T/P PROCESSING - AN ADVANCED P/M SUPERALLOY TECHNIQUE

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ABSTRACT

An economical route for the manufacture of APK1 (a low carbon P/M superalloy) gas turbine discs involving atomization, powder rolling, canning, consolidation, and forging is described. Considerable emphasis is placed on T/P processing by powder rolling which imparts strain energy to the powder. The strain energy resulting from this contamination-free process causes grain refinement as the powder is heated for consolidation. Argon atomization efficiency is substantially improved by powder rolling, since virtually the entire powder output can be utilized. Hot isostatic compaction of the T/P powder can be achieved at lower temperatures and pressures, and, if desired, the as-HIPed compact can be isothermally forged (i.e., superplastic or slow strain-rate pressing). The ability to consolidate T/P processed powder at lower temperatures also reduces the amount of interstitial phases which form on the powder surface during heating of the powder. The continuous nature of these phases in P/M billet can otherwise lead to poor forgeability and mechanical properties in the HIPed billet. The fine grained condition of the as-HIPed billet made from T/P powder also aids conventional forging or extrusion by lower stresses.

Introduction

P/M production techniques for superalloys were developed to reduce segregation in these highly alloyed, high strength materials, thus making them amenable to hot working. Problems with hot working occurred as the need for stronger materials grew. Superalloys with continually higher volume fractions of γ' were developed, culminating in the mid-1960's with IN-100 which contains 60% γ'. The combination of a high volume fraction of γ' with a highly strengthened matrix makes IN-100 virtually impossible to hot work using conventional forging techniques. Thus, use of this attractive material was limited to relatively small cast parts such as blades for gas turbines.

A P/M technique called thermoplastic processing (T/P) will be described which, when integrated into P/M technology, offers several technical advantages that are expected to greatly improve the economics of P/M superalloys.

T/P Processing Concept

T/P processing is a technique by which superalloy powders can be consolidated and formed at significantly lower flow stresses and temperatures than with conventional P/M practices (U.S. Patent 3,865,575). This is a result of cold working the powder such that its grain size is greatly reduced on recrystallization. The finer grain size of the T/P powder makes it considerably softer at elevated temperatures, thus facilitating consolidation. Imparting strain energy to the powder can be accomplished in many ways, ranging from the use of a mortar and pestle to a rolling mill. Three T/P processing techniques, ball milling, attriting and rolling, will be compared.

T/P Processing Methods

Ball Mill

A ball mill can be used to impart strain energy to a powder by entrapping the particles between the colliding balls. The amount of strain energy imparted is a complicated function of the momentum of the colliding balls, the roughness of the ball surfaces, the number of particles trapped, and the size and hardness of the powder. Upon heating, the material adjacent to the powder surface recrystallizes to a fine grain size. The grain size of material at the core of the powder particle, in this case, is unchanged from that of the as-atomized condition (see Figure 1).

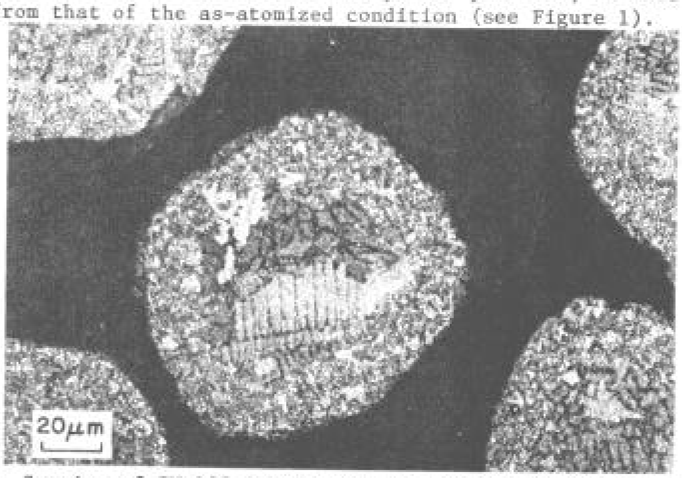


Figure 1: Section of IN-100 Powder Ball Milled for 50 Hours and Heat Treated at 1038°C for One Hour

Each of the powders shown in Figure 1 has been involved in many collisions but only the material near the powder surface has been sufficiently strained to recrystallize.

