Net-Shape HIP Powder Metallurgy Components for Rocket Engines

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Abstract True net shape consolidation of powder metal (PM) by hot isostatic pressing (HIP) provides opportunities for many cost, performance and life benefits over conventional fabrication processes for large rocket engine structures. Various forms of selectively net-shape PM have been around for thirty years or so. However, it is only recently that major applications have been pursued for rocket engine hardware fabricated in the United States. The method employs sacrificial metallic tooling (HIP capsule and shaped inserts), which is removed from the part after HIP consolidation of the powder, by selective acid dissolution. Full exploitation of net-shape PM requires innovative approaches in both component design and materials and processing details. The benefits include: uniform and homogeneous microstructure with no porosity, irrespective of component shape and size; elimination of welds and the associated quality and life limitations; removal of traditional producibility constraints on design freedom, such as forgeability and machinability, and scale-up to very large, monolithic parts, limited only by the size of existing HIP furnaces. Net-shape PM HIP also enables fabrication of complex configurations providing additional, unique functionalities. The progress made in these areas will be described. Then critical aspects of the technology that still require significant further development and maturation will be discussed from the perspective of an engine systems builder and end-user of the technology.

1   INTRODUCTION

Net-shape powder metallurgy (P/M) by hot isostatic pressing (HIP) has been vigorously pursued by both the aircraft engine and rocket engine industries for more than 40 years. The method effectively "casts" complex shapes with solid metal powder rather than with liquid metal (Figure 1). This eliminates expensive machining and provides maximum performance, equal or better than a wrought version of the alloy. The P/M HIP method also avoids welds, which typically introduce added costs of inspection and repair and limit fatigue life. The method may also be exploited to allow extensive use of very high strength alloys, which often have poor producibility, by conventional processes and environmental compatibility limitations. Net-shape P/M technology is attractive for liquid fueled rocket engines, where production rates are low and where performance, life and reliability requirements are very high. This situation is especially evident in reusable, man-rated, launch and space transportation systems.

Since the early 1990's, when the quality of powders was substantially improved, Boeing-Rocketdyne has been pursuing these selectively net-shape P/M capabilities, in partnership with first VILS and more recently with LNT. Boeing-Rocketdyne initially focused attention on P/M HIP for rotating components in high performance turbopumps. Cost and schedule savings are possible, especially where turbine rotors and pump impellers are designed with integral shrouds that severely restrict access for machining of the propellant flow paths. Several innovations have been demonstrated which exploit the increased design freedom enabled by application of the P/M HIP and tool dissolution method. This includes integral damping features and bimetallic structures for enhanced

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environmental compatibility, both of which improve life and reliability while allowing increased performance (typically higher tip speeds).
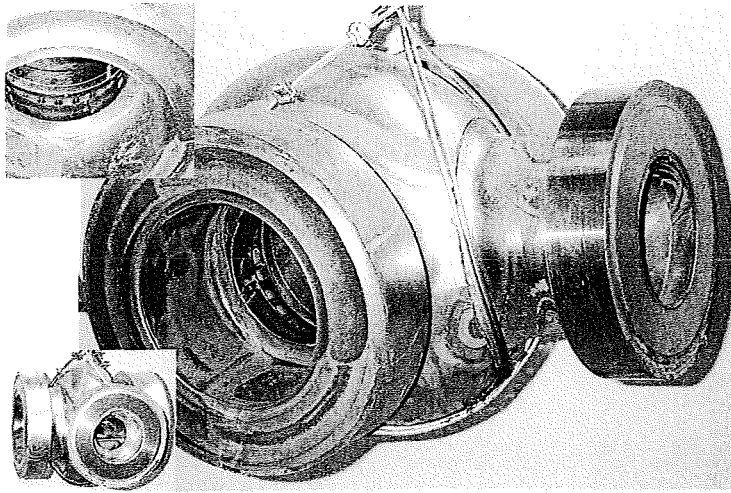
Figure 1. Net-Shape PM-HIP Rocket Engine Turbopump Housing in Final Stages of Leaching Out Tooling

As familiarity with the technology increased, a much broader range of applications was considered. A major step forward was achieved by application of the technology to very large, complex, non-axisymetric, static components, such as the monolithic turbine housing, nozzle, manifold and flanges shown in figure 1. Figure 2 shows the numerous candidates for major structures which, in addition to the turbine housing, were identified as baseline options for a large, developmental booster engine.
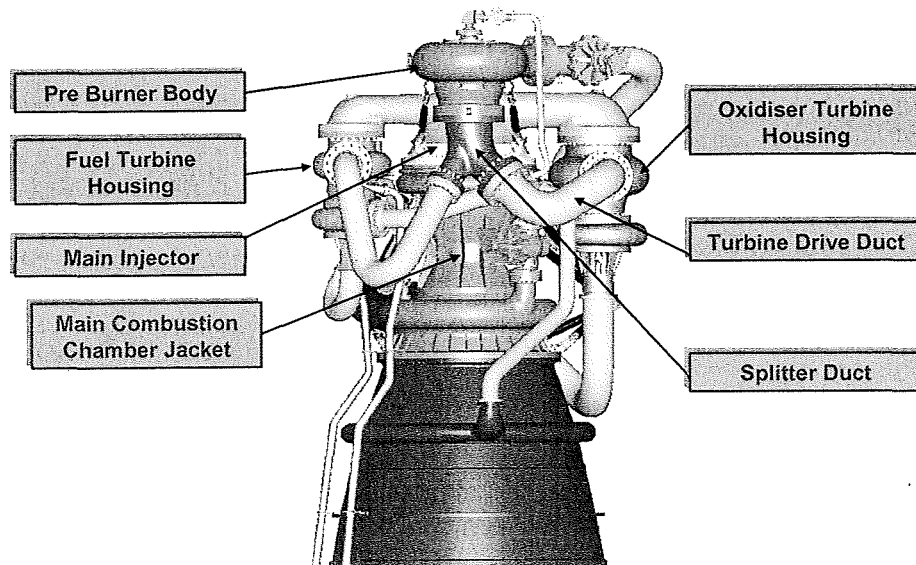
Figure 2. Candidate selectively net-shape P/M components on a large booster engine.

Figure 3 shows a processing flow chart which attempts to identify all aspects of the design, analysis, manufacturing and inspection for flight hardware. Not included is the currently mandated hot-fire testing of the hardware, prior to every launch of an engine. The black boxes represent the major steps in the overall process. The red boxes represent special issues, or procedures, that enable these steps to be consistently achieved. In several of the red boxes, significant issues are still in development.
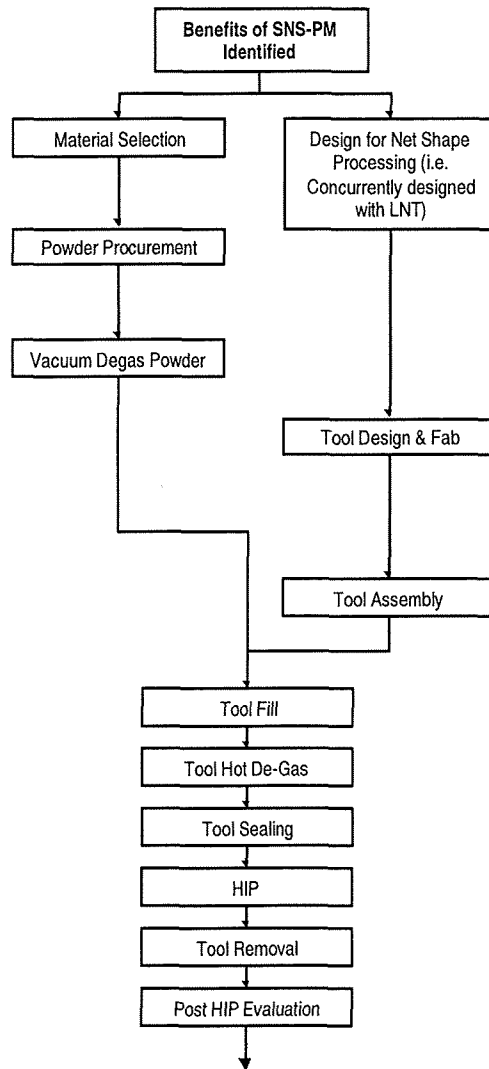
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                    ┌─────────────────┐
                    │ Benefits of SNS-PM│
                    │   Identified    │
                    └────────┬────────┘
              ┌──────────────┴──────────────┐
    ┌─────────────────┐         ┌─────────────────────┐
    │ Material Selection│         │ Design for Net Shape │
    └────────┬────────┘         │   Processing (i.e.   │
             │                  │ Concurrently designed│
    ┌─────────────────┐         │      with LNT)      │
    │Powder Procurement│         └──────────┬──────────┘
    └────────┬────────┘                     │
             │                              │
    ┌─────────────────┐         ┌─────────────────────┐
    │Vacuum Degas Powder│        │  Tool Design & Fab  │
    └────────┬────────┘         └──────────┬──────────┘
             │                              │
             │                  ┌─────────────────────┐
             │                  │   Tool Assembly     │
             │                  └──────────┬──────────┘
             └──────────────┬──────────────┘
                    ┌─────────────────┐
                    │    Tool Fill    │
                    └────────┬────────┘
                    ┌─────────────────┐
                    │ Tool Hot De-Gas │
                    └────────┬────────┘
                    ┌─────────────────┐
                    │  Tool Sealing   │
                    └────────┬────────┘
                    ┌─────────────────┐
                    │      HIP        │
                    └────────┬────────┘
                    ┌─────────────────┐
                    │  Tool Removal   │
                    └────────┬────────┘
                    ┌─────────────────┐
                    │Post HIP Evaluation│
                    └────────┬────────┘
                             ▼
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Figure 3. Process flow chart for implementation of the selectively net-shape P/M technology in rocket engines.

While there are many critical aspects to implementation of the technology, such as: part design concepts; tool design and fabrication and computational modeling, this article is focused on identification and discussion of materials and processing issues typically encountered during application to large, complex rocket engine structures. Some discussion of future developments is also included.

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2   MATERIALS AND PROCESSING CONCERNS

The issues discussed here are generally those that affect microstructures and mechanical behaviors.

2.1   Prior Particle Boundaries

Figure 4 shows the utility of scanning electron microscopy (SEM) to reveal important microstructural information in HIP-consolidated alloy powders. The two modes of imaging with SEM: secondary electron imaging (SEI) and back-scatter electron imaging (BEI) of highly polished (un-etched) surfaces, provide complementary information. In figure 4(a), SEI very clearly highlights the prior particle boundaries (PPB's), primarily by electron charging of non-conducting oxides. The BEI image of the same field-of-view, shown in figure 4(b), does not show PPB oxides so clearly. The black PPB particles in figure 4(b) are not visually distinguishable from metal carbides. The BEI image in figure 4(b) does, however, show much clearer crystallographic contrast; primarily matrix grain structure and second phase particles (Laves phase in the case shown). All of this information is critical for optimizing the microstructure for the desired balance of properties. Comparison of figures 4(a) and 4(c) indicates a much higher degree of powder surface oxidation, prior to HIP consolidation, in the 4(a) sample than in the 4(c) sample.
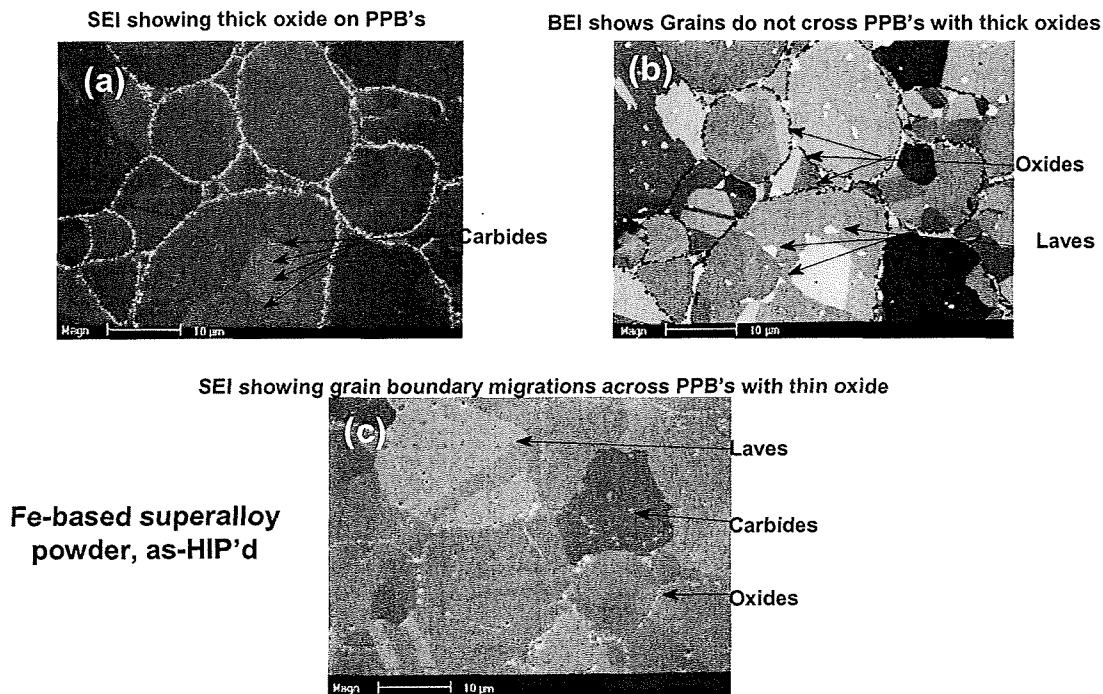
Figure 4. Scanning Electron Microscopy of HIP'd superalloy samples.

