Powder Metallurgy Techniques Applied to Superalloys

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Introduction

NICKEL-BASE superalloys are used for parts which are subjected to relatively high temperatures under severe stress and corrosion conditions. Originally these alloys were designed for ulitization in aircraft gas turbine engines. They have also found wide use in marine and land-based power systems and, to a smaller extent, in the area of metal-working tools.

The first-generation superalloys were basically nickel with chromium for oxidation resistance and Al + Ti to form an $Ni_3(Al, Ti)$ precipitate called γ', which imparts high strength at elevated temperatures. These alloys were usually fabricated by casting and working. As the need for stronger materials grew, superalloys with continually higher volume fractions of γ' precipitate and stronger matrices were developed. These alloys, such as IN-100, are virtually impossible to hot-work using conventional forging techniques. Direct production of large parts by casting leads to severe segregation resulting in the formation of deleterious acicular phases. Metal powder technology not only offers methods to form these alloys into a variety of components, but can also improve mechanical properties and reduce costs. This paper will discuss three techniques which are in or entering production stages for use in aircraft gas turbine engines: conventional Powder Metallurgy (P/M), Thermoplastic Processing (T/P), and Mechanical Alloying.

Conventional P/M Superalloys

The use of P/M techniques to reduce segregation in superalloys is a relatively old idea[1] but was not technologically feasible until powders could be produced with an oxide layer less than 30 nm thick. In addition, superalloy powders cannot be consolidated by cold-pressing and sintering. To form monolithic bodies for further processing, they are hermetically sealed in a can and consolidated by such processes as hot isostatic pressing (HIP), hot extrusion, or hot forging.

Atomization Techniques and Powder Characteristics

Three different powder production techniques which yield sufficiently clean powders are currently being used: *argon atomization,*[2] *vacuum atomization,*[3] and the *rotating electrode process.*[4] These techniques have been reviewed extensively in the literature[5] and only a brief description will be given here.

Argon atomization is accomplished by impinging high-pressure argon gas on a molten metal stream (see Fig. 1). The powder has a relatively fine distribution of monocarbides (MC) and small dendritic spacings (see Fig. 2). A powder particle typically has about 5 grains across its diameter. The spherical holes in the powder particles are the result of argon entrapment. Argon atomization is generally regarded as the least expensive method for superalloy powder production.[6]

The second method, *vacuum atomization,* involves charging a superalloy melt with pressurized hydrogen and imploding it into a vacuum chamber (see Fig. 3). A typical powder particle made by this process (see Fig. 4) has a grain size and dendritic arm spacing which are generally larger than that of the argon atomized powder. It also contains a considerably higher degree of porosity and a relatively rough surface. The porosity of the powder is interconnected and is due to hydrogen gas evolution during atomization rather than entrapment.

The third commercial atomization technique is the *rotating electrode process* wherein the powder is produced by spinning and melting a superalloy rod simultaneously (see Fig. 5). The spherical particles (see Fig. 6) are formed in a He atmosphere in order to prevent oxidation and to accelerate cooling. Virtually no inert gas is entrapped during this process. A section of this powder clearly shows MC carbides which are much larger than those in either the argon or vacuum atomized material. The large MC particles which originally formed on solidification of the vacuum induction melting stock are virtually unaffected by remelting during powder production. This process is considered the most expensive for producing superalloy powders.[6]

Superalloy Powder Problems

Although P/M superalloys have solved many problems associated with their cast and wrought counterparts, they do have some unique problems of their own. Even under the most controlled atomization conditions an oxide film 15 to 30

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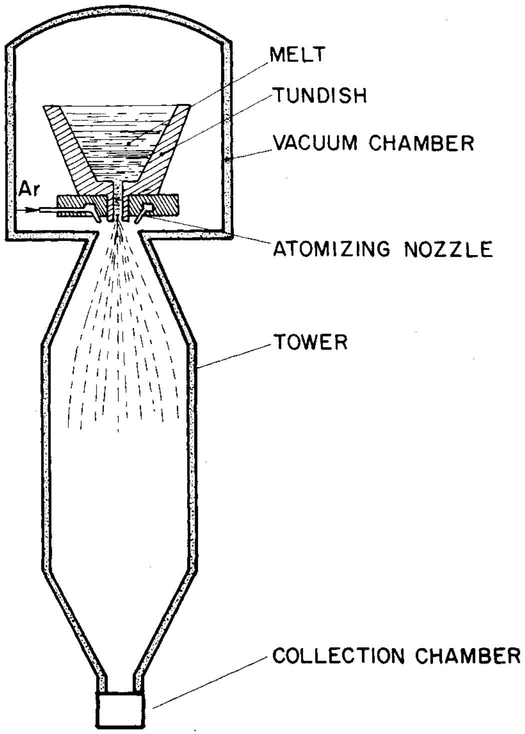

Fig. 1 Argon atomizer.

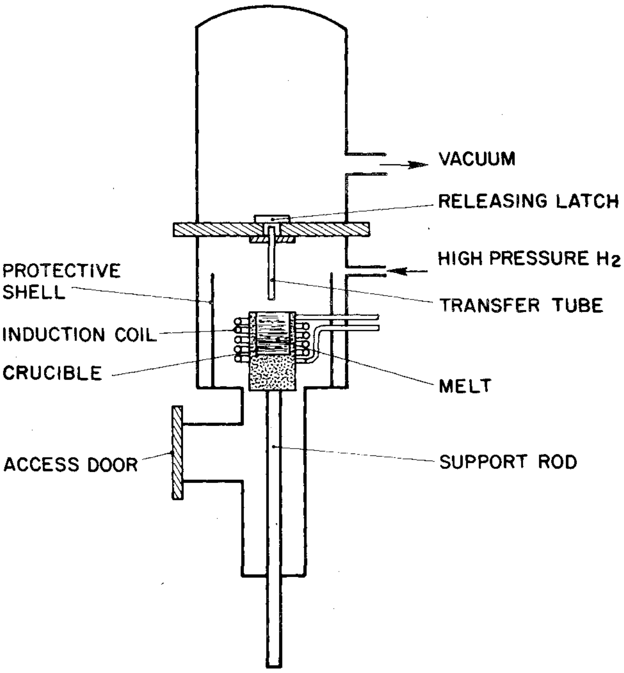

Fig. 3 Vacuum atomization technique using H_2.

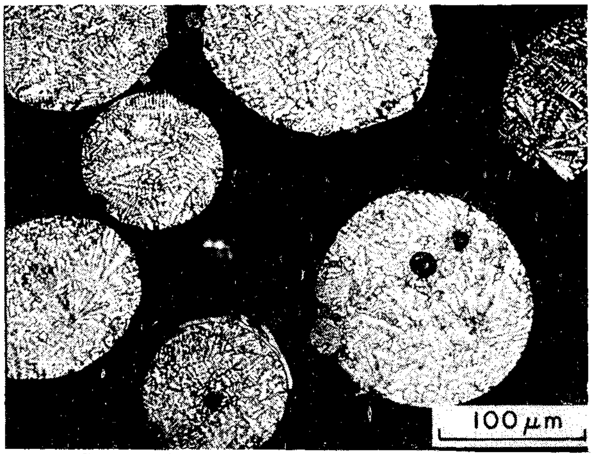

Fig. 2 Cross section of IN-100 produced by argon atomization.

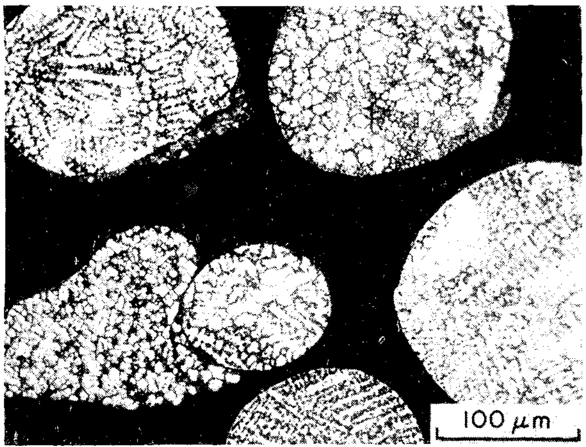

Fig. 4 Cross section of IN-100 powder produced by vacuum atomization.

nm thick forms on the powder surface. In the case of consolidation by hot isostatic pressing (HIP), where each powder particle sees little shear action, this powder surface oxide can lead to a relatively continuous brittle network. The effects of this oxide network are minimized by keeping the oxygen contamination low through careful atomization control and by good degassing procedures prior to canning the powder.

