SUB-SOLIDUS HIP PROCESS FOR P/M SUPERALLOY CONVENTIONAL BILLET CONVERSION

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Abstract

Recent studies have identified thermo-mechanical conditions for powder UDIMET®alloy 720[1] which resulted in significantly higher ductility of HIP compacts[1]. In this process, the compact is produced by hot isostatic pressing (HIP) at temperatures just below the solidus of the alloy (slightly sub-solidus HIP or SS-HIP). The workability of the resulting as-HIPed material is high enough to allow billet conversion using conventional forging. This new process has the potential of reducing the cost of powdered metal (P/M) billet manufacturing. The results of the present study provide some insight on the mechanisms leading to the enhanced forgeability of the SS-HIP processed UDIMET alloy 720. In addition, a full-scale trial has shown that this new processing route has potential application for future production of high performance turbine disks, with equivalent microstructural and mechanical properties as parts manufactured with the conventional compact plus extrude plus isothermal forging route.

Introduction

UDIMET alloy 720 is a high strength nickel base

[1] UDIMET is a registered trademark of Special Metals Corporation

superalloy currently produced in cast and wrought and P/M form for turbine disk applications. UDIMET alloy 720 was developed originally as a wrought turbine blade material for power plant turbines[2]. A new generation of the alloy was developed in order to increase its high temperature stability and fatigue resistance to make it suitable for turbine disk applications. The modified cast and wrought alloy, UDIMET alloy 720LI, has since been recognized as having outstanding strength and fatigue resistance compared to other advanced turbine disk materials[3].

UDIMET alloy 720LI is one of the most highly alloyed cast and wrought disk materials, making the production of billet larger than 6.5" diameter very challenging. On the other hand, the powder metallurgy route allows the production of very homogeneous material, without the segregation caused by melting and re-melting processes. The grain size of P/M material is in general finer and more uniform than cast and wrought material, resulting in better defect detection capability by ultrasonic inspection. In addition, the P/M version of the alloy in the compact plus extrude plus isothermal forge form offers greater microstructural flexibility and control during post forging heat treatments[4].

Powder metal superalloy billets are conventionally processed by hot compaction of powder in sealed cans followed by an extrusion process. During the hot compaction process, phases precipitate at the particle surface, and the resulting continuous second phase network or prior particle boundaries (PPB), was found to be responsible for the observed low ductility of the compact[3, 4].

The effects of PPB's in P/M superalloys have been extensively reviewed by Thamburaj et al.[5]. Their review showed that the PPB's particles form as a result of carbon segregation during consolidation and particle contamination at the PPB's. The PPB particles were identified as carbides, nitrides or oxy-carbo-nitrides, and the PPB particle volume fraction was found to depend greatly on the atomization and handling method. Alloy designers have attempted to reduce the amount of PPB precipitation by chemistry and HIP cycle modification in alloys such as Astroloy, but the benefit of putting the PPB particles in solution was offset by an increase in grain growth and reduced workability during final forging[6].

The detrimental effects of the PPB particles in the microstructure can be greatly reduced if the material goes through a heavy warm working process which provide high strains within the component to ensure the dispersion of the prior particle boundary phase network. Specialized extrusion processes were developed to induce the required large strains without extensive cracking. The extrusion process also develops a fine grain microstructure suitable for the slow strain rate, isothermal forging of high performance jet engine turbine disks[3].

One disadvantage of the conventional compaction plus extrusion route to manufacture P/M superalloys billet is its cost. The process calls for specialized equipment to provide the high tonnage necessary for extrusion, and the setup and maintenance of the specialized tooling requires significant costs relative to initial procurement, on-going maintenance and operations. Contrary to the compaction plus extrude consolidation route, the consolidation of powders using a hot isostatic pressing (HIP) route can be performed using a single piece process flow. In addition, HIP consolidation is readily available throughout the world with over 1,000 units operating[7].