This can have serious consequences since grain boundaries may not be able to migrate through a heavily oxidized PPB. Such a situation may be prone to embrittlement by easier fracture along the common grain and prior particle boundaries. Some alloys tend to from PPB's decorated with metal carbide particles. The detailed mechanism for this preferred carbide growth on powder particle surfaces is not known. Some alloys are more tolerant of these PPB carbides (regarding ductility and toughness) than others.

Presumably dependent on the types, shapes and sizes of the carbides. The powerful capability of the SEM-BEI microscopy is illustrated in the very high magnification image in figure 5. This micrograph shows clearly γ', in the γ grain interiors and Al_2O_3, metal carbides and microporosity in the grain/prior particle boundaries.
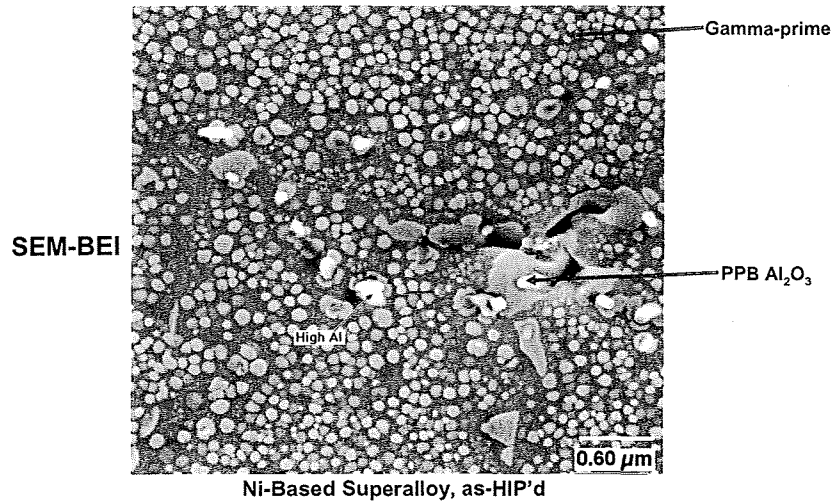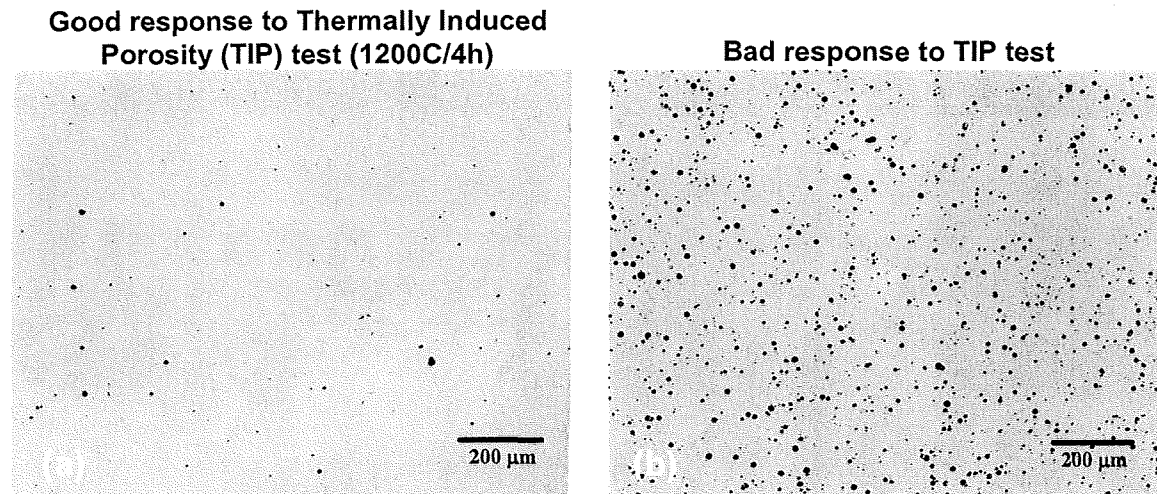

Figure 5. *High magnification BEI image showing γ', in the grain interiors and Al_2O_3, metal carbides and microporosity in the grain/prior particle boundaries.*

2.2   Porosity

Figure 6 shows the tendency to develop porosity, especially during high temperature exposure in a low pressure furnace.

Figure 6. *Thermally Induced Porosity tests on two HIP'd Ni-based superalloys samples.*

The most common cause is a small leak in the capsule which is slow enough to allow HIP consolidation but entraps argon gas from the HIP vessel in the PPB triple points.
This inert gas, highly compressed by the HIP, may be barely detectable as HIP'd. However, a standardized Thermally Induced Porosity (TIP) test will expand the small

pores to easily observed larger pores, as seen in the example in figure 6(b). This is an important quality control technique.

2.3   Surface Contamination

The most commonly used tooling material is a standard, low carbon (mild) steel. While this has obvious cost, availability and machineability advantages, interdiffusion with the consolidating powder may be excessive. Figure 7 shows the effect of carbon diffusion from the tooling in to the consolidated Ni-based superalloy (mild steels typically have more than an order of magnitude higher carbon content than superalloys). Metal carbides are formed most heavily on the PPB's with carbide particles also precipitated inside the prior particles.

Ni-diffusion layer in steel tooling from Ni-based superalloy

Low carbon steel (tooling)

Ni

C

Steel Tooling

Carbon contamination (carbides) from steel tooling

Ni-based superalloy, HIP'd powder

125μm

60μm

Uncontaminated Ni-based superalloy, as-HIP'd

Optical metallography - etched
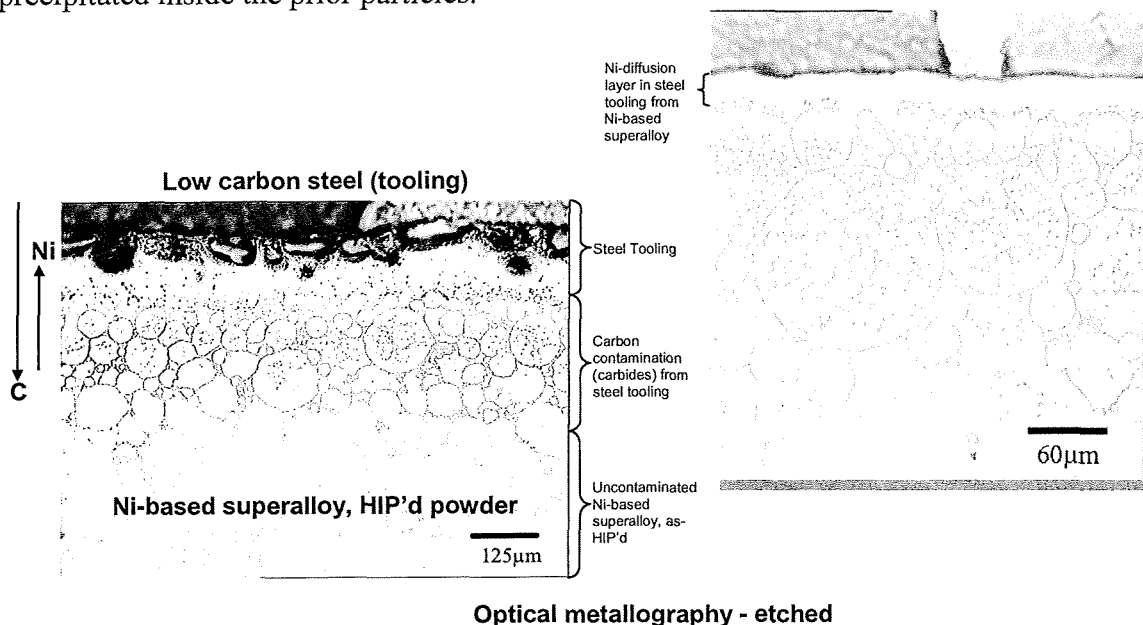
Figure 7. Interdiffusion effects between mild steel tooling and Ni-based superallloy powder during HIP consolidation.

The example shown in figure 7 is a typical case, showing a carbon diffusion depth of about 250 μm. Such carbide precipitation may seriously reduce fatigue life of the component. Simple methods of alleviating this problem include: removal of the contamination layer by chemical milling; or by using special iron alloys with similar carbon contents to the powder alloys. Other, more complex, methods have been demonstrated using coatings or interlayers.

2.4   Surface Roughness

Figure 8 shows an example of powder particles which have indented the tooling surface during HIP consolidation. This example shows the indenting effect so clearly because the powder particle size was relatively large, (~ 250 μm diameter) compared with superalloy powders (typically ~50 μm diameter). The titanium alloy powder particles show negligible deformation at the tooling interface, as a result of its higher strength. This degree of indentation is undesirable since, on selective removal of the tooling, the titanium part surface has large dimples with sharp crevices between the

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dimples. These crevices may initiate early fatigue. Also, the increase in interface area and the disruption of surface oxide on the tooling both lead to enhanced interdiffusion during the HIP cycle. One solution to this problem is to use a tooling material with higher hardness. The hard alloy must still be amenable to selective dissolution, which limits the choice of alloys. A more sophisticated solution has been demonstrated with the HIP bonded surface layer method (discussed in section 3.3 below).
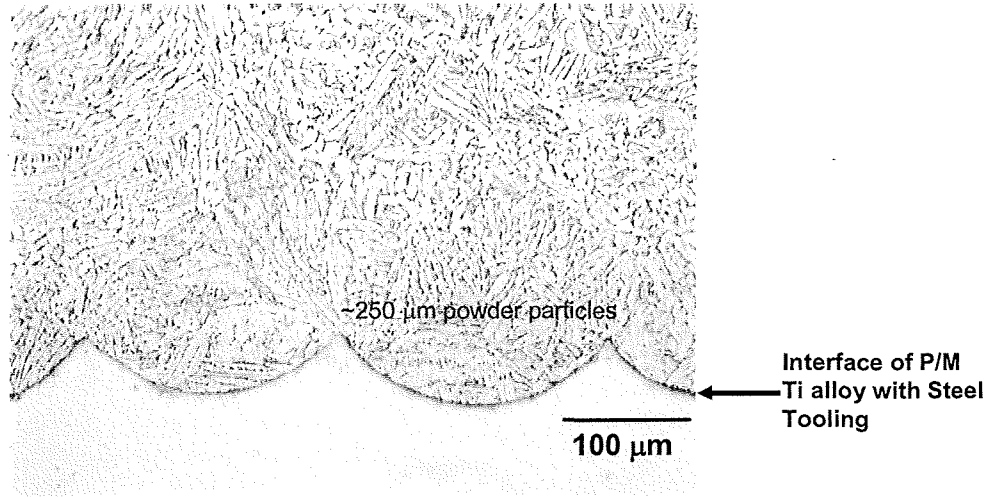
Figure 8. Titanium alloy powder particle indentation of steel tooling during HIP consolidation.

2.5   Selective Acid Dissolution

Figure 9(a) shows remnant nodules of tooling material (mild steel) that quite commonly become passivated during the last stages of selective acid dissolution. Increasing the aggressiveness of the acid to remove the remnant nodules may cause significant etching of the P/M part surface.