Attritor

The attritor is a very effective device for imparting strain energy to metal powder. This occurs by entrapment of powder particles between grinding balls, as in conventional ball milling; however, the kinetic energy in attritor processing is considerably higher. The powder particles are severely deformed into irregular particles. The result, upon heating, is a fine grain size throughout the powder particles, as shown in Figure 2.

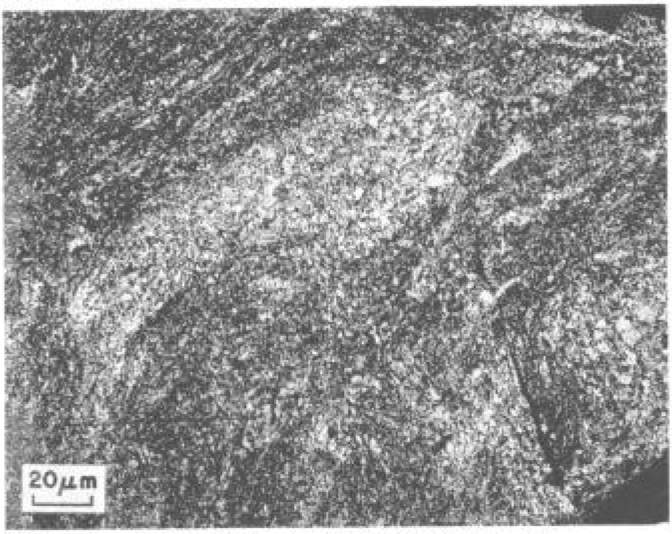


Figure 2: Section of IN-100 Powder Attrited for 10 Hours and Heat Treated at 1038°C for One Hour

Because of the statistical nature of this collision deformation process and the wide variety of powder sizes, several hours of processing are required before all the powder has been sufficiently strained to recrystallize to a fine grained condition.

Rolling Mill

T/P processing can be accomplished more efficiently by passing the powder through a vertical feed rolling mill, shown in Figure 3. This experimental mill is preferably equipped with carbide sleeves to minimize wear and achieve a uniform powder thickness. The spherical powder is rolled into discs by passing once through the mill. An extremely fine grain size is achieved in the powder upon heating (see Figure 4). In order to achieve the finest grain sizes on recrystallization, the particles should be deformed at least 40% in thickness. If it is desired to have a completely uniform recrystallized grain size in all powder sizes, the atomized powder is cut into three to five mesh size ranges and roll each mesh size range separately. The smaller argon-atomized powder particles (typically -240 mesh) have grain sizes fine enough to exhibit superplasticity in the as-atomized condition and do not require T/P processing. The powder is then blended.

Results of conventional ball milling, attritor processing and rolling show that interstitial pickup in T/P processing is highest in the attritor and in ball mill processed material. This is shown in Table 1.

