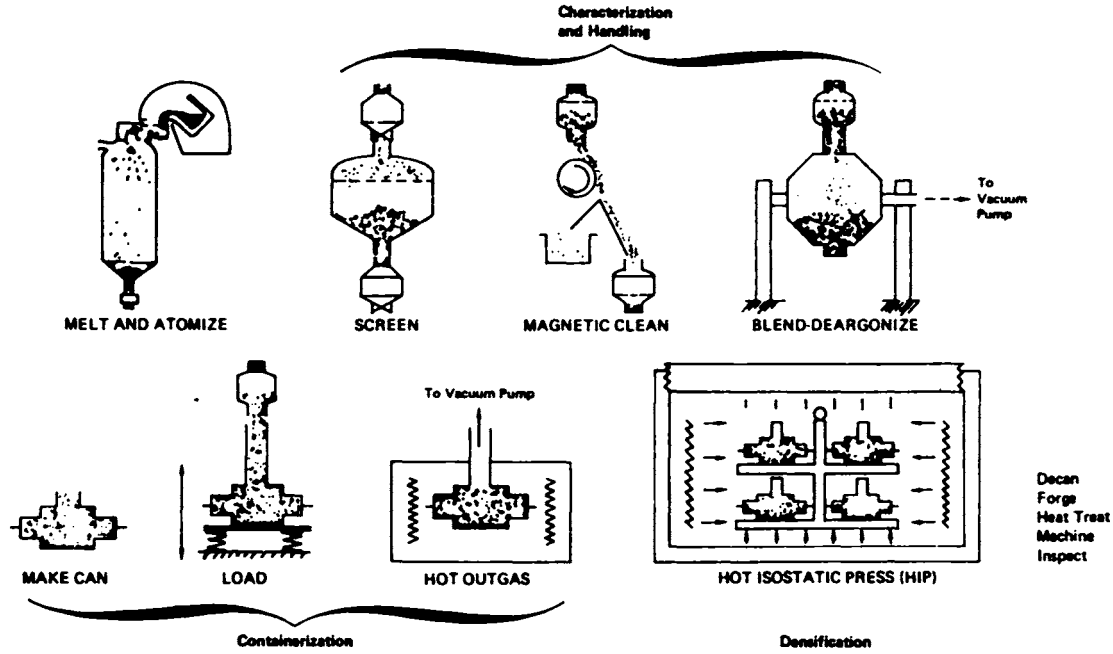
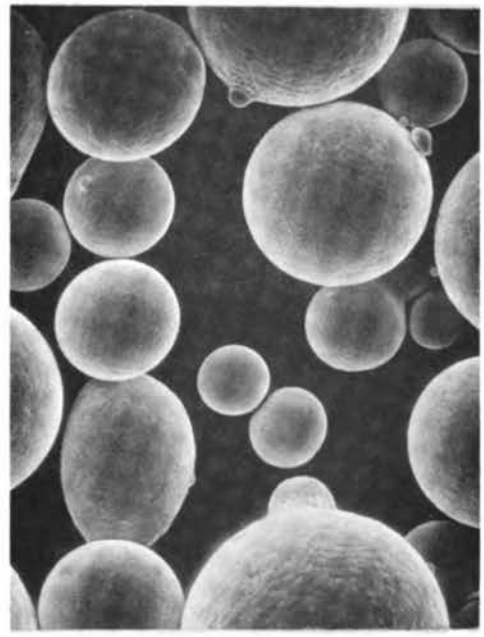


FIGURE 1 Example of commercial P/M superalloy processing operation from powder to fully dense material.



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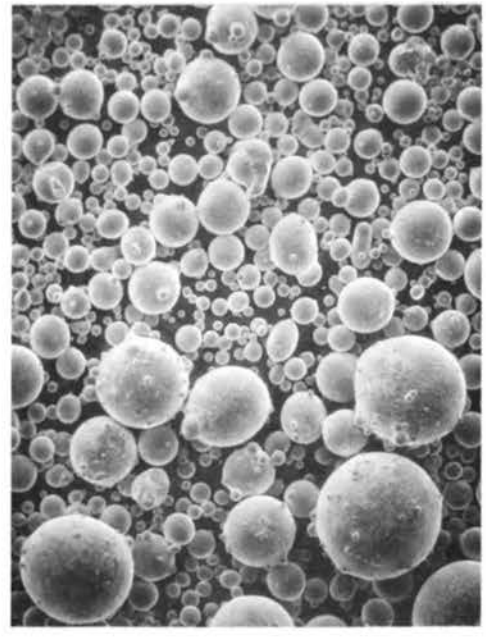

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FIGURE 2 Argon-atomized Rene 95 powder.

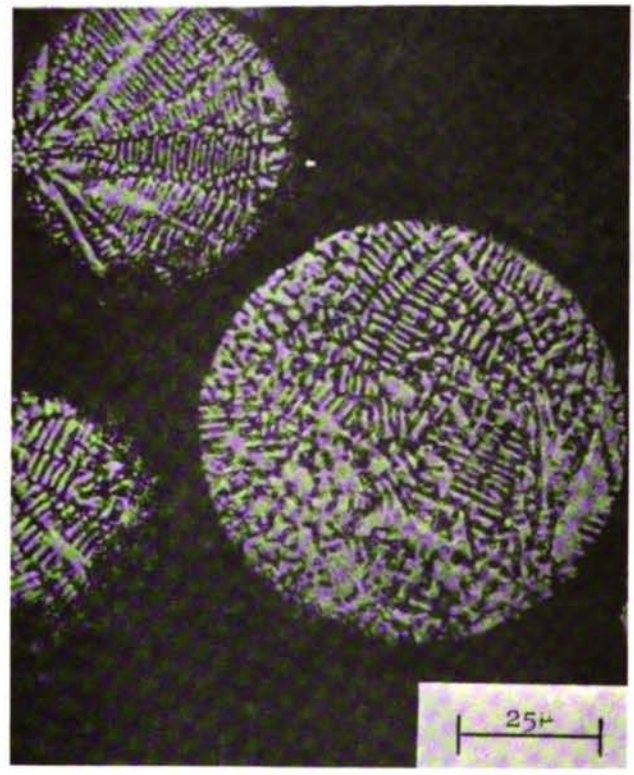


FIGURE 3 Internal microstructure of argon-atomized Rene 95 powder. Source: Crucible, Inc.

Engineering alloys solidify over a range of temperatures and liquid compositions, and, hence, the elements that are combined to make up an alloy segregate during solidification. Segregation in conventional ingot technology occurs on two different scales--long-range segregation (macrosegregation) and short-range segregation (microsegregation). The most important advantages of P/M technologies over the conventional ingot approach include the following: First, long-range segregation in the final product is avoided because of the small size of the cast powders. Second, the large surface-area-to-volume ratio of atomized droplets permits high rates of heat extraction during solidification. Consequently, significant reductions in the scale of the second type of segregation (microsegregation) and more homogeneous microstructures are achieved with attendant beneficial effects on formability and properties. In addition, larger quantities of strengthening alloy elements can be added due to the increased metastability of the very rapidly cooled powder particles. Thus, a new unique approach for designing new high-performance superalloys is now available that was not possible by conventional ingot practice. Finally, the P/M route lends itself to the production of net or near-net shapes of high-strength, highly alloyed materials.

Chapter 3

POWDER ATOMIZATION OF SUPERALLOYS

INTRODUCTION

As a first step in assessing the status and potential of superalloy powder alloys, it is necessary to have an overview of powder production techniques. Although there are several approaches of commercial importance (e.g., chemical, electrolytic, comminution, atomization), liquid-metal atomization has emerged as the primary technique for the production of powder from reactive metals and alloys (Lawley 1978, Lawley 1977, Clark 1975). Atomization enhances powder quality and reproducibility, particularly with respect to chemistry, cleanliness, size, and shape.

Commercial production methods of atomization are considered first, followed by a description of other techniques. The latter range from prototype pilot-scale processes to those still in the early stages of development. Included are summaries of powder properties and characteristics and the advantages or limitations of each atomization method.

Apart from powder production per se, it is important to consider the effects of process variables in atomization on heat-flow conditions and the resulting microstructures of atomized powders. Unfortunately, the complex heat-flow and solidification conditions in the atomized droplets of complex alloys are not well understood. The literature contains much confusing and sometimes erroneous data on cooling rates. These have been incorrectly deduced from: pre-established relationships between segregate (dendrite arm) spacings and cooling rates, oversimplified heat-flow and solidification models, and assumption of unreasonably high values for heat-transfer coefficient at the powder-environment interface. This problem is addressed in appendix B in a summary of existing information concerning heat-flow and limitations on the rate of heat extraction from atomized droplets.

PRODUCTION METHODS OF ATOMIZATION

At present, the bulk of commercial capacity for superalloy powder production resides in inert-gas and vacuum (soluble gas) atomization. The only other production method is the rotating-electrode process.

Gas Atomization

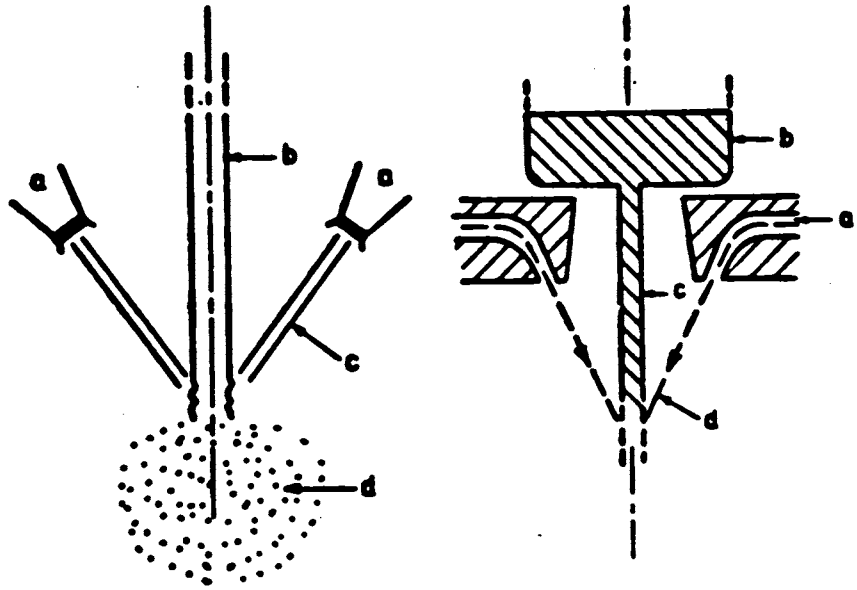
The basic gas-atomization process (Lawley 1977 and 1978, Clark 1975, Benjamin and Larson 1977, Wallis 1976) involves initial melting of the charge in vacuum (e.g., vacuum induction melting). When the appropriate temperature of the molten bath is reached, the liquid metal is tapped into a tundish that contains a nozzle at the base; the liquid alloy flows through the nozzle into the atomizing chamber below as a continuous stream.

The principle of gas atomization is simple--the continuous stream of liquid metal is broken down into droplets by means of a subsonic or supersonic gas stream or jet. Atomization is due to kinetic energy transfer from the atomizing medium to the metal. In practice, nitrogen, argon, or air can be used; mixtures of these gases and helium also are effective. The number and geometry of gas-metal configurations are unlimited (Klar and Shafer 1972); typical arrangements involve multiple jets or an annular ring (Figure 4).

There are several interrelated processing and material variables involved in gas atomization: gas jet distance and pressure, gas velocity and mass flow rate, metal velocity and mass flow rate, nozzle geometry, angle of impingement, superheat, metal surface tension, and metal melting range. As a consequence, numerous empirical relationships have been documented or proposed for the prediction of particle size and shape and the distributions of size and shape. It is always found that as gas pressure and gas-mass flow rate increase and/or as the jet-to-metal stream distance decreases, the average particle diameter decreases. Typical operating ranges of some of the above variables in gas atomization are:

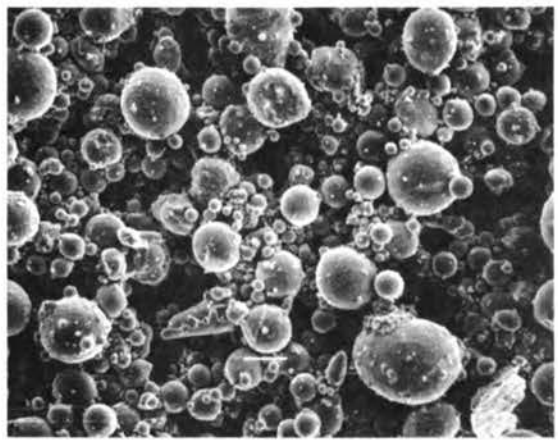
Gas pressure	$14 \times 10^5 - 42 \times 10^5$ Pa (200-600 psi)
Gas velocity	50-150 m/s (164-490 ft/sec)
Superheat	210-390°F (100-200°C)
Angle of impingement	15-90°

Gas-atomized powders typically are spherical and have smooth surfaces. A typical gas-atomized powder is illustrated in Figure 5(a). Cooling rates depend on particle size and the thermal properties of the atomizing gas. The interplay of these parameters is discussed in appendix B. A particle-size distribution curve for gas-atomized superalloys is shown in Figure 6. These powders typically are screened to -80 mesh (<177 μm) prior to use. Thus, the overall yield of usable powder varies with process. The vacuum-atomization process appears to give significantly more useful powder than either the argon- or rotating-electrode atomization process. Inherently, vacuum-atomization and the REP processes are limited in ability to vary powder-size distribution. In contrast, gas atomization provides reasonable flexibility in selecting process parameters for controlling and changing powder-size distribution.



(a) (b)

FIGURE 4 Representative gas-atomization configurations: (a) two-jet configuration with: a = jets, b = liquid-metal stream, c = gas stream, and d = atomized powder; (b) annular-ring configuration with: a = ring orifice, b = liquid-metal reservoir, c = liquid-metal stream, d = gas stream (Lawley 1978).



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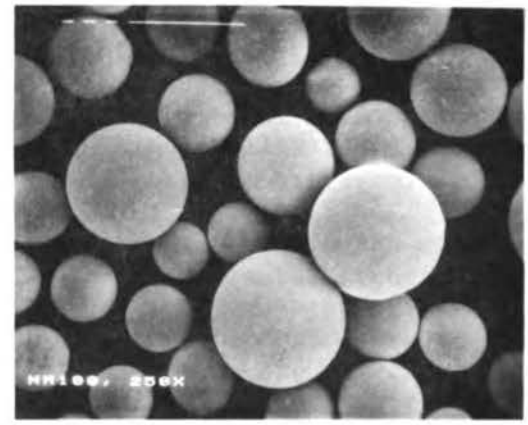

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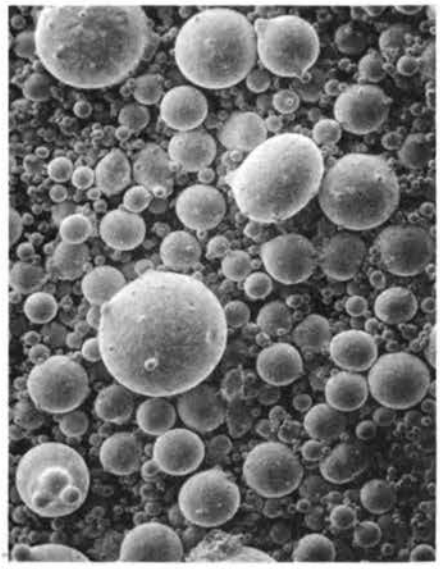

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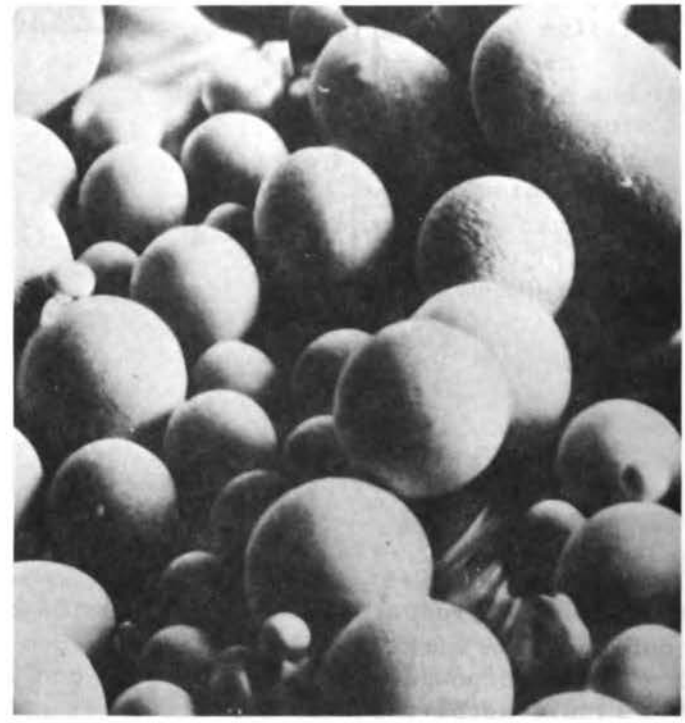


(d) 500X

FIGURE 5 Representative superalloy powders: (a) soluble-gas atomized, (b) rotating electrode, (c) gas atomized, and (d) RSR.

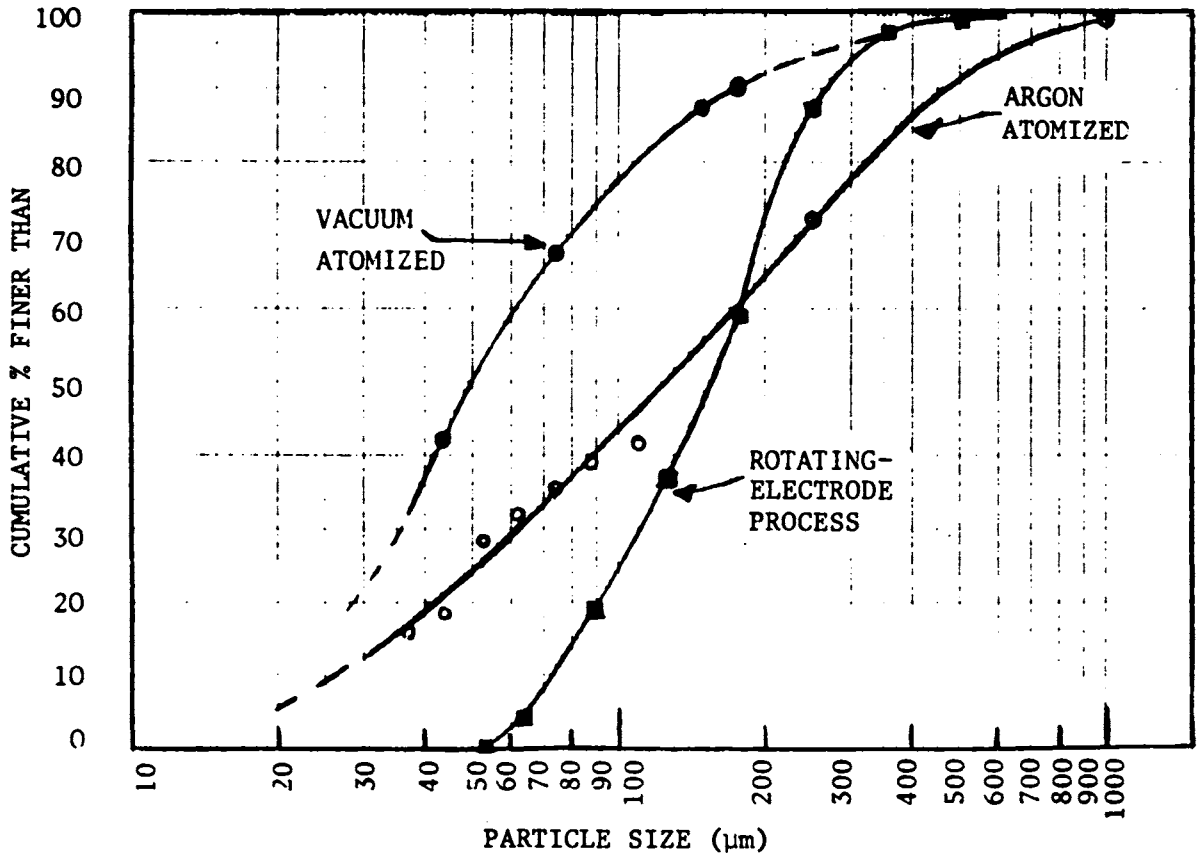


FIGURE 6 Representative particle-size distribution of commercially atomized superalloy powders (size distribution data provided by powder manufacturers).

Figure 7 shows the distributions of the three types of powders remaining after such a screening operation and is more typical of the powders actually used to produce hardware. Argon is most frequently used as the atomizing gas in superalloy powder production; however, nitrogen is now being evaluated as an atomizing medium.

Gas atomization occurs via a three-stage process--primary break-up of the liquid stream, secondary disintegration, and particle solidification (Figure 8). Details of the break-up of the metal stream are now reasonably well understood (See and Johnston 1978). Understanding of these details suggests that some improvements in powder yield, smaller particle diameters, narrower size distributions, and refined structures can be achieved in gas atomization.

A major concern in gas atomization is the appearance of nonmetallic inclusions, particularly oxides, in the superalloy powder. These originate in the melting and/or pouring operation (e.g., pick-up from the refractory crucible walls or nozzle erosion). The detrimental effect of inclusions on property levels is discussed in other sections of the report. Possible solutions to the problem are the use of a water-cooled hearth or melting without direct contact with refractory materials.

Soluble-Gas (Vacuum) Atomization

Vacuum or soluble-gas atomzation (Benjamin and Larson 1977, Wentzell 1974) is a commercial batch process based on the principle that when a molten metal supersaturated with gas under pressure is suddenly exposed to vacuum, the gas expands, comes out of solution, and causes the liquid metal to be atomized. The set-up for soluble-gas atomization is shown in Figure 9. Alloy powders based on Ni, Cu, Co, Fe, and Al have been vacuum atomized with hydrogen as the gas. Powders generally are spherical (Figure 5b) with an average particle diameter and size spread smaller than for gas-atomized or rotating electrode powders. A size-distribution curve for soluble-gas-atomized powder is given in Figure 6.