(a)   (b)
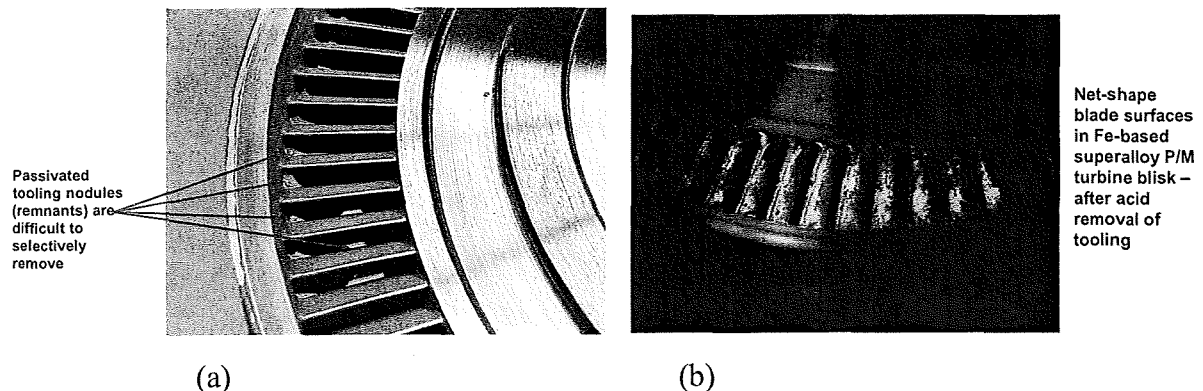
Figure 9. (a) Passivated tooling remnants resisting dissolution on the net-shape blades of a P/M superalloy turbine blisk. (b) Fluorescent (UV) penetrant inspection response on net-shape turbine blades indicating grain boundary attack during tooling selective dissolution.

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Figure 9(b) shows this result on the same part as shown in figure 9(a). The net-surfaces on the rotor blades have suffered grain boundary attack (etching) which shows as the blue areas in figure 9(b) in the ultra violet fluorescent visual inspection method. This degree of grain boundary etching may reduce fatigue life. The selective dissolution process thus requires careful development for each P/M alloy and tooling combination and strict process controls.

2.6   Non-Destructive Inspection

Powder synthesis and handling inevitably result in a population of non-metallic foreign particles in the alloy powders. These are most commonly ceramic particles, such as Al_2O_3. Strict process controls can ensure that non-metallic particles are sized no larger than the largest of the alloy powders: < 100 μm for typical superalloys and < 300 μm for typical titanium alloys. The Heavy Liquid Separation (HLS) method has confirmed this assumption (largest ceramic particle measured in a sample batch of Ni-based superalloy = 50 x 100 μm). The HLS technique has also shown that typically the population of these largest non-metallic particles are less than 1 ceramic particle per 6 million metal particles. Since commercially available ultrasonic inspection systems are limited to detecting features > 1500 μm, there is no non-destructive inspection method available to quantitatively analyze the non-metallic inclusion population in P/M HIP'd parts. Conventional C-scan methods are also severely limited by the complex net-shapes typical with P/M HIP'd parts, although the highly uniform and relatively small grain sizes enhance the signal:noise ratio of convention ultrasonic inspection compared with the case of ingot/wrought versions of the same alloys.

3   FUTURE DEVELOPMENTS

3.1 Probabilistic Life Prediction.

With the current limitations of non-destructive inspection techniques (discussed above) application of the traditional deterministic life prediction analytical methods becomes excessively conservative. While the net-shape aspect of the P/M HIP technology presents a non-destructive inspection challenge, the inherent consistency and homogeneity of the starting raw material (powder) and of the resulting HIP-consolidated microstructure, offer a highly attractive solution: probabilistic life prediction (PLP). PLP will allow designs that are much more efficient (lower weight) than with the current deterministic method of life prediction while providing the desired statistically safe life. Progress toward routine implementation of PLP requires the following development steps:
- Characterize powder & component defect populations
- Characterize fatigue crack initiation
- Characterize short crack growth rates
- Develop PLP models
- Validate on coupons, including "seeded" samples and apply to P/M HIP components.

3.2   Integral Damping

Figure 10 shows one of a number of concepts for highly robust and effective damping of resonant frequencies in a selectively net-shape P/M turbine blisk (ref. 1, 2).
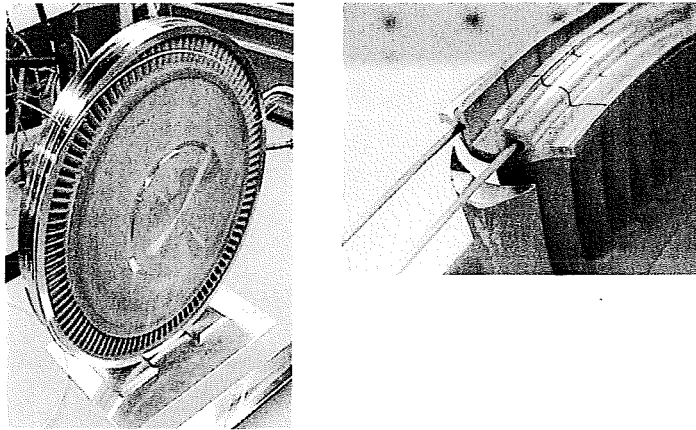
Figure 10. A novel PM HIP processing concept enables fabrication of an integral damping feature in the shroud of a turbine blisk.

Here the P/M HIP technology enables embedding of loose wires in the blisk shroud which provides highly effective friction damping of most resonant frequency vibration modes in the blades and disk.

3.3   HIP-Bonded (Bi-Metallic) Surface Layers

The state-of-the-art method for manufacturing rocket engine turbine disks is to fabricate from either conventional, high-strength superalloys and coat for environmental protection or fabricate from moderate-strength alloys, which are fully compatible with the hostile environment. Coatings introduce reliability and cost issues, while the moderate-strength alloys limit turbine performance. Boeing-Rocketdyne, supported by the U.S. Air Force Research Laboratory, fabricated a high, specific-strength blisk using a novel selective net shape P/M HIP process to provide an environmentally compatible HIP-bonded surface layer (HBSL) (ref. 3). Figure 11 shows tooling segments and a net-shape P/M turbine blisk processed with a HBSL for compatibility (burn-resistance) with high pressure oxygen gas. As discussed earlier, both surface contamination and surface micro-roughness are inherent on parts fabricated with the net-shape P/M HIP method. These defects are related to powder indention and diffusion bonding with the soft sacrificial tooling during HIP. The addition of an HBSL not only solves the micro-roughness problem of the net-shape P/M HIP surfaces but also offers a highly robust approach to protect very-high-performance alloys in turbine environments normally hostile to these alloys. The environment in which a turbine disk must operate is often hydrogen-rich or oxygen-rich gas. Hydrogen-rich gas embrittles and oxygen-rich gas burns high-strength superalloys and titanium alloys. In the HBSL process, a layer of an alloy, selected for its environmental resistance and compatibility with the core alloy, is deposited on the machined surface of the steel tooling. After tool assembly, powder filling, degassing, sealing, and HIP, the core superalloy powder consolidates and HIP bonds to the outer surface of the environmental alloy on the sacrificial tooling. This durable and damage

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tolerant HBSL provides environmental compatibility and improves the as-processed surface finish by replication of the tooling machined surfaces.
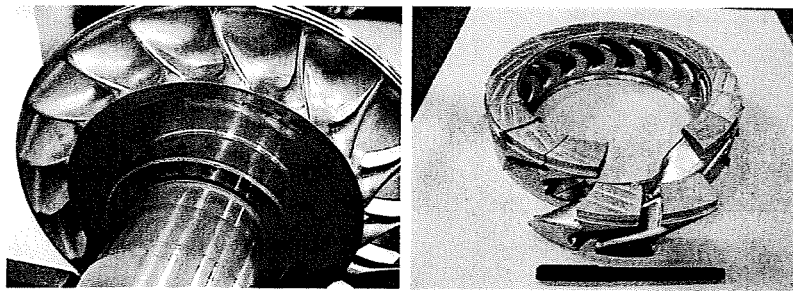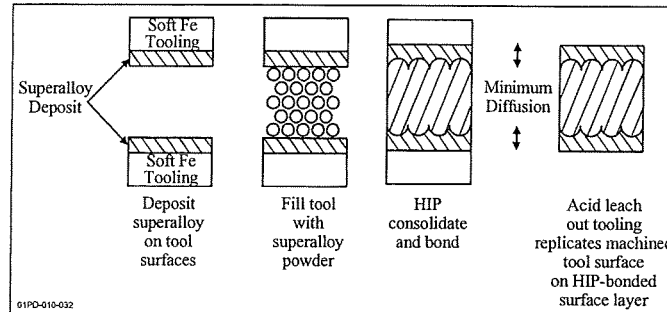

Figure 11. Hip-Bonded Surface layers for Environmental Compatibility.

4   SUMMARY

Processing methods and variables are so numerous that the part fabricator must become involved with the part designer and the systems designer in an effective integrated product team. The technology is ready for major structural applications, although qualification of flight hardware will be a laborious point-design approach for some time to come. Many technology improvements are possible and in some cases required, to enable full exploitation.

5   REFERENCES:

1 T.J.VanDaam, T.J.Hosking, United States Patent No. 6,482,533; 19Nov 2002
2 T.J.VanDaam, T.J.Hosking, United States Patent No. 6,547,526; 15Apr 2003
3 C.C.Bampton, V.Samarov, U.S. Pat. Pending, App No.20040081572

6   ACKNOWLEDGEMENTS:

Net-Shape, HIP Powder Metallurgy Components for Rocket Engines

**Cliff Bampton, Wes Goodin, Tom Van Daam,
Gordon Creeger, Steve James**

Rocketdyne Propulsion & Power, The Boeing Company

**International Conference on Hot Isostatic Pressing
(HIP'05)**

Paris, France

May 22-25, 2005

Rocketdyne

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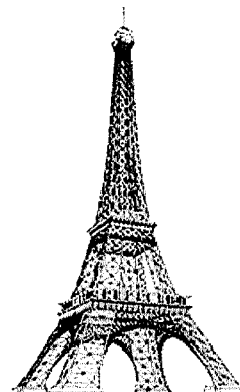
Acknowledgments

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- US Air Force Research Laboratory, Integrated High Performance Rocket Propulsion Technology (technical point of contact, John Barnes)

- Boeing Internal Application Development

Technical support was supplied by:

- LNT/Synertech; Crucible; Special Metals; Carpenter; Rockwell Scientific Company

- Boeing-Rocketdyne, RS83 and RS84 Programs, Integrated Product Teams

Rocketdyne

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Agenda

- **Focus on:**
  processing issues for large, complex, rocket engine structures

- **Minimal attention to other important issues:**
  part design concepts;
  tool design & fabrication;
  process deformation models
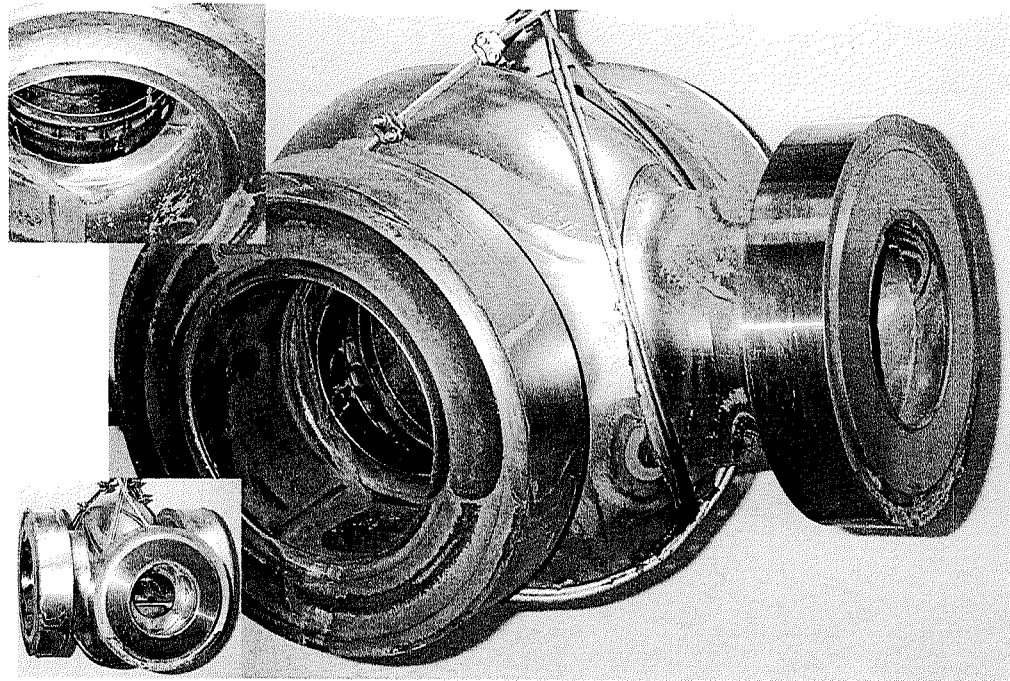
Selectively Net-Shape Powder Metal Processing
Provides Cost, Performance & Life Benefits