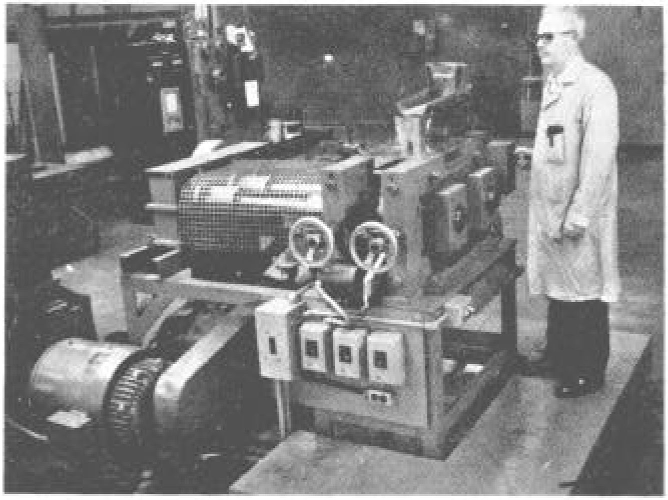


Figure 3: Experimental Vertical Feed Rolling Mill

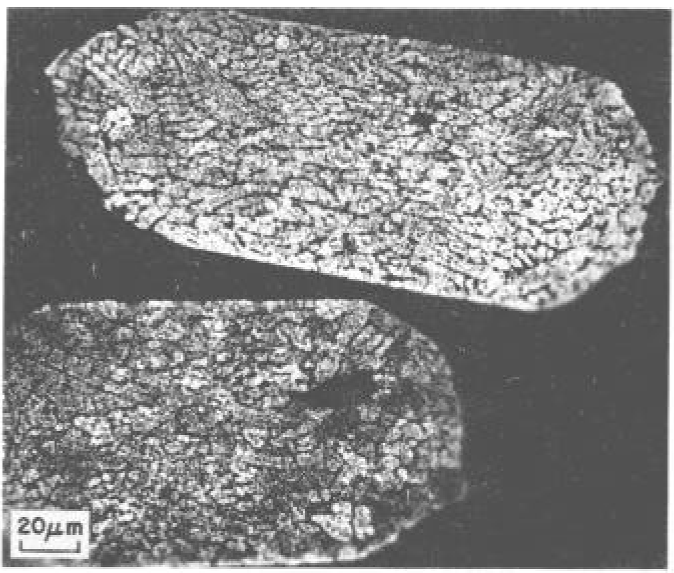


Figure 4: Section of Rolled IN-100 Powder Which Has Been Heat Treated at 1038°C for One Hour

Table I

Interstitial Pickup of IN-100 Powders During T/P Processing (Wt. %)

Attritor	O	N
Before Processing	.0059	.0014
After Processing for 10 Hours	.0275	.0074
Ball Mill		
Before Processing	.0105	.0010
After Processing for 50 Hours	.0290	.0033
Rolling		
Before Processing	.0050	.0012
After One Pass Through Rolls	.0060	.0012

Hot Hardness and Flow Stress Data

Hot hardness data on IN-100 (see Figure 5) shows that T/P processed powders are considerably softer than conventional P/M IN-100 at elevated temperatures. The composition of IN-100 is shown in Table II.

Table II

Composition of Materials Used in This Study (Wt. %)

	Cr	Co	Mo	W	Ta	Al	Ti	B	C	Ni	Fe	Zr
IN-100	10	15	3	--	--	5.4	4.5	.014	.06	Bal.	--	.06
IN-792	12.5	10	2	3.9	4	3.2	4.3	.017	.04	Bal.	--	.10
IN-744	26	--	--	--	--	.01	.2	--	.06	6.6	Bal.	--
APK1	15	17	5.0	--	--	4.0	3.5	.02	.025	Bal.	--	.05

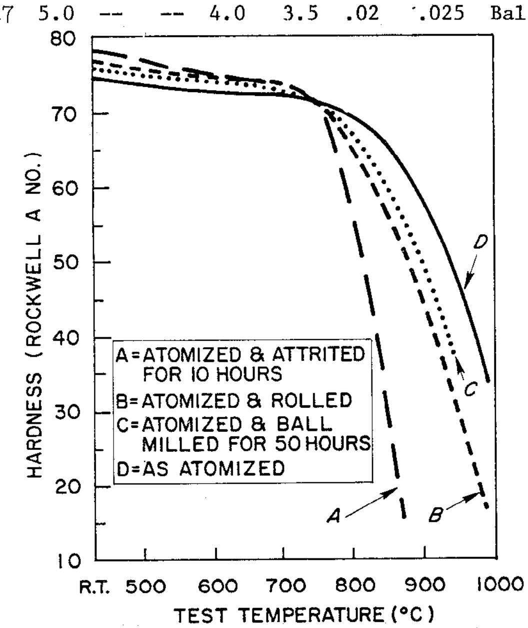


Figure 5: Effect of Temperature on Hardness (R_A) of Consolidated IN-100 Powder Prepared by Various Techniques

IN-100 compacts of as-atomized, ball milled, rolled, and attrited powders were canned and consolidated against a blank die at 1308°C, using a 750-ton extrusion press. Hardness measurements were made between room temperature and 982°C. Below 760°C the T/P processed materials are harder than as-atomized powder. The reverse is true above 760°C. This effect is related to grain size. The attrited material has the finest grain size (.4 to 2μm), followed by the rolled powder (1 to 6μm), ball milled powder (predominantly 60μm) and finally, the as-atomized powder (predominantly 70μm). This same ranking in hot hardness at a given temperature above 760°C is apparent in Figure 5.