Initial attempts to convert as-HIPed P/M superalloys on conventional press conversion equipment were unsuccessful due to poor workability of the as-HIPed material compared to ingot material, making conventional press forging with the available press speeds, reduction and thermal control not cost effective. Special Metals Corporation has recently identified thermo-mechanical conditions for powder UDIMET alloy 720 which resulted in significantly higher ductility of the HIP compact[1, 8]. The compact was produced by hot isostatic pressing at temperatures just below the solidus of the alloy (slightly sub-solidus HIP or SS-HIP). The SS-HIP temperature range is define as $T_{solidus} - T_{SSHIP} < 28°C$. The as SS-HIP material possessed enough workability to be processed using conventional press conversion equipment. A 3000kg, 50cm diameter, SS-HIP consolidated UDIMET alloy 720 ingot was successfully press forged in Special Metals Corporation forging facility in Dunkirk, NY, to produce a 16.5cm billet.

In order to understand the micro-mechanisms leading to the enhanced workability of the SS-HIP material, a study of the effect of HIP temperature on microstructure and mechanical properties was undertaken on sub-scale containers filled with -140 mesh gas atomized powder. In addition, material from the SS-HIP consolidated and press forged billet was sent to Ladish Co. Inc.. Forged components were produced by Ladish Co. Inc. to compare the properties of the SS-HIP UDIMET 720 material with conventional P/M and cast and wrought material.

Sub-scale study

Experimental Procedures

Lab-scale experiments consisted of a detailed study of the HIP temperature effects on microstructure and mechanical properties of the as-HIPed and HIP plus rolled UDIMET alloy 720. The composition of the alloy can be found in Table I.

Table I: UDIMET alloy 720 P/M composition

	Ni	Co	Cr	Al	Ti
Wt.%	57.59	14.4	16.15	2.55	4.9
	C	B	Zr	Mo	W
Wt.%	0.022	0.018	0.03	3	1.34

Sub-scale 304 stainless steel 4.6 cm diameter containers were filled with -140 mesh gas atomized powder. HIP was performed under 103.4 MPa pressure for 3 hours. The HIP temperature was varied from 1120°C (sub-solvus) to 1270°C (slightly below the solidus temperature of the alloy) with a controlled cooling rate of 0.8°C /min.

The microstructure was examined using optical, scanning and transmission electron microscopy techniques (SEM and TEM respectively). In order to observe the PPB's by

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optical microscopy, the material was first heat treated for 5 min. at 1170°C (just above the γ' solvus), removing most of the γ' precipitates which mask the fine PPB particles.

Extraction and analysis of the secondary phase particles were performed by Micro-Met Laboratories, Inc[2]. The extraction was done using a 10% HCl in methanol electrolytic solution. This solution dissolves the γ and γ' phases, leaving in the extracted residue the carbides, nitrides, borides, oxides and oxy-carbo-nitrides. Extraction of secondary phase particles from the HIP compact was performed on material which was solution heat treated for metallographic observation of the PPB networks. The particles in the extracted residue were observed using SEM techniques and phase identification was performed by X-ray diffraction.

Workability was evaluated by performing elevated temperature tensile tests (1093°C) at a strain rate of 2 sec^{-1} and measuring the reduction of area at failure. In addition to testing the as-HIPed material, HIP materials were rolled down and tested along the rolling direction to study the effect of microstructural refinement possible by press forging and its affect on workability. Roll down operations were performed at 1093°C , for a final height reduction of 30%.

Microstructural analysis

PPB particles could clearly be observed in the microstructure of the as-HIPed materials, Figure 1. The particles are believed to precipitate as a result of surface contamination during the atomization, and carbon segregation to the particle free surface in the first stage of the consolidation process[6]. The PPB particles formed a network of secondary phases in the consolidated material throughout the microstructure, following the location of the powder particle surfaces. In this study, the particle density on the PPB's was found to decrease with increasing HIP temperatures, Figure 1. This is in accord with the second phase extraction investigation, which found that the amount of residue collected decreased for increasing HIP temperatures.