A serious problem in some P/M superalloys is the formation of MC on prior powder particle boundaries (PPB)[5] in the consolidated material. The MC at PPB in the consolidated material is a result of dissolving the finely dispersed MC particles during the heat-up before consolidation and reprecipitating MC on the powder surface. The carbide network on PPB is similar to the oxide network (the oxide film may act as nucleation site for MC), but is generally much thicker and more deleterious to the forgeability and mechanical properties of the superalloy. This problem has not been compeltely solved, particularly in alloys that form monocarbides based on TiC [IN-100, Astroloy (registered trademark of Wyman-Gordon Co.)], but its effects have been greatly minimized by lowering carbon contents and developing special low-temperature sintering treatments.[7] It has been found that alloys which contain Nb, Ta, and/or Hf form more stable MC carbides and greatly reduce the formation kinetics of the MC at PPB.[8-12]

Another potential problem unique to argon atomized powders is argon retention. Argon can be carried over into the final part either by entrapment during atomization or improper degassing prior to consolidation. If the argon content is too high, the forgeability of the HIP billets can be seriously degraded. The argon is widely distributed in pores which are compressed during consolidation and is not especially noticeable in as-consolidated material. Coalescence into large visible pores can occur if the material is subsequently solution-treated at a high temperature in order to increase grain size.[13] These pores are generally referred to as thermally induced porosity (TIP).

The P/M approach can radically alter the carbides in superalloys. In the intermediate temperature range the MC tends to decompose and form the complex cubic carbide $M_{23}C_6$.[8] As discrete particles, $M_{23}C_6$ inhibits grain boundary sliding and improves creep strength. A film of $M_{23}C_6$ fomed at the grain boundaries, however, lowers mechanical properties.[14] The accelerated dissolution of the very fine dispersion of MC carbides in P/M superalloys promotes film formation of $M_{23}C_6$ during the aging procedure (see Table 1).

Table 1 Carbide distribution and heat treatment for IN-792

Condition	MC interparticle spacing, μm	$M_{23}C_6$ morphology after 2050°F/2 hr, 1550°F/8 hr (1121°C/2 hr, 843°C/8 hr)	Average stress rupture life, h, at 1400°F/90 ksi (760°C/620 MPa)
cast	1000-100	globular at GB	225
cast and wrought	100-10	tended towards film	32
P/M	10-1	continuous film	11

Table 2 P/M vs cast and/or wrought compositions

	C	Cr	Ni	Co	Mo	W	Cb	Ti	Al	Ta	B	Zr
Astroloy												
C/W	.06	15	Bal.	15	5.25	–	–	3.5	4.4	–	.03	–
P/M	.03	15	Bal.	17	5.0	–	–	3.5	4.4	–	.03	.05
IN-100												
Cast	.18	10	Bal.	15	3.0	–	–	4.7	5.5	–	.01	.06
P/M	.06	10	Bal.	15	3.0	–	–	4.7	5.4	–	.01	.06
IN-792												
Cast	.21	12.7	Bal.	9	2	3.9	–	4.2	3.2	3.9	.02	.1
P/M	.04	12.5	Bal.	10	2	3.9	–	4.3	3.2	3.5	.02	.1
René[a] 95												
C/W	.15	14	Bal.	8	3.5	3.5	3.5	2.5	3.5	–	.01	.03
P/M	.05	12.7	Bal.	8	3.5	3.5	3.5	2.5	3.5	–	.01	.03

[a] Registered trademark of Allvac, a Teledyne Company.

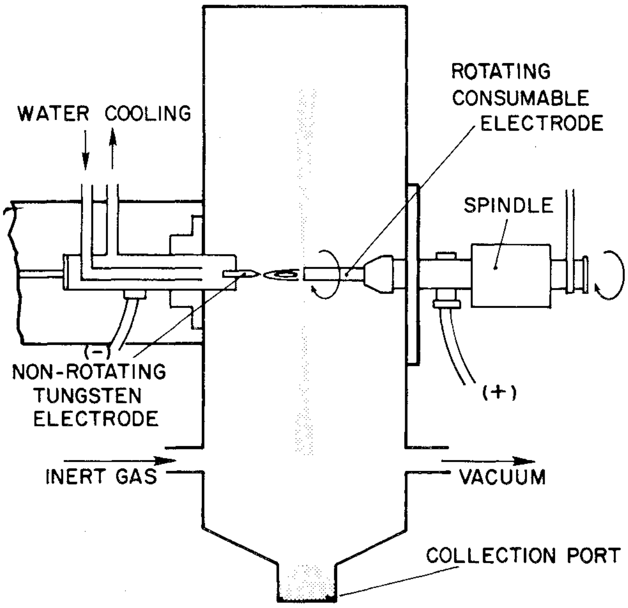

Fig. 5 The rotating electrode process.

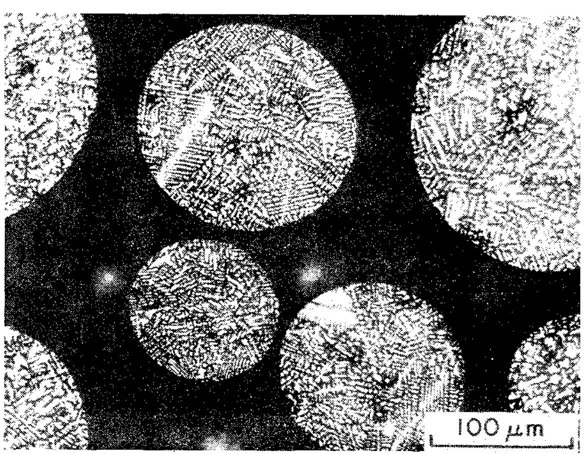

Fig. 6 Cross section of IN-100 powder produced by the rotating electrode process.

This problem can usually be solved by lowering the carbon content of a cast superalloy composition (see Table 2 for a comparison of P/M versus cast and/or wrought compositions).

Structural Control

The study of conventional P/M superalloys has led to several heat-treatment techniques which improve their mechanical properties. The first technique alters the grain boundaries of alloys such as P/M IN-792 from a smooth to a serrated interface (see Fig. 7).[7,15] The interlocking boundaries inhibit sliding and promote intragranular deformation. The result is improved rupture life and ductility (see Table 3).

Another structural control technique used in some P/M superalloys is thermomechanical processing of the material to obtain a necklace structure.[16] This structure, shown in Fig. 8, is composed of large warm-worked grains surrounded by a thin network of fine-grained material. This morphology offers high tensile strength with adequate stress rupture properties and has been applied to several materials including René 95 and Astroloy. It should be pointed out that the necklace structure was developed for cast and wrought materials, but is particularly suitable to P/M superalloys because of the more homogeneous nature of these materials.

P/M Processing Routes

Two processes, Gatorizing[17] and HIP + Forge,[16] are used to make P/M superalloy gas turbine discs. Gatorizing, shown schematically in Fig. 9, involves a hot-working step, usually extrusion, to develop a fine-grained structure (less than 5 μm) which can subsequently be superplastically formed into a disc sonic shape. (Sonic shape refers to a right cylindrical shape which can be nondestructively tested for internal defects.) Gatorized material can be formed into extremely complex shapes, such as an intergal wheel containing both disc and blades. Although Gatorizing is not confined to powder materials, the P/M approach greatly facilitates the hot-working step to set up the fine grain size.