For superalloys, vacuum induction melting is essential. Vacuum- and gas-atomized powders have similar impurity levels. Alumina, hafnia, and zirconia inclusions are present in small amounts in all commercial powders, and these limit low-cycle-fatigue life.

Rotating-Electrode Process

The rotating-electrode process (Clark 1975, Benjamin and Larson 1977, Loewenstein and Roberts 1979) is in commercial operation; however, production rates are low. Primary application is for mild steel and titanium alloy powders. Some superalloy powders are produced by this process for special applications. Until recently (Loewenstein and Roberts, 1979) normal practice involved the use of a water-cooled stationary tungsten cathode. The bar of the metal or alloy to be atomized was rotated about its

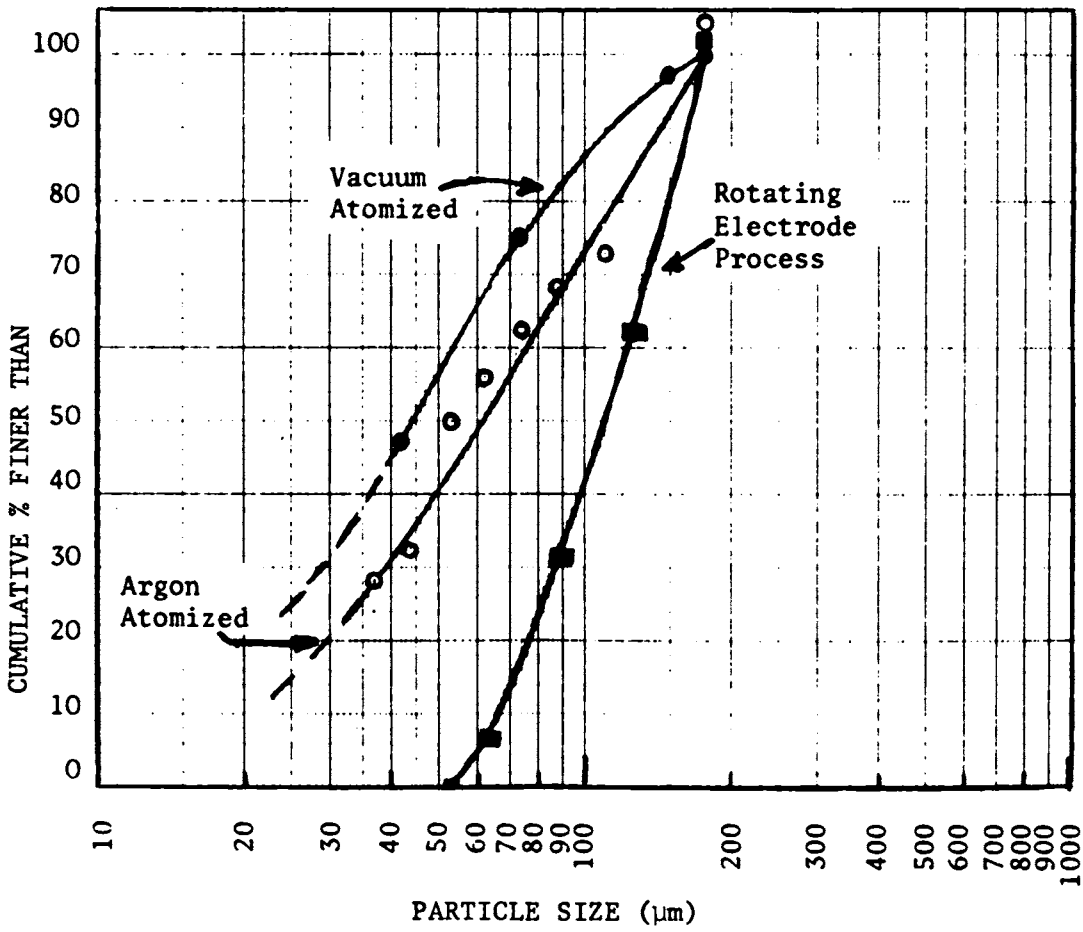


FIGURE 7 Particle-size distributions of -80 mesh portions of commercially atomized superalloy powders.

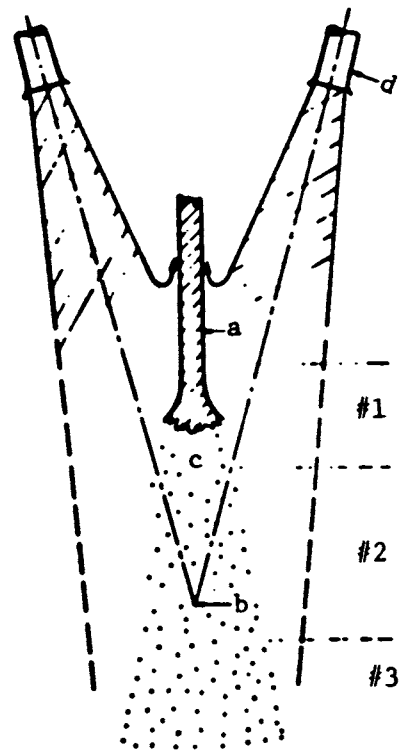


FIGURE 8 Three stages of liquid-metal-stream disintegration in gas atomization: a = liquid stream, b = focal point of gas jets, c = powder particles, d = gas jet, #1 = primary liquid-stream break-up, #2 = secondary disintegration, #3 = particle solidification (See and Johnston 1978).

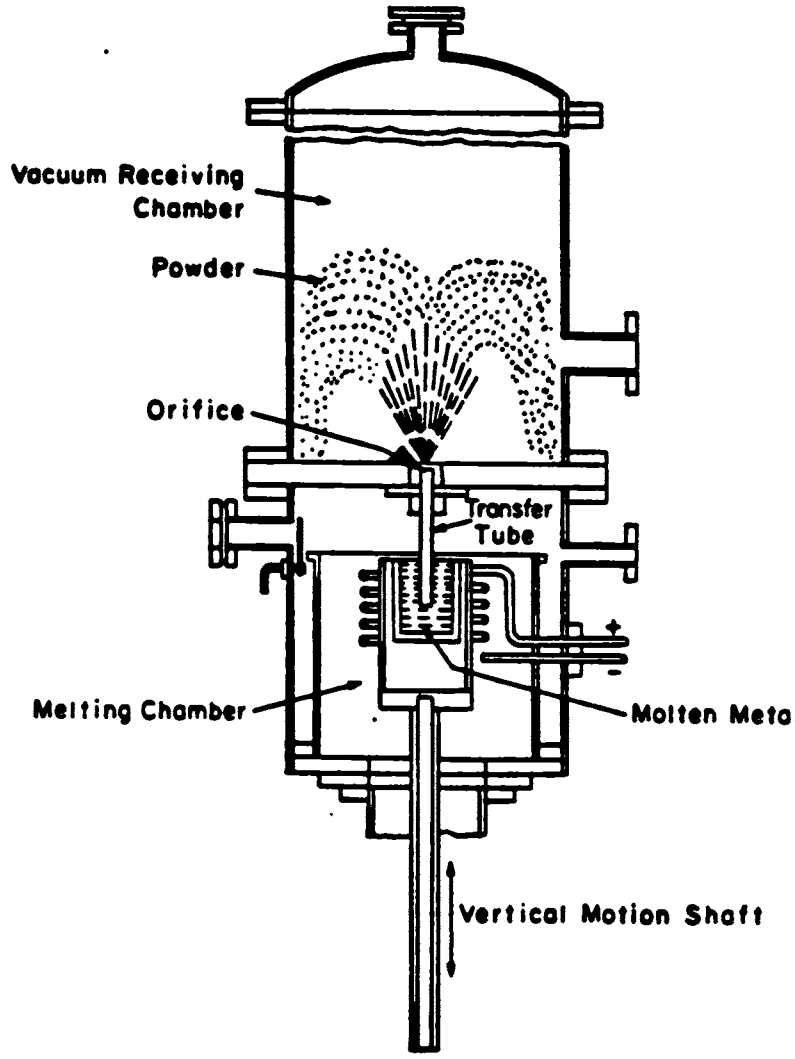


FIGURE 9 Soluble-gas atomization.

axis at 15,000. The end of the bar was then melted by electric arc, plasma arc, or electron beam and the molten metal centrifugally ejected in the form of molten metal droplets (Figure 10) that solidified before hitting the walls of the inert-gas-filled chamber.

With the conventional electrode configuration shown in Figure 11a, tungsten contamination of the powder occurs with deleterious effects on the fatigue life of the consolidated material. Attempts to eliminate contamination by the use of a double titanium electrode system resulted in other serious problems. In the most recent form of rotating-electrode atomization, melting is accomplished by means of a transferred arc, plasma (helium) torch (Figure 11b). Preliminary indications are that the tungsten tip in the plasma gun loses exceedingly small amounts of tungsten and that the tungsten level in the powder is comparable to that in the starting electrode stock (Loewenstein and Roberts, 1979).

Rotating-electrode powders usually are spherical and of high surface quality, but they have a large average particle diameter of 150 μm. A typical size distribution is given in Figure 6. For comparable size particles, cooling rates in REP are less than those occurring in gas atomization. Superheating is limited in the rotating-electrode process and this results in carbides coarser than those in gas-atomized powders.

PILOT-SCALE METHODS OF ATOMIZATION

Centrifugal Atomization

In centrifugal atomization (Sutcliffe and Morton 1976, Stephan 1976, and DeCours et al. 1976), molten metal is ejected in the form of droplets from a rapidly spinning crucible plate or disc. Particles either cool in the environment of the collection chamber or can be gas-quenched as they leave the rotating vehicle. Erosion, dissolution, oxidation, and creep of the spinner pose severe materials problems; the advent of improved ceramics has been a key factor in the development of this process. Energy requirements are relatively low.

Three centrifugal-atomization configurations are shown schematically in Figure 12. In each, particle shape can be varied from spherical to flake by altering speed of rotation, geometry of disk, and superheat (Sutcliffe and Morton 1976, Stephan 1976, DeCours et al. 1976). Spherical powders produced by these methods of centrifugal atomization are relatively coarse with average particle diameters of 200 μm.

The rapid-solidification-rate process (designated by Pratt and Whitney Aircraft as RSR) for making superalloy powders is a form of centrifugal atomization (Cox et al. 1976 and 1978). The process employs a high-speed (400-600 rev/sec) water-cooled rotating disk that breaks up the molten metal stream. The droplets are hit by high-pressure helium gas as they leave the periphery of the rotating disk. High cooling rates

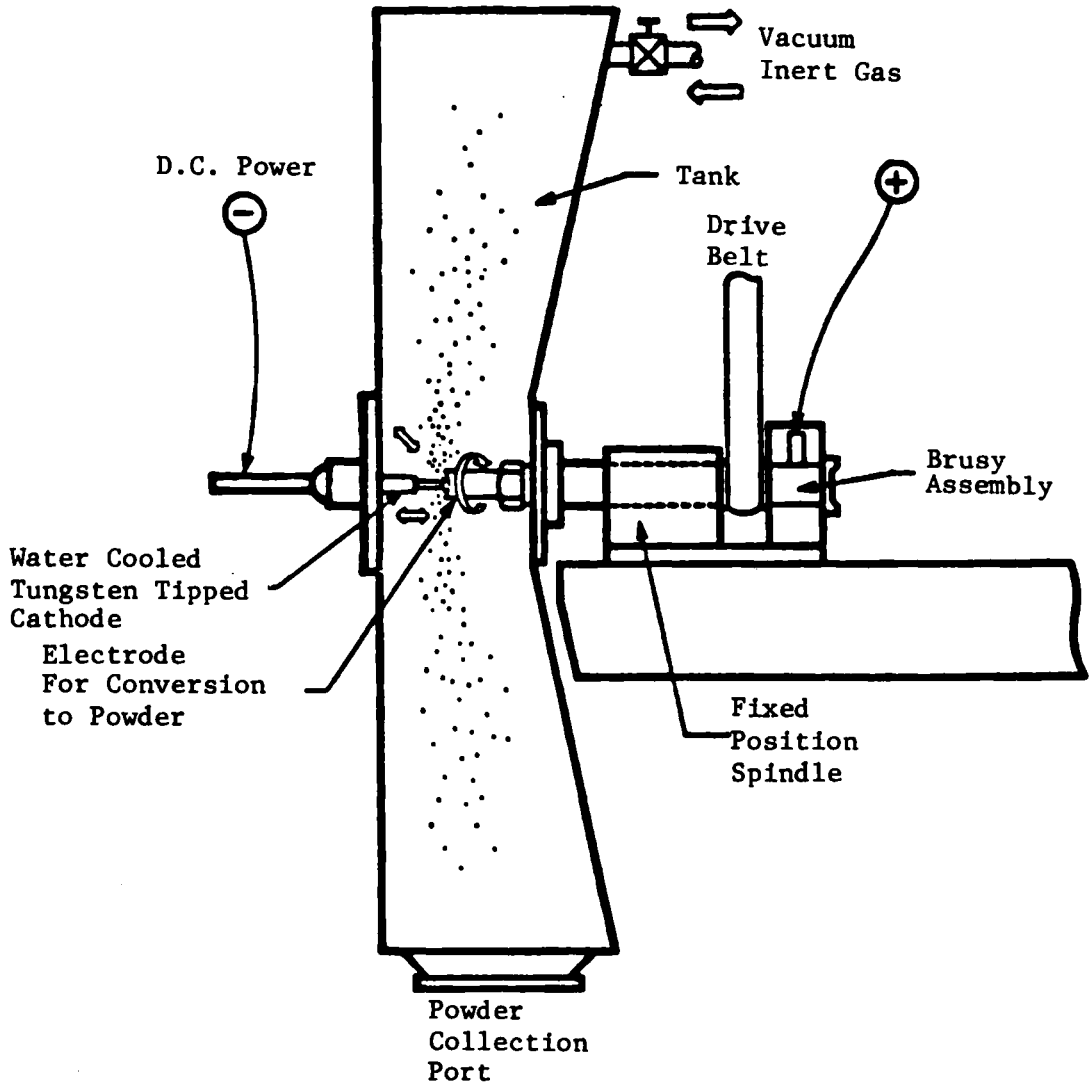
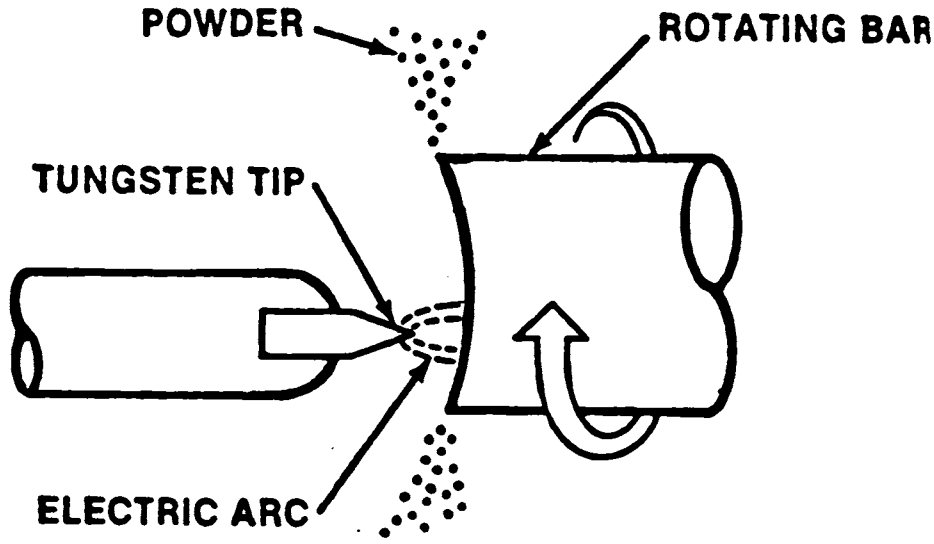
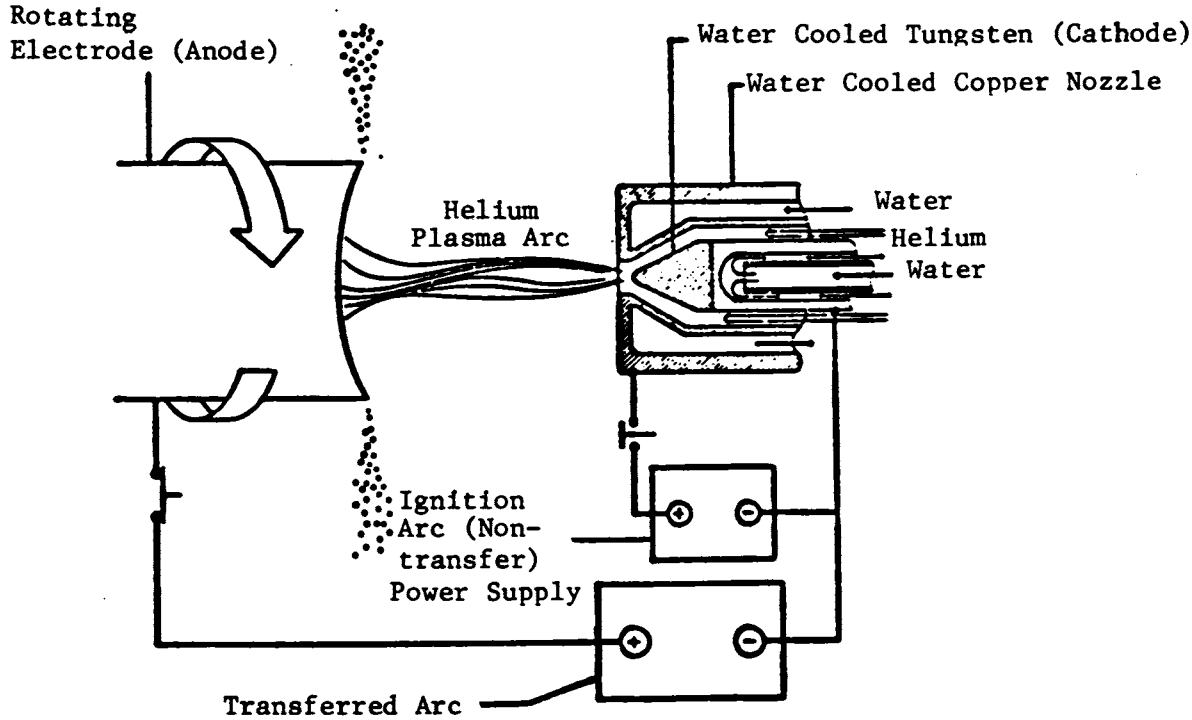


FIGURE 10 Schematic of the rotating-electrode process (Loewenstein and Roberts 1979).



(a)



(b)

FIGURE 11 Rotating-electrode process: (a) conventional method, and (b) plasma electrode (Loewenstein and Roberts 1979).

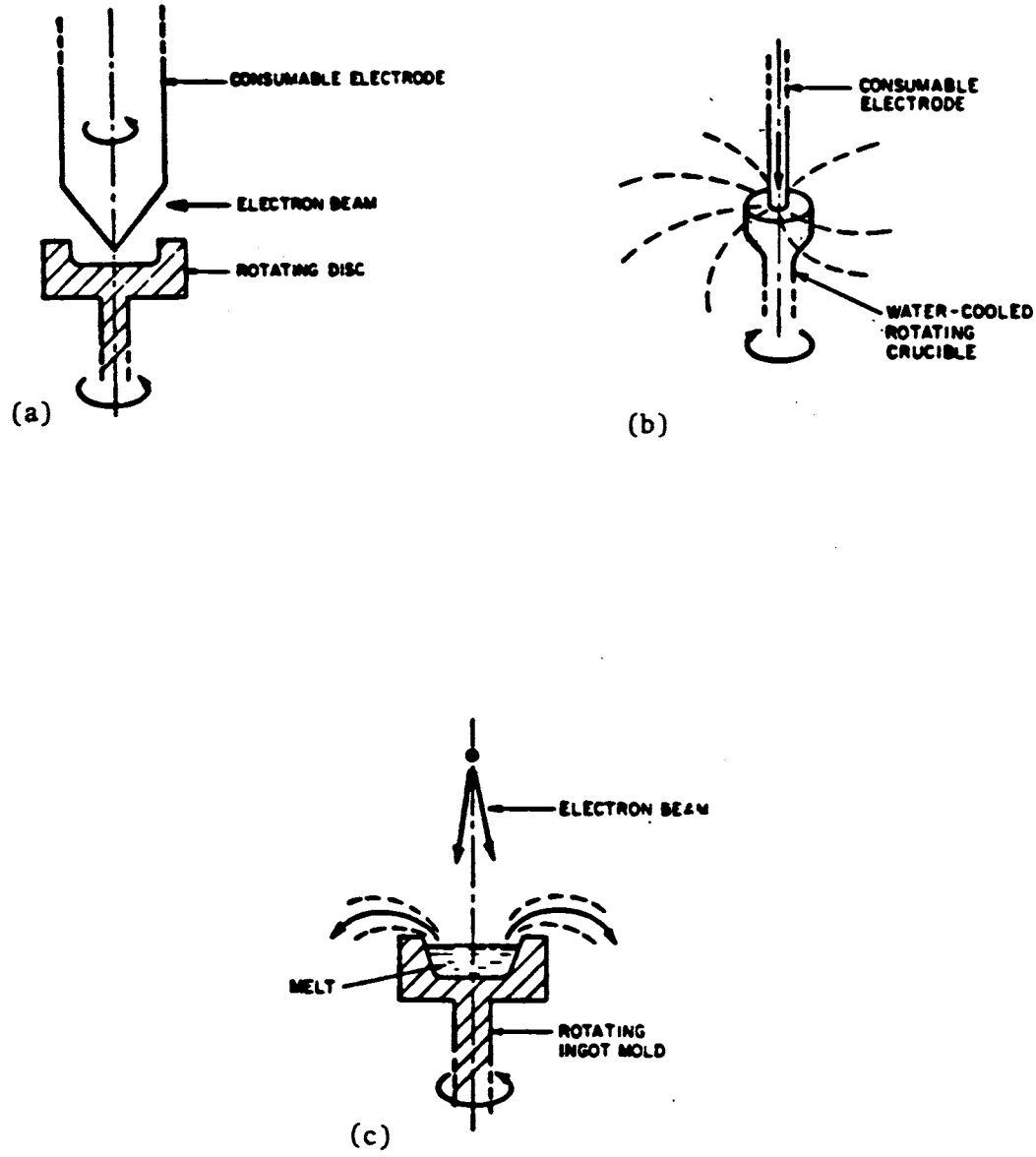


FIGURE 12 Schematic representation of centrifugal-atomization configurations: (a) rotating disk (Stephan 1976), (b) rotating crucible (Sutcliffe and Morton 1976), and (c) rotating ingot mold (DeCours et al. 1976).