The particle size in the collected extraction residue from the HIP trial materials was approximately 0.2 μm. The few particles extracted from the SS-HIP material were on average larger than for the other HIP conditions. The larger particles in the SS-HIP material were up to 0.5 μm diameter. Those larger particles are believed to be TiC which have precipitated inside the grains but not at the PPB's. X-ray diffraction performed on the residue
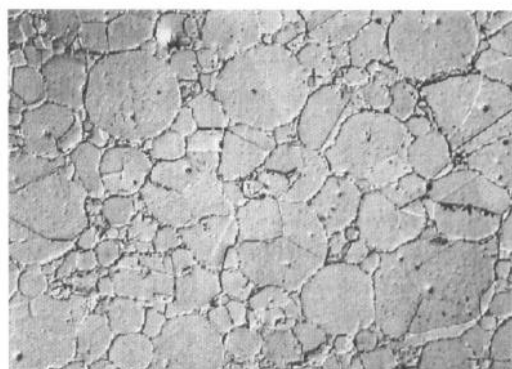
(a) 1120°C

(b) 1265°C

Figure 1: View of the PPB particle network and grain size after a 3 hour HIP cycle at (a) 1120°C and (b) 1265°C .

extracted from material HIPed at 1120°C (below solvus), 1230°C and 1265°C (SS-HIP condition) gave rise to diffraction patterns fitting MC carbides, and zirconium oxide ZrO_2 or possibly ZrCaTiO. The lattice parameter of the MC carbides was 42.8 nm. EDAX on the residue observed by SEM showed that the particles were enriched in Ti, indicating that the MC carbides should be TiC.

The TEM study of the microstructure of the as-HIPed materials showed discrete particles aligned along what could have been the free surface of a prior powder particle, Figure 2. No continuous phase or film was observed at the PPB's. Grain boundaries were not always associated with the particles. However, some particles were observed to pin grain boundaries and seem to impede grain growth. EDAX analysis performed in the TEM showed that some of the particles were enriched in Ti or in Zr, in accord with the extraction results. In the case of SS-HIP material, very few particles could be found in the TEM thin foils.

These results indicate that, in the HIP temperature range investigated, the majority of the particles forming on the PPB's were Ti rich MC carbides and Zr rich oxides.

Similar studies[9] performed on Rene' 95, a P/M nickel based superalloy containing 3.5 wt.% Nb and 0.05 wt.% Zr, have found Nb and Ti rich MC carbides and ZrO_2 oxides at the PPB's. The precipitation of Zr oxides rather than similar common superalloy oxides such as Al and Mg oxides could be due to a higher stability of the Zr oxides or to a faster segregation of Zr to the free surface of the powder particle in these alloys. As the HIP temperature increased, some second phase particles went into solution. It seems unlikely that the oxides, due to their inherent stability, could go in solution at the HIP temperatures investigated, but the MC carbides seem to be substantially dissolved.


(a)
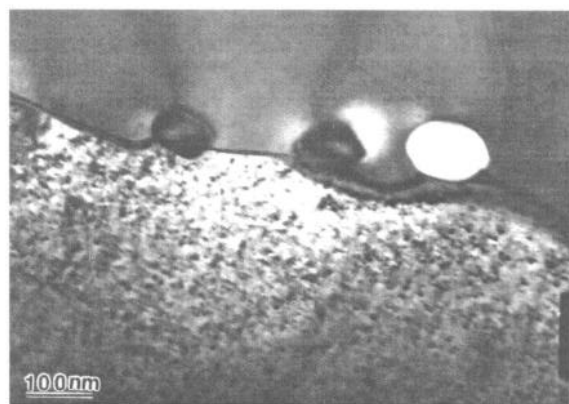
(b)

Figure 2: TEM bright field micrographs showing (a) the discret particles along the PPB's, and (b) particles pinning a grain boundary.

Figure 3 shows that the secondary γ' precipitates in material HIPed below the solvus temperature was much finer than the secondary γ' observed in material HIPed at temperatures above solvus. This is due to the very different precipitation conditions of the γ' phase in those two cases. For super solvus HIP, the γ' phase precipitates as the material cools slowly in the HIP unit at the end of the HIP cycle. This results in large γ' precipitates, occasionally forming a fanlike structure[10]. In the case

of sub-solvus HIP, very fine, well dispersed primary γ' precipitates in the supersaturated matrix quenched from the liquid during the atomization process, Figure 3.