An alternative commercial P/M processing route, known as HIP + Forge (see Fig. 10), is accomplished by consolidating the powder by hot isostatic pressing (HIP) in the 2100-2300°F (1150°C-1260°C) temperature range, followed by conventional forging to a disc sonic shape. The grain size of the HIP'ed P/M product (≈40 μm) is less than that of a casting and decreases forging loads to as much as one-third of that of

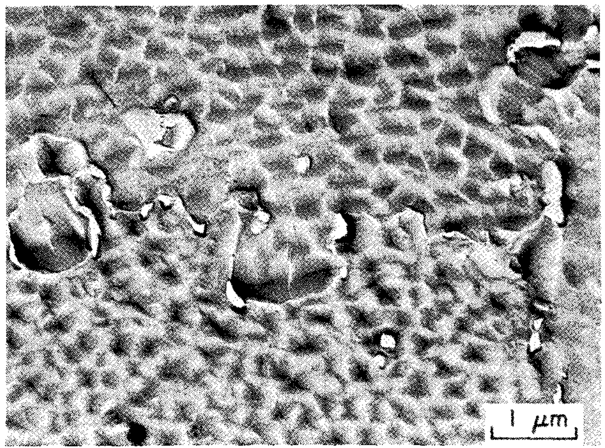

Fig. 7 P/M IN-792 heat treated at 1220°C/2 hr/AC, 845°C/16 hr/AC. Note that the grain boundary tried to move around heterogeneously nucleated gamma prime during cooling through the gamma prime solvus resulting in a serrated boundary.

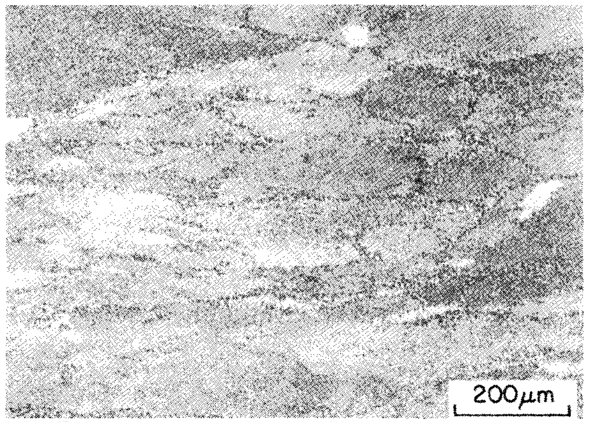

Fig. 8 The necklace structure of P/M René 95. Note large grains surrounded by a thin network of fine grains.

cast and wrought superalloys. The lower raw materials and machining cost associated with the P/M route improve the economics of disc manufacture and also result in a more reliable product. This process, which is considered more cost effective than Gatorizing, was originally developed for René 95 discs and is also being used to produce P/M components of other superalloys.[19-22]

Thermoplastic Processing (T/P)

The original impetus for P/M superalloys was reduction of segregation but the sustaining driving force has been cost reduction. Much effort is being expended on making less expensive superalloy parts. Special techniques which allow the powder to be HIP'ed directly to sonic shape, avoiding the extrusion and forging steps, are being developed. Thermoplastic processing,[18] denoted T/P, offers an excellent means to achieve this goal.[23]

T/P takes advantage of the fact that when a superalloy powder is cold-strained, its grain size is greatly reduced on recrystallization and the powder is considerably softened at elevated temperatures. The most commercially attractive method of straining powders is by passing them through a vertical-feed rolling mill. Each particle is flattened into a minature disc (see Fig. 11), with the largest coarsest-grained particles which are the most difficult to hot-deform receiving the most plastic strain. When this rolling operation is integrated into the conventional routes, as shown in Fig. 12, it is possible to HIP directly to final form.

T/P offers other technical and economic advantages over the conventional P/M techniques.[19] Atomization efficiency is expected to be enhanced since more of the output of the atomizer can be utilized. The larger powder (+100 mesh, 149 μm) is not generally used in conventional P/M processing because of its large grain size which inhibits consolidation and forging. These larger powders are rendered workable by the T/P process, however.

Hot isostatic compaction of T/P-treated powder can be achieved at lower temperatures than conventional powder. This should lead to improved HIP furnace life and lower energy consumption. Consolidation by HIP also results in a finer grain size (≈ 5 μm) in the consolidated billet (see Fig. 13) and, if desired, the as-HIP'ed compact can be superplastically formed. Forging of T/P billet can be done at 30% lower pressures than that of conventional P/M billet, making the production of larger parts feasible using presently available forging equipment. In addition, die filling is better during conventional forging of T/P billet.

The HIP-to-final-form capability has been demonstrated with IN-792 and Astroloy. The mechanical properties of these materials are comparable to those of materials made using more conventional P/M processing techniques (see Table 4). Expensive extrusion and forging steps have been avoided.

Mechanical Alloying

A major disadvantage of conventional superalloys is that the phases upon which they depend for their strength at high temperature coarsen and/or dissolve with increasing temperature. It has been known for some time that the strength of metals at high temperatures could be increased by the addition of a fine dispersion of insoluble refractory oxides.

The Process

The mechanical alloying process[24] was developed to introduce a fine inert oxide dispersion into superalloy matrices containing reactive alloying elements. Mechanical alloying is a high-energy dry-milling process in which a mixture of metal and/or nonmetal powders is subjected to constant fracturing and rewelding in an energetic grinding-ball charge. During each collision of the grinding balls, many powder particles are trapped and plastically deformed (see Fig. 14a). Along the line of centers between the colliding balls sufficient deformation can occur that the adsorbed surface contaminant film on the metal powder particles is ruptured, exposing atomically clean metal surface. Where metal particles overlap, cold-welds are formed, building up composite metal particles such as that outlined by the dark line in the figure. At the same time other powder particles are fractured. Two metallic constituents have been indicated by light and cross-hatched particles.

Table 3 Effect of cooling rate on P/M IN-792 on serration wavelength and stress rupture life[a]

Rate of cooling	Wavelength, μm	Stress rupture life at hours at 1400°F/90 ksi (760°C/620 MPa)
Water quench to room temperature	.05	11
Air cool to room temperature	1.8	78
Furnace cool to 2100°F (1149°C) followed by air cool to room temperature	4.3	168

[a] All materials heated 2192°F (1200°C)/1 hr/WQ, AC, or FC; 1562°F (850°C)/16 hr. Each material had a grain of ASTM 10.

Table 4 Mechanical properties of extruded and T/P + HIP P/M IN-792[a]

	Extruded + heat treatment	T/P + HIP + heat treatment
0.2% YS (ksi/MPa)	140/965	144/994
UTS (ksi/MPa)	203/1420	206/1460
Elong. (%)	16	15
RA (%)	18	16
	Stress rupture at 1400°F/90 ksi (760°C/620 MPa)	
Life (hrs)	168	185
Elong. (%)	6.0	7.0
RA (%)	8.0	9.0

[a] HT at 2192°F (1200°C)/1/3 hr/FC to 2125°F (1163°C), AC; 1550°F (843°C)/16 hrs.

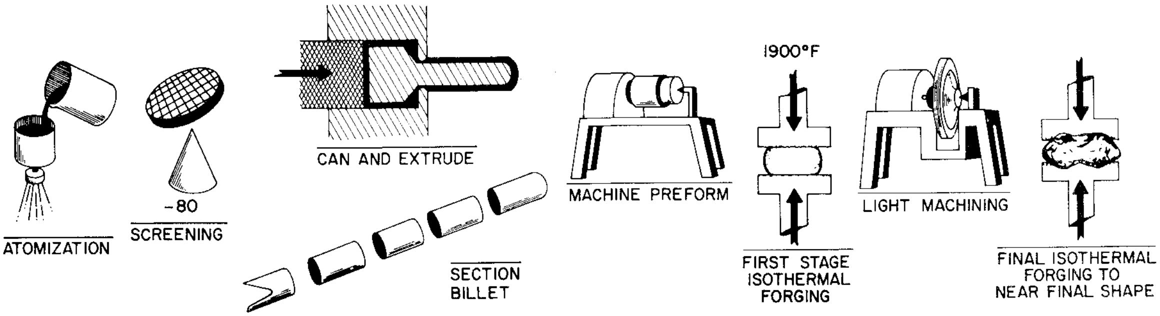

Fig. 9 Schematic flow chart of the Gatorizing process.