($\geq 10^{5}$°Csec^{-1}) can be achieved and this is reflected in a high degree of compositional homogeneity and fine-scale microstructure in each powder particle; dendrite-arm spacings have been reported in the range 0.1 to 1 μm. A schematic of the Pratt and Whitney facility is given in Figure 13.

RSR powders are spherical (Figure 5d). A comparison of the size distribution of RSR powders with those of commercially atomized superalloy powders is shown in Figure 14. The RSR process produces powders with very narrow size distribution similar to but finer than that of REP powders. The yield of -80 mesh powder (177 μm) is also relatively high. The average size of the RSR powder (∿100 μm) is smaller than argon-atomized powder. However, it is important to appreciate that RS and inert-gas atomization techniques permit flexibility in processing parameters and it is entirely possible that the curve can be shifted either way. In addition, data for the 3 curves was obtained from different sources and perhaps are not strictly comparable. Since melting takes place in a ceramic crucible and the liquid superalloy is spun off a ceramic-coated disk, the potential for inclusion contamination exists.

LABORATORY-SCALE METHODS OF ATOMIZATION

Ultrasonic Atomization

Ultrasonic atomization (Grant 1972) appears attractive for the production of small-diameter (<50 μm) particles with a narrow size distribution at high quench rates (∿10^{5}°C/s). Hartman shock wave tubes are used to deliver high-velocity pulses of gas (up to Mach 2) at frequencies in the range of 60,000 to 80,000 cps. There is no contact between the ultrasonic "die" and the liquid-metal stream, and the configuration is similar to that in gas atomization. Yields in the 80 to 90 percent range have been achieved for -325 mesh powder of stainless steel. Ultrasonic atomization has been applied to low-melting-point alloys and, more recently, interest has centered on superalloys. No attempt yet has been made to scale-up the process.

In contrast to gas atomization, ultrasonic atomization appears to be a single-step process. Because of the high velocity of the gas pulse, the liquid-metal stream responds on impact as though it were a solid with low shear resistance. This is believed to be responsible for the small powder sizes achieved. Gas retention is expected to be lower in powders produced by ultrasonic atomization than in those produced by gas- or vacuum-atomization methods.

Roller Atomization

In roller atomization (Singer and Roche 1977), a stream of molten metal is fed between rapidly rotating rolls (up to 200 rev/sec). The roll gap is ∿50 μm. Heat transfer to the rolls is minimized by a suitable insulating coating so that the atomization event actually takes place by break-up of the molten sheet of metal below the roll gap. A median

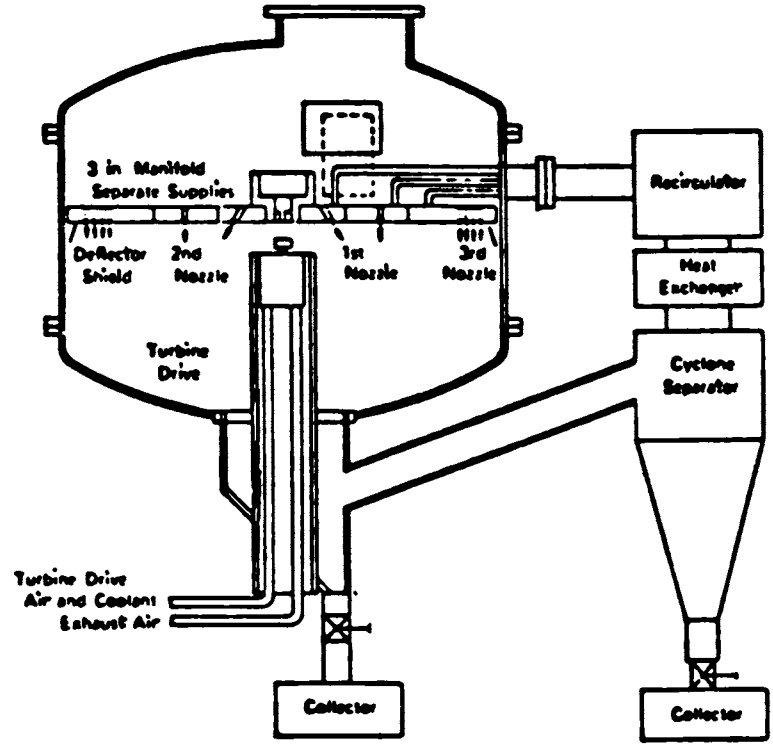


FIGURE 13 RSR facility for centrifugal atomization (Cox et al. 1976).

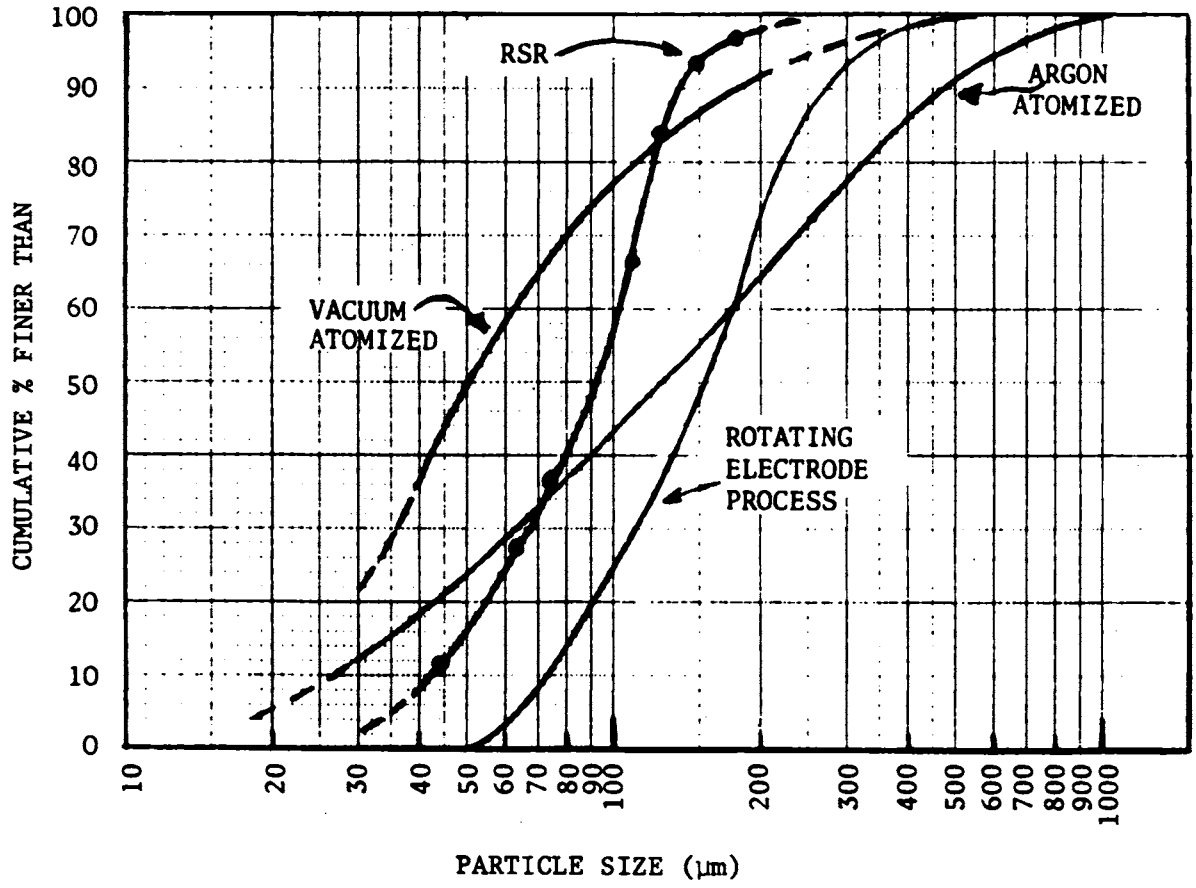


FIGURE 14 Distribution comparison of RSR powder and commercially atomized superalloy powders.

particle size of 220 μm has been obtained for tin powders with a yield ~70 percent. Median particle size is inversely proportional to roll speed. A wide spectrum of forms can be atomized: flake, acicular, irregular, spherical. Cooling rates have not been determined. To date, roller atomization has not been applied to superalloys.

POWDER SIZE AND STRUCTURE

Measured segregate size, in terms of dendrite-arm spacings, for atomized droplets of three alloys (IN-100, Mar-M-509, and VM-300 maraging steel) produced by different atomization processes, are summarized in Table 1. Note that dendrite-arm spacings correspond to the specific powder sizes listed. It must be emphasized that the dendrite-arm spacing is a sensitive function of alloy composition and purity.

It has been established that the commercial prototype and laboratory techniques are capable of producing unique microstructures, high levels of supersaturation, and a reduced tendency for incipient melting. The degree to which such features are carried through to the final fully dense form is influenced by subsequent consolidation and thermal treatment, which are discussed later in this report.

TABLE 1 Measured Dendrite-Arm Spacings (DAS) in Atomized Powders

Alloy	Process	Avg. Powder Size (μm)	Dendrite-Arm Spacing (μm)
Cobalt base alloy MAR-M-509	Vacuum atomized	110	2.5 to 3.0
	Steam atomized (coarse powder)	2000	1.5 to 5.0
Iron base alloy VM-300 maraging steel	Vacuum atomized	300	6.0 to 7.0[a]
	REP atomized	180	2.0 to 3.0
	Steam atomized (coarse powder)	2000	4.5 to 11.0
	Argon atomized (coarse powder)	2500	5.0 to 12.0
Nickel base alloy IN-100	Vacuum atomized	300	5.0 to 7.5[a]
	REP atomized	170	∿3.0
	Argon atomized (fine powder)	75	∿2.0
	Centrifugal atomization (helium)	85	∿0.7

Source: Joly and Mehrabian 1974.
[a]Dendrite-arm-spacing values cover the powder-size range of 100 to 830 μm.

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Chapter 4

POWDER HANDLING AND CONTAINERIZATION

POWDER HANDLING

Extreme care in powder handling at every step is required to avoid or to minimize the two types of defects that can occur in P/M superalloys (i.e., porosity and/or foreign particles). Contamination by a relatively small amount of material does have a significant effect on the properties of the final superalloy part. Foreign particles in the final product to be made into engine parts are primarily fine-sized nonmetallics that may originate during melting, the atomization process, or the powder-handling stage. Minimizing and limiting the maximum size of the nonmetallic inclusions is important in achieving the required performance characteristics of rotating engine parts.

A very small amount of porosity forms in the final solid product and this can lead to thermally induced porosity (TIP) after heating to a high temperature such as is sometimes used for solution heat treatment. TIP is the result of inert gas entrapped inside hollow particles, absorbed on the particle surfaces, or pumped into the partially consolidated material during compaction. Special care in making and processing the powder is required to minimize TIP.

The powder produced by the inert-gas (primarily argon) process of atomization initially requires an atomization vessel to be designed so that locations where powder or debris can accumulate are avoided and to permit rapid and thorough cleaning so that no foreign matter is added to the powder generated by the atomization process. The powder is maintained under an argon atmosphere in the atomizing vessel and is collected at the bottom of the vessel, usually in a clean-room-air atmosphere. One method is to discharge the powder into a previously evacuated or inert-gas-filled transfer container through a valve coupling system between the base of the atomizer and the container. Other methods involve discharging the powder into a transfer container in a clean-room-air atmosphere. The use of a vacuum or inert-gas atmosphere insures against possible contamination from the surrounding atmosphere. However, if stringent handling steps in clean-room atmospheres are followed, powder quality is maintained in air handling. An appropriate sample of the powder is obtained for chemical analysis to determine if the composition meets specification requirements. The inert-gas, vacuum, and air-atmosphere transfer containers are sealed after being loaded with powder.

Powder processing involves screening to below a maximum size fraction to be used in the final product. Screening today is used primarily

to eliminate nonmetallic inclusions that have a significant effect on pertinent mechanical properties. The powder usually is screened to sizes in the range of -60 to -150 mesh (-250 μm to -100 μm). A strong trend in production is toward screening to a very fine maximum size of -100 mesh (-150 μm) and finer prior to hot-isostatic compaction. Screening out large contaminants by this process is the only current means of eliminating them in production. However, several difficulties are encountered in handling very fine-sized powder, e.g., -150 mesh (-100 μm) and finer. During screening, considerable screen blinding occurs, powder flow rate is decreased, and more vibration is required during container loading.

CLEANLINESS AND DEFECTS

The powder then may be subjected to a further cleaning method such as magnetic treatment to eliminate small quantities of magnetic material. The screening step then is followed by hot-vacuum outgassing (static or dynamic) and blending, both of which can be performed in one step. At this point, a sample of the powder is subjected to various quality-control tests including powder cleanliness, flowability, and tap density. The powder is then directly charged into the container for hot-isostatic pressing (HIP) or returned to the transfer vessel for subsequent loading into a HIP container.

As part of a NASA/General Electric project on powder metallurgy Rene 95 rotating parts (Pfouts et al. 1979), Crucible Research Center prepared Rene 95 powder compacts with material defects that were intentionally added to the powder. The defects consisted of four size ranges of oxides (SiO_2 and Al_2O_3) with about 70 oxide particles per pound of powder, foreign alloy contaminants (low-carbon Astroloy and M2 tool steel), and argon entrapment. For this laboratory study, the effects of oxide inclusions (Figure 15) could be determined more readily by exaggerating the amounts and sizes of the oxides over that normally found in powder production. The finest contaminant added in this study (0.15-0.25 mm) is much larger than the largest inclusions that could occur in commercial practice where powder is screened to -150 mesh (0.1 mm). The defect-containing compacts were made with normal-size-distribution powder and with normal HIP cycle and heat treatment. A summary of the effects of defects on as-HIP mechanical properties is as follows:

1. Oxide inclusions
   - o Degrade all mechanical properties
   - o Degree dependent on defect size and/or location

2. Foreign alloy contaminants
   - o M2 tool steel--no significant effect on tensile properties, reduces rupture life/ductility, acts as fatigue initiation site
   - o LC Astroloy--no significant effect on properties

3. Argon entrapment
   - o Properties decrease with increasing argon content
   - o 0.3 percent TIP is an acceptable limit
   - o Pores act as fatigue initiation sites

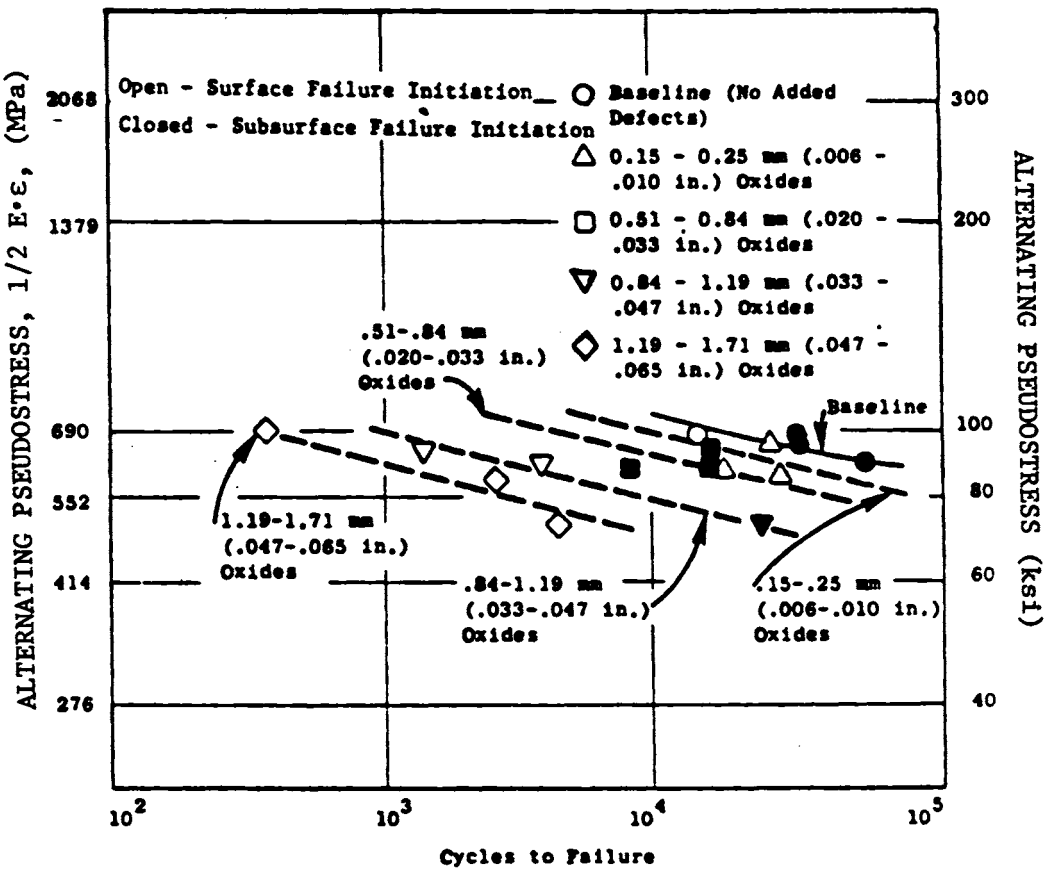

FIGURE 15 Effect of oxide inclusions of argon-atomized Rene 95 on 1000°F (538°C) strain control (Pfouts et al. 1979). Note that R = 0(A=1,K_t=1)LCF.

The effect of different-sized oxide particles on the important property, low-cycle fatigue, is shown in Figure 15. LCF decreases as the size of oxides increases. Thus, minimizing or eliminating larger oxides is important for good LCF properties. At present, eliminating larger oxides is accomplished in commercial production by screening out the larger oxide particles and powder. (Appendix C discusses, in more detail, the dependence of fatigue life on defect size.)

Because of the large surface-area-to-volume ratio, compared to bulk material, and the extremely small quantities of harmful contaminants that could be involved in superalloy powder production, any handling step is a potential source of new contamination. Thus, the ideal solution to powder contamination is to eliminate the source rather than to treat the powder. To provide improved control and to upgrade currently produced P/M superalloy parts, both the elimination of contamination of the powder at the various sources and powder treatment are being pursued.

Methods for removing contaminants from superalloy powder are being investigated extensively so that powder quality can be upgraded. Methods of removing nonmetallic inclusions by electrostatic processes (Miles and Rhodes 1978) are in the development stage. For outgassing, other than the currently used production methods of static and/or dynamic vacuum outgassing, an electrodynamic degassing process (Miles and Rhodes 1978), developed by Kelsey-Hayes, shows promise.

To upgrade superalloy powder cleanliness so that the fatigue life of critical engine components can be increased, two major programs are being jointly sponsored by the Army Aviation Research and Development Command and the Air Force Materials Laboratory.* One program is being conducted by the General Electric Aircraft Engine Group with emphasis on investigating all the processing steps and equipment currently being used to produce P/M Rene 95 parts. General Electric is investigating all the steps starting with powder atomization to determine where contaminants may be picked up. The other program is being carried out by Pratt and Whitney with the objective of improving the quality of the remelt stock for powder atomization. The approach is to investigate the use of electron-beam melting using a copper hearth as opposed to induction melting in a ceramic crucible.

CONTAINERIZATION

The main types of containers for P/M superalloy production are metal. Ceramic containers are being evaluated extensively because complex shapes can be made economically by this method.

Metal

Metal containers are made of stainless steel, low-carbon steel, and electroformed nickel or nickel-base alloys. The shape of metal containers

*General Electric program, Contract No. F33615-78-C-5225, and Pratt and Whitney program, Contract No. F33615-79-C-5006.

ranges from a simple cylinder to a relatively complex net shape made by electrodeposition. At present, commercial production of P/M superalloy parts involves the use of metallic shaped containers, cylindrical cans, or pipes that have welded ends. The powder-filled container or can is then either hot-isostatically pressed (HIPed) or forged and extruded. Currently, emphasis in production is on the as-HIPed superalloy powder near-net shapes (Bartos 1978, American Metal Market 1980, and Metals Progress 1979). The containers for these shapes are fabricated from metal sheet by spinning, hydroforming, or pressing and then welding sections together. The near-net-shape dimensions of the container allow for the contraction that occurs during hot-isostatic pressing since the tap density of the powder as loaded is in the order of 65 percent. Gas-tight welding is important to avoid leaks during subsequent processing.

After fabrication of the container, it is "purged" with powder similar to that which is to be used for the compact. The container then is filled with powder and vibrated so that the required tap density is obtained. The powder-filled container may or may not be further vacuum outgassed to insure removal of possible gas contaminants. The containers then are sealed and are ready for hot-isostatic pressing. Striations that correspond to the macroscopic interface between coarse and fine powders caused by powder segregation and that prevent grain growth have been observed in forgings produced from P/M preforms and are believed to be the result of excessive or improper vibration during container loading. Forge cracking was attributed to the presence of these striations. Striations have not been observed in as-HIPed parts.