Fuel Turbine
Housing
Design

Tooling Assembly
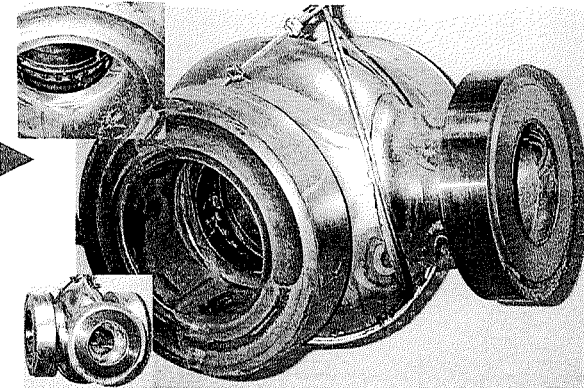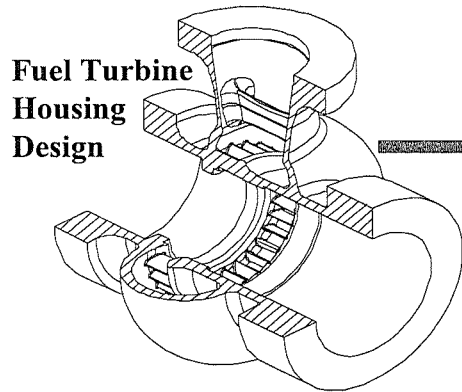
Ni-based
superalloy,
single-piece
housing
& nozzle
after selective
acid
dissolution of
tooling
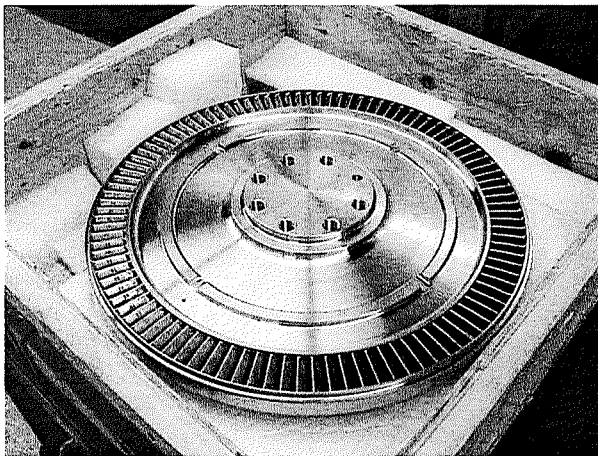
Single piece shrouded turbine blisk with net-shape blades – Fe-based superalloy
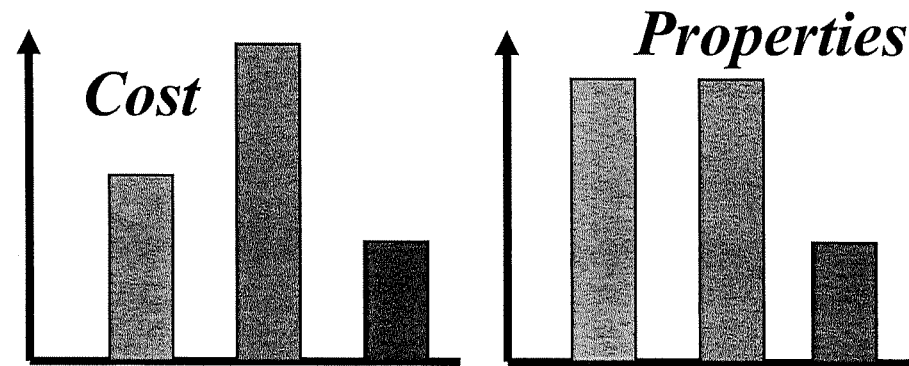
Cost

Properties

☐ Net-Shape PM
☐ Machined & Welded Forgings
■ Investment castings

- *Avoids welding, casting, forging and complex machining*
- *Provides uniform microstructures in very large components*

Net Shape P/M Improves Reliability & Cost

- Properties typically equivalent to wrought
  - Proven in many alloy systems

- Shape complexity equivalent to investment castings

- Allows for lighter weight structures

- Eliminates / minimizes weldments
  - Can produce net shape structures

- Eliminates scale-up issues
  - Provides uniform fine grain structure independent of hardware geometry

- Cost effective for low volume aerospace production requirements
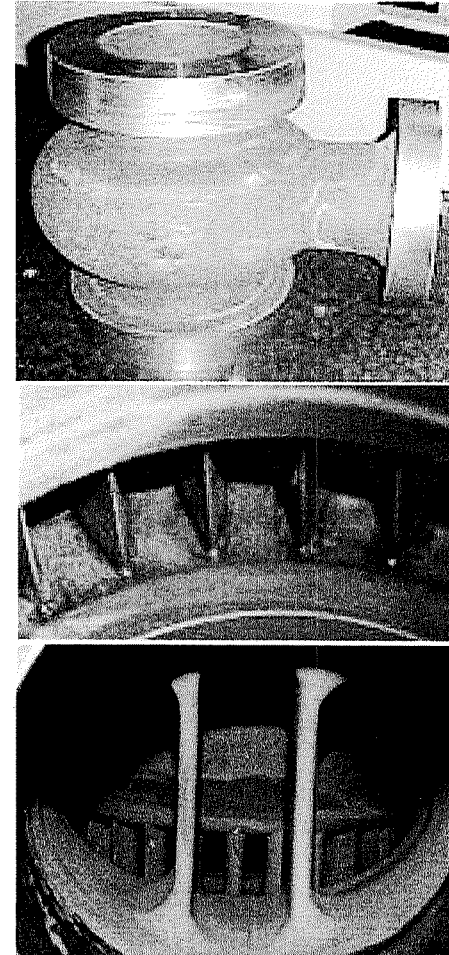
Fuel Manifold

Candidate Net-Shape P/M Components
Hydrocarbon/LOx Booster Engine

Pre Burner Body

Fuel Turbine Housing

Main Injector

Main Combustion Chamber Jacket

Oxidiser Turbine Housing

Turbine Drive Duct

Splitter Duct

Rocketdyne

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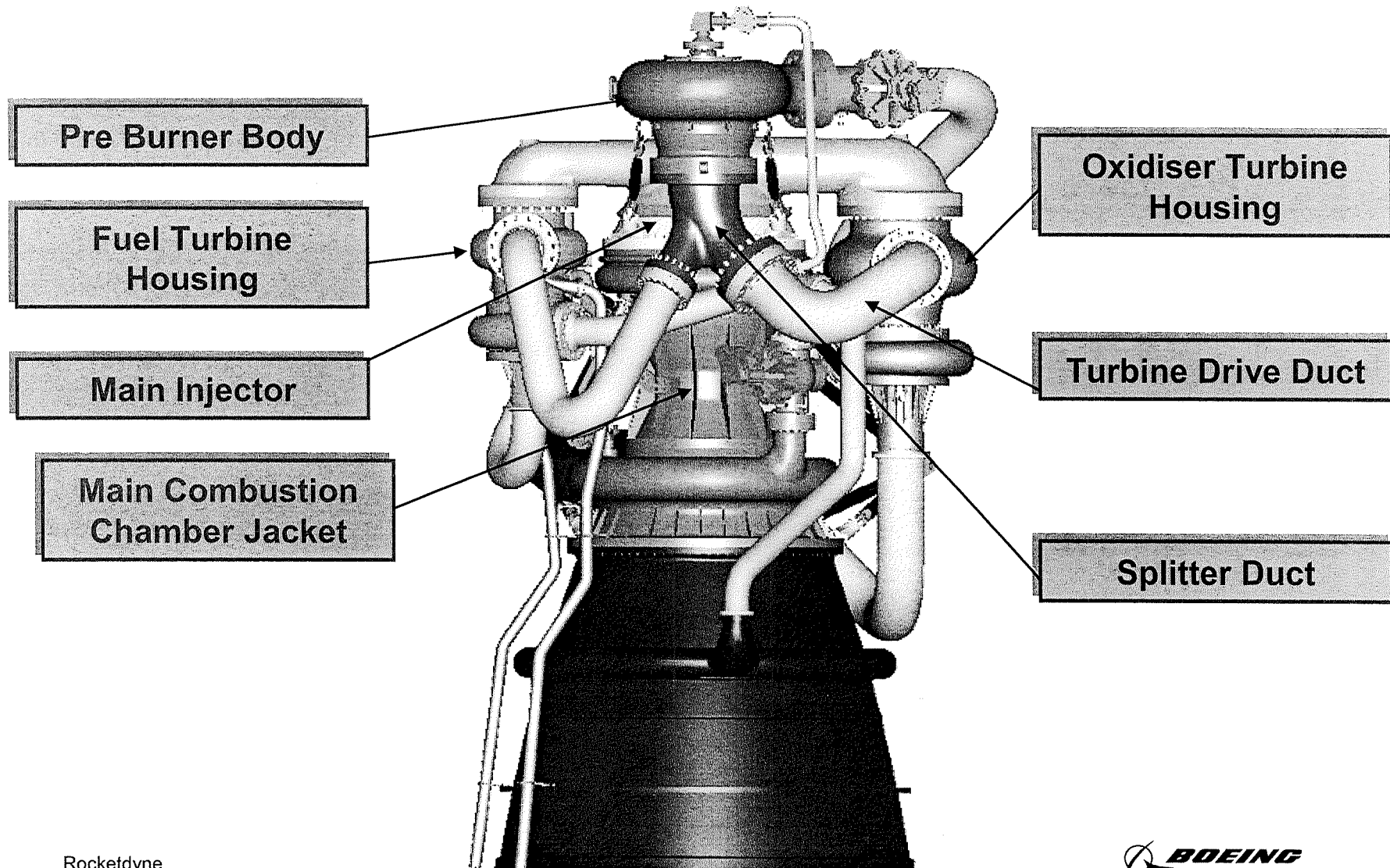
Net Shape P/M Manufacturing
Process Flow

Benefits of SNS-PM Identified

Material Selection → Powder Procurement → Vacuum Degas Powder

Design for Net Shape Processing (i.e. Concurrently designed with LNT) → Tool Design & Fab → Tool Assembly

Tool Fill → Tool Hot De-Gas → Tool Sealing → HIP → Tool Removal → Post HIP Evaluation
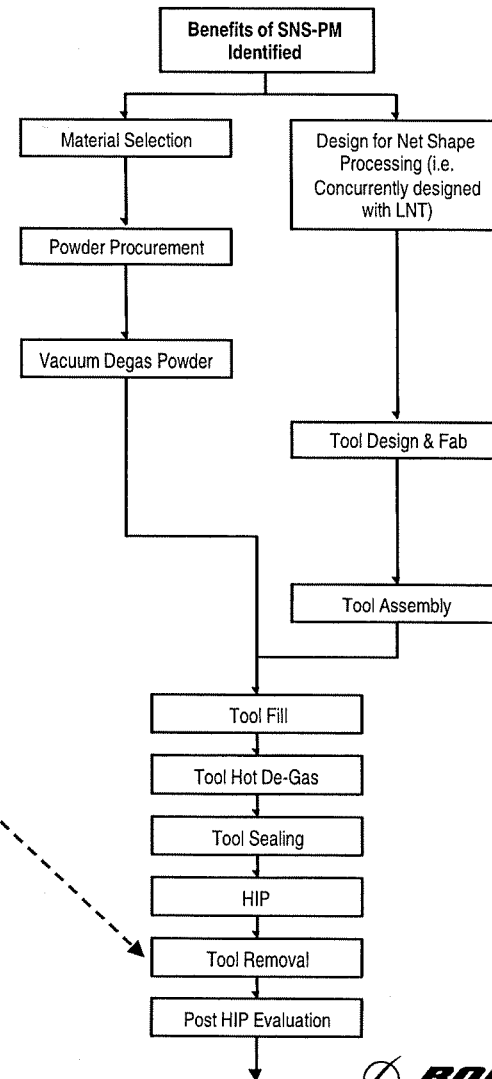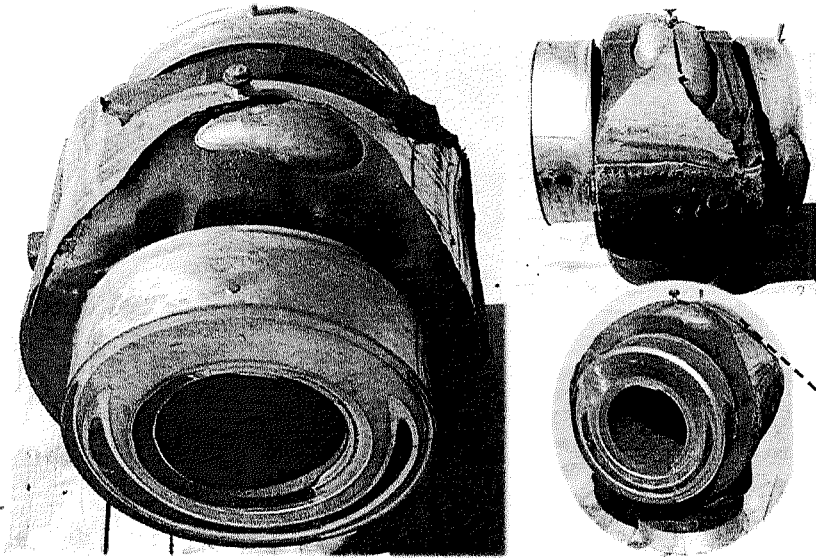
HIP-Consolidated Metallography