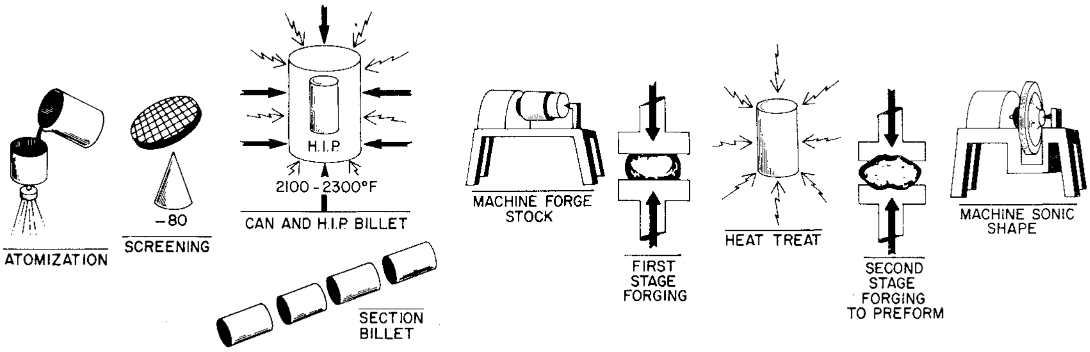

Fig. 10 Schematic flow chart of HIP and forging processing.

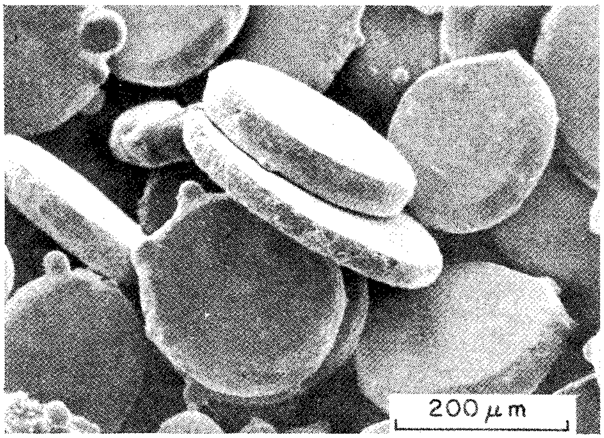

Fig. 11 IN-100 powder (−60 mesh) T/P processed by powder rolling.

Figure 14b shows the situation later in the process when most of the particles consist of composites similar to the one produced in the collision of Fig. 14a. The diagram in Fig. 14b shows the rewelding of composites with further plastic deformation and refinement of structure. Several composite particles, delineated by dotted lines representing their fractured surface films, are being welded together into a larger composite. Other composite particles, less able to withstand the plastic deformation required for cold-welding, are being fractured. The cold-welding and the fracturing of the particles reach a steady-state balance, leading to a relatively coarse and stable overall particle size. The internal structure of the particles is continually refined by the repeated plastic deformation.

Production of Oxide-Dispersion-Strengthened Superalloy Powders

The commercial manufacture of oxide-dispersion-strengthened superalloys requires a powder mixture more varied in composition and particle size than that indicated schematically in Fig. 14. A typical powder mixture, shown in Fig. 15, consists of fine (4-7 μ) nickel powder, −100 mesh light chromium and −100 mesh gray master alloy. About 2 v/v% of very fine Y_2O_3 (25 nm) is also present but not resolved.

Establishment of an Oxide Dispersion

During the production of dispersion-strengthened superalloy powders, a mixture of powder such as that in Fig. 15 is charged into a high-energy grinding mill – for example, a Szegvari Attritor Grinding Mill (registered trademark of Union Process, Inc.). During the mechanical alloying of this mixture the nickel, which is the softest constituent in this mix, acts as the cement which binds the other constituents together and forms a continuous matrix (see Fig. 16a). The original constituents of the raw materials powder mix can be identified within the composite particles. The chromium tends to form flake or platelike fragments. The master alloy, containing aluminum and titanium along with some nickel, breaks up

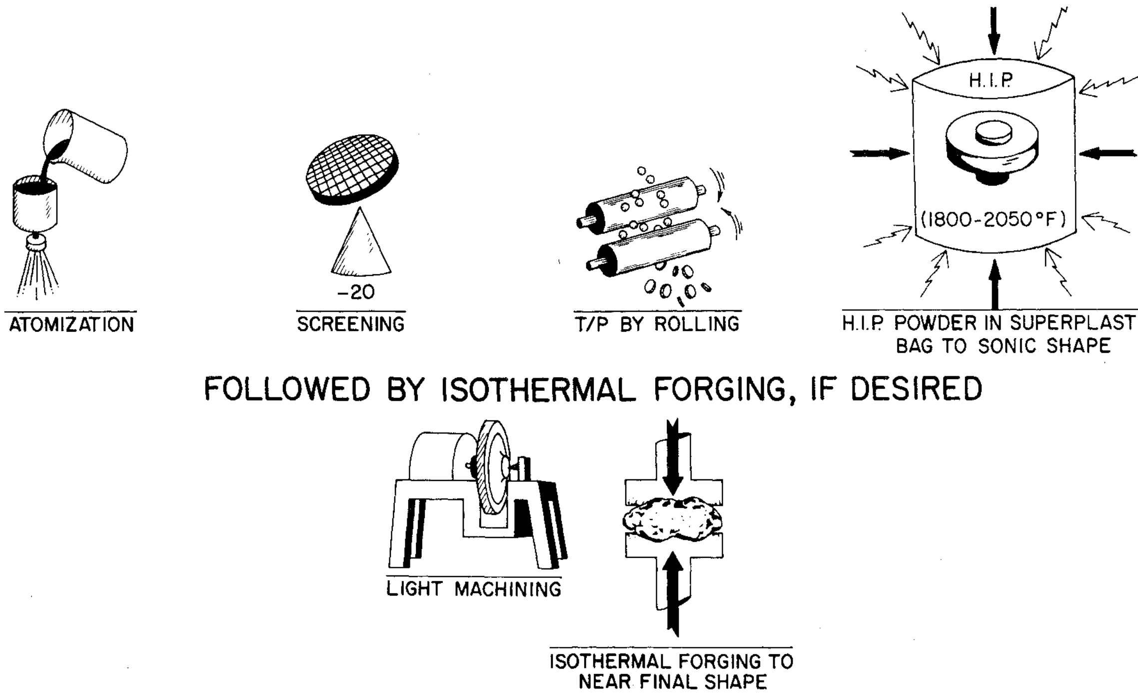

Fig. 12 Schematic flow chart showing use of T/P processing.

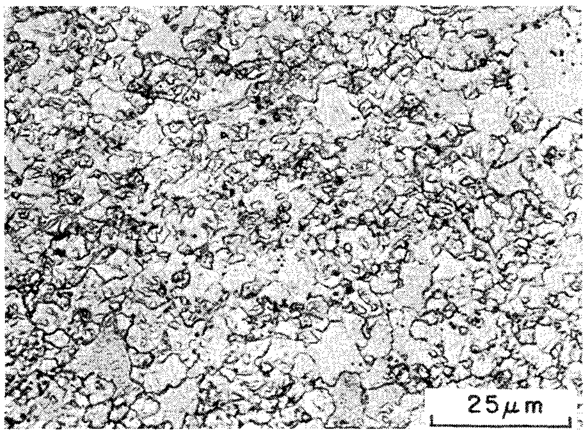

Fig. 13 Structure of IN-792 consolidated from −40 mesh T/P processed powder by HIP'ing at 1960°F (1070°C) and 15,000 psi (103 MPa).