Other types of metallic-container processes that are currently in the research and development stage include the following:

1. Electroforming metal molds using processes developed and patented by United Technologies Corporation and Messerschmitt-Bolkow-Blohm (United Technologies Corporation 1976, 1977, and 1978, and Messerschmitt-Bolkow-Blohm 1977). These processes make use of wax, plastic, or low-melting-point metals for the starting shape design (somewhat similar to the lost-wax investment-casting process). However, rather than a ceramic shell being made as the container, an electroformed metal is deposited on the shaped substance, which then is removed to leave a metallic shell. The metallic shell is filled with the powder to be hot-isostatically compacted with its accompanying shrinkage in size from tap density ($\sim$65 percent) to full density (100 percent).

2. The Kelsey-Hayes "fluid die" process whereby the shaped cavity in two mild-steel blocks is prepared by machining, casting, or forging, followed by welding the blocks, evacuating, and filling with powder for subsequent HIPing or die pressing (Kelsey-Hayes 1979).

3. The United Technologies Corporation "soft can" process whereby the space between the outer cylindrical can and inner shaped cavity is a sintered mild steel with the same density as the

powder in the cavity to be compacted by hot-isostatic pressing (United Technologies Corporation 1976).

Ceramic

The method of producing ceramic-shell containers by a process analogous to the lost-wax investment-casting process has been developed by Crucible, Inc. (1972). With this process, intricate superalloy powder net shapes are producible. Superalloy disks as large as 25 in. (63 cm) in diameter and weighing 600 lb (270 kg) have been produced by this process. The process is somewhat similar to the fabricated-metal can-shape process except that a granular ceramic substance is used to transfer the pressure during hot-isostatic pressing. A metallic cylindrical can is used as the outer container and the powder-filled ceramic shell is positioned in this outer metallic can and supported by the granular ceramic material. This material transfers the pressure from the outer container to the ceramic shell during hot-isostatic pressing. After HIP, the metallic can is opened and the near-net-shape part is removed. This process is being thoroughly evaluated for commercial application since it offers many advantages in near-net-shapemaking capability. At this stage, it is not in commercial production for superalloy parts.

Another ceramic process involves the Kelsey-Hayes glass-bag container in which a vitreous evacuated container of a predetermined shape corresponding to the shape of the desired densification product is prepared (Kelsey-Hayes 1975). The powder-filled container then is subjected to a temperature and pressure during HIP so that the powder is compacted to full density. The glass-bag process is no longer being pursued because of various problems encountered.

Still another ceramic shapemaking process developed by Crucible, Inc., involves the use of a core of ceramic material that is resistant to size change upon compressive loading at elevated temperatures (Crucible, Inc. 1974). The surface contour of the core corresponds to the desired configuration of a cavity of a HIPed powder superalloy. The outer sealed container is metallic and is a cyliner or shape. Excellent dimensional control is possible by compressing the powder against the nondeformable ceramic core during HIP. This process is still in the development stage.

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Chapter 5

CONSOLIDATION

INTRODUCTION

Despite the advantages of atomization (e.g., improved homogeneity), the P/M superalloy process presents problems not found with conventional casting and working. In particular, some means must be employed to consolidate the powders into a monolithic body. This body can be a simple shape such as a right cylinder, a complex sonic shape, or a near-net shape, close to the engineering shape of the finished article (Figure 16).*

The most obvious method for consolidation, and potentially the cheapest, is the press-and-sinter technique used for engineering alloys such as brasses and steels; however, early work showed that certain characteristics of superalloy powders, mainly their hardness and spherical shape, effectively precluded use of this method.

Emphasis then was placed on methods employing simultaneous application of pressure and temperature. One of these, hot-isostatic pressing, has assumed the major role in commercial operations because of its flexibility and ability to produce near-net shapes most easily.

The increased homogeneity of P/M superalloys compared to their cast counterparts has permitted more highly alloyed materials to be used for turbine disks by rendering them forgeable. As the ability to consolidate powders to a shape close to that of the final part is developed, the need to have workability will be eliminated. This will permit the considerations of even more highly alloyed compositions with higher strengths and temperature capabilities.

HOT-ISOSTATIC PRESSING

HIP is performed in autoclaves with working volumes of up to 1.2 m in diameter by 3 m in length (4 by 10 ft) and capacities of up to 6.8 metric

*A sonic shape is a relatively simple shape that can be inspected with ultrasonic techniques. Curved surfaces and re-entrant angles produce scatter that impedes interpretation of the returned signal. A near-net shape is one that is as close to the final shape as is possible, allowances being made for distortion and for any surface contamination which must be removed.

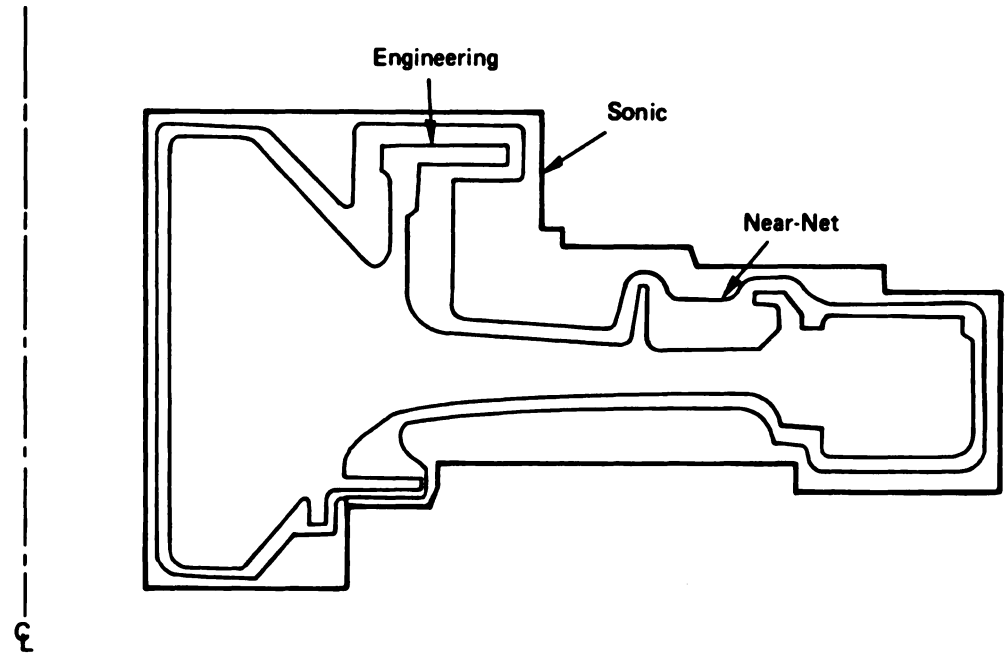

FIGURE 16 Relationship of sonic, near-net, and engineering shapes for a typical P/M turbine disk.

tons (6.7 tons) per load (Hanes et al. 1977). Other units have typical working volumes of 0.6 m in diameter by 1.5 m (2 by 5 ft) long. A typical autoclave is shown in Figure 17 and a schematic diagram for the overall system is shown in Figure 18.

HIP temperatures range from 2050°F (1120°C) to 2300°F (1260°C) depending on alloy and intended subsequent processing. Lower temperatures are used for fine-grain-size retention when superplastic forming is planned. More effective use of the HIP vessel itself can be made by use of preheat furnaces to bring the billets to about 1832°F (1000°C). The time in the autoclave can be cut to nearer the amount required to accomplish consolidation.

Pressures ranging from 15 ksi (100 MPa) to 30 ksi (205 MPa) are employed for up to three hours with argon as the working fluid. At these pressures, argon has a viscosity and density similar to water and is a very effective heat-transfer medium. Convection of argon was a major problem to be overcome since the pressure vessel itself is operated with a "cold wall." Thus, very high thermal gradients exist within the vessel.

A typical HIP cycle involving use of preheat furnaces would have a total cycle time of about 12 hours (Figure 19). Following the preheat at 1800°F (982°C), the containerized powder is transferred to the autoclave. Pressure is raised to 15 ksi (100 MPa) and the temperature to 2230°F (1200°C) over 1.5 hours. Pressure and temperature are held for three hours. The pressure then is reduced over a period of 1.5 hours and the charge cooled before unloading.

Much work has been done to develop isothermal forging to fairly complex shapes (Walker and Fuss 1977, Pfouts et al. 1979). In this process, molybdenum dies are heated to the working temperature (in a protective environment) and slow strain rates are employed to take full advantage of the low stresses and high strains offered by superplasticity.

Early use of isothermal superplastic forming started with simple cylindrical forging stock and aimed at final shapes, such as sonic envelopes, which still left considerable metal to be removed. More recent work has started with more complicated preforms and aimed at finished forgings with a near-net shape, with the ultimate goal of eliminating forging altogether.

There are several technical and economic trade-offs to be considered. In processes that start out with simple shapes, the manufacture of HIP containers and forging dies is relatively cheap and simple. The amount of expensive material scrapped is high as is the cost of the final machining. On the other hand, if the initial preform and/or final part is made more complex to reduce scrap loss and machining, preform and forging die production become much more difficult and expensive. With the present understanding of the HIP process, it is not possible to predict the final shape of a complex piece (after HIP) or to design can shapes analytically. When it is realized that typical packing densities for atomized powders are

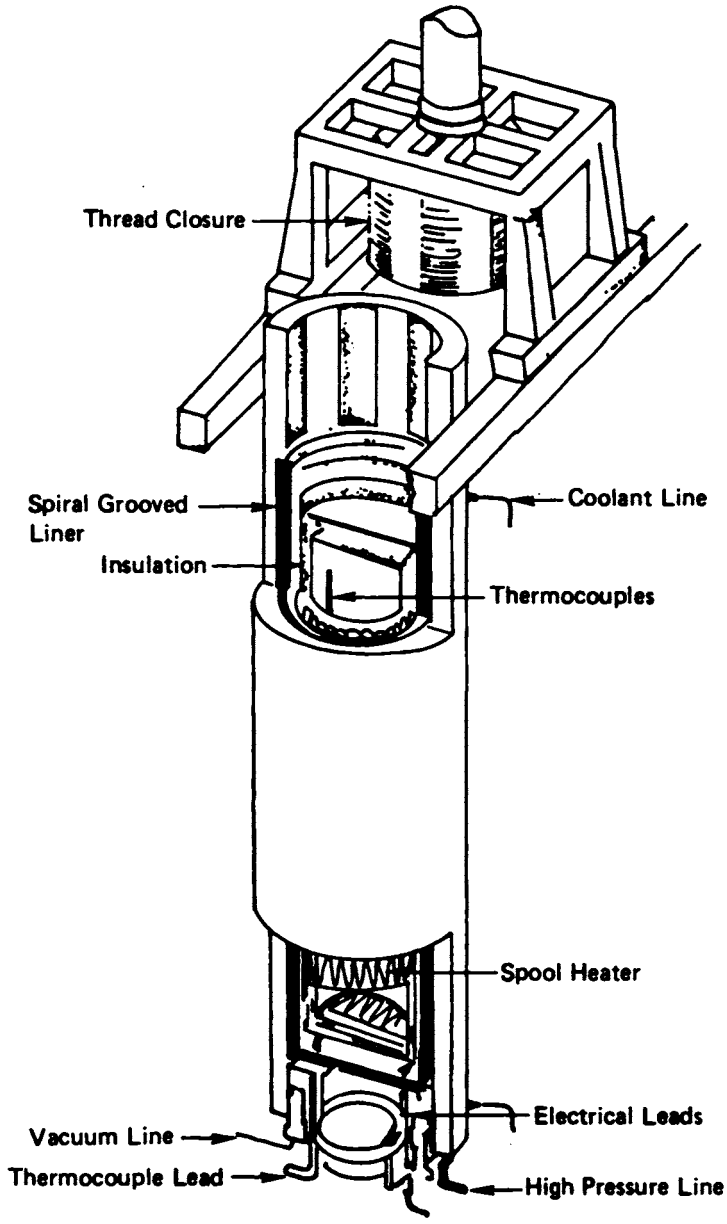

FIGURE 17 Schematic diagram of a high-temperature, cold-wall autoclave (from Hanes et al. 1977).

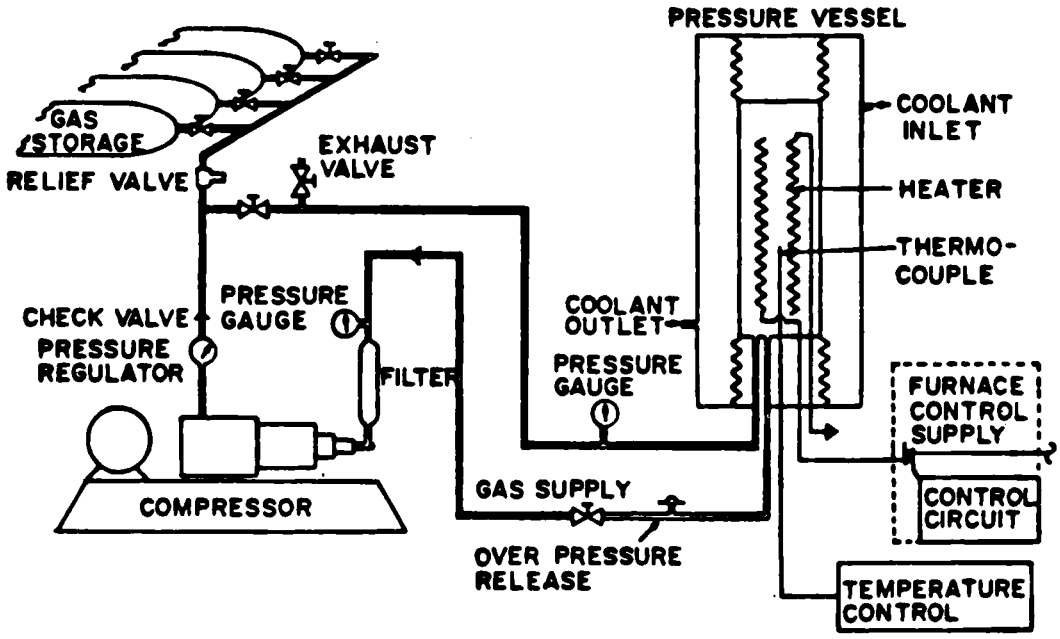

FIGURE 18 Simplified schematic for a HIP system (from Hanes et al, 1977).

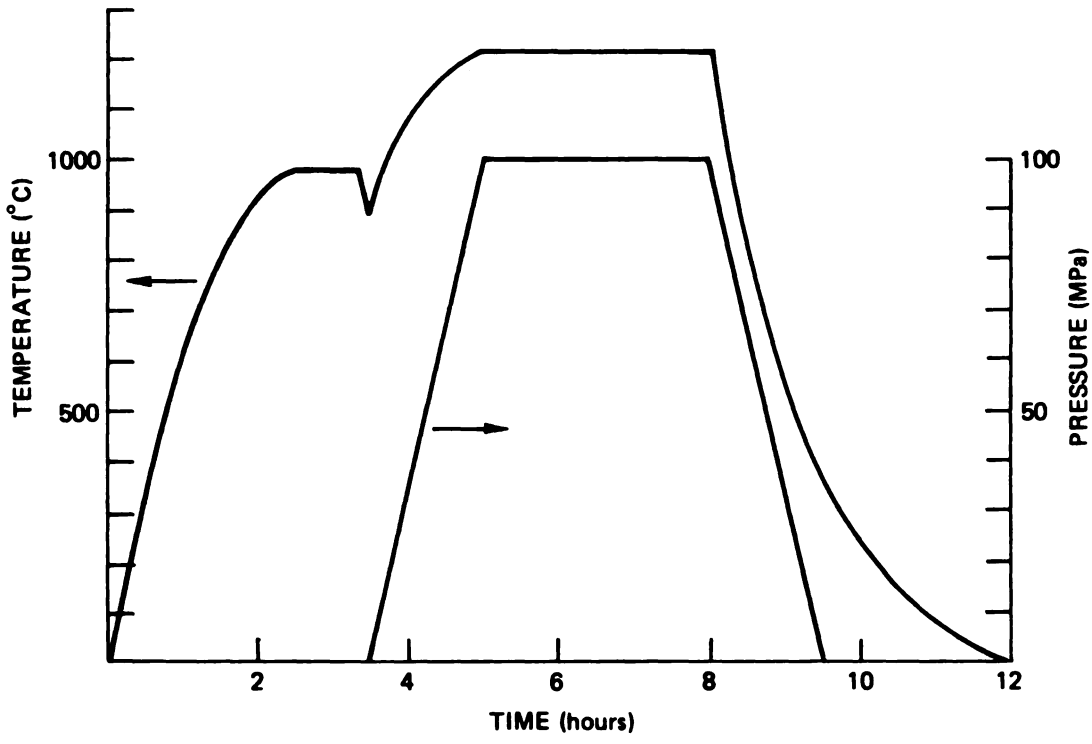

FIGURE 19 Typical HIP cycle.

60 to 70 percent of theoretical, considerable shrinkage is inevitable during HIP. This can occur in a nonuniform and nonrectilinear manner causing gross distortion in shape.

An example of this in an early attempt to obtain a sonic shape is shown in Figure 20. Clearly, the HIPed compact is not suitable for producing the target sonic shape in this example. This effect can be more severe for a more complicated near-net shape. Several "iterations" of shape usually are required in preform design. The aim is to produce a shape with an envelope at least 1.3 mm (0.05 in.) larger than the target but otherwise as close as possible. With experience, empirical rules have been developed to permit even very complex shapes to be produced. For example, Figure 21 shows the result obtained on the third iteration of a HIP-to-sonic-shape part. Similarly, cracking, lapping, and die-fill problems make die design difficult for forging complex shapes. Once these problems are solved, metal requirements and waste are reduced greatly.

As an alternative to HIP consolidation, Universal Cyclops is examining a process involving isostatic consolidation at atmospheric pressure (CAP) (Buzzanell and Lherbier 1980, Kent 1980). It is not used commercially at this time.

UNIAXIAL HOT PRESSING, FORGE COMPACTION, AND SINTERING

Hot pressing, mentioned in early experimental reports, is not used commercially. Forge compaction of cans has been used to consolidate prior to the extrusion step used to "set up" the structure required for Gatorizing ® (superplastic deformation).

P/M superalloys do not lend themselves to conventional pressing and sintering techniques. Cold pressing of atomized powders is not practical due to their regular spherical shape and high hardness. Because of the coarse size of atomized superalloy powders, solid-state sintering rates are relatively low. In order to consider sintering, some modifications of the traditional P/M approach must be made. These have included use of shaped ceramic containers to overcome poor pressibility and surface treatments to increase sintering rate.

Techniques such as nickel plating of the powders have been explored but generally lead to unacceptable heterogeneity. Kiefer et al. (1975) report that some liquid phase must be present to sinter as-atomized superalloys to a reasonable density in short times. This technique has been explored as a means of eliminating canning during HIP (Profant and Fiedler 1978). The amount of liquid phase produced was minimal with only slight amounts of metallic carbonitride chains showing the location of liquid areas. Following sintering, the uncanned compacts were HIPed to full density. Although reasonable properties were obtained compared to direct HIP, considerable problems were encountered in trying to produce shapes with re-entrant angles such as would occur in near-net shapes. The ultimate potential of this technique will depend on the difference between the costs of sintering versus those of canning and, possibly, the economic sacrifice in not being able to achieve near-net shapes.

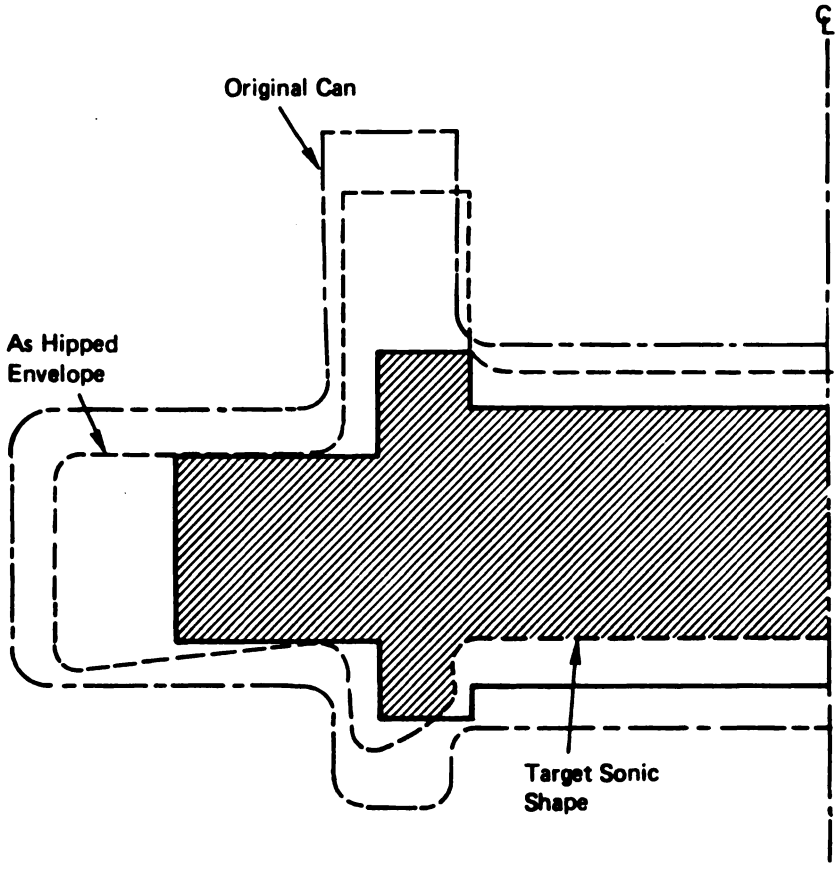

FIGURE 20 Nonuniform shrinkage during HIP.