Different modes of Scanning Electron Microscopy (SEM) reveal different microstructural features:
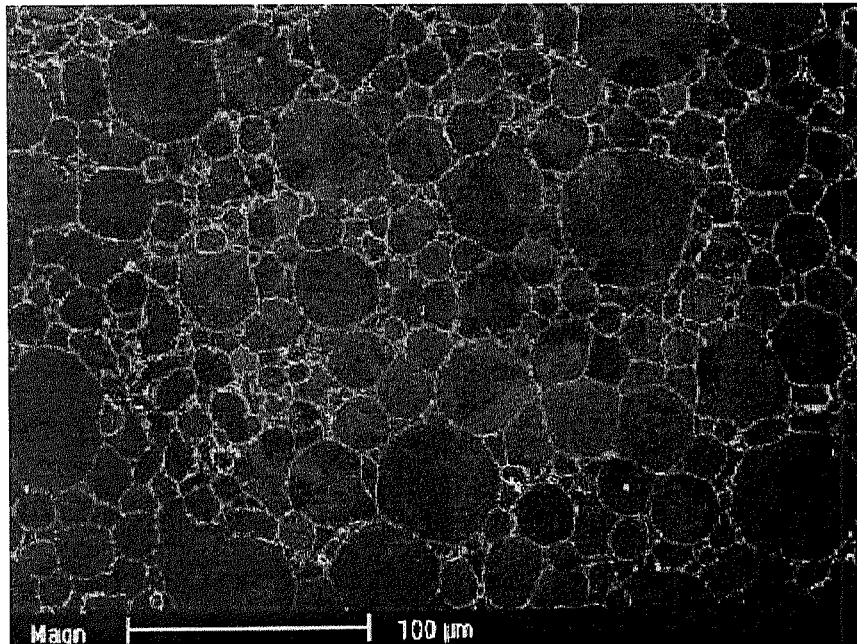
Secondary Electron Imaging (SEI) shows Prior Particle Boundaries (PPB's) decorated by oxide particles
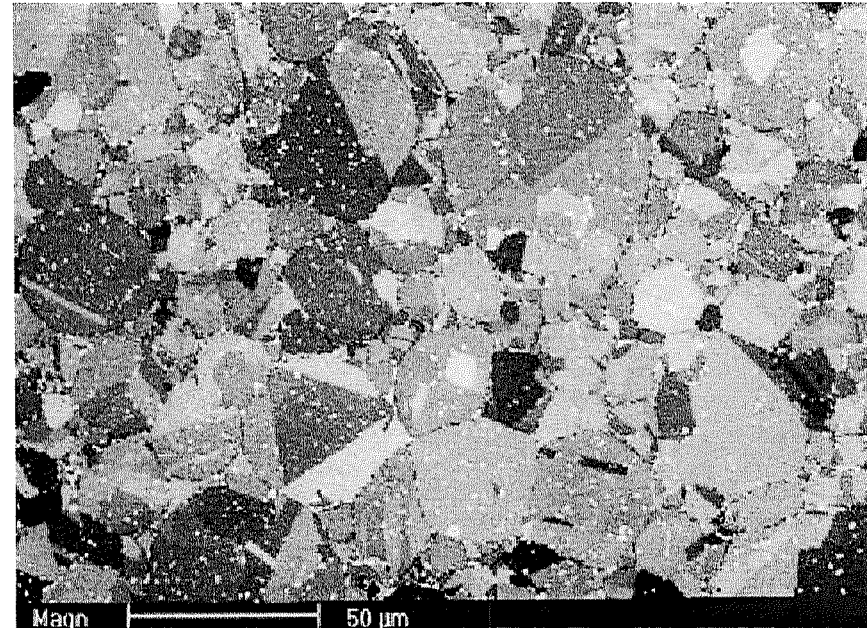
Back-Scatter Electron Imaging (BEI) shows matrix grains & finely dispersed second phase (Laves)

Fe-based superalloy powder, as-HIP'd

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Thick Oxides on PPB's Prevent Grain Boundary Migration

SEI showing thick oxide on PPB's
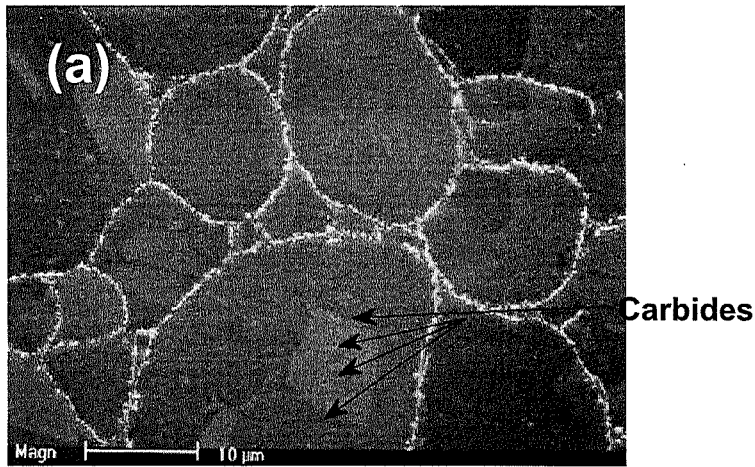
BEI shows Grains do not cross PPB's with thick oxides

Carbides

Oxides

Laves
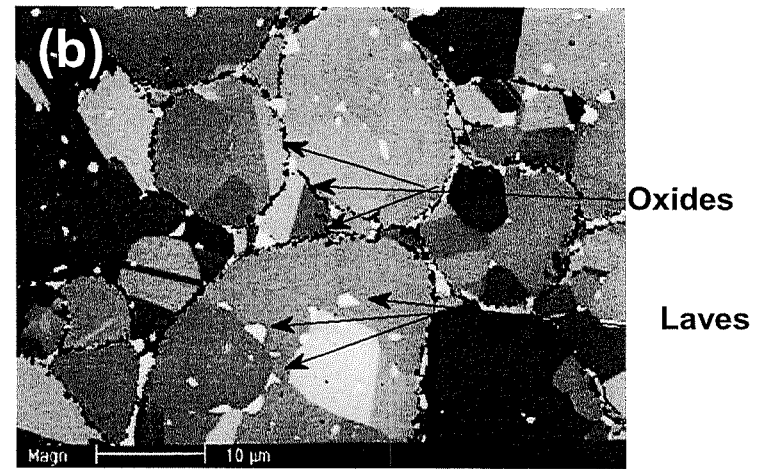
SEI showing grain boundary migrations across PPB's with thin oxide
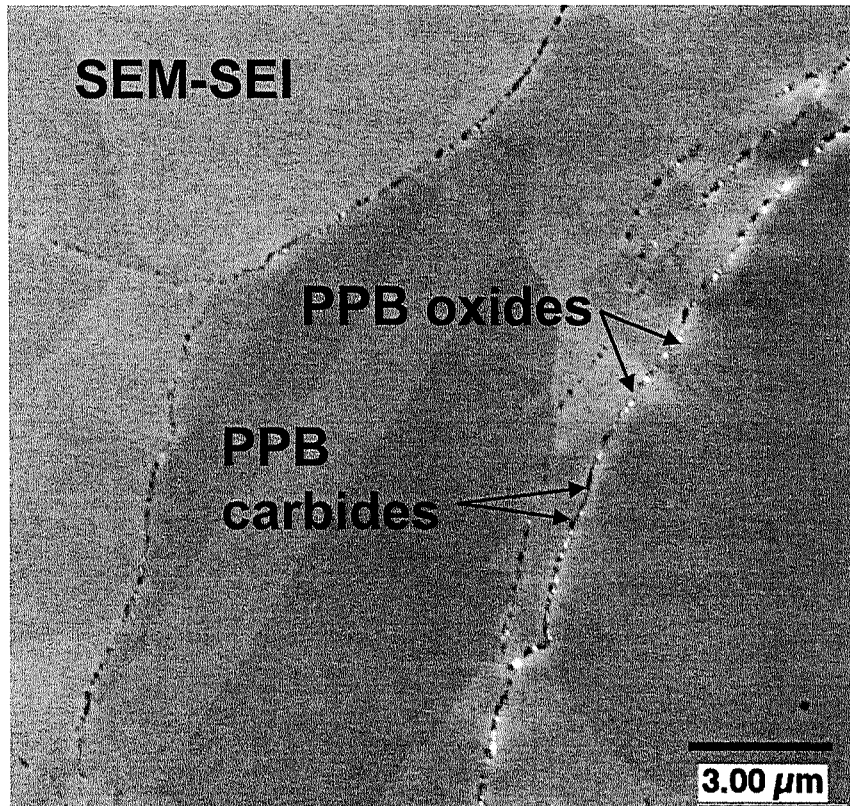
Laves

Carbides

Oxides

Fe-based superalloy powder, as-HIP'd

Rocketdyne

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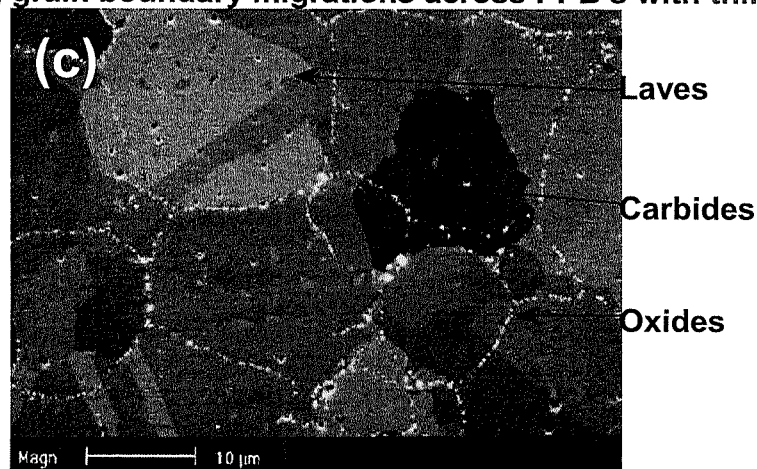
Oxide & Carbide Particles may both Decorate PPB's