The effect of HIP temperature on the grain size of P/M UDIMET alloy 720 is presented in Figure 4. The results suggest that grain growth occurs in stages, corresponding to temperatures where grain growth barriers, such as γ', nitride, carbides or oxy-carbo-nitrides, go in solution. The driving force for grain growth in the case of HIP should be primarily grain boundary surface minimization, as after consolidation, the isostatic pressure would not induce sufficient plastic strain to provide enhanced driving force for grain growth.
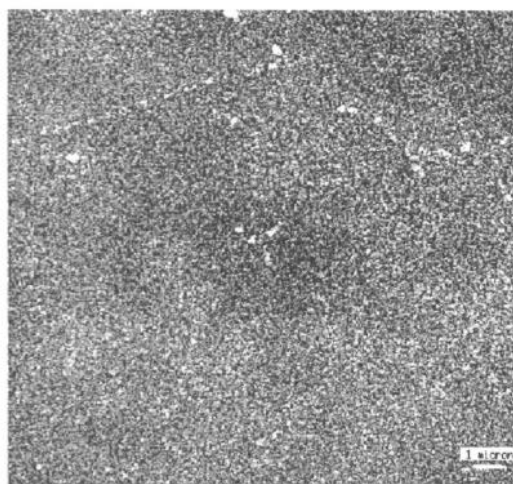
(a) 1120°C
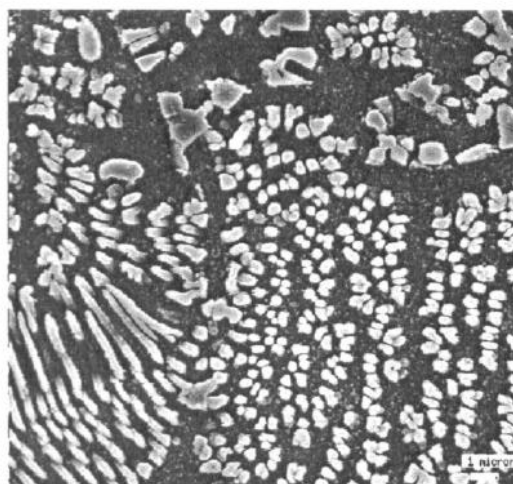
(b) 1265°C

Figure 3: γ' precipitates after HIP at (a) 1120°C and (b) 1265°C

Below the γ' solvus, grain growth was restricted within the prior particle by the γ' precipitates and the PPB

particles. The resulting grain size was in general smaller than the prior powder particles. Above γ' solvus, grains started to coarsen as a result of γ' solutioning but the PPB network was still present, preventing extensive grain growth. As the HIP temperature was increased, some of the PPB particles dissolved, and the grains were able to grow to larger sizes before being pinned again on clusters of larger or more densely packed PPB particles. It was only in the SS-HIP temperature range, where extensive dissolution of the PPB's network was observed, that grains coarsened clearly beyond several prior particles to reach ASTM 3-4, Figure 1b. The drastic increase in grain size in the SS-HIP temperature range indicates that the rate of dissolution of the MC carbides increased readily at those temperatures.

(a)

(b)