Table III shows that flow stress for the T/P processed materials is lower than that of the as-atomized powder at 1038°C and .01 $min.^{-1}$ strain rate. The ranking of flow stresses is consistent with the hot hardness results above 760°C. Note that the flow stress of the attrited and rolled powder materials is only about one-half of that of the conventional P/M IN-100.

Table III

Flow Stress of T/P Processed IN-100 Powder Compacts at 1039°C at 0.01 Min^{-1} Strain Rate

Processing	Flow Stress ksi/MPa
Atomized Powder	9.8/68
Ball Milled for 50 Hours	8.2/56
Rolled	5.2/36
Attrited for 10 Hours	4.8/33

Experiment Results of Processed Superalloy Powder

Enhanced Extrudability

The extremely fine grain size of T/P processed powder (processed in this case in an attritor) greatly increases the attainable extrusion ratio. Table IV shows that extrusion ratios as high as 50 to 1 were obtained on IN-100 powder at 1120°C. Conventional P/M material would be difficult to consolidate completely using a 16:1 ratio at this temperature. Following extrusion, the 50:1 material was found to be fully dense. When given an 1177°C/4 hrs/OQ, 650°C/24 hrs, 760°C/8 hrs heat treatment, this T/P processed material exhibits mechanical properties which are virtually identical to P/M IN-100 consolidated by extrusion, as shown in Table V. The room temperature tensile properties of the T/P material are above those of the conventionally processed P/M IN-100, while the 704°C properties are slightly lower. This latter effect is attributed to a finer grain size in the T/P material. The smooth bar stress rupture lives of the T/P IN-100 at 732°C/689 MPa ranged from 39 to 85 hours, which is above the 30-hour requirement, and the material is notch ductile with a $K_t = 2.0$.

Table IV

Extrudability of Thermoplastic (Attritor) and Conventional P/M IN-100 Powder

Powder	Extrusion Ratio	Extrusion Temp. (°C)
As-Atomized	16:1	1149
T/P Powder (Attritor)	50:1	1121
	32:1	1066
	8:1	1038

Table V

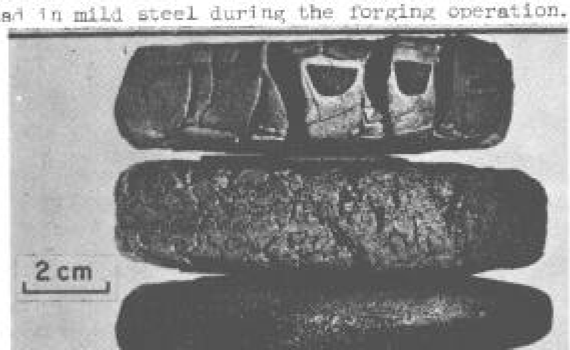
Comparison of T/P and Conventional P/M IN-100

Mechanical Property*	P/M IN-100 (ksi/MPa)	T/P IN-100 (ksi/MPa)
UTS at RT	215/1480	230/1585
.02% YS at RT	150/1030	165/1140
Elong. at RT	10%	17%
RA at RT	10%	15%
UTS at 704°C	190/1310	186/1280
.02% YS at 704°C	155/1070	155/1070
Elong. at 704°C	10%	10%
RA at 704°C	10%	12%
Time for 0.2% Creep at 704°C/606 MPa	150 Hrs.	230 Hrs.
Stress Rupture Life at 732°C/689 MPa	30 Hrs.	39-85 Hrs.
Notched to Smooth Rupture Life (K_t = 2.0)	>1.0	>1.0

*With 1177°C/4 Hrs/OQ, 650°C/24 Hrs, 760°C/8 Hrs.
T/P powder produced in attritor.