Oxides on PPB's inhibit grain boundary migration more strongly than carbides

SEM-SEI

PPB oxides

PPB carbides

3.00 µm

SEM-BEI

3.00 µm
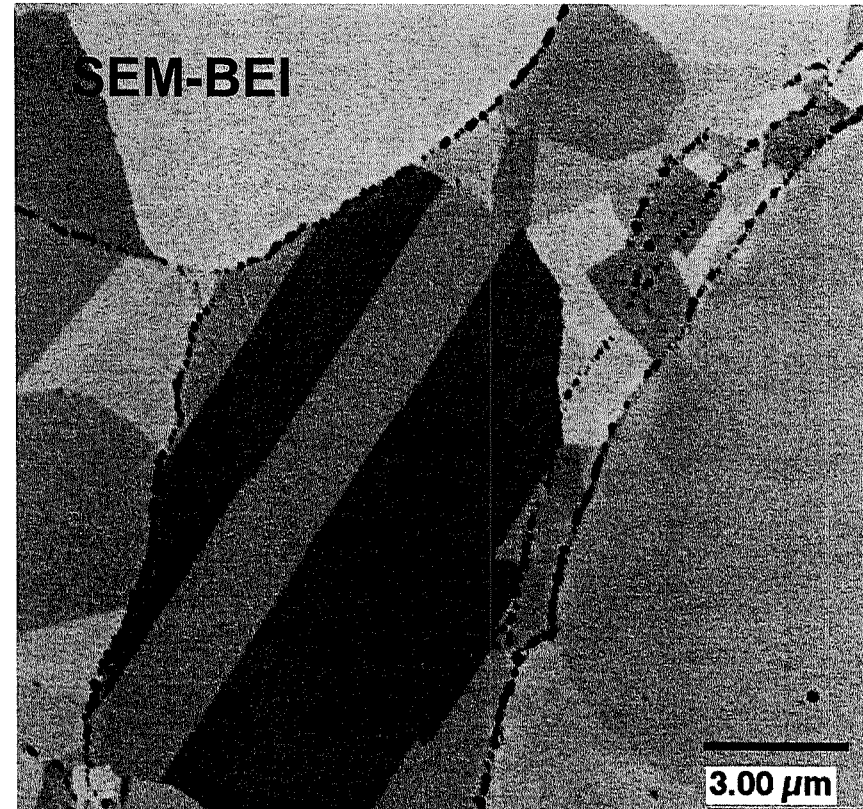
Ni-base superalloy, as-HIP'd

High Magnification SEM shows Gamma Prime Precipitates
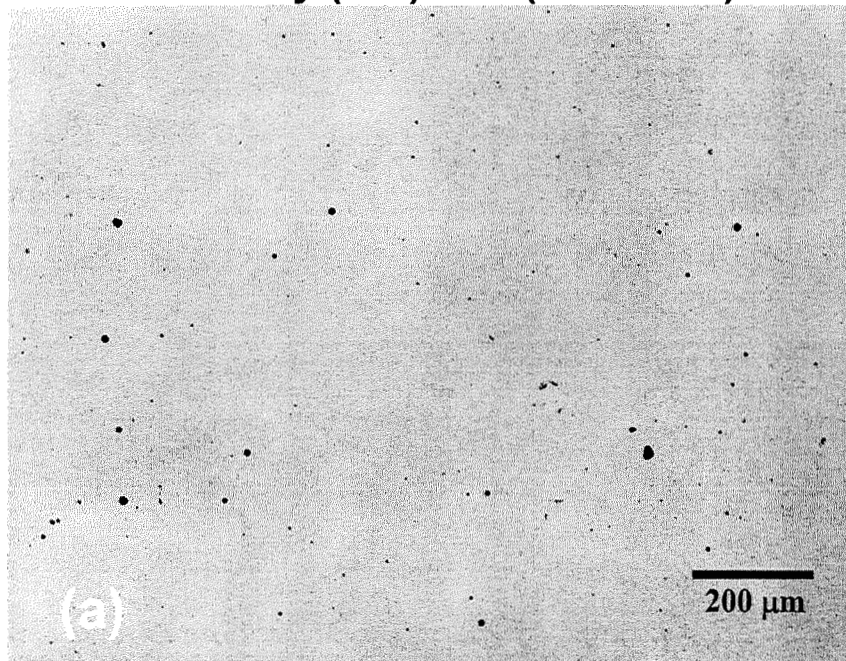
SEM-BEI

Gamma-prime

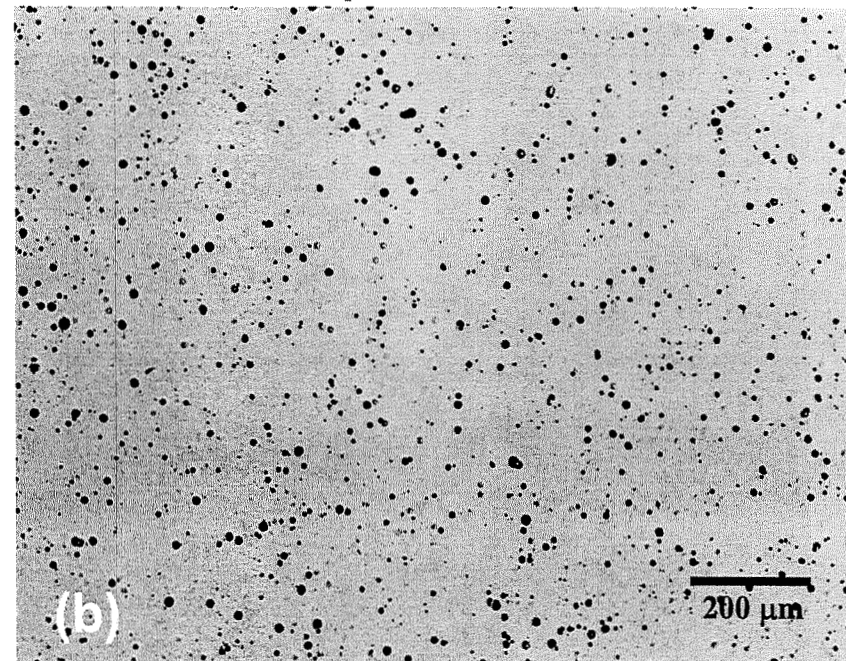PPB Al$_2$O$_3$

High Al

0.60 μm
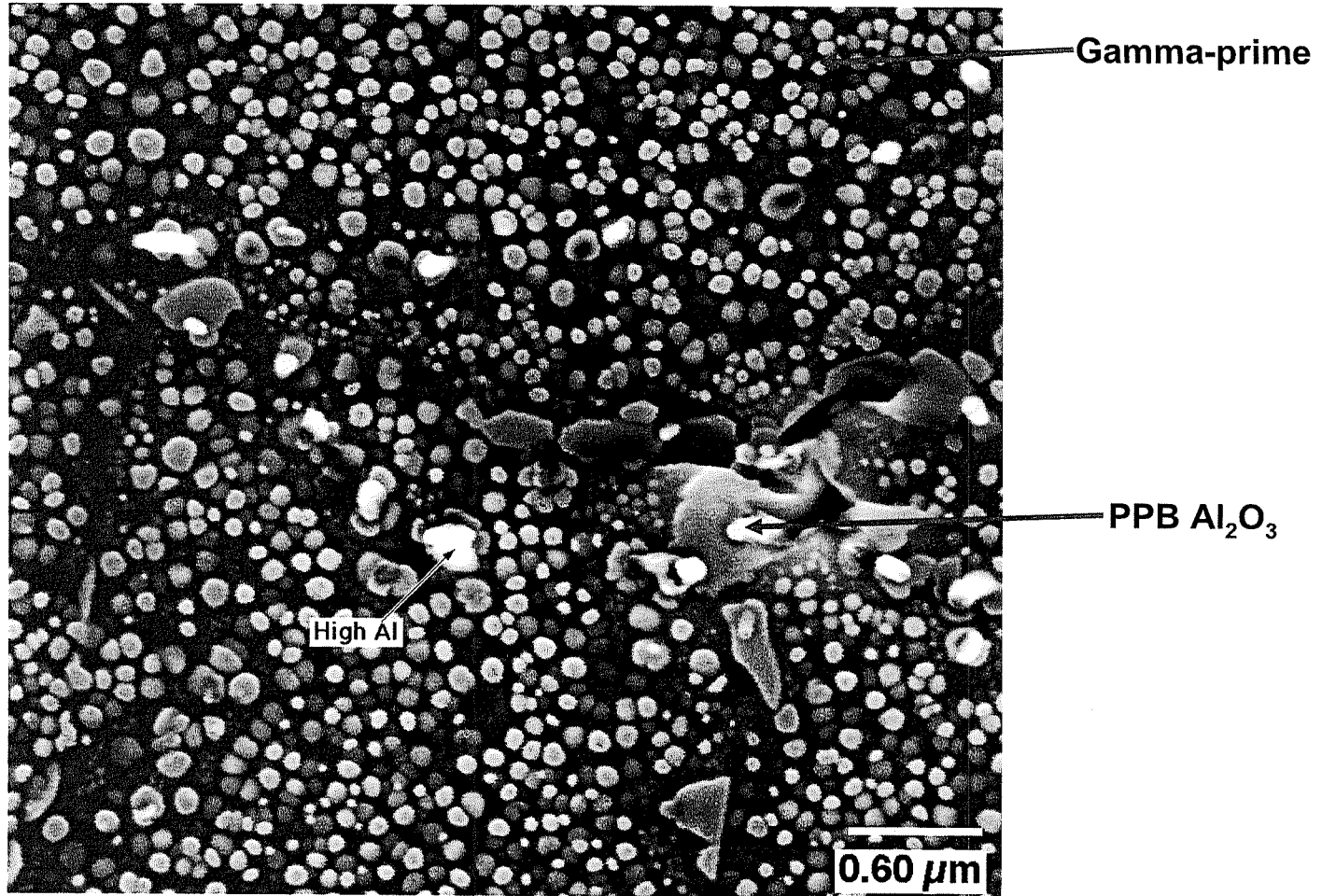
Ni-Based Superalloy, as-HIP'd

Leaking HIP Capsule can Entrap Argon Gas

Good response to Thermally Induced Porosity (TIP) test (1200C/4h)

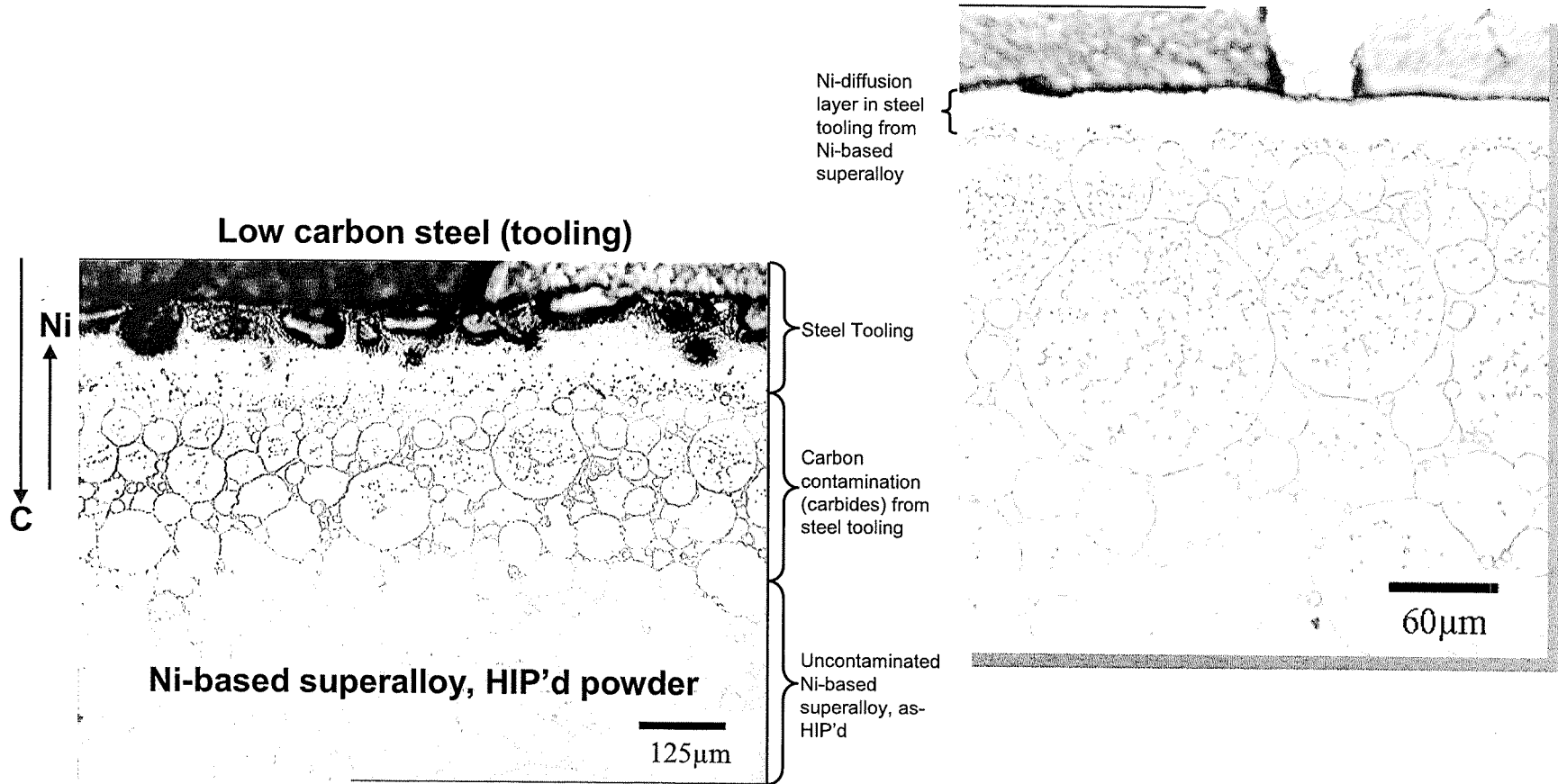**Bad response to TIP test**

(a) 200 μm   (b) 200 μm

Ni-based superalloy powders, as-HIP'd & TIP-tested

Steel HIP Tooling Contaminates Superalloy Surface Region

Low carbon steel (tooling)