Figure 5: Effect of HIP temperature on (a) workability and (b) flow stress. Testing was done at 1093°C with a strain rate of 2 sec^{-1}.
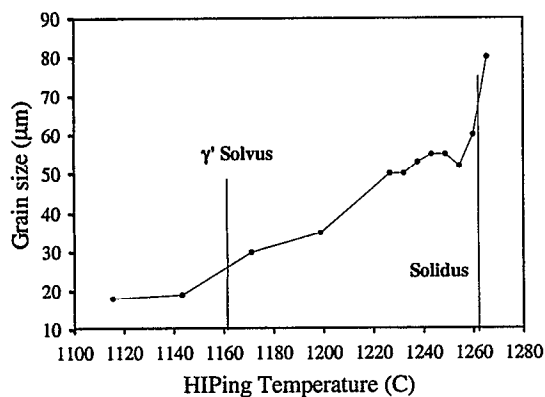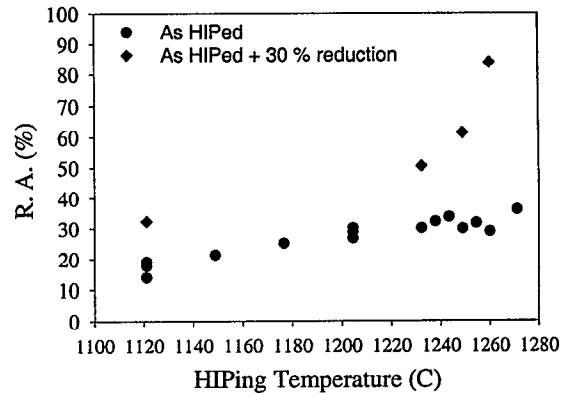

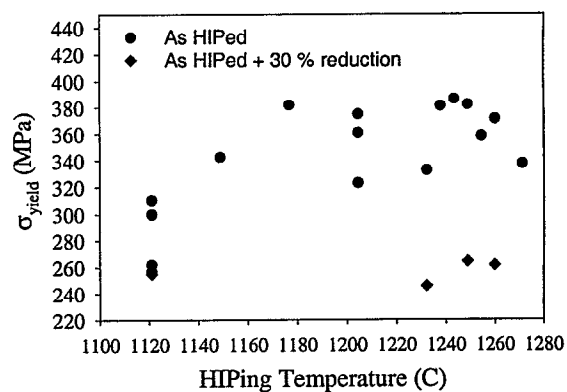Figure 4: Effect of HIP temperature on grain size of as-HIPed microstructure.

Aging treatments of the SS-HIP material were performed for 3 hours at temperatures below the SS-HIP temperature in order to study the eventual re-precipitation of the carbides at the PPB's. No new PPB particles re-precipitated in the microstructure after aging. However aging at temperatures above approximately 1200°C at atmospheric pressure created thermal induced porosity (TIP), i.e. the entrapped argon in the material during gas atomization and the consolidation expands and formed visible pores.

Mechanical testing

Figure 5 shows the evolution of the workability of the as-HIPed material as a function of HIP temperature. The workability at 1093°C increased steadily with the HIP temperature and reached 30% R.A. for HIP temperatures above 1230°C . For HIP temperatures above the solidus, the workability drops as incipient melted areas start to form at grain boundary triple points in the microstructure.

The fractured specimens were sectioned such that the fracture initiation sites under the fracture surface could be observed, Figure 6. Fracture initiated at voids found around PPB's particles in SS-HIP and non SS-HIP material. In the non SS-HIP material, the PPB particle network and the grain boundaries were in general close to each other as grain growth during HIP was contained by the PPB particles. As a result, occasionally a void initiated at a PPB particle could initiate a crack into a neighboring grain boundary. For SS-HIP material, the voids were generally isolated, coalescing with neighboring voids where the PPB's particle inter-spacing was low. Some voids were also observed at grain boundary triple points.

After rolling, the grain size (ASTM 11-12) was independent of HIP temperature and uniform. The workability increase that could be obtained by press forging was found to be highly dependent on the HIP temperature,

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Figure 5. For HIP temperatures just above the γ' solvus, the workability after rolling increased to just above 30%. However, as the HIP temperatures get closer to the SS-HIP condition, the workability of the press forged material increased steeply and exceeded 85% RA for the SS-HIP material. The 1093°C flow stress at high strain rate (2 sec^{-1}) of the as-HIPed and as-HIPed plus rolled material can be seen Figure 5. For HIP temperatures between the γ' solvus and the solidus, the flow stress of the as-HIPed and as-HIPed plus rolled material did not vary significantly. Rolling of the as-HIPed material reduced the flow stress in the rolling direction from approximately 350 MPa to 242 MPa.
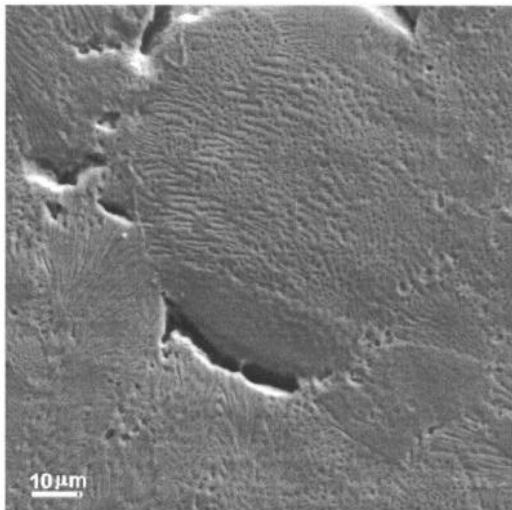

