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PROCESSING, MICROSTRUCTURE AND MECHANICAL PROPERTIES OF PM GAMMA TITANIUM ALUMINIDES

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Abstract

Gamma titanium aluminide alloys (TiAl) are receiving increasing attention as candidates for advanced structural materials in high temperature aerospace applications. Inert gas atomization of prealloyed powder followed by hot isostatic pressing (HIP) to full density is a viable approach for the production of forging and rolling performs as well as as-HIP components. The prealloyed powder metallurgy (PM) production route has the capability of producing a uniform and fine microstructure in preforms/components of any size. This paper will describe gamma titanium aluminide powder production, processing and characterization. Alloys such as Ti-48Al-2Nb-2Cr, Gamma Met (Ti-46.5Al-4(Nb,Ta,Cr,B), and 395MM (Ti-46Al-4(Nb,Mo,Cr, B,C) have been produced in quantities exceeding 500 kg. Microstructure and processing of consolidated material will be discussed. Thermally induced porosity (TIP) is a phenomenon, which occurs in any inert gas atomized powder. The results of a study to evaluate the effect of processing and heat treatment conditions on the microstructure and occurrence of TIP in gamma titanium aluminide powders will be presented. Mechanical properties of consolidated product will also be presented.

Introduction

Titanium aluminide alloys based on the TiAl intermetallic compound have attractive properties particularly for structural applications in high performance aircraft and automotive engine components as well as other applications [1,2]. The primary advantages of titanium aluminide alloys are low density, high strength to weight ratio at elevated temperatures, a higher modulus than conventional titanium alloys and good oxidation resistance. While gamma titanium aluminide alloys have several attractive properties industrial scale ingot production and wrought processing of these alloys has proven to be challenging. Some of the quality issues related to production of large ingots are centerline porosity and pipe, grain size variations and regions of varying density related to melt homogenization [3]. Powder metallurgy (PM) offers the potential for minimizing many of the problems associated with large ingot production and the potential for reducing overall cost of the final component. Another advantage of the PM process is that it may enable the development of alloys that cannot be made by conventional ingot metallurgy. Gas atomization of prealloyed powder followed by hot isostatic pressing (HIP) or consolidation by extrusion to full density is a viable approach for the production of forging and rolling performs, as-HIP components and extruded shapes. This paper will describe the prealloyed PM process for production of gamma titanium aluminide and characterization of the product.

Powder Production and Processing

A schematic of the system developed by Crucible [4] for production of prealloyed gas atomized titanium powder is shown in Figure 1. This system consists of a melting chamber, an atomization tower and a cyclone collector. The starting charge material is melted in a cold wall induction crucible within the melt chamber. The cold wall induction crucible is shown in Figure 2.

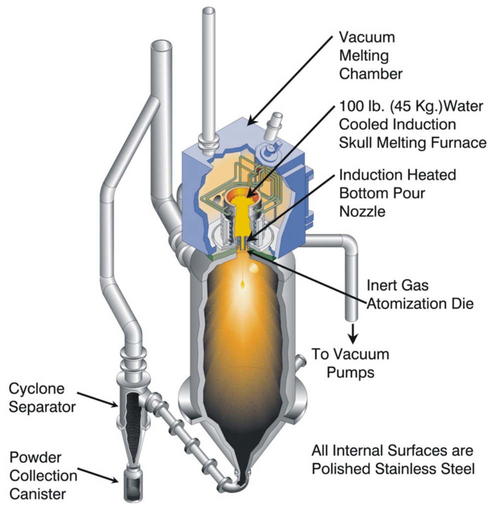


Figure 1. Titanium gas atomization system. (100lb)

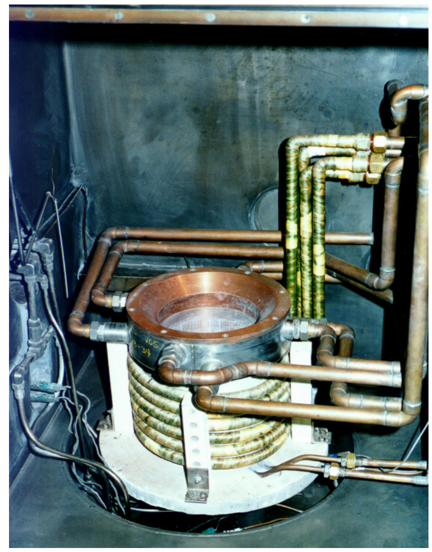


Figure 2. Bottom pour 45kg cold wall induction crucible.