Improved Forgeability

T/P processing of IN-100 powder also improved the upset forgeability of P/M IN-100 compacts. Containers of IN-100 powder in the as-atomized, ball milled, and attrited conditions were consolidated at 1065°C against a blank die in an extrusion press (∿827 MPa). The compacts were then conventionally press forged at a strain rate of about 2 to 10 cm/sec at 1093°C. Figure 6 shows the upset pancakes following acid leaching of the canning material. The as-atomized IN-100 disc shows severe radial cracking, while the attrited material shows only a few small surface cracks. It should also be noted that in this particular case, the attrited material was decanned prior to forging. No cracks at all were observed on the attrited material when it was clad in mild steel during the forging operation.



ATOMIZED POWDER

ATOMIZED & BALL MILLED FOR 50 HOURS

ATOMIZED & ATTRITED FOR 10 HOURS

Figure 6: P/M IN-100 Discs Forged at 1093°C from Various Types of Powder Compacted at 1065°C

Improved HIP to Near-Final-Form Capabilities

The fine grained structure of recrystallized T/P powder can be utilized to HIP superalloys to a near-final form. Using superplastic bag techniques, a stainless steel (IN-744) container was inflated into a typical gas turbine disc shape. A container before and after inflation is shown in Figure 7. One of the containers was filled with as-atomized IN-792* powder (-40 mesh) and another with T/P processed powder (-40 mesh) of the same composition.

*See Table II for composition.

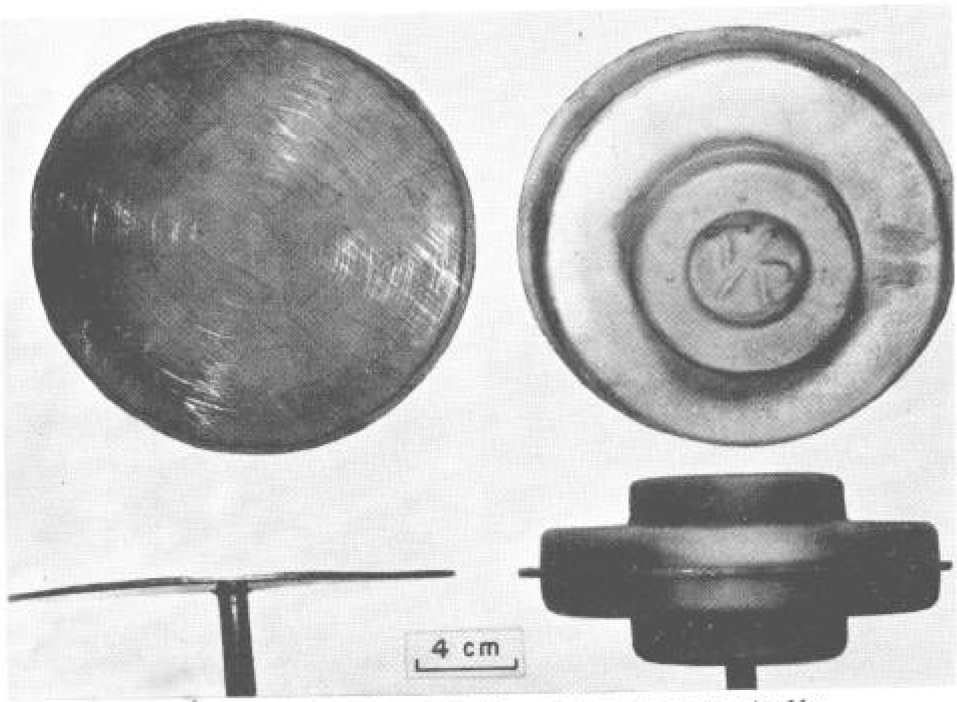
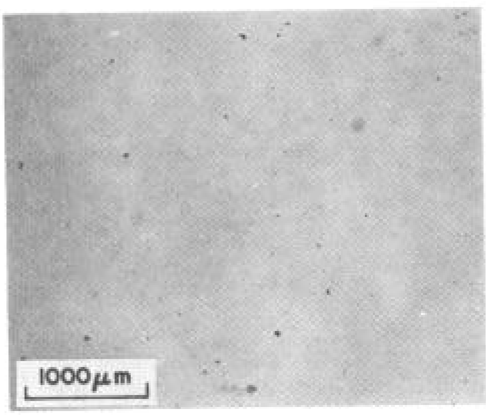
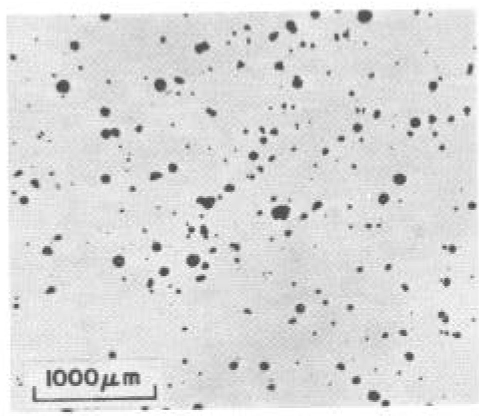