Figure 6: View of the fracture initiation sites at the PPB particle under the fracture surface for material HIPed at 1260°C and tested in tension at 1093°C with a strain rate of 2 sec^{-1}.

Workability is highly controlled by grain size, and usually increases as grain size decreases. In the case of P/M superalloys, the workability is also influenced by the PPB particles, at which voids initiate during high temperature and high strain rate deformation. In the case of P/M UDIMET alloy 720, as HIP temperatures increase, the grain size increases and the PPB particle volume fraction decreases. Figure 5 indicates that for increasing HIP temperature up to the solidus, the workability stayed unchanged at 30%, showing that the detrimental effect of grain growth on the workability was balanced by the beneficial dissolution of some of the PPB particles. After refining the microstructure by rolling, all the HIP materials had the same grain size, i.e. ASTM 11-12, and the workability would only be controlled by the PPB particles. This is indeed what can be observed Figure 5, where the workability increases with the HIP temperature, i.e. as the PPB particle volume fraction decreases.

For SS-HIP UDIMET alloy 720, despite a grain size of ASTM 3-4 in the as-HIPed condition, the material had sufficient workability to be thermo-mechanically processed using conventional forging techniques used to break down ingot structure. After some grain refinement, the workability of the SS-HIP material plus press forge was comparable to cast and wrought material. Those results indicated that a UDIMET alloy 720 SS-HIP powder can could be forged to produce a highly homogeneous and workable billet using conventional open die press forging equipment.

Large Scale Trials

A 3000 kg, 50 cm diameter SS-HIP consolidated UDIMET alloy 720 ingot was press forged in Special Metals Corporation forging facility in Dunkirk, NY. The billet was forged at 1080-1110°C to 17.5 cm using a direct draw conversion sequence. The sonic inspectability of the 16.5 cm diameter billet was tested and exhibited an ultrasonic noise level of 15-18% on a 0.4 mm diameter reference hole (#1FBH), a much better sonic response than what can be accomplished with a cast and wrought billet. The microstructure was very uniform, ASTM 10-11 grain size, across the billet cross section.

Small lot quantities were provided to Ladish in diameters of 16.5 and 23 cm for forging and characterization studies. Producing two billet diameters with a relatively small lot size represents a distinct advantage of the SS-HIP process relative to standard powder metal conversion by extrusion for which minimum lot sizes are quite large.

A desirable attribute of powder metal superalloys is the ability to tailor the microstructure over a large range of grain size. In this fashion, a single alloy can meet widely varied engine disk design requirements by altering the specific TMP cycle and subsequently the grain size. In contrast, cast and wrought materials have less grain size flexibility and often require working within a difficult or impractical process window to obtain desired grain size requirements. The grain coarsening response of the SS-HIP UDIMET alloy 720 material was investigated.

Ladish Co. Inc. performed controlled sub-solvus and super-solvus (1107°C - 1177°C range) thermo-mechanical processing trials in which a variety of grain size targets were successfully achieved in forged components. The results of this study showed that the SS-HIP material revealed good grain size flexibility and control. Table II shows grain size results from this study.