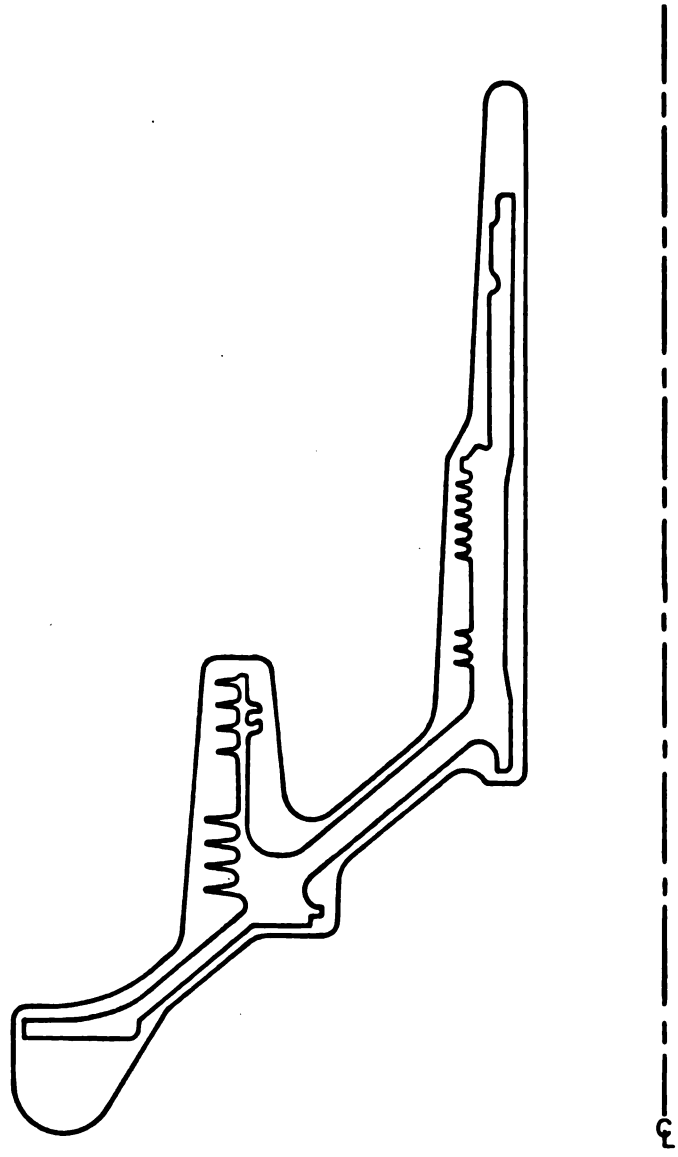

FIGURE 21 Result of third shape trial showing sonic envelope and final part shape.

Another way that has been used to circumvent the poor sintering characteristics of P/M superalloys is boron-activated sintering (DiGiambattista and Reilly 1974). A small amount of boron is put on the powder surfaces, and the powder then is encapsulated in ceramic; sintered densities of ~99.8 percent are achieved. Densification is completed by closed die forging. Tensile and stress-rupture properties generally exceed requirements for the alloys. The effects of residual boron on more stringent cyclical properties and an economic comparison of HIP to near-net shape would need to be determined to assess the true potential of this technique.

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Chapter 6

POST-CONSOLIDATION THERMOMECHANICAL PROCESSING (TMP)

INTRODUCTION

Post-consolidation TMP can consist of mechanical working and/or heat treatment. By reducing segregation and grain size, powder-atomization processes allow the working of alloys considered unworkable as conventional castings. At the same time, this removes a major aim of forging for conventionally processed superalloys--namely to produce a uniform metallurgical structure in a complex shape. Thus, mechanical working of P/M superalloys after consolidation can be limited in extent to that required to produce full properties such as by breaking down prior particle boundaries.

Many of the alloys utilized as P/M materials are not heat treated or are given only minor aging treatments when produced as castings. A grossly segregated cast alloy cannot be fully homogenized before serious liquation occurs. P/M alloys, by virtue of their homogeneity, do not suffer from this drawback. Production of a P/M version of even a cast-and-wrought alloy requires re-optimization of the heat treatment.

FORGING

The fine uniform structure of consolidated P/M superalloys greatly facilitates forging. In fact, the fine grain size (1-10 μm) of P/M superalloys HIPed below their recrystallization temperature permits superplastic forming (Reichman and Smythe 1971). Subsequent heat treatment can be used to adjust grain size and structure to develop desired properties.

Conventional forging of consolidated P/M billets and shaped preforms also has been employed (Evans et al. 1973). Acceptable P/M Astroloy disks were produced from both starting forms by forging at 2030°F (1107°C).

HOT EXTRUSION

Hot extrusion has been explored experimentally as a method for consolidating powders. In commercial operations, however, the powder billet is first hot compacted to full density. Strictly speaking, therefore, extrusion is used, not as a consolidation technique, but as a post-consolidation working process. For example, it is used to produce superplastic forging stock for near-net-shape isothermal forming operations.

Recognizing inevitable differences in composition and testing procedures which preclude one-to-one comparisons, it would appear that, in general, the mechanical properties of P/M disk forgings match or exceed those of ingot metallurgy counterparts. Some specific comparisons are made in the following section.

Free powder surface is eliminated by the hot-compacting operation which occurs at a relatively low temperature (1830°F or 1000°C). This prevents the formation of the continuous brittle carbide film that can occur in some alloys when loose powders are exposed to the higher temperatures required for extrusion. Severe cracking can occur in material that has such a carbide film.

HEAT TREATMENT

The unique characteristics of P/M superalloys lead to differences in heat treatment compared to cast or cast-and-wrought versions of the same alloys. For example, a simple three-step treatment instead of a conventional five-step treatment can be employed with P/M Astroloy (Podob 1977) because of the better homogeneity of the P/M material. As-HIPed Rene 95 must be heat treated at 62°F (17°C) below the γ' solvus to minimize grain growth and maximize the amount of γ' put into solution for subsequent aging (Bartos and Mathur 1976). The heat-treatment temperature for the P/M material is about 2080°F (1140°C) compared to 1990°F (1093°C) for cast-and-wrought Rene 95.

Grain morphology, as controlled by heat treatment, has been found to critically affect the properties of P/M superalloys. P/M IN 792 benefits by the development of a grain size of ASTM 10 with a serrated structure (Larson and Floreen 1977).

The serrated structure with an optimum "wave length" of about 4 μm is developed by slow cooling through the γ' solvus where grain boundaries try to move around heterogeneously nucleating γ'. A subsequent lower temperature carbide aging treatment is used to pin the boundaries. Similar benefits are found for cast-and-wrought material.

Development of the capability to produce near-net-shape parts either by precision isothermal forging or by direct HIP has introduced an additional problem in heat treating due to the vast differences in section size in the same piece. The cooling rate required for a superalloy following solutionizing is affected by two factors: The rate must be fast enough to retain adequate γ' in solution to develop required strength upon subsequent aging but slow enough to retain adequate ductility and prevent warping or cracking in the complex part. This may require a study of the use of special quenching methods to give cooling rates within the required range for all sections of a complex shape. Use of hot-salt-bath quenching to an elevated temperature gives cooling rates intermediate between air cooling and water or oil quenching to room temperature. This has been found to overcome the problem in work on Rene 95 near-net-shape parts (Pfouts et al. 1979).

Experimental work on extrusions of rapidly solidified powders (RSP) of MAR M 200, IN-100 (Cox et al. 1976), and many other compositions has shown that directional recrystallization can be employed to produce elongated grain structures analogous to those obtained by the same process in oxide-dispersion-strengthened alloys or by directional solidification of

cast superalloys. Such behavior is not confined to RSP powders. It has previously been noted in extruded rod produced from conventionally atomized alloy 713 C (Miner 1972). Properties comparable to those obtained in directionally solidified castings have been obtained. Thus, the production of directionally structured P/M turbine blades or dual structured P/M integral wheels may be possible.

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Chapter 7

PROPERTIES OF POWDER NICKEL-BASE SUPERALLOYS

This chapter examines the differences between the properties of superalloy P/M products and the properties of cast and worked forms of these alloys. Since the emphasis is on the differences, the general properties of the cast and worked forms are not reviewed.

The nickel-base superalloys are divided into two groups. In the first are the alloys that either can be cast and wrought or utilized in powder form; they are used in powder form when it offers economic or manufacturing advantages. The second group is comprised of alloys that can be utilized only in powder form because of a severe segregation of the solute when they are cast into ingots; the high-solute alloys fall into this category.

These two groups are considered separately here, not only because of the differences already noted but also because their properties differ. Alloys in the first group are referred to as the conventional superalloys and those in the second group, as the advanced superalloys.

P/M PRODUCTS OF CONVENTIONAL NICKEL-BASE SUPERALLOYS

The work of a number of investigators has shown that the P/M products of low-carbon derivatives of conventional superalloys (e.g., Astroloy, Waspaloy, IN-100, and Rene 95) have properties that are comparable to, or better than, the wrought products of these alloys (Bartos et al. 1974, Bartos and Mathur 1977, Cowles et al. 1978). This can be seen from the results obtained with Rene 95 (Figure 22).

As the solute contents of these superalloys are increased to improve their strength and elevated-temperature performance, the powder products look even better than the cast-and-wrought products because of the differences in their microstructures. The microstructures of the powder products are uniform while those of the cast-and-wrought products show relatively severe freezing segregation.

Table 4 and Figure 22 show that P/M products after HIP and P/M products after HIP and forging have tensile strengths that are as high or higher than those of the wrought forms of the same alloy. Active test data for HIPed-and-forged parts are compared to minimum specified values for cast-and-wrought parts. Inadequate information is available to provide statistically sound minimum values for the P/M products. Table 4 and Figures 22 and 23 show that the elevated-temperature creep strength and

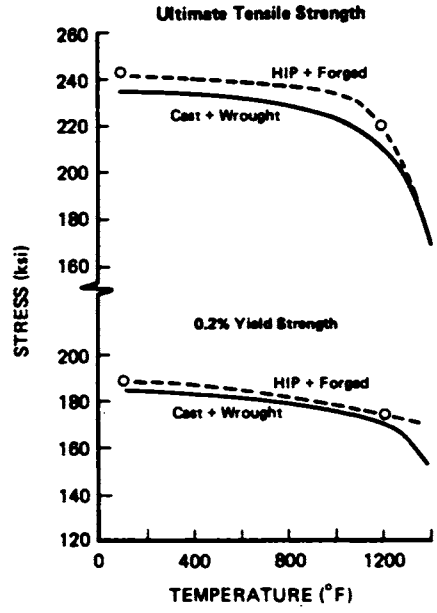


FIGURE 22 Tensile properties of cast-plus-wrought and P/M Rene 95 forged hardware.

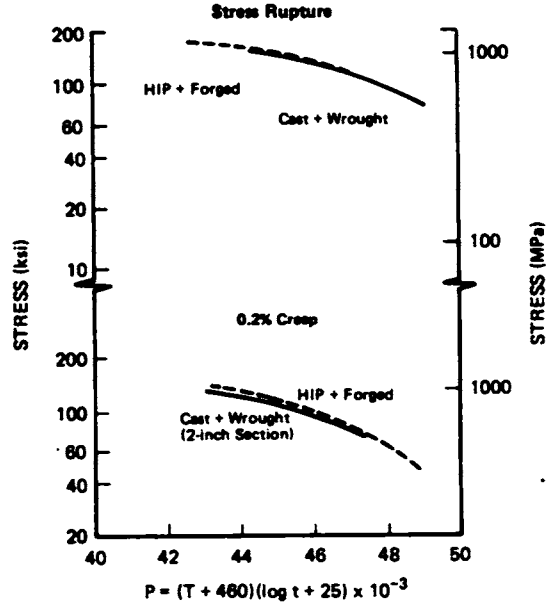


FIGURE 23 Stress-rupture and creep properties of cast-plus-wrought and P/M Rene 95 forged hardware.

TABLE 2 Properties of Rene 95

Condition	Room Temperature				1200°F (650°C)				1200°F (650°C)/150 ksi	
	YS (ksi)	UTS (ksi)	Elongation (%)	RA (%)	YS (ksi)	UTS (ksi)	Elongation (%)	RA (%)	Rupture Life (h)	Elongation (%)
HIP plus[a] forge	186	236	12	14	176	218	11	16	195	5
	186	228	11	14	174	217	10	15	167	7
	191	235	11	13	177	219	12	16		
As-HIPed[b,c]	167	233	16.3		157	216	16.1		68	5.2
	177	239	17.2	20.5	165	220	13.8	16.5	88.8	6.5
Specified[d] values	163	225	10.0		153	203	8.0		50	3.0

[a]Bartos et al. 1974.
[b]Bartos and Mathur 1977.
[c]Dulis et al. 1980.
[d]Based on wrought-product experience.

stress-rupture behavior of P/M products also are as good as or better than those of the wrought forms. Creep and stress-rupture data for Rene 95 in the as-HIPed and HIPed-plus-forged conditions are shown in Figures 24-26.

The low-cycle-fatigue life of an alloy must also be considered before it is used for a particular product. This property, rather than the creep or stress-rupture characteristic of an alloy, may prevent its use for products such as turbine disks. The LCF characteristics of P/M products are much more variable than those of the cast-and-wrought forms of the same alloys. Available data suggest that the LCF behavior of as-HIPed powder products can be as good as HIPed-plus-forged material. This is shown in Figures 27-29. Discontinuities such as inert-gas pores and nonmetallic inclusions act as "metallurgical notches" that can, under certain combinations of location and loading, lead to the premature initiation of fatigue cracks (Cox 1979), reducing the allowable design stresses and loads to unacceptably low levels.

Since LCF is a process that is subject to a "first failure criterion," any such discontinuities within the stressed region of a test specimen or, more importantly, a real component will lead to early crack initiation and fracture by low-cycle fatigue. This reduces the design-allowable property values to unacceptably low levels. The cleanliness of the powder therefore becomes a critical issue and paces its application for many components.

Since LCF failures follow the weakest-link criterion, it becomes important to assess the statistics of defect occurrence. In order to do this, substantial effort has been devoted recently to the effects of specimen size and geometry on the apparent life of powder products. The general trend among the major users of conventional superalloy powder products is not to test LCF specimens that have a large stressed volume. This enhances the probability of including a defect in the gauge length and, therefore, of finding a "weak link." One drawback associated with this approach is related to the statistics of powder-product defect location. In a large stressed-volume test bar, the stress field is homogeneous and, thus, any defect can initiate a fatigue crack. In a disk, the stress field is not uniform. This introduces an additional statistical variable. It seems appropriate to examine the data base for conventional alloys tested using the large LCF bar specimens as a comparison. The occurrence of microstructural inhomogeneities in conventionally produced superalloys also will scale with test bar volume.

There has been considerable discussion concerning the best fatigue test for determining the presence of defects. Arguments have been made for high-cycle fatigue and others for low-cycle fatigue. This point is discussed in greater detail in appendix C.

ADVANCED P/M ALLOYS

In addition to the conventional alloy compositions that now are being atomized and used for P/M products, alloys of very high solute

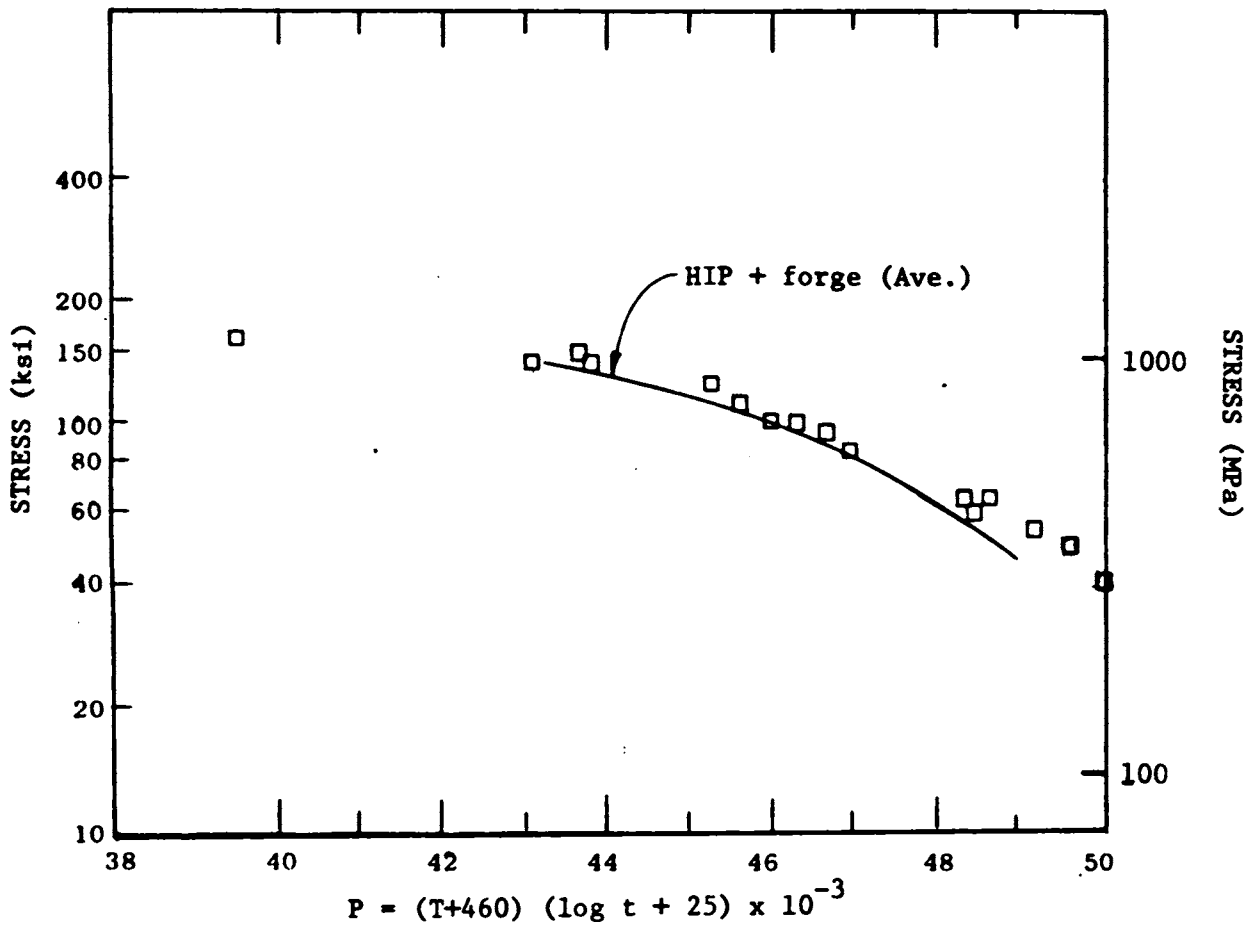


FIGURE 24 0.2 percent creep data for as-HIPed and HIPed-plus-forged Rene 95. Data points are as-HIPed.

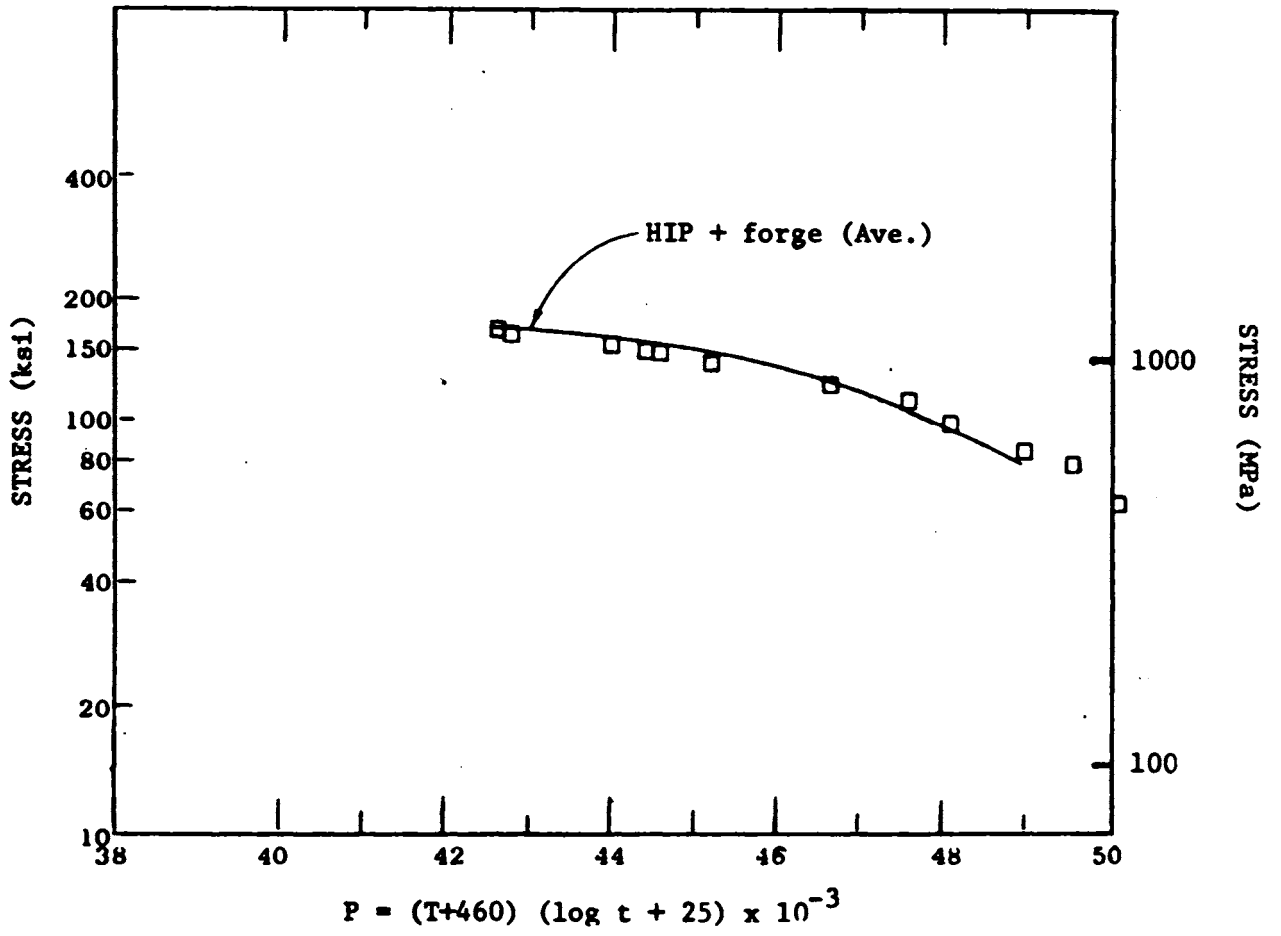


FIGURE 25 Stress-rupture data for as-HIPed and HIPed-plus-forged Rene 95. Data points are as-HIPed.