The starting stock for melting may be virgin raw materials or clean prealloyed stock such as ingots or bars. As in conventional vacuum induction melting, no special preparation of the melt stock is required. As the charge is melted a skull forms at the crucible bottom and wall so the molten charge is always contained in a solid skull of the same composition. After the charge is fully molten it can be held in the molten state for an extended period to assure complete homogenization of the melt. The active stirring produced by the induction field and the ability to hold the metal in the molten state for an extended time are particular advantages of this type of melting system. After the starting charge is fully melted it is bottom poured into a heated graphite susceptor. The pour is controlled by the power applied to the inductively heated graphite susceptor and the primary power applied to the crucible. The bottom of the susceptor contains a nozzle which forms, nominally, a 4mm (0.160inch) diameter stream that free falls into high pressure argon gas jets which atomize the molten stream. For gamma titanium aluminde alloys the nozzle material is typically high quality graphite. The atomization gas carries the powder to the cyclone where the powder is collected in a removable canister. After cooling under inert gas to room temperature, the powder is removed for further processing.

Powder cleanliness is an important consideration in any powder that is to be used in aerospace applications or other fatigue critical applications. This aspect has been taken into consideration in design of the atomization system. The atomization tower is constructed entirely of stainless steel and all internal surfaces of the tower, which may come in contact with the powder, are polished. The atomization tower, transfer piping and cyclone collector are made up of multiple sections joined by O-ring flanges to permit disassembly and thorough cleaning of all internal surfaces. The cleaning is done whenever the alloy is changed to prevent cross contamination of alloys. The atomization tower and all sections of the atomizer below the melt deck are contained in a positive pressure room with a dedicated air handling system to prevent air borne contamination from the general shop atmosphere.

Two processing rooms are located adjacent to the atomization tower room. These rooms contain powder processing equipment such as screeners and blenders. The rooms are under positive pressure with dedicated air handling systems. In addition to operations such as screening and blending, all loading of containers for shipment or consolidation are done in these rooms. Only one alloy is processed in a room at any time and before another alloy is processed the equipment and the room are thoroughly cleaned.

Approximately 40 different gamma titanium aluminide compositions have been made in the atomizer to date. Several of these alloys are listed in Table I. The majority of the alloys have been proprietary compositions made for outside customers. Approximately 5445 kg (12,000 lbs) of gamma titanium aluminide powder have been produced in the last five years. The estimated annual capacity of the atomizer is 4,990 to 27,223 kg (11,000 to 60,000 lbs) depending on the number of shifts it is operated.

Table I. Gamma Titanium Aluminide Compositions Produced by Gas Atomization

Ti-50Al at%
Ti-48Al-2Nb-2Cr at%
Ti-48Al-2Nb-2Mn at%
Ti-50Al-2Nb at%
Ti-45Al-5Nb at%
Gamma Met (Ti-46.5Al-4(Nb,Ta,Cr,B) at%
K5 (46.2Al-3Nb-2Cr-0.2W-0.2B-0.2C at%)
395MM (Ti-46.2Al-4(Cr,Nb,Mo,B,C) (at%)
More than 30 proprietary compositions.

Powder Characteristics

Control of chemical composition is another important consideration in any powder making operation. Typical chemistry tolerances for gamma titanium aluminide powder are given in Table II.

Some alloys, such as K5 and 395MM require higher levels of carbon in the alloy. For these alloys, carbon is added to achieve the required carbon level. In melting and atomization there is

Table II. Typical Chemistry Tolerances For Gamma Ti Aluminide Powder

Element	Wt %	At %
Aluminum	$\pm$ 0.50	$\pm$ 0.56
Minor	$\pm$ 0.25	
Iron	0.08 max	
Oxygen	0.08 max	
Nitrogen	0.02 max	
Carbon	0.05 max	
Hydrogen	0.005 max	

essentially no increase in carbon, nitrogen or hydrogen over the starting melt stock. The increase in oxygen content over the starting melt stock is typically 100 to 200 wppm for –100 mesh (<150µm) powder.

Gas atomized gamma titanium aluminide alloy powder is predominantly spherical with some satellite particles, it has good flow characteristics and it has a packing density of about 65% of solid density. Cross sections of as-atomized Ti-47Al-2Nb-2Cr (at%) powder are shown in Figure 3. The powder exhibits a cellular structure resulting from the rapid solidification. The cell size is 2 to10µm and does not change significantly with powder particle size. Studies by other workers have shown that in the as-atomized state, the powder is predominately alpha phase which then transforms to gamma plus alpha two phase on subsequent heating[5].

Figure 4 shows typical size distributions for gas atomized gamma titanium aluminide powder. The mean particle size for the –35 mesh powder is 90µm. Mean particle size for the –100 mesh powder is 70µm.