Ni

C

Ni-based superalloy, HIP'd powder

125μm

Steel Tooling

Carbon contamination (carbides) from steel tooling

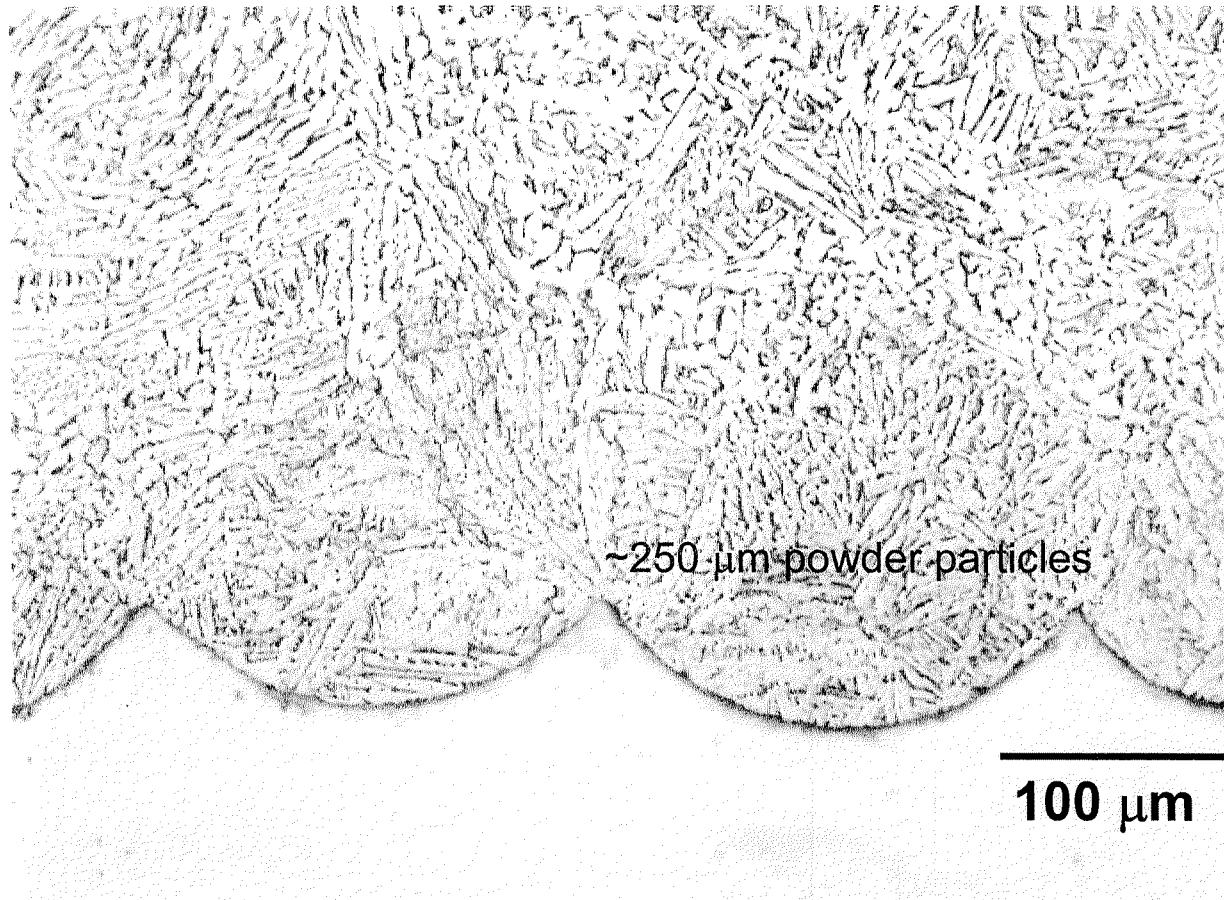Uncontaminated Ni-based superalloy, as-HIP'd

Ni-diffusion layer in steel tooling from Ni-based superalloy

60μm

Optical metallography - etched

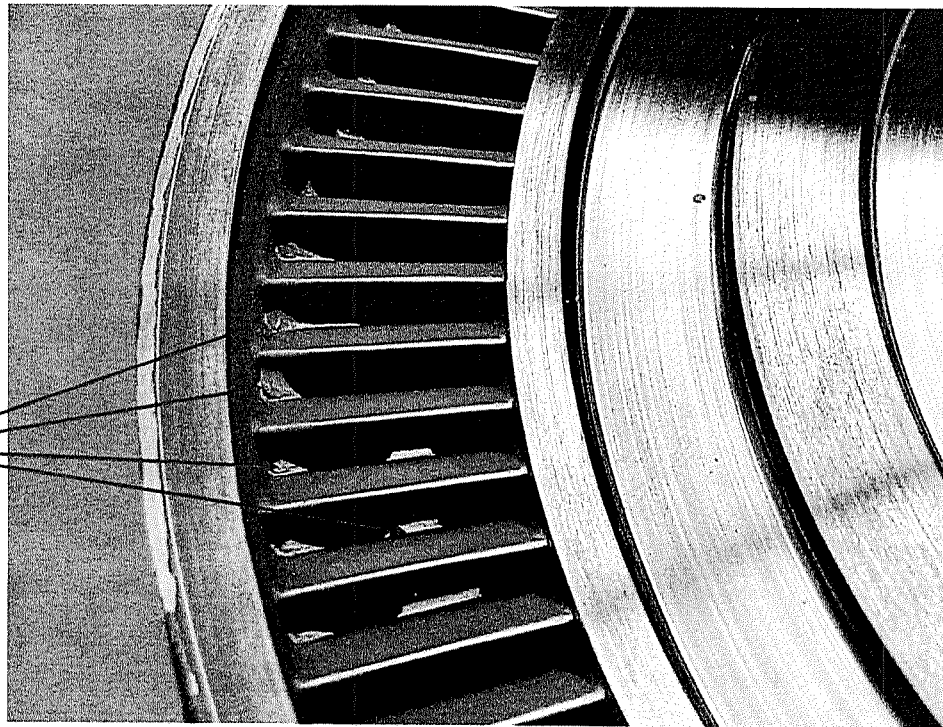# Powder Particles may Indent Tooling during HIP Consolidation

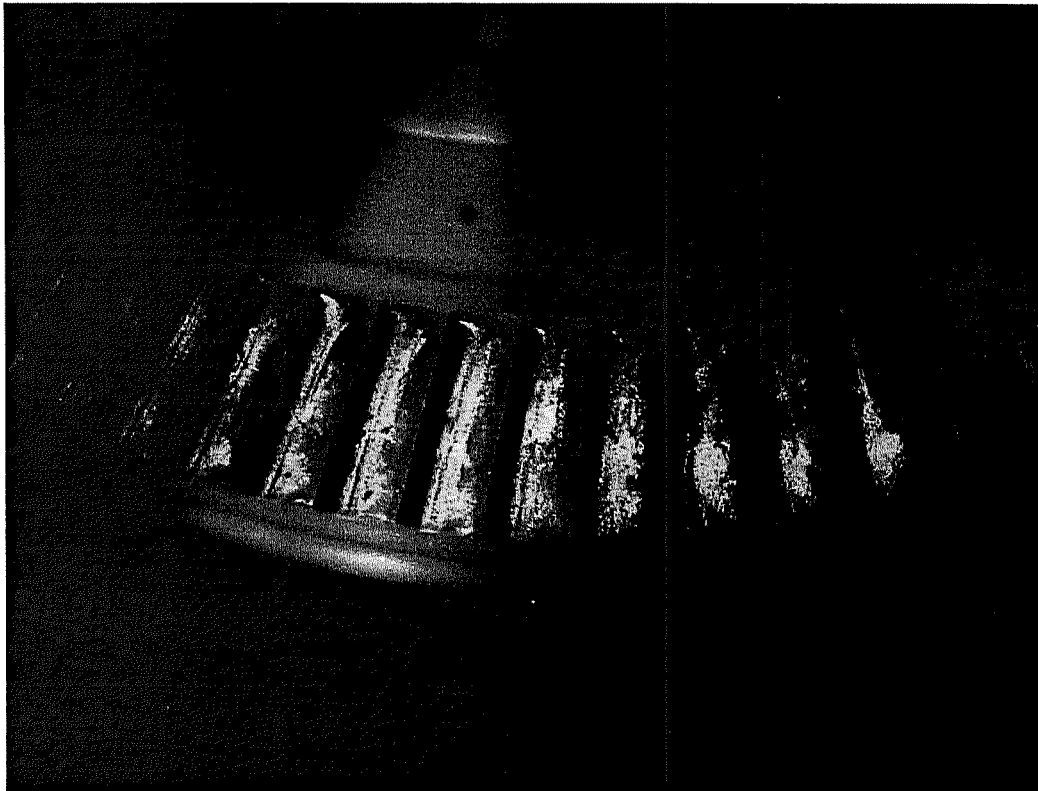~250 μm powder particles

Interface of P/M Ti alloy with Steel Tooling

100 μm

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Passivated Tooling Remnants are Difficult to Remove

**Passivated
tooling nodules
(remnants) are
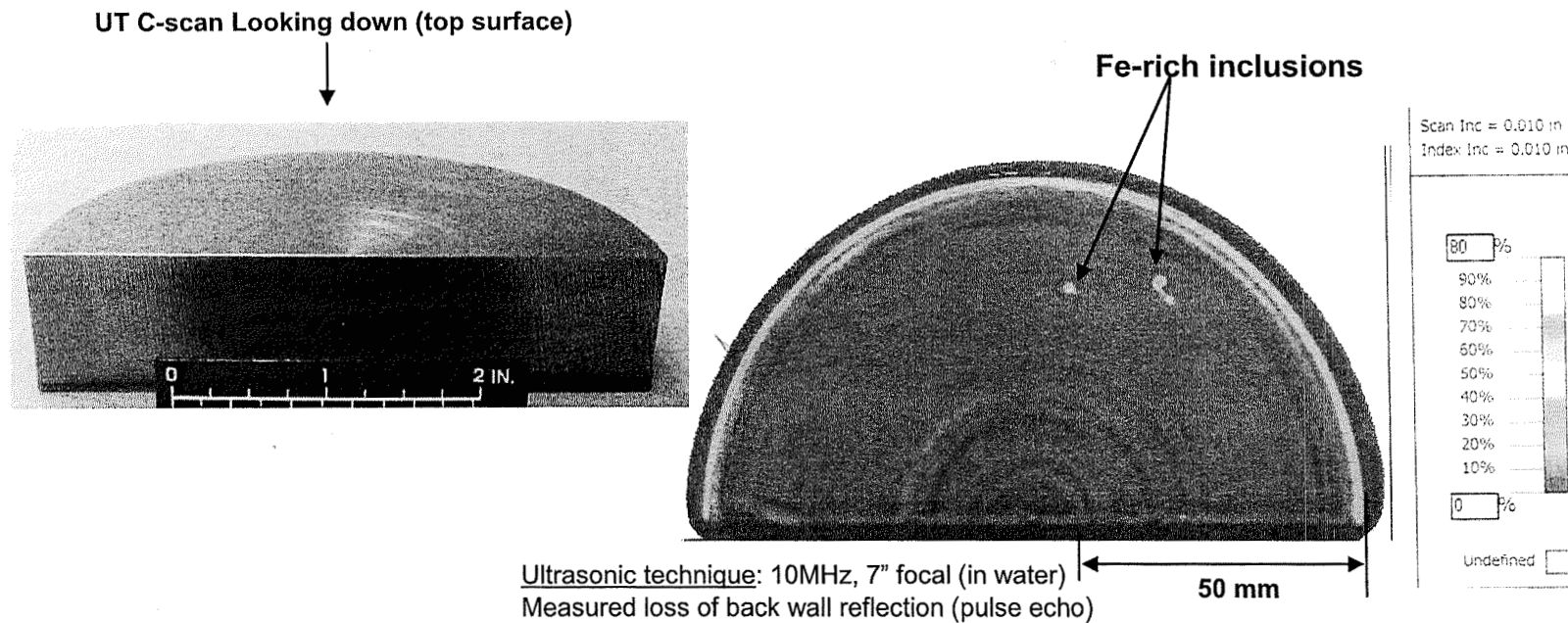difficult to
selectively
remove**

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Etching may Damage Part Surface