Figure 7: HIP Container Before and After Being Superplastically Formed into a Disc Shape

The cans were evacuated and hermetically sealed. The as-atomized powder was HIPed at 1180°C at 103 MPa; whereas, the T/P processed IN-792 powder was HIPed at 1070°C. The T/P processed material was fully dense. However, this was not the case for the container of as-atomized powder, as shown in Figure 8. Test specimens were cut from the T/P processed material and heat treated to develop a serrated grain boundary morphology, which has been shown to improve stress rupture properties. The results of these tests are compared with conventional P/M IN-792 data in Table VI. Both tensile and stress rupture properties are equivalent to those of P/M IN-792 consolidated by extrusion techniques.



(a)



(b)

Figure 8: IN-792 Powder HIPed at 15 ksi (103 MPa) for One Hour
(a) Atomized, T/P by Rolling, Canned and HIPed at 1070°C
(b)Atomized, Canned, and HIPed at 1180°C

Table VI

Mechanical Properties of Extruded and T/P + HIP P/M IN-792

	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 760°C/620 MPa	
Life (Hrs)	168	185
Elong. (%)	6.0	7.0
RA (%)	8.0	9.0

HT at 1200°C/1/3 hr/FC to 1163°C, AC; 843°C/16 hrs.

Pilot Scale Results on Processing of APK1

A production atomization and HIP facility has recently been commissioned at Henry Wiggin & Company, Ltd., Hereford, England, and work is being undertaken to explore the advantages of T/P processing in the production of APK1* discs. APK1 is a low carbon superalloy composition which has been developed specifically for the powder production routes. Initial work used attritors for processing the powders. Although excellent reductions in hot flow stresses were achieved, the final disc properties were affected by oxygen contamination from attrition. A small rolling mill was therefore modified and within the past year a semi-production scale rolling unit has been installed.

T/P processed APK1 powders have rather unique characteristics. Argon atomized powders contain spherical pores which contain argon. The rolling of powder reduces this entrapped argon and lends itself to a totally enclosed system which is utilized on the powder production and consolidation facility. The tap density of T/P powder was 62.5%, while that of the as-atomized powder was 60%. The flow rate for 50 g of the T/P powder was 21 seconds, and that for the as-atomized powder was 16 seconds. These results indicate that the canning characteristics of as-atomized and T/P powder should be very similar.

Both as-atomized and T/P processed powders have been processed through three basic routes, including HIP to direct shape, HIP + isothermal forging, and HIP + conventional forging.

Hot Isostatically Pressed and Heat Treated T/P APK1

The direct HIPing of shaped components appears to offer the greatest potential cost saving via the powder route. Typical as-HIPed plus heat treated properties of atomized material are shown in Table VII. Since T/P powder was found to have much reduced elevated temperature flow stresses, consolidation of the powder can be made to occur at lower HIP temperatures, thus enabling a partially recovered structure to be obtained in as-HIPed material. Typical tensile properties are given in Table VII and show improvements over as-atomized powder in 0.2% yield and tensile strength while retaining an acceptable level of ductility. The grain size for as-HIPed T/P materials was 1 to 1.5 microns, while for the as-atomized powder it was of the order of 50 microns. It has been shown that improved tensile and low cycle fatigue properties can be obtained by thermomechanical processing.

*See Table II for composition.

A more comprehensive survey is at present under way to determine the response to LCF and creep rupture.