Table II: Grain size control evaluation of the SS-HIP processed billet

Target grain size ASTM, average	Resulted Grain Size ASTM (average, ALA)
	11, 5 (input billet)
4-6	4.5, 2
6-8	7, 5
8-10	9.5, 8
10-12	11, 9

Component Demonstrations

Large, complex, closed-die shapes were produced with the SS-HIP material and examined for forgeability, microstructure and mechanical properties. Two shapes that were produced are shown in Figure 7. These components were produced by isothermal forging to a uniform fine grain size requirement. The turbine disk shown in Figure 7a was further machined to a rectilinear shape and ultrasonic tested. The test showed substantially improved ultrasonic transparency when compared to the same part produced from cast and wrought UDIMET alloy 720LI material.

The forging shown in Figure 7b is a multi-part disk in which seven individual turbine disks are excised from one forging. This contoured near-net shape forging measuring approximately 70 cm. in diameter was produced in an efficient single-step isothermal forge process. The excellent chemical and microstructural homogeneity associated with the powdered metal product enabled an isotropic product on this non-axisymmetric forging[11].

The results previously shown in Table II reflect individual forgings each produced to a single microstructure, *i.e.* uniform grain size throughout the volume of the forging. The same grain coarsening behavior of the SS-HIP plus press forged product can also be locally applied and controlled to produce a component with dual microstructure and property characteristics. Figure 8 shows a cross-section of a disk that was processed to a dual microstructure. It is the combination of the forge and the post forge thermal practices that control the grain coarsening response.

Another example of a disk processed to provide a dual microstructure is shown in Figure 9. The component was solution heat treated such that the rim maintained a nominal temperature of γ' solvus + 28°C , and the bore maintained a nominal temperature of γ' solvus - 28°C . The disk was then stabilized at 760°C for 8 hours followed by an air cool, and aged at 649°C for 24 hours followed by an air cool. The forging was cross-sectioned and specimens were removed from the
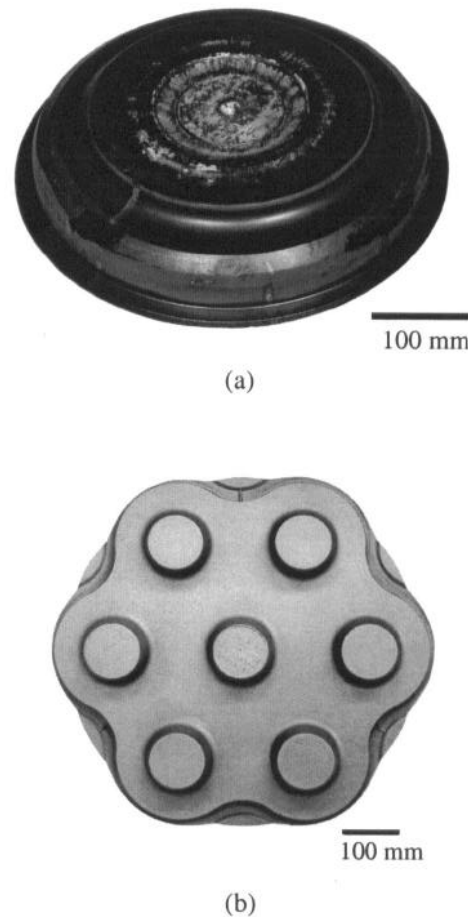

(a)

(b)

Figure 7: Closed die forging shapes were produced with SS-HIP processed material, (a) and (b).

coarse grain rim region and the fine grain bore region. The tensile properties for this P/M dual microstructure forging are shown on Table III, with a comparison to cast and wrought forgings also made into this forged shape.

A machineability trial was performed on SS-HIP plus press forged and cast and wrought billet utilizing a well established finish machine production plan at Rolls-Royce Allison. Two AE3007 second stage turbine disks from SSHIP plus press forged material were successfully machined to blueprint requirements as shown in Figure 10. The machineability of powdered metal U720 was shown to be better than cast and wrought due to a more homogenous chemistry and microstructure.

Table III: Average tensile properties from a turbine wheel forging utilizing SS-HIP and cast and wrought UDIMET alloy 720 billet processed to a dual fine grain (FG) and coarse grain (CG) microstructure.