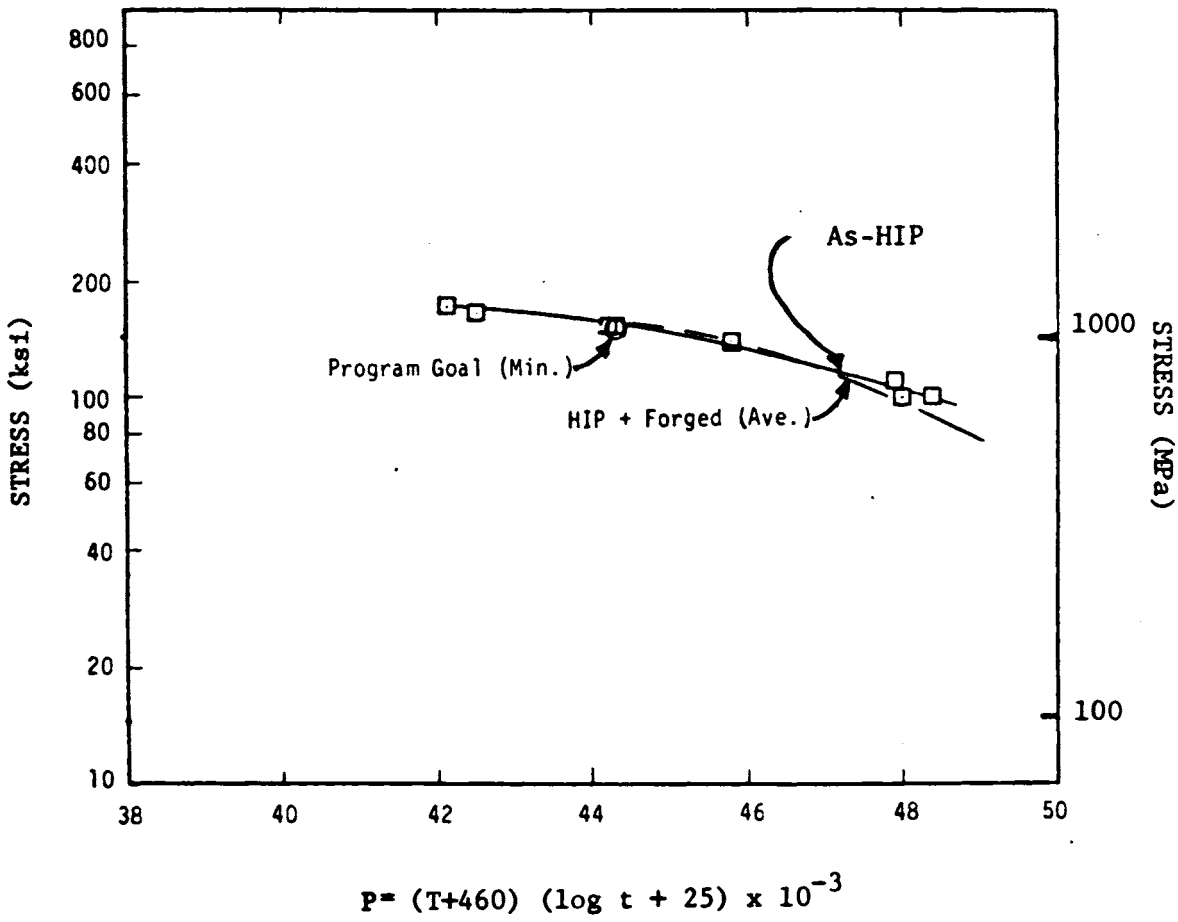
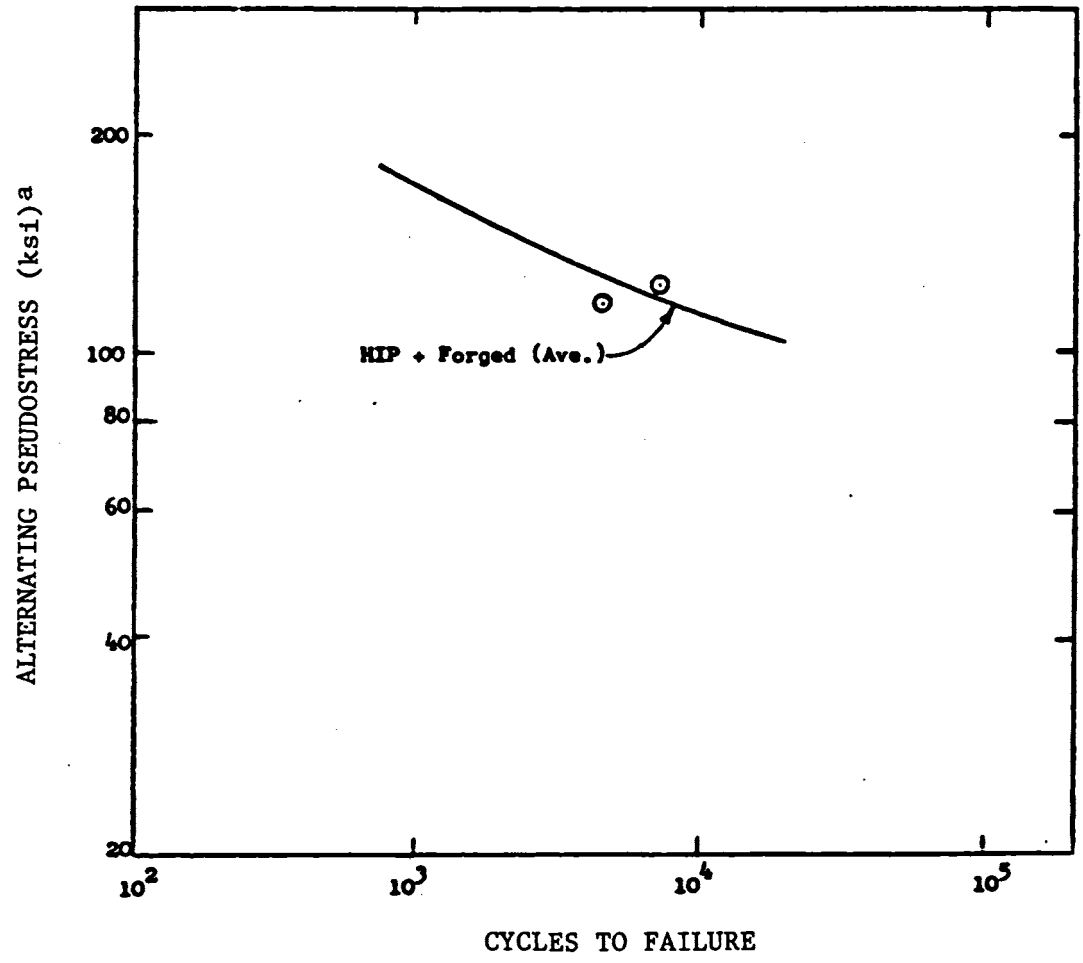


FIGURE 26 Additional stress-rupture data comparing as-HIPed with HIPed-plus-forged Rene 95. Data points are as-HIPed.



[a]Pseudostress = $\Delta\varepsilon_t \cdot E_y$,

where $\Delta\varepsilon_t$ = total strain amplitude and

E_y = Young's modulus.

FIGURE 27 Strain control 1200°F (650°C) LCF data at A = 1 for as-HIPed (data points) and HIPed-plus-forged Rene 95. Data points are as-HIPed.

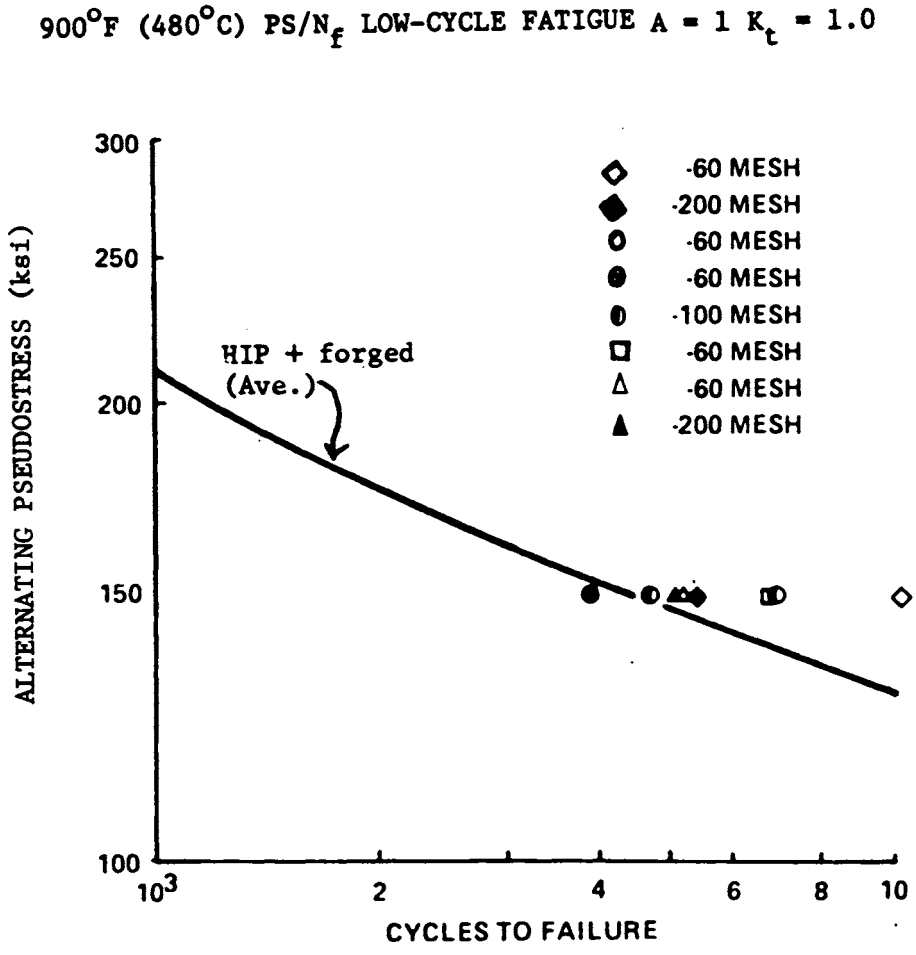


FIGURE 28 Fatigue data for as-HIPed and HIPed-plus-forged Rene 95. Data points are as-HIPed.

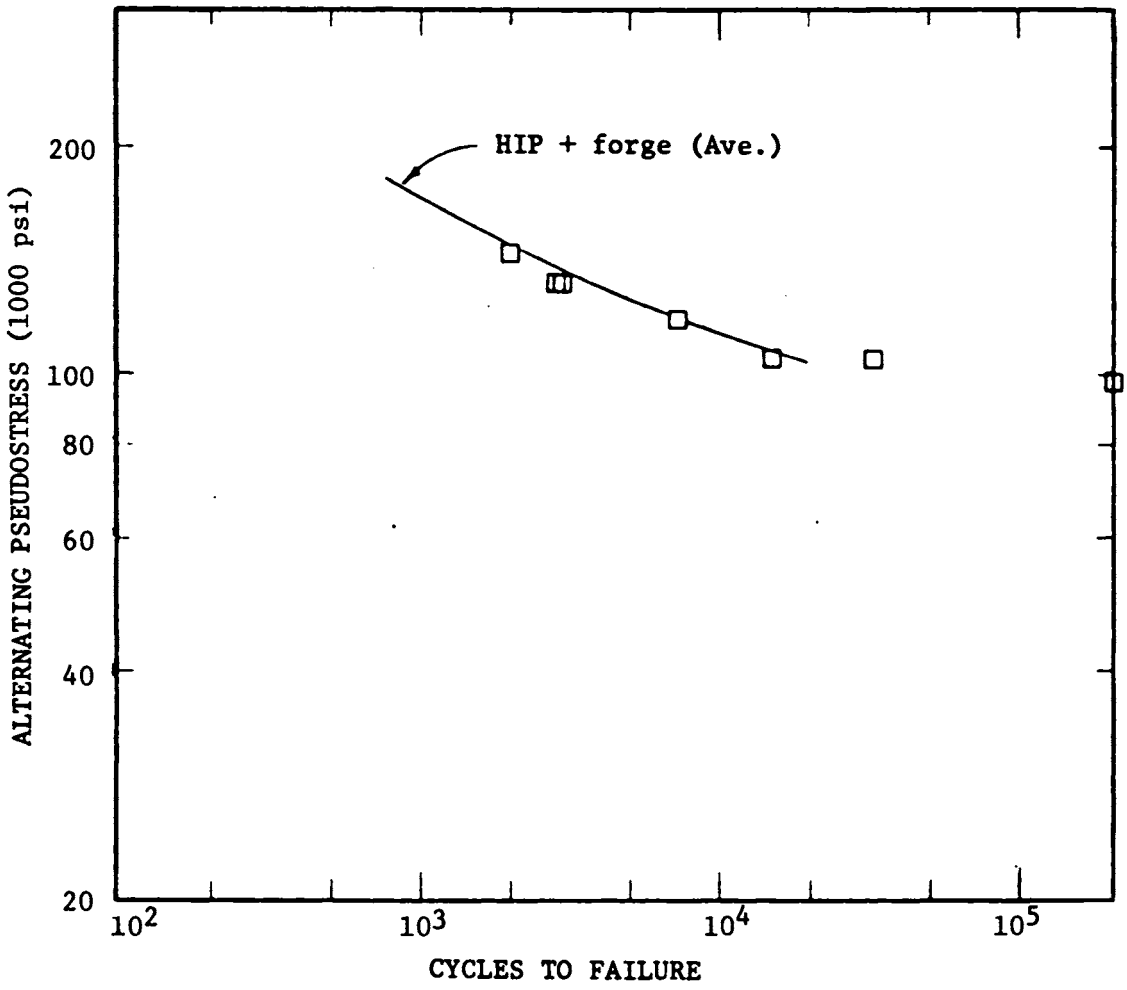


FIGURE 29 Fatigue data at 900°F (480°C) for as-HIPed and HIPed-plus-forged Rene 95. Data points are as-HIPed.

content, for example, 14 percent Mo, have been produced by a new rapid-solidification-rate method of powder manufacture. Based on 100h rupture-life data, these new compositions appear to have very attractive properties. For example, their stress-rupture and creep resistance is as much as several Larson-Miller parameters better than any of the cast or wrought conventional alloys.

It must be emphasized that the production of high-solute-content alloys that also are macroscopically homogeneous must be produced using P/M techniques. However, an important remaining question still under discussion is whether these high-solute-content alloys can be manufactured successfully by conventional atomization techniques or whether the new rapid-solidification-rate process is required. Another important factor regarding solidification-rate requirements during powder production is assessment of the extent to which the as-solidified powder structure influences the structure after consolidation and, therefore, the properties of the powder product. The alloys with exceptionally high solute content must be microstructurally stable, not only through fabrication, but also in use. This point is still under investigation but is critical to the determination of cooling-rate requirements for powder production.

Most of the powder of the very-high-solute alloys manufactured to date has been made by the RSR process. An alloy such as RSR 185* (Ni-14.3 Mo-7 Al-6 W-.04 C) in the directionally recrystallized condition has 34 ksi (234 MPa), 1900°F (1040°C), 100h rupture strength after consolidation. This is approximately 1.8 times the life of DS Mar-M 200+Hf (Cox 1979). The lower-temperature properties (∿1400°F or 780°C) of these alloys also are attractive and, thus, they have potential for improved turbine disks.

The application of high-solute alloys to disks that tend to be LCF limited as opposed to creep limited has not been evaluated as carefully. Here it seems that defects such as those found in conventional alloy products will be very detrimental. If these alloys are stronger, the applied stress also will be higher and will activate even small defects if the analysis in appendix C is correct.

QUALITY CONTROL OF P/M PRODUCTS

The evaluation of product quality for P/M components includes the same procedures as are used for wrought products, but certain additional precautions need to be taken because of the powdered-metal origin of the products.

Careful scrutiny of specifications for premium-quality P/M Rene 95 shows that the two principal additional evaluation procedures are to determine the presence and extent of gas porosity and foreign particles. In the case of the former, a thermally induced porosity (TIP) test is used. One such test entails a maximum density reduction of 0.3 pct after a 4h 2200°F (1200°C) exposure in air.

*Not considered a disk alloy.

In the case of nonmetallic inclusions, special LCF bars that have a large stressed volume are used. As mentioned above, increasing the volume of the gage section increases the probability that a foreign particle will be contained therein. It is not good practice to use special small-volume test specimens since to do so might decrease the probability of finding defects. In addition to LCF testing, a metallographic technique for measuring nonmetallic-inclusion content also exists. This technique relies on the availability of an etchant that attacks these inclusions without etching the carbides and other minor phases present in Ni-base alloys. Such an etchant exists for Rene 95 but might have to be developed for other alloys.

Sonic inspection has been a standard inspection procedure for disks of the type discussed here virtually since inspection to detect internal forging bursts and other such manufacturing defects was found necessary. This tradition for sonic inspection has carried over into P/M disks even though there is some reason to believe that a whole new type of defect may be present. Moreover, there is some question whether sonic inspection can find pores and nonmetallic inclusions because of their relatively small size. In this regard, the sensitivity of sonic inspection should be greater in P/M products because the finer, more homogeneous microstructure causes less spurious scattering and "noise" in the reflected signal.

An associated concern is the insistence of turbine manufacturers on the availability of a sonic shape for inspection. The requirement for such a shape represents a major barrier to incorporation of near-net shapes. This is, the sonic shapes are larger and simpler than the near-net shapes because they must have a relatively simple geometry to be inspectable by sonic methods. Thus the P/M shape-making capability has currently "outstripped" the inspection capability. The result of this is to place an unfortunate economic penalty on the P/M technique. The solution to this problem is either to demonstrate that sonic inspection is inappropriate for P/M parts or to incorporate newer, more sophisticated sonic inspection techniques. In this latter regard, some work on acoustic holography (Bartos and Mathur 1977) and computerized sonic inspection (Doherty 1978) is underway. Both of these techniques appear to hold promise but neither are currently state-of-the-art for routine production. It thus appears that development of better inspection methods for near-net shape P/M parts is required before a major pay-off of improved material utilization and more attractive "buy-to-fly" ratios can be achieved.

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Chapter 8

ALLOYS FOR P/M APPLICATIONS

INTRODUCTION

Efforts to obtain strength and fatigue improvements by further alloying additions to conventionally processed alloys led to increased microsegregation in cast-and-forged components, which in turn restricted or negated the effect of these additions. In addition to economic advantages associated with near-net-shape or as-HIPed technology, consolidated prealloyed superalloy powders possess the advantages of compositional homogeneity, equiaxed fine-grain size, and uniform distribution of precipitated γ' $\{Ni_3(Al,Ti)\}$ phase particles and carbides. The P/M approach also offers the potential for creating new alloy compositions that are not castable but that can be produced by atomization techniques and that provide opportunities for the study of new types of alloy systems.

Although there is some concern that alloy development studies should involve a reduction in the concentrations of strategic elements such as chromium or cobalt, it is believed that in the long term the availability of these elements will be assured for critical applications (National Materials Advisory Board 1978a and 1978b, Charles River Associates, Inc. 1979). Therefore, alloy development should not ignore the achievement of optimum properties by unduly restricting alloy compositions.

ALLOY COMPOSITIONS

A partial list of alloys that have been produced by conventional atomization techniques is presented in Table 3. Although this report is concerned with disk alloys, Table 3 includes alloys that have been used for other P/M parts or for hardfacing powder feed. Several of these alloys represent low-carbon versions of conventional alloys normally used as castings or cast-and-wrought material. Low carbon contents are used to avoid the precipitation of carbides on powder-particle surfaces during consolidation. These precipitates generally lead to premature failure; the fracture follows the inter-particle boundaries.

Included in Table 3 are four Ni-base alloys that were developed in the Soviet Union for conventional processing but that subsequently have been reported to be used for P/M processing of parts. These compositions suggest that the alloys are equivalent to the lower-strength alloys used in the United States. It should be noted that the published data on superalloys in the Soviet Union tend to be several years behind current research and development status.