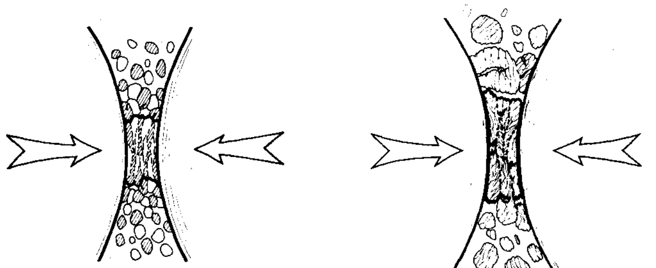

Fig. 14 The mechanical alloying "event": a) formation of composites early in process; b) rewelding of composites leading to refinement of structure.

into small equiaxed fragments which become imbedded in the nickel. The Y_2O_3 becomes entrapped along the weld interfaces between different fragments in the composite metal powders (see schematic diagram on right of Fig. 16a).

After 4 hours of processing, the concentration of the oxide particles within the welds is decreased as the welds move closer together (see Fig. 16b). Finally, after about 20 hours (see Fig. 17c), the powder has been processed to the point where its optical structure has been largely eliminated. At this point (see schematic diagram on right of Fig. 16c), the distance between the oxide particles along the weld interfaces is approximately equal to the spacing between the welds and both are less than 0.5 μm. This spacing also coincides with the random interparticle spacing of oxide calculated on the basis of the average particle size and volume fraction of oxide added to the original powder mix. Further mechanical alloying beyond this point physically could not improve the dispersion.

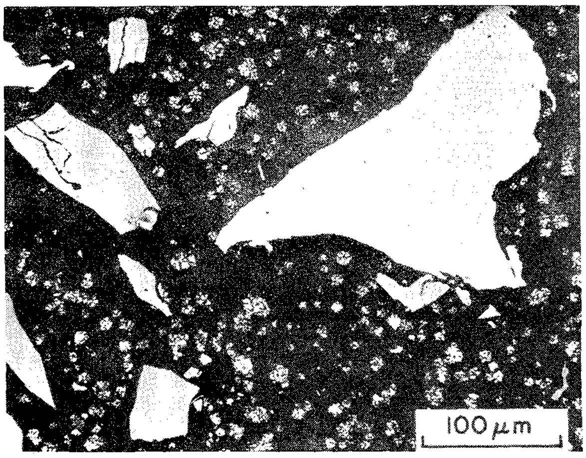

Fig. 15 Typical raw materials mix for the production of an oxide dispersion strengthened superalloy. Note the presence of fine nickel, coarse white chromium, and gray master alloy. Very fine Y_2O_3 is also present but unresolved.

Consolidation and Property Establishment

In order to achieve superior high-temperature properties, the powders must first be consolidated and worked under conditions which leave them with a high level of stored energy. Material is then given a secondary recrystallization heat treatment to form coarse grains which are highly elongated in the direction of extrusion and working. This structure is necessary for achievement of maximum elevated temperature properties[25] (see Fig. 17). Grain aspect ratio, the average grain dimension parallel to the applied stress divided by the average grain dimension perpendicular to the applied

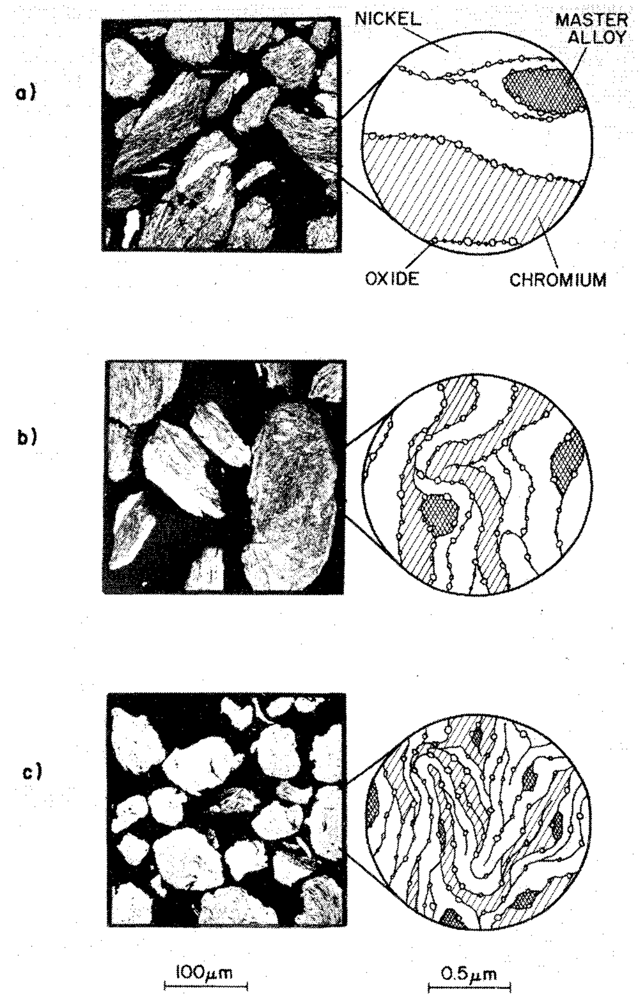

Fig. 16 Establishment of an oxide dispersion in a mechanically alloyed superalloy powder after a) 1 hr, b) 4 hr, and c) 20 hr.

stress, has a strong effect on elevated temperature rupture stress.

INCONEL Alloy MA 754

The first mechanically alloyed oxide-dispersion-strengthened superalloy to be produced on a large scale is INCONEL (registered trademark of The International Nickel Company, Inc.) alloy MA 754.[26] This material is basically a nickel-20% chromium alloy strengthened with about 1 v/v% of Y_2O_3 (see Table 5). This relatively simple alloy, which is comparable in its characteristics to TDNiCR but with a nonradioactive dispersoid, is suitable for such applications as gas turbine vanes.

Structure

The macrostructure of commercially produced rectangular bar, hot-rolled, heat-treated, and etched to show the grain structures of three faces, is shown in Fig. 18. The elongation of the grains along the direction of working is obvious. The grain width in the plate plane is somewhat greater than the grain thickness. This reflects the tendency of grain structures in oxide-dispersion-strengthened materials to mirror the symmetry of the mill product shape.

Table 5 Nominal composition of INCONEL alloy MA 754

Weight, %					
Cr	Y_2O_3	Al	Ti	C	Ni
20	0.6	0.3	0.5	.05	Bal.

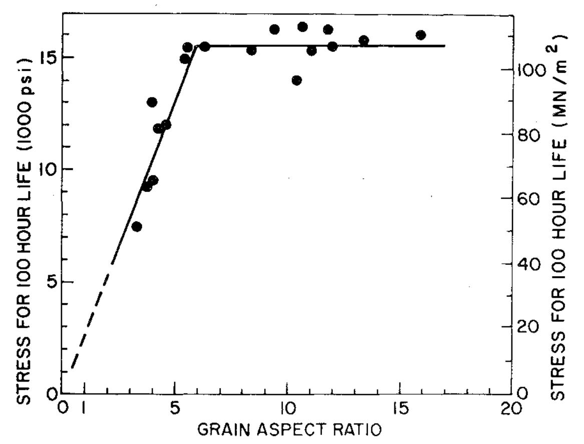

Fig. 17 Relationship between stress for 100-hour life at 1900°F (1038°C) and grain aspect ratio.

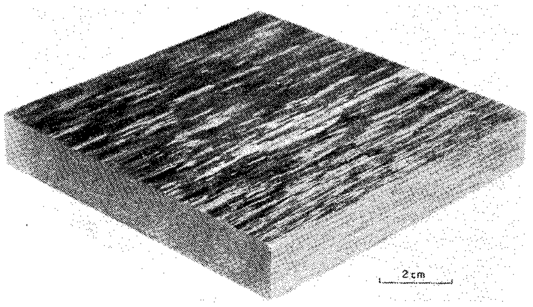

Fig. 18 Microstructure of INCONEL alloy MA 754 bar. Note different grain shapes in principle planes.