Net-shape
blade surfaces
in Fe-based
superalloy P/M
turbine blisk
after over-
aggressive
acid
dissolution of
tooling

- Fluorescent (UV) penetrant inspection shows over-etching on net surfaces
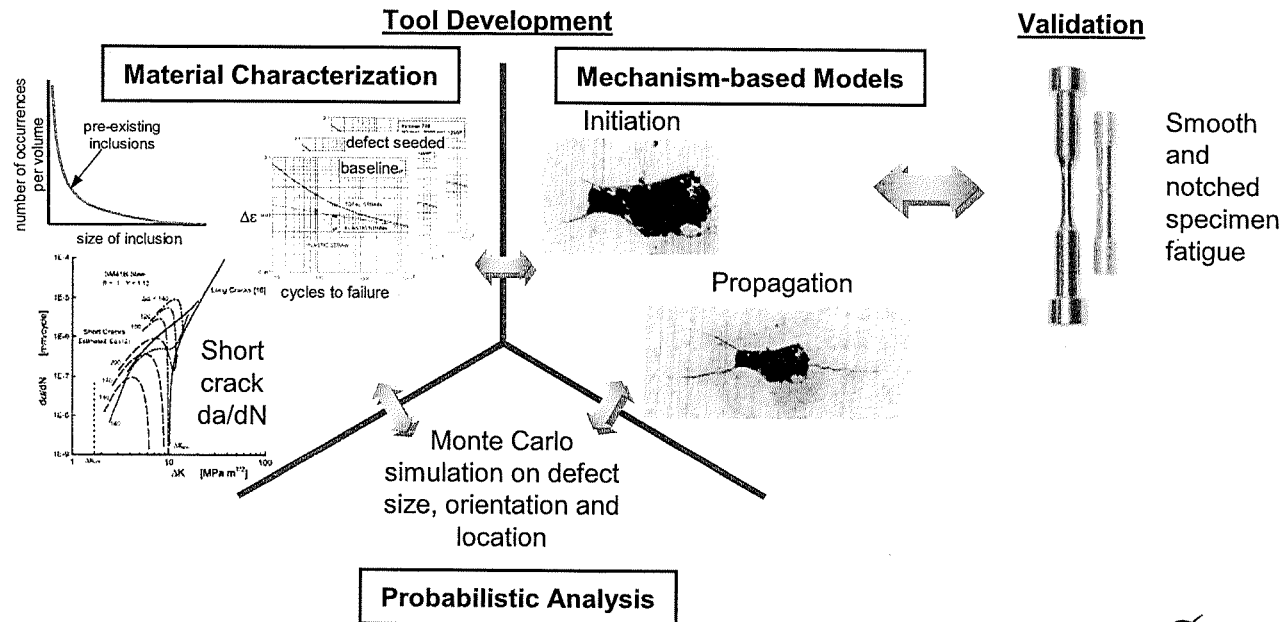
- Grain boundary attack may compromise fatigue life

Ultrasonic Evaluation of Ti-Alloy P/M Sample Finds Gross Defects (> 1500µm)

UT C-scan Looking down (top surface)

Fe-rich inclusions

Scan Inc = 0.010 in
Index Inc = 0.010 in

80 %
90%
80%
70%
60%
50%
40%
30%
20%
10%
0 %
Undefined

0   1   2 IN.

Ultrasonic technique: 10MHz, 7" focal (in water)
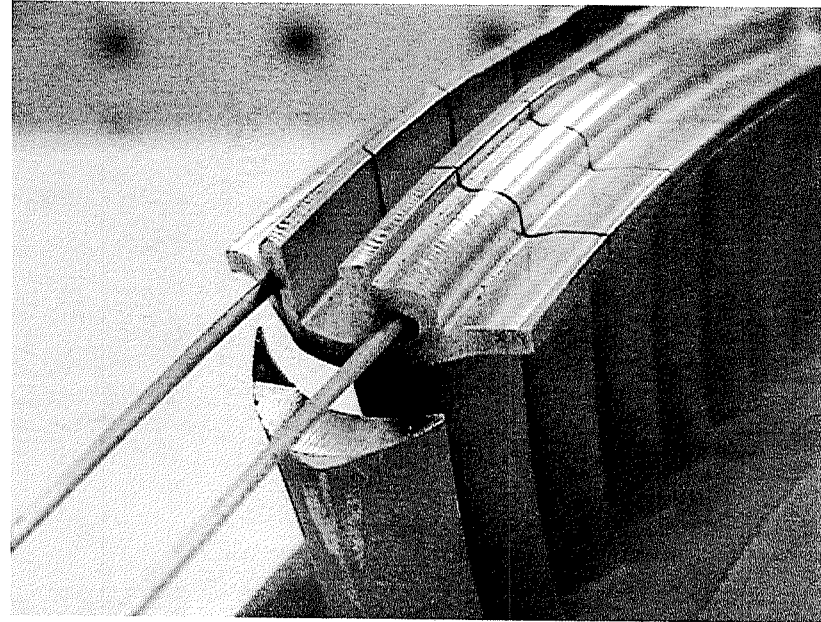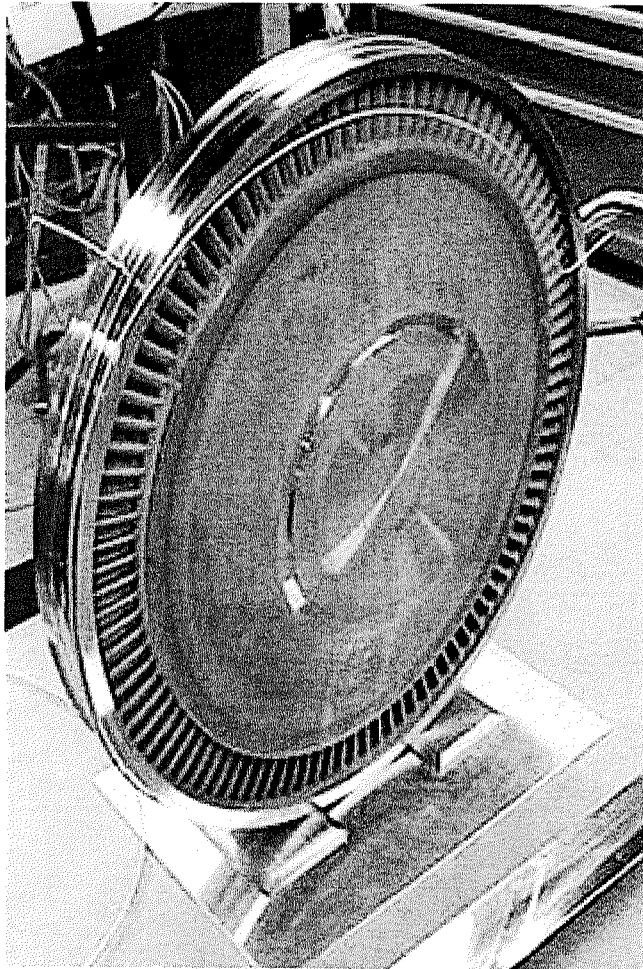Measured loss of back wall reflection (pulse echo)

50 mm

No method for non-destructive inspection of complex, net-shapes for very small (<100µm), cross-contamination & non-metallic inclusions

Probabilistic Life Prediction will Enable More Efficient Designs

Analysis Need	Current Deficiencies	Tool Development
• Improved life modeling for net-shape P/M parts	• Current non-destructive inspection is ineffective for complex shapes & small defects • Deterministic life prediction becomes excessively conservative	• Characterize powder & component defect populations • Characterize fatigue crack initiation • Characterize short crack growth rates • Develop models • Validate on coupons & apply to P/M components

Tool Development

Validation

Material Characterization

pre-existing inclusions

size of inclusion

$\Delta\varepsilon$

defect seeded

baseline

cycles to failure

Short crack da/dN

Mechanism-based Models

Initiation

Propagation

Monte Carlo simulation on defect size, orientation and location

Probabilistic Analysis

Smooth and notched specimen fatigue
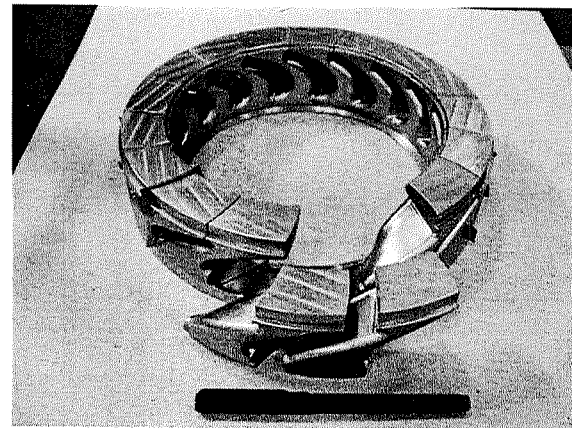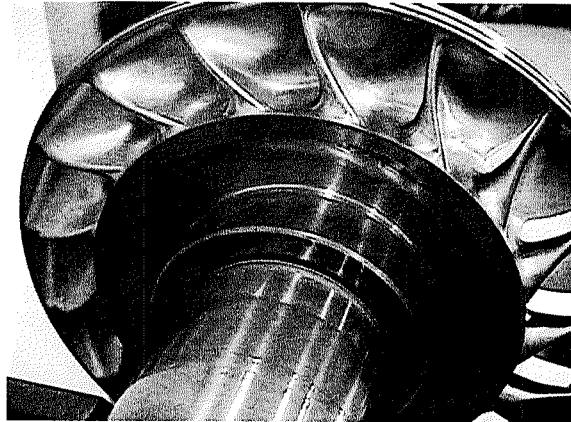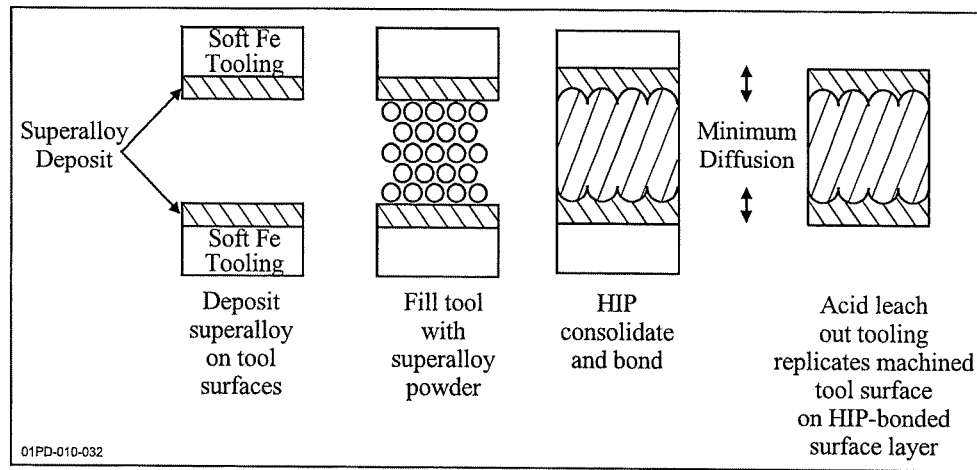
number of occurrences per volume

P/M HIP Processing Enables Novel Concepts such as Integral Damping of Turbine Blisks

HIP-Bonded Surface Layer Concept Provides Smooth Surfaces and Robust Environmental Protection

Superalloy Deposit

Soft Fe Tooling

Soft Fe Tooling

Minimum Diffusion

| Deposit superalloy on tool surfaces | Fill tool with superalloy powder | HIP consolidate and bond | Acid leach out tooling replicates machined tool surface on HIP-bonded surface layer |

01PD-010-032

Summary

- **Processing methods and variables are so numerous that the part fabricator must become involved with the part designer and the systems designer in an effective integrated product team**

- **The technology is ready for major structural applications, although qualification of flight hardware will be a laborious point-design approach for some time to come**

- **Many technology improvements are possible and in some cases required, to enable full exploitation.**

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