TABLE 3 Alloy Compositions Produced as Prealloyed Powders

Alloy		C	Mn	Si	Cr	Ni	Co	Mo	W	Ta	Cb	Hf	Al	Ti	B	Zr	Other	Reference
Alloys Used for Commercial Powders																		
Astroloy (LC)	D[c]	0.04	-	-	15	Bal	17	5	-	-	-	-	4	3.5	0.02	0.05	-	ASM 1978
Rene 95 (LC)	D	0.06	0.15[a]	0.2[a]	13	Bal	8	3.5	3.5	-	3.5	-	3.5	2.5	0.01	0.05	0.5[a] Fe	ASM 1978
IN 100 (LC)	D	0.07	-	-	12	Bal	18.5	3	-	-	-	-	5.5	4.3	0.014	0.06	0.8 V	ASM 1978
Alloys Developed Specifically for Powder Metallurgy																		
AF-115[b]	D	0.155	-	-	10.9	Bal	15.2	2.8	6.4	-	1.7	2.0	4.1	3.9	0.02	0.05	-	Bartos 1974
XN-312Hf		0.08	-	0.01	11.5	Bal	20	3	3	2	2	1.3	2.9	4.3	0.02	0.05	-	Bauerle and Whelan, 1976
PA-101	D	0.16	-	-	12.2	Bal	9	2	4	4	-	1	3.4	4.1	0.015	0.11	-	Ewing et al. 1972
MERL 76	D	0.02	-	-	12.5	Bal	18.5	3.2	-	-	1.6	0.7	5.0	4.3	0.02	0.06	0.03[a] Fe	
RSR 116		0.05	-	-	9.2	Bal	-	-	9.4	-	-	-	8.3	-	-	-	-	Bourdeau and Moore 1977
RSR 143		-	-	-	-	Bal	-	14.3	-	6.0	-	-	6.0	-	-	-	-	
RSR 185		0.04	-	-	-	Bal	-	14.3	6.0	-	-	-	7.0	-	-	-	-	
Conventional Alloys Evaluated for Use in Powder Metallurgy																		
IN 792 (LC)		0.04	-	-	12.4	Bal	9	1.9	3.8	3.9	-	-	3.1	4.5	0.02	0.10	-	Larson 1976
MAR M200		0.15	-	-	9	Bal	10	-	12	-	1	-	5.0	2.0	0.015	0.05	-	ASM 1978
713 (LC)		0.05	-	-	12.5	Bal	-	4.5	-	-	2	-	5.9	0.6	0.01	0.10	-	Sims and Hagel 1972
U 700	D	0.08	-	-	15	Bal	18.5	5.2	-	-	-	-	4.3	3.5	0.03	-	-	ASM 1978
B 1900		0.10	-	-	8	Bal	10	6	-	4	-	-	6.0	1.0	0.015	0.10	-	ASM 1978
B 1950		0.006	-	-	8.6	Bal	9.9	-	12.1	-	1.1	-	4.8	2.1	0.2	0.06	-	Holiday et al. 1977
NASA IIb-11		0.06	-	-	8.5	Bal	9	2	7.1	6.6	-	0.7	4.5	0.75	0.01	0.05	0.5 V	Kent 1972
NASA-TRW VI-A		0.15	-	-	6	Bal	7.5	2	5.8	9	0.5	0.5	5.4	1.0	0.018	0.10	0.5 Re	Sims and Hagel 1972
AF2-1DA[b]	D	0.35	0.1[a]	0.1[a]	12	Bal	10	3	6	1.5	-	-	4.6	3.0	0.015	0.10	0.5[a] Fe	ASM 1978
HS-31		0.5	1[a]	1[a]	25	Bal	10.5	-	7.5	-	-	-	-	-	-	-	2[a] Fe	Sims and Hagel 1972
Waspaloy	D	0.07	0.5[a]	0.75	19.5	Bal	13.5	4.3	-	-	-	-	1.3	3.0	0.006	0.06	2[a] Fe	ASM 1978
Conventional Nickel-Base Alloys Evaluated for Use in Powder Metallurgy in the Soviet Union																		
KhN55VMTFKYu (EI 929)	D	0.003	-	-	13.2	Bal	13.1	5	5	-	-	-	3.3	1.7	-	-	-	Lavrent'ev 1974
EI 698	D	0.08	-	-	15	Bal	-	2	-	-	-	-	1	2.5	-	-	-	Hdbk. Sov. Alloy Comp. 1975
Kh20N80T	D	0.12	0.7	0.8	22	Bal	-	-	-	-	-	-	0.2	0.4	-	-	-	"
ZhS6K	D	0.14	-	-	10.8	Bal	4.5	3.8	4.9	-	-	-	5.3	2.8	-	-	-	"

[a]Maximum.
[b]Alloys available for application.
[c]Designates use as disk alloy.

Two alloys, AF-115 and XN-312Hf, were developed specifically as P/M alloys and approach the limit of alloy compositions that are stable to the precipitation of topologically close-packed (TCP) phases such as Sigma and Laves. These TCP phases are regarded as being undesirable when present in any significant amount.

Low-carbon Astroloy, IN-100, and Rene 95 are three commercially produced powder alloys that satisfy many, but not necessarily all, of the property requirements for current-generation gas-turbine disks. It is logical that these most widely used powder alloys should have evolved from established wrought or cast alloys. New powder alloys must meet existing minimum property levels for creep-rupture strength, low- and high-cycle-fatigue strength, surface stability (corrosion resistance), and microstructural stability. These properties become mutually exclusive as higher alloy levels are employed and have been balanced in the established alloys as best they can. Use of powder, however, increases the opportunities for improvements in low-cycle-fatigue resistance and structural stability while also giving more freedom from processing restrictions. Several stronger alloys (e.g., AF-115) are undergoing extensive evaluation.

Almost all of the compositions of the superalloys used at this time for P/M processing originally were developed for fabrication by other methods (i.e., casting or forging of blades). Since properties are dictated by processing history as well as composition, it would be fortuitous that a given composition would give rise to optimum properties by alternate processing methods. It follows that optimum properties can be obtained from P/M parts only if compositions are developed specifically for such processing. It has been estimated by one aircraft-engine manufacturer that the cost of developing and qualifying a new disk alloy is $8 to 10 million with the alloy-definition phase constituting only 10 percent of this amount. Clearly it is difficult to justify isolated compositional evaluation or development programs without clearcut end objectives.

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Chapter 9

ECONOMIC CONSIDERATIONS

INTRODUCTION

The attainment of military or commercial levels of performance, reliability, and efficiency requires judicious use of available technology and a determination of not only the level of performance or reliability that must be attained but also when and at what cost. Performance, schedule, and cost considerations must be addressed in a total life-cycle context at the aircraft-system level for the particular aircraft system and engine subsystem of interest. Performance and reliability are linked to the desired schedule because of the trade-off between increased performance and/or improved reliability with regard to the available technology at a specific time. If improved levels of performance and reliability beyond the present state of the art are desired, additional technology is necessary and time is required to achieve that higher level of technological maturation. This becomes evident in a comparison of performance-driven military and reliability-driven commercial experiences although commercial objectives recently have been approaching military objectives concerning some aspects of performance and reliability.

One incentive for use of P/M technology, especially when used to produce near-net shape, is an economic one. There are at least three sources of potential cost savings: decreases in raw material costs, elimination of working steps and less machining. Decreases in raw material weight requirements are indeed realized. Setting the raw material requirements for conventional melting and forging at 1.0, the calculated requirements for various P/M alternatives range from about 0.35 to 0.6 for isothermal forging of a P/M billet or preform, 0.2 to 0.4 for HIP to sonic shape, and 0.2 for HIP to near-net shape (Reichman, 1975; Bartos and Mathur, 1976). The "sonic shape" is a fairly simple cylindrical shape which can be inspected ultrasonically. More complex shapes with curved surfaces or reentrant angles severely scatter ultrasonic pulses. While such signals could be analyzed in principle with the aid of computers, the necessary programs have not been developed and proven. Near-net shape is one as close to that of the final part as possible with allowances for distortion during HIPing and for material contaminated by reaction with the container.

Raw materials costs are not necessarily decreased by P/M technology (Profant and Fiedler, 1978). The higher processing costs for powder production, as opposed to melting and casting, are factors. Overall part costs may be beneficially impacted, however, by savings in machining and inspection. Other less obvious savings may arise through the ability to produce a single complex part where several simpler parts had to be joined when using conventional casting and forging.

These figures represent early attempts to calculate cost advantages of P/M technology. In reality, the problem is a complex one involving overall life cycle cost analyses rather than simple data on weight savings or even final part costs.

Industry uses two approaches in assessing the value of a particular new technological improvement. At one level, estimates are made parametrically of the performance gains and costs that might be expected at the macrosubsystem or system level concerning an engine's overall performance, reliability, efficiency, and installation in a particular new aircraft system and the benefits that might be achieved by applying the technological improvements if they were available. At a more detailed microcomponent level, studies and tests are conducted concerning the impact of a particular part or material in a component and the component's impact on the engine and, thus, on the aircraft.

Parametric analysis is used at the macrosubsystem or system level so that designers can select the appropriate design point for a new system prior to extensive engineering design. The available data base of historical engine programs is utilized to obtain parametric relationships from which the new technological improvement is extrapolated. At the microcomponent level, detailed engineering analysis usually is employed in evaluating how great a benefit an anticipated improvement in cycle performance or materials or a new design technique for a particular part or component can be expected to provide. If a particular technology looks very promising at the part level, a piece of hardware is manufactured so that it can be tested in an existing engine to determine performance, reliability, or cost differences relative to the part being replaced. It is very important to obtain experience in the new technology as early as possible. In both approaches, engineering value judgments play a large role.

This chapter discusses one particular technological improvement--superalloy powder metallurgy for turbine disks--and its potential benefits and costs. A brief summary is presented of the short-term and long-term benefits and costs to be expected from this new technology. Many new ideas for improving aircraft turbine engines in some way are developed over a period of time, but the government and industry cannot fully fund all the basic research and early exploratory and advanced developmental programs needed. This is, in a sense, encouraging from an overall technology standpoint in that it implies that aircraft-turbine-engine technology is dynamic and does not lack for new ideas to improve the state of the art. However, some selection criteria must be employed to establish priorities among many potentially useful ideas. This selection criteria takes the form of some type of benefit-cost analysis.

POTENTIAL BENEFITS AND COSTS

The use of superalloy powder metallurgy for high-temperature turbine disks is receiving increasing support by industry and the military because of the potentially significant benefits to be attained from this new technology. The benefits include:

1. Higher temperature levels and/or improved life for disks.

2. A stronger and/or lighter part.

3. A need for less input material in the manufacturing process.

4. Lower final rejection rate during and after machining (because the material is more homogeneous).

5. Fewer steps in the manufacturing process for complex shapes.

6. Potentially lower overall manufacturing costs (because of lower material and labor requirements).

The overall cost savings for a particular disk will depend on its size, complexity of shape, application, and production quantity. Very limited data made available to the committee by several manufacturers indicate that cost savings currently expected for a direct substitution of a powder disk for a forged disk in a current inventory engine are about 10 to 20 percent. Expectations of savings for newer programs in which the powder disk could be designed for the engine initially could be about 40 to 50 percent. No one in the industry has provided specific figures in terms of dollar savings except for the TF30-P-414 example presented below. There are no data concerning the investment required in facilities and in gaining experience. For example, there are no data concerning the cost of powder development, the cost of facilities for powder development, the cost of manufacturing facilities and tooling for parts development and production, or the cost of the development of expertise in turbine engine design, development and production industry. In addition, it has not been determined whether energy requirements make a difference. Nevertheless, everyone seems to agree that over the longer time period and for reasonable production rates, significant cost savings and performance and reliability enhancements are possible.

Pratt and Whitney provided some data comparing a TF30-P-414 Waspaloy first-stage turbine disk that was redesigned for improved LCF life utilizing IN-100 powder metal. Pratt and Whitney employed the Gatorizing® process in this particular example. The substitution of IN-100 for Waspaloy was intended to achieve higher strength and a doubling of the LCF life for the TF30-P-414 engine in the F-14A. All cost data provided by Pratt and Whitney are in relative percentages. Waspaloy is the baseline with an index of 1.0 in terms of cost. The final IN-100 finished part cost was 0.92 for an 8 percent saving. The part was dimensionally identical to the Waspaloy part but was stronger and of lower density and, thus, provided weight savings as well as life improvement at lower cost. In this particular case, the substitution of a disk in an existing engine-aircraft combination did not provide a weight saving for the aircraft system as would be the case if the part were for a new engine in a new aircraft.

The IN-100 part has higher yield and ultimate strengths, material utilization is improved, less labor and fewer machining operations are needed, and cost is lower. It is more inspectable and more homogeneous in terms of nondestructive testing and is manufactured closer to the near-net shape in terms of the material utilization. For instance, the Waspaloy forging as it comes from the vendor weighs 337 lb whereas the IN-100 part weighs 155 lb. Thus, the weight ratio of the IN-100 to the Waspaloy is 0.46 prior to machining. The Waspaloy is 13-1/2 percent cobalt (45 lb) whereas the IN-100 part, even though it has more cobalt per pound (18 percent), utilizes less of the strategic material (about 29 lb). The cost ratio of the semi-finished part from the vendor is 0.87 since the IN-100 is more expensive per pound and its processing cost is higher. Although the IN-100 is lighter, there is not much difference in labor cost for the vendor-delivered product. The cost of machining at Pratt and Whitney appears to be about the same since the IN-100 is harder to machine and requires special tooling (it cannot be cut with carbide tools). The IN-100 also requires extra polishing since it is sensitive to notches. The overall final cost is 0.92.

Pratt and Whitney also is utilizing a significant number of P/M IN-100 parts in the F100 engine, which is in the F-15 and F-16 aircraft. Approximately 1000 lb of billet material goes into the F100 engine. Nine parts are involved: three compressor disks, four turbine disks, and two spacers. However, since this engine was not initially designed to use other materials and processes, no cost comparison exists as in the case of the TF-30-P-414.

General Electric (GE) provided a rough comparison for the T700 engine, which utilized six parts made from Rene 95 powder, including a seven-in.-diameter disk. GE also cited data for the F404, which utilized 12 P/M parts including a 17 to 20 in. disk. The cost saving in the T700 is expected to be 20 percent and the F404 is expected to save 40 percent. Again, the machinability improvement did not seem to provide much cost saving. The real payoff appears to be in the ability to eliminate the excessive scrap losses associated with conventional Rene 95. GE also cited an input-material savings of about 50 percent. Ease of machinability and ease of sonic inspection also were cited as advantages. GE specifically noted increasing alloy temperature capability and increasing complexity in part shape as the incentives for the continuing research in P/M superalloys.

EVALUATION OF POWDER METALLURGY

In attempting to evaluate a new technology improvement, one can compare a specific P/M part to the present conventional part in an existing engine or in a new engine design. It is possible, given appropriate data, to calculate a return-on-investment based on the savings from the P/M part relative to the cost of developing the part. Calculations have been employed in NASA studies by GE, Pratt and Whitney, and AiResearch, but these are estimates only. In NASA's Materials for Advanced Turbine Engines (MATE) program (Bisset 1976, Comey 1977, Hillery and Johnston 1977), the benefits and costs were estimated in terms of relative value. In this case, a net-return-on-investment (development cost) was the measurement used.

Sensitivity analyses were employed to determine the probability of success and costs were estimated for a variety of materials and parts for new engines. Again, it must be emphasized that all of these estimates are hypothetical; no actual data from past experience were provided.

Over the long term, the manufacturers are interested in understanding what the parts actually can do and, thus, are trying to get experience by replacing specific parts in engine programs. This was the case at Pratt and Whitney with the TF30 and F100 and several commercial programs (JT8D, JR9D) and also at GE for the T700 and F404 and certain commercial programs (CF6, CFM56).*

It is important to continue to consider the impact of the long-term engine improvements at the system level and what the ultimate benefits and costs might be of continuing evolutionary technology improvements. It is at the system level that the greatest net savings can be achieved even though the new technology could result in a more expensive engine for a new engine design in a new aircraft. It has been shown that although the cost of turbine engines for fighter aircraft has increased in real terms over the past decades at the aircraft-system level the new engine technology provided payoffs in improved performance and/or reliability and in reducing the overall life-cycle cost of the total aircraft system (Nelson 1979).

In summary, an economic analysis of superalloys from powder must await accumulation of a sufficient data base concerning material-development, facilities, manufacturing, and learning costs. In any analysis of technological improvements regarding the benefits and costs to be expected, it is important to maintain both a short-term and a long-term perspective regarding the direct substitution of parts in current engines for the experience to be achieved (even though in some cases not necessarily immediately cost-effective), and the long-term impact of improved technology on new engine designs for new aircraft systems. Overall, new technology for aircraft turbine engines still is apparently well worth the cost.

*No commercial program data were supplied to the committee by either company.

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Appendix A

FOREIGN DEVELOPMENTS

The information contained in the body of the report pertains primarily (although not exclusively) to P/M superalloy activities in North America. To provide an international perspective of the current technology, developments in Europe, the Soviet Union, and Japan are summarized here.

EUROPE

Through the mid-1970s Sutcliffe (1976a and 1976b) has assessed the state of advanced powder metallurgy in European NATO countries with particular reference to nickel and titanium alloys for high-integrity aero-engine components. More recently, Evans (1979), under contract to the United States Air Force (European Office of Aerospace Research and Development), made an extensive review of European powder metallurgy superalloys. Input also was obtained from the Office of Naval Research (Diness, 1979). These studies served as the basis for the following summary and conclusions vis-a-vis P/M superalloy technology in Europe.

Several atomization methods have been developed for the production of superalloy powders suitable for eventual incorporation into disk components. These involve gas atomization and various modes of rotating electrode or centrifugal atomization. In light of the ability of the Henry Wiggin (U.K.) argon-atomizing facility to handle scrap and its high production rate, proven technical capability, and commercial availability, it is predicted that argon atomization will be the only commercially viable process needed in Europe for the foreseeable future. Powder-handling procedures are similar to those adopted in North America.

In terms of consolidation, a number of different alloys and consolidation routes are under evaluation. HIP to sonic shape and HIP plus isothermal forging are considered most useful. As is the case in North America, disks are limited to sonic rather than near-net shape because of limitations in nondestructive testing; therefore, maximum materials savings cannot be realized. Concern exists with respect to the level and reproducibility of ductility and low-cycle-fatigue properties in powder-consolidated disk materials.

Some P/M disks presently are undergoing engine trials in the United Kingdom although it is realized that considerably more research and development work is needed. It appears that no serious effort is being made in Europe to explore or develop new nickel-base alloy compositions. Limited work in Europe suggests that P/M cobalt alloys do not offer advantages over nickel-base compositions.

The various forms of spray forming are considered attractive since the method combines the benefits of powder metallurgy with those of rapid solidification processing while eliminating powder handling. In North America, the corresponding technology is the Pratt and Whitney Laserglazing® approach.

SOVIET UNION

Although less extensive and direct than for Europe, there is sufficient evidence to show a strong interest on the part of the Soviet Union in superalloy powder metallurgy technology. The Soviets are felt to have made considerable progress over the past five years (Marley 1979) through a judicious effort to advance nickel-base powder metallurgy. This is reflected in the areas of powder production and powder consolidation (Marley 1979 and private communication, N.J. Grant, Massachusetts Institute of Technology, 1979). Displays of powder-processed materials at the Paris Air Shows in 1975, 1977, and 1979 indicate a major commitment has been made to the fabrication of nickel-alloy turbine disks.

Electrolytic and carbonyl powders have been produced for a number of years, but since the early 1970s, attention has been directed to gas-atomization and rotating-electrode production methods. It recently has been reported that rapid-solidification work has been initiated at the Central Scientific Research Institute for Ferrous Metallurgy (CSRI) in Moscow and that this is an area of increasing interest and importance to the Soviets (private communication, N.J. Grant, Massachusetts Institute of Technology, 1979).

Little information exists with which to quantify Soviet powder-production capabilities. By far, the majority of research reported in the open literature pertains to electrolytic or carbonyl powders. According to Grant (private communication, 1979), pilot-scale vacuum melting and argon atomization are practiced at Zaporozhy; attempts are being made to vacuum degas nickel-base powders by electron-beam bombardment.

There is a strong interest in HIP and isothermal forging. "Can" technology and know-how would appear to lag behind current North American practice. The Soviets use heavier and thicker cans than the Americans so that the practice of approaching near-net shape is precluded, at least for the time being. Recent Soviet literature reports on work on superplastic forming with reference to the mechanisms of plastic flow. Other consolidation methods that have received attention in the USSR are roll compaction and explosive compaction (hot explosive pressing).

JAPAN

Although there has been considerable growth in the Japanese P/M industry over the past 15 years, most of it pertained to nonmilitary industries. This trend now is changing as the benefits of nickel P/M technology become more apparent (Marley, 1979). This previous (apparent) lack of interest in nickel powders for aerospace applications may be attributed, in part, to a reluctance to initiate costly small-volume production runs. There now are clear indications that the Japanese are attempting to expand their capabilities in nickel P/M as they seek a larger share of the international market.

The leading proponents of nickel P/M in Japan are Kobe Steel, Ltd. and the Daido Steel Company, Ltd. Kobe is engaged in nickel P/M research

and development as well as limited production. Nickel powders are made by atomization techniques. The company is Japan's premier developer and producer of hot-isostatic-pressing units. In 1980, Kobe announced plans to introduce a new HIP facility capable of operating at 3630°F (2000°C) at pressures in excess of 32 ksi (221 MPa).

Similarly, Daido is conducting research on powder production and consolidation. Currently 90 percent of the company's P/M products are sold within Japan, but the company anticipates a significant growth in exports--principally to Europe and North America.

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Appendix B

HEAT FLOW DURING ATOMIZATION

During solidification of metal powders, heat is extracted from the droplets by both convection and radiation at their surface. However, there are no accurately established values for the combined radiative and convective heat-transfer coefficient, h, and direct measurement of the cooling rate or heat flux during solidification of an atomized droplet would be extremely difficult, if not impossible. In gas atomization, the convective heat-transfer coefficient is overriding and an upper limit of $\sim 10^5$ W/m^{2}K can be estimated from existing expressions for h under the most favorable conditions (Mehrabian 1978).

Indirect estimates of heat-transfer coefficients in various atomization processes also have been made by comparison of measured segregate (dendrite arm) spacings in crystalline alloy powders with predetermined relationships between these spacings and average cooling rates during solidification.

In general, the fineness of microstructure during dendrite solidification can be directly related to local solidification time, t, or average cooling rate during solidification, ε_{Avg}:

$$d = a\varepsilon_{Avg}^{-n} = bt_f^{n} \quad , \tag{B-1}$$

$$\varepsilon_{Avg} = \frac{\Delta T}{t_f} \quad , \tag{B-2}$$

where ΔT is solidification temperature range; d is dendrite-arm spacing; and a, b, and n are constants.