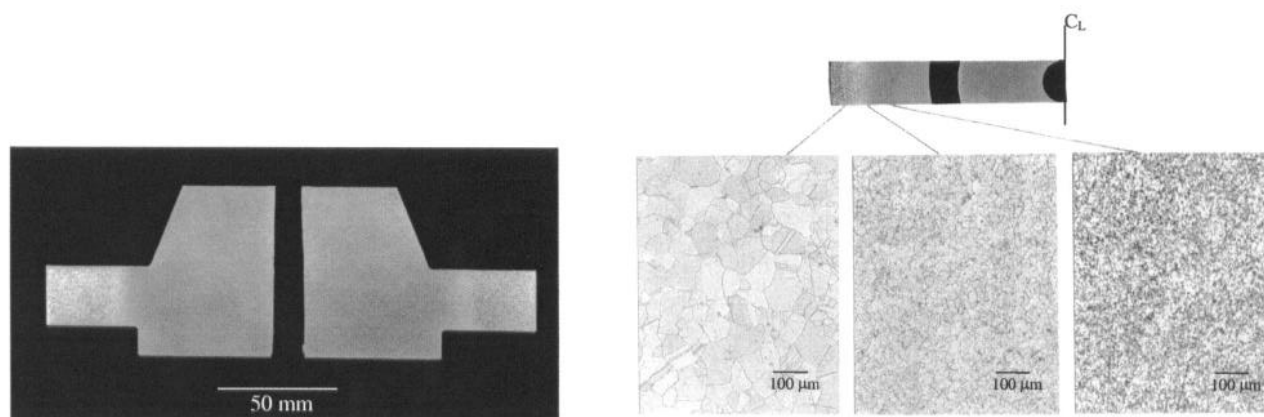| Billet	Grain size	Temp.	UTS (MPa)	σ_{Yield} (MPa)	Elong. (%)	R.A. (%)
SS-HIP	FG Bore	Room	1631	1190	22	23.5
SS-HIP	CG Rim	Room	1537	1082	21	25
SS-HIP	FG Bore	538°C	1564	1131	20.5	23.5
SS-HIP	CG Rim	538°C	1376	989	18.5	23
C & W	FG	Room	1634	1203	21	22
C & W	FG	538°C	1558	1128	19.5	24

Figure 8: A closed die forging shape produced with SS-HIP processed material and thermomechanically processed to a dual microstructure.
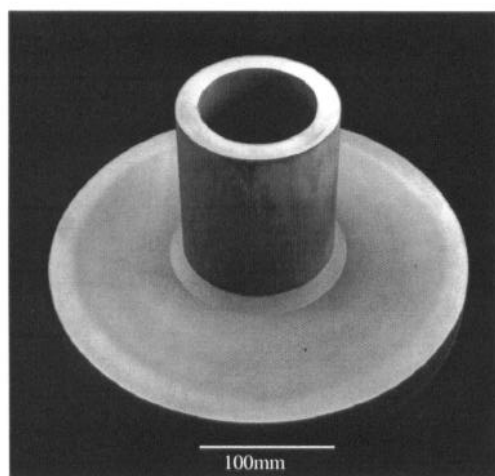

Figure 9: A turbine wheel produced with SS-HIP processed material and thermomechanically processed to a dual microstructure.

Figure 10: Finish machined turbine wheel, P/M SS-HIP + press forged input stock

Conclusions

1. In UDIMET alloy 720, a large fraction of the PPB particles formed during powder atomization and HIP can be eliminated by HIP in the temperature range $T_{solidus}-T_{SSHIP} < 28°C$, well within HIP unit capabilities.

2. SS-HIP powder metal UDIMET alloy 720 can be converted to billet using conventional press conversion practices.

3. SS-HIP material has superior ultrasonic inspectability compared to cast and wrought material.

4. Conventional press conversion practices employed with SS-HIP processed materials allow for increased production flexibility and reduced billet cost.

5. Grain size response of the SS-HIP processed material can be controlled using a combination of forging and heat treatment processes.

6. Turbine disks were successfully produced by Ladish Co., Inc. for Rolls-Royce Allison from the SS-HIP processed billet.

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