Table VII

650°C Tensile Properties for HIPed As-Atomized and T/P APK1

Material	HIP Temp. (°C)	0.2% YS (ksi/MPa)	UTS (ksi/MPa)	Elong. (%)	R.A. (%)	NTS (ksi/MPa)
As-Atomized	1150	141/ 970	194/1338	25.0	23.8	249/1717
		141/ 975	195/1348	19.6	17.4	250/1722
T/P	1050	156/1077	199/1375	13.0	14.1	247/1700
		156/1080	199/1376	13.0	20.5	--

HT at 1080°C/4 Hrs/OQ, 650°C/24 Hrs/AC, 760°C/16 Hrs/AC.

Hot Isostatically Pressed Plus Isothermal Forging

T/P APK1 material was produced from -36, +100 mesh powder T/P processed via rolling and HIPed at 1050°C at 103 MPa. In the as-HIPed condition this material is superplastic because of its very fine grain size (1-1.5μm). Values of the strain rate exponent from 0.26 to 0.69 have been obtained from tensile tests performed at 1000 and 1050°C at strain rates of .002, .01 and .02 per minute. T/P processing therefore offers forgers superplastic billet which is limited in size only by the currently available hot isostatic presses. There is also the possibility of producing superplastic forging preforms of complex shapes.

A HIPed billet of T/P material was isothermally forged at 1050°C. Forging loads were approximately the same as for atomized material except in the superplastic temperature range where forging loads were decreased by up to 60%. Tensile properties at 650°C after the three-stage heat treatment were typical of as-atomized material isothermally forged below the γ' solvus temperature; i.e.:

0.2% YS	1097 MPa
UTS	1390 MPa
Elong.	14.3%
RA	16.3%

Hot Isostatically Pressed Plus Conventional Forging

Because of the availability of conventional forging equipment a large proportion of our powder development work has been aimed at this route. Small discs 20 cm in diameter have been produced by pressing on a 350-ton hydraulic press and finally stamped on a 750-ton screw press.

Comparing forgeability of as-atomized and T/P billet, it was immediately evident that the T/P material required much lower forging loads. On average, a 30% reduction over as-atomized material was obtained. Along with the reduced forging loads, it was found possible to reduce the number of upsets and that the T/P material offered superior die-filling on the final die forging operation.

Typical properties of forged discs are shown in Table VIII. More recent results indicate that by optimizing HIP, forging, and heat treatment variables, improvements can be obtained in the ductility and notched properties of T/P processed material.

Table VIII

650°C Tensile Properties of Conventionally Forged As-Atomized and T/P APK1 Discs

	As-Atomized		T/P	
	Radial Mid-Radius	Tangential Periphery	Radial Mid-Radius	Tangential Periphery
0.2% YS (ksi/MPa)	148/1019	152/1045	164/1132	162/1118
UTS (ksi/MPa)	198/1362	202/1395	202/1394	201/1384
Elong. (%)	22.8	25.0	17.4	10.9
RA (%)	27.9	28.3	15.3	12.9
NTS (ksi/MPa)	262/1809	264/1824	243/1676	230/1583

HT at 1080°C/4 Hrs/OQ, 650°C/24 Hrs/AC, 760°C/16 Hrs/AC

Applicability

T/P processing is applicable to any metal or alloy powder which is difficult to hot consolidate and can be recrystallized to a finer grain size. It is expected to be an aid in the fabrication of P/M tool steel and titanium alloy components. T/P processing can also be applied to irregular powder particles such as water atomized stainless steel powders.

Conclusions

The experimental findings of this study have shown that the workability of T/P processed superalloy powders is substantially improved during extrusion and forging over conventional P/M materials. T/P processing also can be used to HIP superalloy powders to a sonic shape and obtain good mechanical properties. T/P processing is potentially useful to HIP to final shape when adequate non-destructuve testing techniques are available.

Pilot scale results on APK1 confirm the potential for T/P processing in presently envisaged disc processing routes. Along with the ease of forging and improvements in mechanical properties, there are other advantages which have not yet been exploited fully including reduction in size of non-metallic inclusions, decreased screening of powders, and opportunity for better structural control.