The details of the grain structure in a longitudinal section are shown in Fig. 19. This view shows the maximum and minimum average grain dimensions. Note the extreme irregularity of grain boundaries, which is typical of oxide-dispersion-strengthened materials, and the presence of numerous fine twins.

The distribution of the oxide dispersion in MA 754 is shown in Fig. 20. The very fine dark particles are the primary dispersion of yttrium-aluminates formed by reaction between the added Y_2O_3, excess oxygen in the powder, and a trace of aluminum added to getter oxygen. The formation of yttrium-aluminates has been shown to have no practical effect on the stability of oxide-dispersion-strengthened materials.[27] The large dark particles are titanium carbonitrides. A few stacking faults or microtwins are also visible. The distribution of the primary dispersoid is quite uniform.

Elevated Temperature Strength

The 2000°F (1093°C) longitudinal stress rupture properties of INCONEL alloy MA 754[28] are compared to those of TDNiCr, thoriated nickel bar, and two conventional alloys having a composition similar to the matrix of MA 754, in Fig. 21. MA 754, in common with other dispersion-strengthened materials such as TDNiCr and thoriated nickel, has a very flat log-stress/log-rupture-life slope compared to conventional alloys. The absolute strength values for MA 754, about 19,000 psi (131 MPa) for 100 hours for example, are higher than both of the other dispersion-strengthened materials, which range from 10,000 to 13,000 psi (69 to 90 MPa) and about an order of magnitude greater than conventional materials such as alloy 80A and NIMONIC (registered trademark of The International Nickel Company, Inc.) alloy 75.

The effect of testing direction on the elevated temperature stress rupture properties of MA 754 is indicated in Table 6.

Table 6 Longitudinal and long transverse stress rupture properties of INCONEL alloy MA 754 at 2000°F (1093°C)

	Stress to produce rupture in:					
	20 hr		100 hr		1000 hr	
Direction of tests	ksi	MPa	kis	MPa	ksi	MPa
Longitudinal	20.0	138	19.0	131	17.9	123
Long transverse	8.0	55	6.1	42	4.2	29

Table 7 Physical properties of INCONEL alloy MA 754

Property	Value
Density	0.3 lb/in^3 (8.3 gm/cm^3)
Melting point (solidus)	2550°F (1399°C)
Modulus of elasticity at 70°F (20°C)	21 × 10^6 psi (144.8 GPa)

Table 8 Nominal composition of INCOLOY alloy MA 956E

		Weight, %		
Cr	Al	Y_2O_3	Ti	Fe
20	4.5	0.5	0.5	Bal.

The rupture stress capability in the longitudinal direction is consistently higher than that in the long transverse direction, reflecting the differences in grain aspect ratio in the two directions.

Physical Properties of MA 754

Some of the important physical properties of INCONEL alloy MA 754[28] are given in Table 7. Its relatively high melting point, 2550°F (1399°C), and the low value of room temperature modulus of elasticity in the longitudinal direction, 21 × 10^6 psi (144.8 GPa), are especially important. The low elastic modulus indicates a predominantly (100) texture in the direction of major grain elongation. This has been shown to give superior thermal fatigue resistance.

INCOLOY Alloy MA 956E

Composition and Structure

The ability to add large amounts of metallic aluminum by mechanically alloying is demonstrated in the production of INCOLOY (registered trademark of The International Nickel Company, Inc.) alloy MA 956E[29] (see Table 8). This is a ferritic iron-chromium-aluminum alloy dispersion strengthened with about 1 v/v% of added Y_2O_3. Sheet of this alloy has been produced by a combination of hot- and cold-working operations which yield "pancake"-shaped grains following heat-treatment. This grain structure insures high and isotropic properties in both directions within the sheet plane.

Mechanical Properties

The 2000°F (1093°C) stress rupture properties of INCOLOY alloy MA 956E in both the longitudinal and transverse direction are given in Fig. 22. The longitudinal and transverse stresses for 1000-hour life in MA 956E at 2000°F (1093°C) are approximately 8000 psi (56 MPa). These properties are both higher and more isotropic than those of TDNiCr sheet and superior to those of TD nickel sheet.

INCOLOY alloy MA 956E possesses excellent room temperature fabricability. It can be bent more than 150° around a diameter equal to twice the sheet thickness. This fabricability is required for production of complex components such as gas turbine burner cans.

Chemical Properties

The results of cyclic oxidation tests performed at 2012°F (1100°C) on INCOLOY alloy MA 956E and TDNiCr are given in Table 9. The superiority of MA 956E is obvious in all categories of data. MA 956E forms a very stable alumina film and continues to undergo parabolic oxidation at times of over 500 hours in this test. TDNiCr, on the other hand, forms a chromium-oxide-based film and loses weight by volatilization of CrO_3 for times over 50 hours. Because of this difference in the identity of the oxide film and the mechanism of oxidation in the two alloys, MA 956E would show increased superiority over a material such as TDNiCr for longer times and at higher temperature. MA 956E, in fact, was designed to have oxidation resistance equivalent to that conferred by aluminide coatings.

MA 956E also has other attractive chemical properties. The results of a 1700°F (927°C) burner rig sulfidation test are shown in Table 10. The alloy shows a marked supriority to TDNiCr, both in weight change and depth of attack. The 2000°F (1093°C) carburization resistance of MA 956E in a

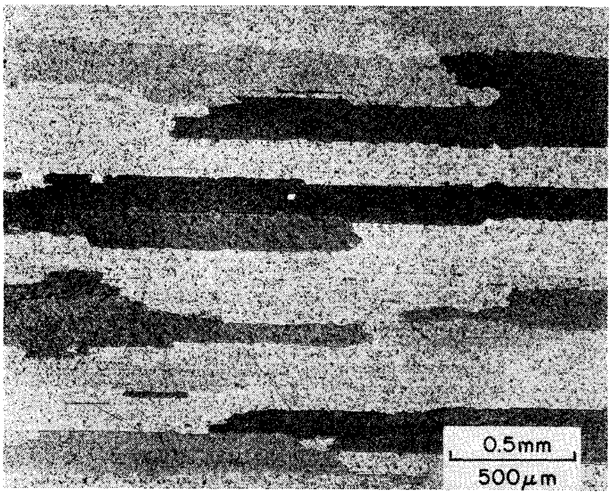

Fig. 19 Longitudinal microstructure of INCONEL alloy MA 754 bar. Note high grain aspect ratio and irregular, serrated grain boundaries.

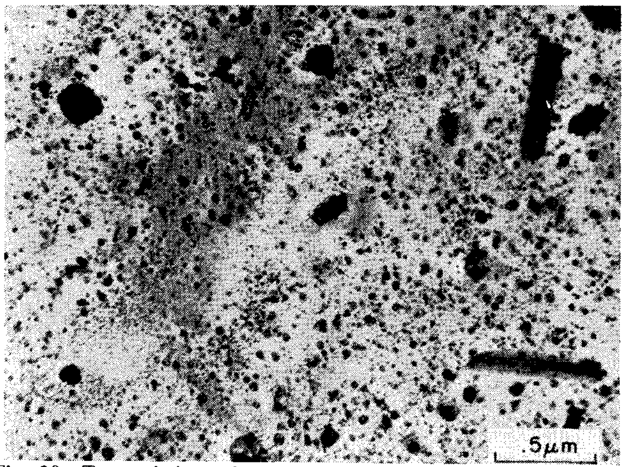

Fig. 20 Transmission microstructure of INCONEL alloy MA 754 showing uniform distribution of fine primary dispersion, presence of coarser carbonitrides, and microtwins.