Data in Figures B-1 and B-2 show the relationships between average cooling rate and dendrite-arm spacing for a maraging steel and a cobalt-base superalloy. Table B-1 shows similar data for a number of alloy systems. Experimental difficulities have limited the range of accurately measured cooling rates to less than $\sim 10^3$K/sec.

It should be noted that caution must be exercised when these exponential correlations are to be extrapolated over many orders of magnitude, especially if the microstructures examined do not exhibit typical dendritic morphologies. Furthermore, the data in Table B-1 show that the constants in eq. B-1 vary from alloy system to alloy system and that even two similar alloys differing in solidification-temperature range, cooled under identical rates of external heat extraction (approximately equal local solidification times), would give considerably different cooling rates during solidification.

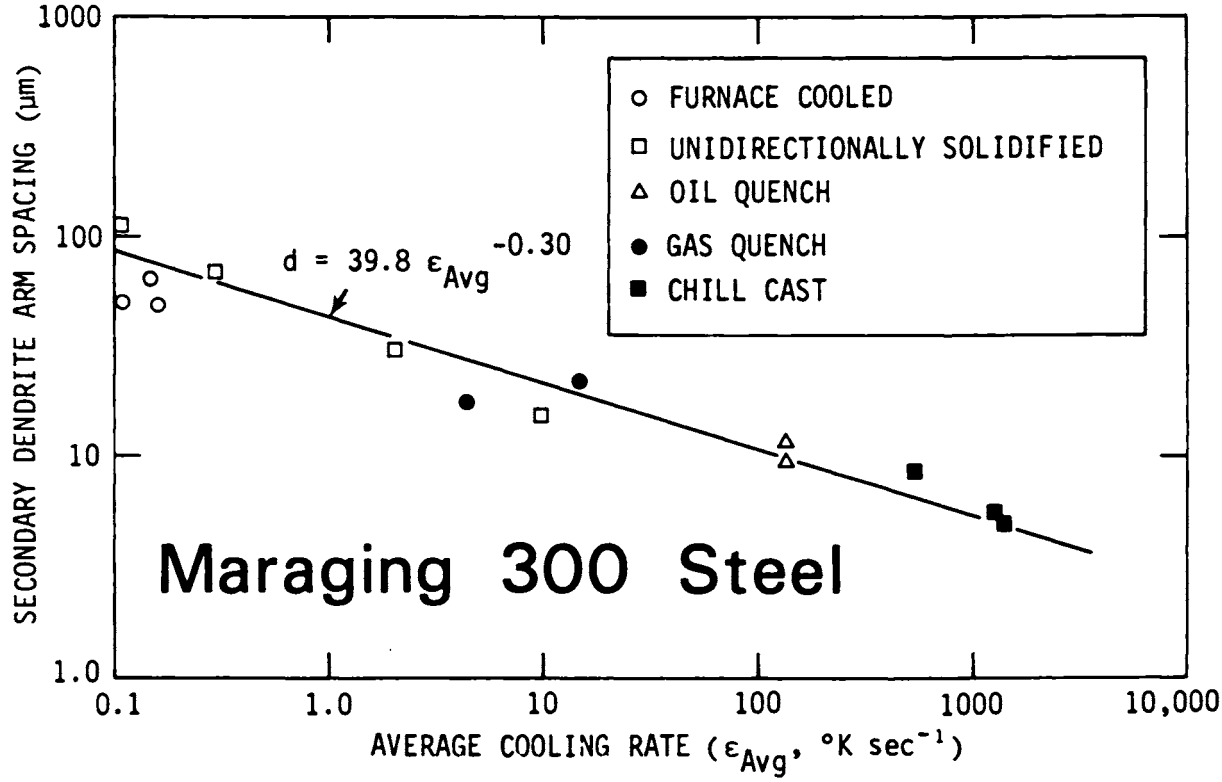

FIGURE B-1 Relationship of average cooling rate during solidification and dendrite-arm spacings in maraging 300 steel (Levi and Mehrabian 1980).

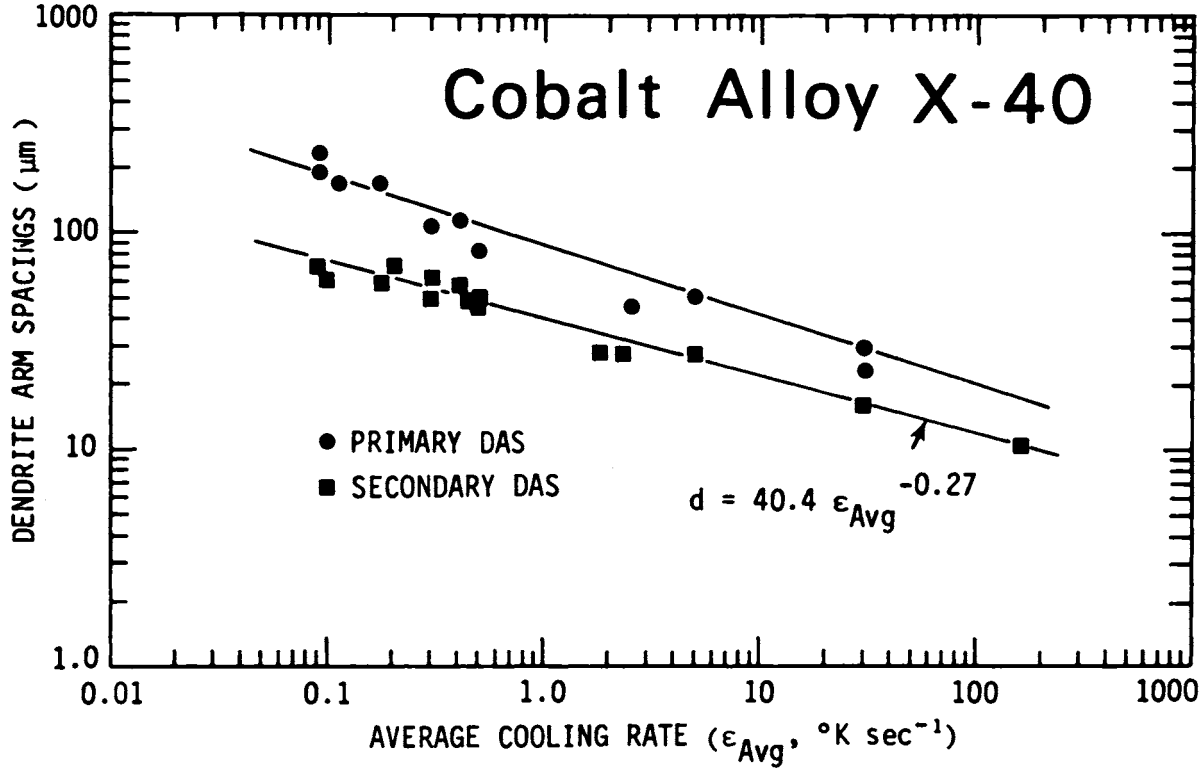

FIGURE B-2 Relationship of average cooling rate during solidification and dendrite-arm spacings in cobalt-base alloy X-40. (R. Mehrabian, National Bureau of Standards, Washington, D.C., unpublished data).

TABLE B-1 Coefficients and Exponents in Equation Relating Dendrite-Arm Spacing to Cooling Rate[a]

Alloy		a	n	Cooling Rate (K sec^{-1})
Inconel 718	primary	141	0.402	10^{-1} to 100
	secondary	33.85	0.338	
Waspaloy	primary	112	0.29	$5x10^{-2}$ to 20
	secondary	32.4	0.4	
Ni-27%Mo	primary	-	-	10^{-1} to 10
	secondary	43.6	0.55	
X-40	primary	90	0.32	10^{-1} to $2x10^{2}$
	secondary	40.4	0.27	
Maraging 300 Steel	primary	-	-	10^{-1} to 10^{3}
	secondary	39.8	0.30	
Al-4.5%Cu	primary	-	-	10^{-5} to 10^{3}
	secondary	40	0.39	

Source: Mehrabian 1978.

$a_d = a(\varepsilon_{Avg})^{-n}$ where d is dendrite arm spacing (μm) and ε_{Avg} is average cooling rate (^{o}K/sec.); a and n are constants.

Table 1 in chapter 3 shows average droplet size and dendrite-arm spacings for atomized droplets of three alloys, IN-100, Mar M-509 and maraging 300 steel, produced by different atomization processes. Heat-transfer coefficients for the various processes can be determined approximately if dendrite-arm spacings and average cooling rate during solidification for the various alloy systems are known. This is done by assuming Newton's law for interface controlled solidification:

$$\varepsilon_{Avg} = \frac{dT}{dt} = \frac{3h(T-T_o)}{\rho C_p R}, \qquad (B-3)$$

where h is the heat-transfer coefficient, R is the droplet radius, T is the droplet temperature, T_o is the temperature of environment, ρ is the alloy density and C_p is the average heat capacity of the alloy during solidification--it is determined by linearizing the heat of fusion over the solidification temperature range. Table B-2 shows the heat-transfer coefficients for the various processes using eq. B-3 and the data for maraging 300 steel. The Newtonian cooling appears justified, at least for the finer powders, from the very small Biot numbers; hR/k, listed in Table B-2.

In recently developed atomization processes such as Pratt and Whitney's centrifugal-atomization process, which employs a high-velocity helium gas as the quenching medium for the high-velocity powder particles, finer structures are observed at equivalent particle sizes. For example, note the measured dendrite-arm spacings for the IN-100 powder in Table 1. The use of higher-conductivity gas, such as helium or even hydrogen, and finer-powder particles may result in estimated heat-transfer coefficients of as high as 10^5 W/m^2K.

In general, then, a limitation on the achievable heat-transfer coefficient at a liquid-metal droplet-environment interface can be translated into a limitation on the important dimensionless variable--Biot number--governing the rate of heat extraction from the droplet. For example, a heat-transfer coefficient of $h < 10^5$ W/m^2K translates to a limitation on the range of Biot numbers of $10^{-3} < Bi < 1.0$ for atomized droplets of liquid nickel in the size range of 1 μm to 1000 μm.

For small heat-transfer coefficients, Biot numbers about 0.01, Newtonian cooling expressions generally are considered to be applicable. However, Levi and Mehrabian (1980) have shown that Biot numbers should be below 0.001 before temperature gradients in a liquid droplet become negligible. For $Bi > 0.001$ numerical heat-flow models are necessary. A short summary of achievable cooling rates and solidification times from one such numerical heat-flow model is presented below (Levi and Mehrabian 1980).

TABLE B-2 Calculation of Heat-Transfer Coefficients from Dendrite-Arm Spacings for Maraging 300 Steel

Atomization Process	Particle Size (μm)	DAS (μm)	ε_{Avg} (K/sec)	h (Calculated) c.g.s.	h (Calculated) S.I.	Biot No. $\left(\frac{hR}{k}\right)^a$
Argon atomized fine powder	75	∿2	$\sim 2.1 \times 10^4$	∿0.23	9.6×10^3	0.0084
Rotating electrode	170	∿3	$\sim 5.5 \times 10^3$	∿0.13	5.4×10^3	0.011
Steam atomized (coarse powder)	1000	∿6.5	$\sim 4.2 \times 10^2$	∿0.06	2.5×10^3	0.029
Vacuum atomized	650	∿6.5	$\sim 4.2 \times 10^2$	∿0.039	1.63×10^3	0.0123

Source: Mehrabian 1978
Data taken from Table B-1 ($d = 39.8 \cdot \varepsilon_{Avg}^{-0.03}$)
[a] R = radius of droplet, K = conductivity of liquid metal, and h = heat-transfer coefficient.

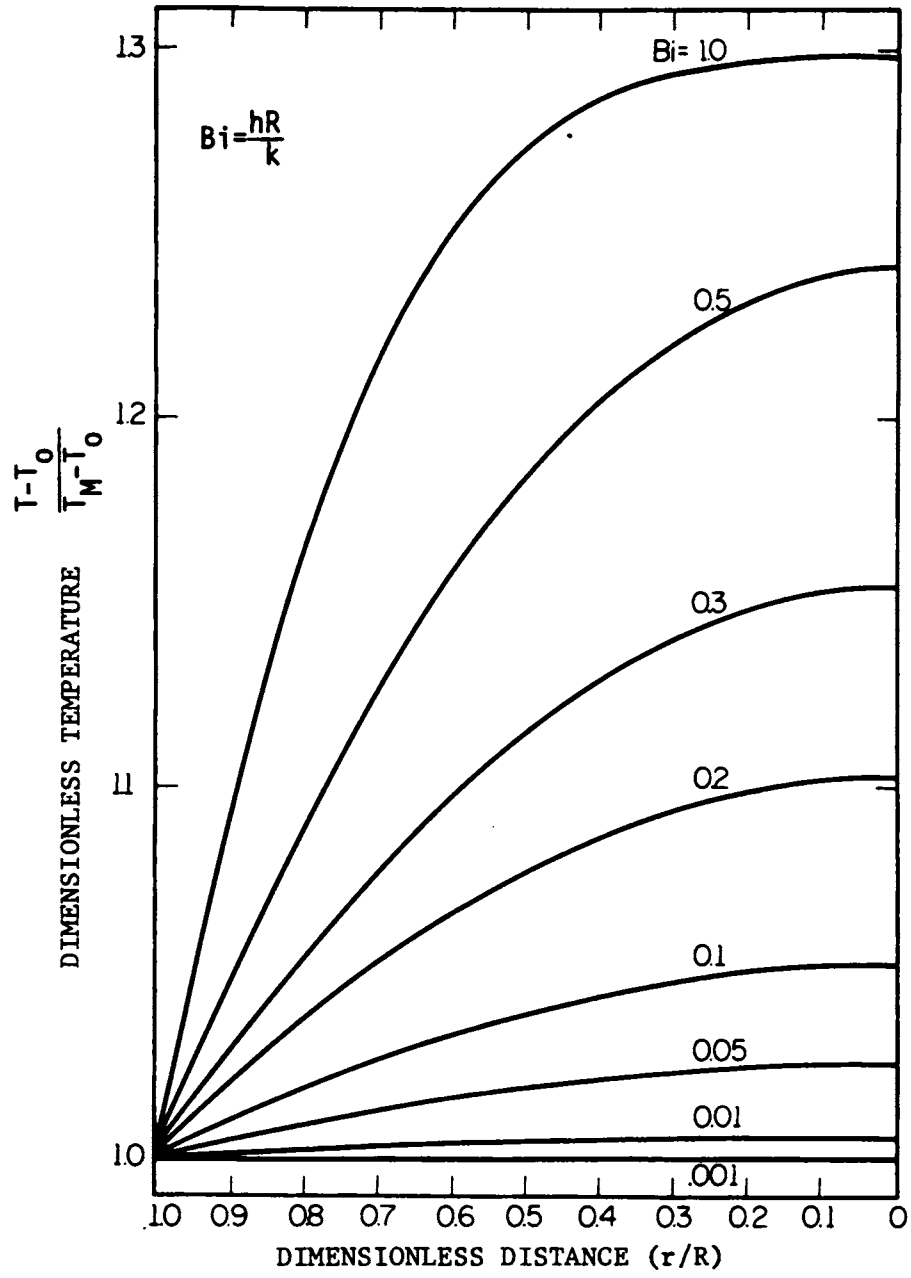

FIGURE B-3 Dimensionless temperature distributions in a liquid droplet when its surface reaches the melting temperature, T_M, for different Biot numbers and dimensionless initial superheat, $T-T_o/T_M-T_o = 1.3$; h is the heat-transfer coefficient at the droplet-environment interface, R is the droplet radius, and r is the location of the liquid-solid interface (Levi and Mehrabian 1980).

Figure B-3 shows calculated dimensionless temperature distribution in a liquid droplet for various Biot numbers and an initial superheat of $T - T_o/T_M - T_o = 1.3$ at the instant the droplet surface reaches its melting point. These data show that for Biot numbers less than $\sim$0.001 there is no significant temperature gradient in the droplet and the simple Newtonian cooling expressions can be used for crystalline and noncrystalline solidification. On the other hand, the results also indicate that even for small Biot numbers, in the range of $\sim$0.01, there may be significant temperature gradients in a metal droplet. For example, in a 20 μm diameter droplet of liquid iron where Bi $\sim$0.01 ($h - 4 \times 10^4$ W/m^2 K), the maximum temperature gradient at the droplet surface when the surface reaches the melting point is $\sim 1.5 \times 10^6$ K/m, and a temperature difference of 7.5 K between the surface and the center of the droplet is calculated. Thus, the Newtonian cooling assumption that temperature differences inside a body are negligible for Bi < 0.01 may not always be justified in atomization processes.

An important variable affecting undercooling prior to crystalline solidification is the cooling rate in the liquid droplet. On the other hand, as noted above, the fineness of a crystalline microstructure (e.g., segregate spacing, size of second-phase particles) usually can be correlated to average cooling rate during solidification or time available for coarsening. Thus, a clear distinction must be made between cooling rates in the liquid (or during noncrystalline solidification) and during crystalline solidification; the latter is significantly lower at equivalent rates of external heat extraction due to the heat of fusion.

Generalized expressions previously derived (Levi and Mehrabian, 1980) indicate that average cooling rate in a liquid-metal droplet is directly proportional to the heat-transfer coefficient at the droplet-environment interface and is inversely proportional to the radius of the droplet. Thus, considering an upper limit for achievable heat-transfer coefficients, the only other method for increasing cooling rate is to decrease particle size. For example, using $h - 5 \times 10^4$ W/m^2 K, average cooling rates of 1.4×10^5 K/s and 1.4×10^6 K/s are calculated for nickel droplets of 500 μm and 50 μm, respectively, when their surface reaches the melting point.

Calculations of cooling rates or times available for coarsening during solidification of metal droplets are much more difficult when Newtonian cooling conditions do not prevail. The calculations are further complicated due to the fact that, in general, engineering alloys solidify over a range of temperatures and that the liquid-"mushy" interface inside a solidifying metal powder usually does not have a spherical geometry. Computer calculations are useful, however, even with the simplifying assumptions of symmetrical solidification of pure metal spheres in that they permit estimation of the range of possible solidification times in atomization processes.

The solidification times for droplets of nickel as a function of Biot number and initial superheat previously have been calculated using a numerical solution of an enthalpy heat flow model (Levi and Mehrabian 1980). It was found that increasing initial superheat during atomization prolongs solidification, and the effect is larger as the Biot number

increases; the sensible heat retained in the liquid portion of the droplet increases resulting in longer times from initiation to completion of solidification. For example, 500 μm droplets of nickel solidifying with an $h = 5 \times 10^4$ W/m^2 K (Bi = 0.32) will require 4.2×10^{-3} seconds to complete solidification with no initial superheat and $\sim 5.0 \times 10^{-3}$ seconds with an initial superheat of $\sim$150 K. The effect of superheat on net solidification times diminishes with decreasing Biot numbers.

As anticipated, decreasing the powder particle size decreases the solidification time, the time available for coarsening of the microstructure. For example, decreasing the nickel powder size to 50 μm, while initial superheat is maintained at $\sim$150 K, results in a net solidification time of $\sim 3.2 \times 10^{-4}$ seconds, which is significantly less than the inverse proportionality predicted by a Newtonian cooling model.

In summary, it should be noted that cooling rates in the liquid and during solidification of metal droplets can be increased by increasing the heat-transfer coefficient at the powder-environment interface or decreasing the powder particle size. As shown above, there is a limitation on achievable heat-transfer coefficients in atomization processes, but the useful size range of superalloy powders cannot be reduced much below 10 μm to 25 μm without paying a heavy penalty in excessive surface contamination, handling, and flowability.

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Appendix C

DEPENDENCE OF FATIGUE LIFE ON DEFECT SIZE

The subsurface crack initiation sites observed in failed LCF bars and earlier S.E.M. identification of these sites as pores and nonmetallic inclusions permit one to assume that a distribution of defect sizes exists in the material. Considering the volume of material contained within the gage section of a fatigue specimen, there is a maximum diameter of inclusions that statistically will be found in any one specimen. If it also is assumed that the largest-diameter inclusion fractures or decoheres from the matrix during the very early stages of the fatigue test, the starting flaw size can be equated with the inclusion size. If the concept of threshold cyclic-stress intensity for crack propagation is used, a relationship between initial-flaw size and critical-stress amplitude for crack propagation can be obtained. To do this, the stress-intensity solution for a circular, embedded, penny-shaped crack can be used:

$$K_I = \frac{2\sigma}{\pi}\sqrt{\pi a} ,$$

where σ is the applied stress, a is the half-crack diameter, and K_I equals stress-intensity factor in mode-one loading (i.e., so that the crack is opened in plane strain). This can be modified to:

$$\Delta\sigma_{crit} = \frac{\pi\Delta K_{Ith}}{2\sqrt{\pi\frac{d}{2}_{max}}} .$$

ΔK_{Ith} equals the threshold for crack growth (i.e., for $\Delta K<\Delta K_{th}$), and the crack will not propagate under the operative conditions. Thus, there is a critical cyclic-stress amplitude below which the inclusion has no effect on fatigue life. Moreover, different microstructures have different ΔK_{th} values and, thus, the d_{max}-$\Delta\sigma_{crit}$ relationship may be microstructure dependent. This would explain the suggested (but unproven) convergence of S-N curves for powder and conventional material at low stresses (high cycles to failure). This analysis also points out the possible danger of using high-cycle-fatigue (HCF) tests to screen the fatigue performance of powder products or any material prone to early crack initiation due to the presence of inclusions or microstructural defects.

In addition to the above analysis, which is based on linear elastic fracture mechanics, there is the further complication of the behavior of short cracks and so-called nonpropagating cracks. In such cases, other factors like net-section stress or crack-opening displacement may determine whether a defect becomes a propagating crack. Thus, defect location becomes important since a near-surface defect will have a small ligament on one side and , therefore, a larger displacement field on one side. This can convert a small pore into a propagating crack, especially in γ' strengthened materials where strain localization is prevalent.