Table 9 2012°F (1100°C) cyclic oxidation resistance of INCOLOY alloy MA 956E

Test time: 504 hr
Atmosphere: air + 5%H_2O
Thermal cycle: to room temperature every 24 hr

	Weight change, mg/cm^2		Metal loss,	Maximum attack,
Alloy	Undescaled	Descaled	μm	μm
MA 956E	0.99	−1.57	<2	15
TDNiCR	−4.66	−12.52	20	33

Table 10 1700°F (927°C) burner rig sulfidation resistance of INCOLOY alloy MA 956E

Test time: 312 hr
Air/fuel ratio: 30/1
Fuel mix: JP-5 + 0.3% sulfur + 5 ppm seawater
Thermal cycle: 58 min at temperature, 2 min cool to room temperature

	Weight change, mg/cm^2		Metal loss,	Maximum attack,
Alloy	Undescaled	Descaled	μm	μm
MA 956E	1.04	−0.17	5	18
TDNiCr	−1.69	−11.57	25	129

Table 11 2000°F (1093°C) carburization resistance of INCOLOY alloy MA 956E

Test time: 100 hr
Atmosphere: H_2 + 2%CH_4

	Weight change, mg/cm^2		Metal loss,	Maximum attack,
Alloy	Undescaled	Descaled	μm	μm
INCOLOY alloy MA 956E	.51	.16	3	3
INCOLOY alloy 800	33.74	29.89	132	1620

dynamic hydrogen-2% methane atmosphere in also quite good (see Table 11). A commercial Fe-Ni-Cr alloy was included in this test for comparison purposes. Weight gain for MA 956E was well below 1 mg/cm^2, while the metal loss and depth of attack were only about 3 μm.

Other Properties

MA 956E has a very high melting point, 2700°F (1482°C), a relatively low density compared to competitive materials, 7.2 g/cm^3, and a relatively low thermal expansion coefficient. This spectrum of properties makes the alloy well suited for sheet applications such as aircraft gas turbine burner cans.

Alloy MA 755E

Alloy Design and Structure

The versatility and breadth of the mechanical alloying process are demonstrated by its ability to produce alloys as complex as the most advanced cast nickel-base superalloys designed for blading applications. The composition of one such material, MA 755E (30), is given in Table 12 and compared to the compositions of some widely used cast nickel-base superalloys.

MA 755E is based on an alloy development philosphy similar to that of the more sophisticated cast alloys, IN-738 and IN-792, in that it contains a critical balance of the refractory elements tantalum, molybdenum, and tungsten. IN-738 and IN-792 were designed to have improved corrosion resistance over earlier alloys 713C and IN-100 at similar strength levels.

The structure of alloy MA 755E is shown in a surface replica in Fig. 23. Note the high volume fraction of γ' (45-50 v/v%) and the very fine dispersoid particles present in both the γ' (dark irregular particles) and lighter matrix. As was the case for the other mechanically alloyed superalloys, MA 755E also contains a secondary dispersion of somewhat coarser carbonitrides.

Stress Rupture Properties

The elevated temperature properties of alloy MA 755E in terms of the stress for 1000-hour life as a function of temperature[31] are given in Fig. 24 and compared with those of IN-792 and thoriated nickel bar. This diagram clearly shows the effect of the two strengthening mechanisms in alloy MA 755E. At intermediate temperatures, around 1450°F (800°C), the strength of alloy MA 755E is comparable to that of a complex and highly alloyed material such as IN-792 and nearly three times that of a simple dispersion strengthened metal such as thoriated nickel. This is due to the presence of γ' formed by aluminum and titanium in MA 755E. At very high temperatures, 2000°F (1093°C), where a conventional nickel-base superalloy such as IN-792 has lost the majority of its strength due to growth and dissolution of its γ' precipitate, alloy MA 755E has useful strength due to the presence of the oxide dispersion. At temperatures between these two extremes, the strength of alloy MA 755E is superior to either the cast complex nickel-base superalloy or the dispersion-strenthened metal because the two strengthening mechanisms supplement one another.

Chemical Properties

The hot corrosion and oxidation resistances of alloy MA 755E[32] are compared with those of cast alloys in Table 13. The sulfidation resistance of alloy MA 755E is somewhat better than that of IN-738, which was specifically developed

Table 12 Nominal compositions of alloy MA 755E and other complex nickel-base superalloys

	Weight, %, balance nickel												
	Al	Ti	Ta	V	Nb	Cr	Alloy Mo	W	Co	C	Zr	B	Y_2O_3
alloy MA 755E	4.5	3.0	2.5	–	–	15.0	3.5	5.5	–	.05	.15	.01	1.0
IN-792	3.0	4.5	4.0	–	–	12.4	2.0	3.8	9.0	.12	.10	.02	–
IN-738	3.4	3.4	1.75	–	.9	16.0	1.75	2.6	8.5	.17	.10	.01	–
IN-100	5.5	4.7	–	1.0	–	10.0	3.0	–	15.0	.18	.06	.014	–
alloy 713C	5.9	.6	–	–	2.0	12.0	4.5	–	–	.05	.10	.01	–

Table 13 Hot corrosion and oxidation resistance of alloy MA 755E

Alloy	100 hr sulfidation rig test 1700°F (927°C) 5 ppm seawater-0.3%S-JP5 Maximum attack, μm	504 hr cyclic oxidation test 2012°F (1100°C) air-5% H_2O Metal loss, mg/cm^2
alloy MA 755E	76	21
IN-738	102	80
IN-100	3302	–
alloy 713C	–	20

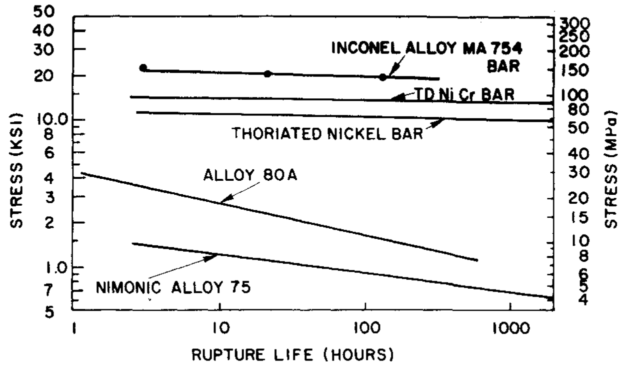

Fig. 21 2000°F (1093°C) Stress rupture properties of INCONEL alloy MA 754 compared to other nickel-based bar materials.

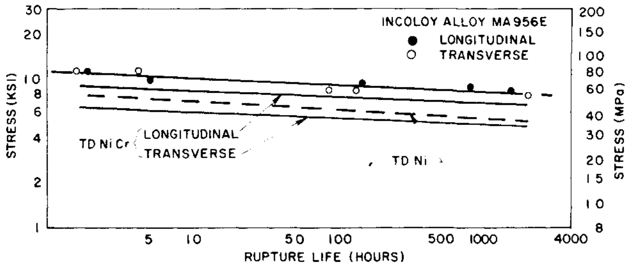

Fig. 22 Stress rupture properties of INCOLOY alloy MA 956E sheet at 2000°F (1093°C).

for this property, and considerably superior to that of IN-100. At the same time, alloy MA 755E possesses cyclic oxidation resistance comparable to that of alloy 713C and significantly better than that of IN-738.

Alloy MA 755E has used the full flexibility of the mechanical alloying process to design simultaneously for a variety of critical material properties. This capability is aided by the reliance on an oxide dispersion for very-high-temperature properties, permitting more freedom in matrix composition design for lower-temperature mechanical and/or chemical properties. A simplified analysis of the effects of the use of MA 755E for advanced aircraft gas turbine blading suggests that significant increases in turbine entry temperature and engine performance could be permitted by this type of material.[33]

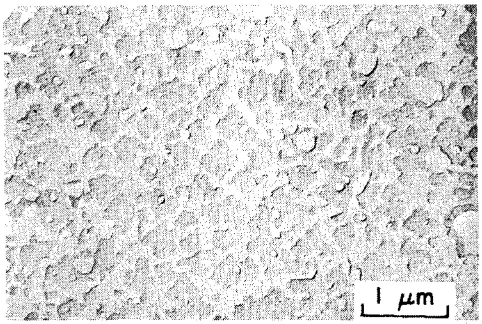

Fig. 23 Structure of fully heat-treated alloy MA 755E showing high-volume fraction γ' and dispersoid phases.

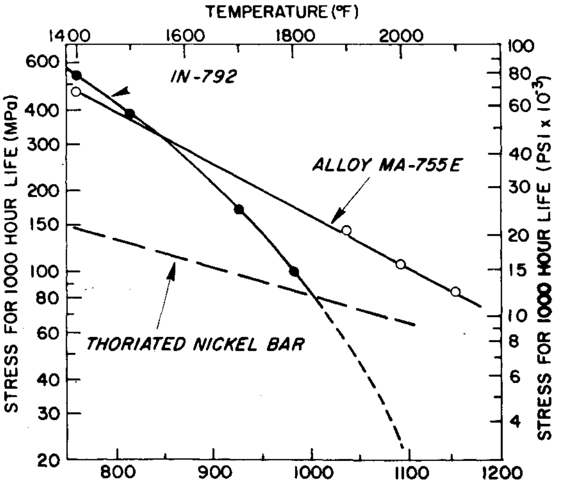

Fig. 24 Stress for 1000-hour life as a function of temperature in alloy MA 755E, IN-792, and thoriated nickel bar.

Summary and Conclusions

P/M technology has been applied to superalloys for the same reasons that it has been applied historically to other systems—to improve the quality of the product, to achieve unique properties, and to effect cost savings. *Conventional powder metallurgy superalloys* offer improved homogeneity and hot-workability over their cast and wrought counterparts in uses such as turbine discs. Research has shown that these alloys can be specifically modified to be produced by powder metallurgy to great advantage. A newer development, *Thermoplastic Processing*, has been applied to powders to further enhance their hot-workability. It appears to offer an excellent chance to achieve the long-sought goal of consolidation to near final shape for P/M superalloy parts. Finally, a variety of oxide-dispersion-strengthened superalloys have been produced by the *Mechanical Alloying* process. These alloys – INCONEL alloy MA 754, INCOLOY alloy MA 956E, and alloy MA 755E – represent materials of progressively increasing complexity which offer potential advantages in aircraft turbine vane, burner can, and turbine blading applications.

References

[1] Poyner, G. T., Tracey, V. A., and Watkinson, J. F., "Sintered High Temperature Alloys," *Powder Metallurgy*, edited by W. Leszynski, Interscience Publishers, New York, 1961, pp. 701-729.

[2] German Patent Applications 2,103,875 and 2,108,978.

[3] U. S. Patent 3,510,546.

[4] U. S. Patent 3,099,041.

[5] Gessinger, G. H. and Bomford, M. J., "Powder Metallurgy of Superalloys," *International Metallurgical Review.*, Vol. 19, June 1974, p. 51.

[6] Peterson, G. P., "Keynote Speaker," presented to AGARD Structures and Materials Panel at the 42nd Meeting, April 4-9, 1976.

[7] Larson, J. M., "Carbide Morphology in P/M IN-792," *Metallurgical Transactions*, Vol. 7A, Oct. 1976, p. 1097.

[8] Larson, J. M., "P/M IN-100-Processing, Microstructures and Mechanical Properties," *Modern Developments in P/M*, Vol. 8, 1974 ('73 International Powder Metallurgy Conference).

[9] Freche, J. C., Walters, W. J., and Ashbrook, R. L., "Evalution of Two Nickel-Base Alloys, Alloy 713C and NASA TAZ-8A, Produced by Extrusion at Prealloyed Powders," NASA TN D5248, March 1969.

[10] Friedman, G. I. and Loewenstein, P., "Processing Techniques for the Extrusion of Superalloy Powders," AFML-TR-68-321, Oct. 1968.

[11] Kotval, P. S., "Superalloy by Powder Metallurgy for Use at 1000-1400°F," NASA CR-72644, Dec. 1969.

[12] Friedman, G. I., "The Powder Metallurgy of Five Nickel Superalloys," Whittaker Co., Nuclear Metals Div., West Concord, Mass., Jan. 1971.

[13] Holt, R. T. and Wallace, W., "Impurities and Trace Elements in Ni-Base Superalloys," *International Metallurgical Review*, Vol. 21, March 1976, pp. 1-24.

[14] Sims, C. T., "A Contemporary View of Ni-Base Superalloys," *Journal of Metals*, Vol. 18, Oct. 1966, p. 1119.

[15] Larson, J. M. and Floreen, S., "Improving the Crack Growth Resistance of a Superalloy," *Metallurgical Transactions*, (accepted for publication).

[16] Barker, J. F. and VanDerMulen, E. H., "Effect of Processing Variables on P/M René 95," *Superalloys-Processing*, Metals and Ceramics Information Center, Columbus, Ohio, MCIC-72-10, Section A-A, Sept. 1972.

[17] U. S. Patent 3,519,503.

[18] U. S. Patent 3,930,841.

[19] Symonds, C. H., "Ni Superalloy Production and Fabrication to Turbine Discs," presented to AGARD Structure and Materials Panel at the 42nd Meeting, April 4-9, 1976.

[20] Allen, M. M., Athey, R. L., and Moore, J. B., "Application of P/M to Superalloy Forging," *Metals Engineering Quarterly*, Vol. 10, Jan.-March 1970, pp. 20-30.

[21] Morris, C. A. and Smythe, J. W., "Comparison of Astrology Powder Consolidation Processes Forging and Testing of Astrology Powder Billet," *Superalloys-Processing*, p. Y, MCIC Report No. 72-10, 1972.

[22] Ewing, B., Rizzo, F., and Zur Lippe, C., "P/M Products for Advanced Gas Turbine Applications," *Superalloy-Processing*, p. BB, MCIC Report No. 72-10, 1972.

[23] Larson, J. M., Thompson, F. A., and Gibson, R. C., "T/P Processing-An Advanced P/M Superalloy Technique," presented at 3rd International Symposium on Superalloys, Sept. 12-15, 1976.

[24] Benjamin, J. S., "Dispersion Strengthened Superalloys by Mechanical Alloying," *Metallurgical Transactions*, Vol. 1, Oct. 1970, pp. 2943-2951.

[25] Benjamin, J. S. and Bomford, M. J., "Effect of Yttrium Oxide Volume Fraction and Particle Size on Elevated Temperature Strength of a Dispersion Strengthened Superalloy," *Metallurgical Transactions*, Vol. 5, March 1974, pp. 615-621.

[26] Benjamin, J. S. and Morse, J. P., "Mechanical Alloying," *New Trends in Materials Fabrication*, American Society for Metals, (to be published).

[27] Benjamin, J. S., Volin, T. E., and Weber, J. H., "Dispersoids in Mechanically Alloyed Superalloys," *High Temperature-High Pressures*, Vol. 6, 1974, pp. 443-446.

[28] Morse, J. P., Huntington Alloys, Inc., Huntington, W. Va., private communication, 1976.

[29] Fischer, J. J., Astley, I., and Morse, J. P., "The Structure and Properties of a Dispersion-Strengthened Fe-Cr-Al Alloy, INCOLOY alloy MA 956E," *Proceedings of the Third International Symposium on Superalloys*, American Institute of Metallurgical Engineers, (to be published).

[30] Benjamin, J. S. and Schultz, J. W., "High Strength Corrosion Resistant Nickel-Base Alloy," U. S. Patent 3,926,568, Dec. 16, 1975.

[31] Curwick, L. R., The International Nickel Company, Inc., Paul D. Merica Research Laboratory, Suffern, N. Y., private communication, 1975.

[32] Schultz, J. W. and Hulsizer, W. R., "Laboratory Development of Corrosion Resistant Nickel-Base Superalloys for Gas Turbines," *Proceedings of Gas Turbine materials in the Marine Environment Conference*, edited by J. W. Fairbanks, and I., Machlin, Metals and Ceramics Information Center, Columbus, Ohio, MCIC 75-27, 1975, pp. 335-356.

[33] Benjamin, J. S., "The Potential of Oxide Dispersion Strengthened Superalloys for Advanced Gas Turbines," American Society of Mechanical Engineers, Gas Turbine Division, Pamphlet 76-GT-118, 1976.