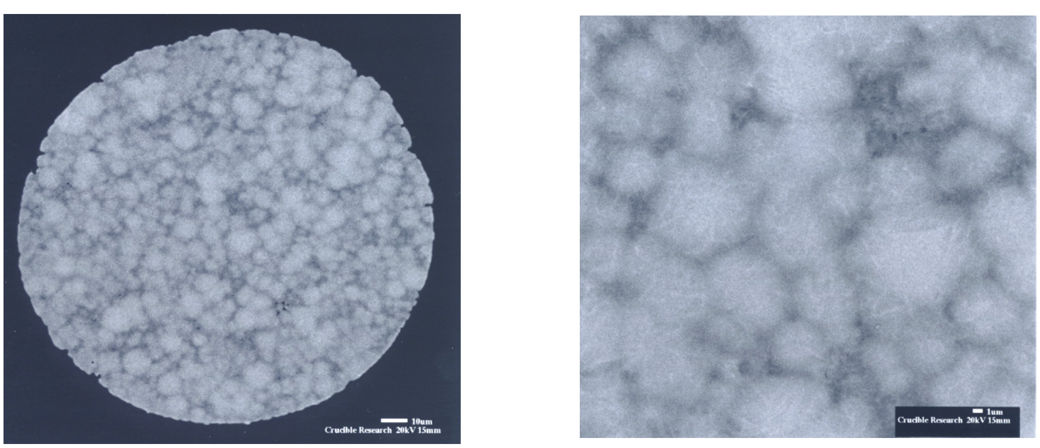


Figure 3. Cross-section of as-atomized Ti-47Al- 2Nb-2Cr (at%) powder.

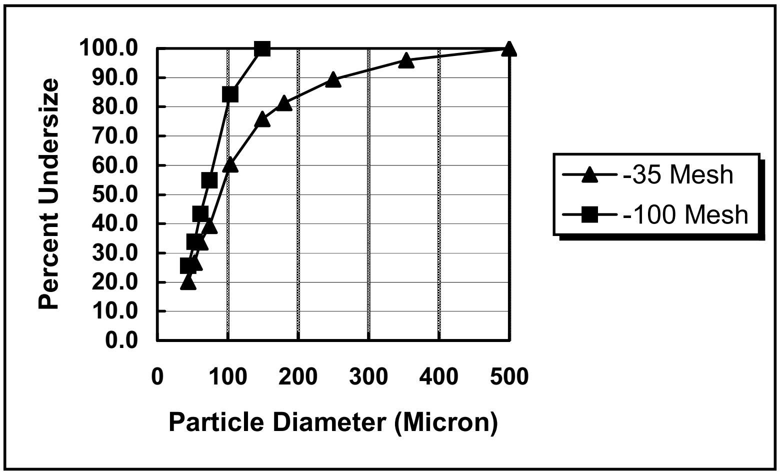


Figure 4. Size distribution of gas atomized gamma titanium aluminide powder.

Powder Consolidation

After atomization, a full chemistry is determined on each heat and physical properties such as size distribution, tap density and flow rate are determined. The powder is then screened to the required particle size. After screening the powder is loaded into suitable containers for consolidation, outgassed, and sealed under vacuum.

Typical methods for consolidation of powder are extrusion and hot isostatic pressing (HIP), with HIP being the most common method. Typical consolidation temperatures for gamma titanium aluminide range from 1000C (1832F) to 1300C (2372F). A significant volume contraction takes place in HIP. With packing densities of over 60% and good can design, the shrinkage is relatively uniform. Two of the largest gamma titanium aluminide compacts that have been hot isostatically pressed to date are shown in Figure 5. The alloy HIPed in these compacts is 395MM. After HIP to full density, the 263 kg (580lb) compacts were 318mm (12.5in) in diameter by 813mm (32in) high.

Microstructure of HIP Consolidated Powder

As-HIP products are fully dense and have fine homogeneous microstructures. Figure 6 shows a typical as-HIP microstructure of gamma titanium aluminide HIPed at 1000C (1832F), which is at the lower end of the range of consolidation temperatures. The microstructure of material HIPed at 1260C (2300F), which is at the upper end of the temperature range, is also shown in Figure 6. Both HIP temperatures result in near-gamma equiaxed microstructures with the expected larger grain size at the higher HIP temperature. These microstructures are from relatively small HIP compacts weighing about 4.5 kg (10 lb). The microstructure developed at given HIP temperature is relatively independent of compact size. The microstructures in Figure 7 were taken from a 263kg (580lb) compact HIPed at 1260C (2300F). As indicated by the photomicrographs, the microstructure is uniform and similar to the smaller compact microstructures HIPed at the same temperature. No major microstructural variations between the top and bottom or center and edge of the as-HIP compact were observed. The fine equiaxed near gamma microstructure is a very good starting structure for subsequent working operations such as isothermal forging or plate/sheet rolling. It is not, however, necessarily the best structure for final applications where good toughness and elevated temperature properties are required. For applications such as gas turbine engines components, a fully lamellar microstructure may be required. The as-HIP material can be heat treated to produce a fully lamellar microstructure by heating above the alpha tranus. A fully lamellar structure in HIP plus heat treated material is shown in Figure 8.

Figure 5. As-HIP 318mm (12.5 in.) diameter by 813mm (32.0 in) 395MM compacts

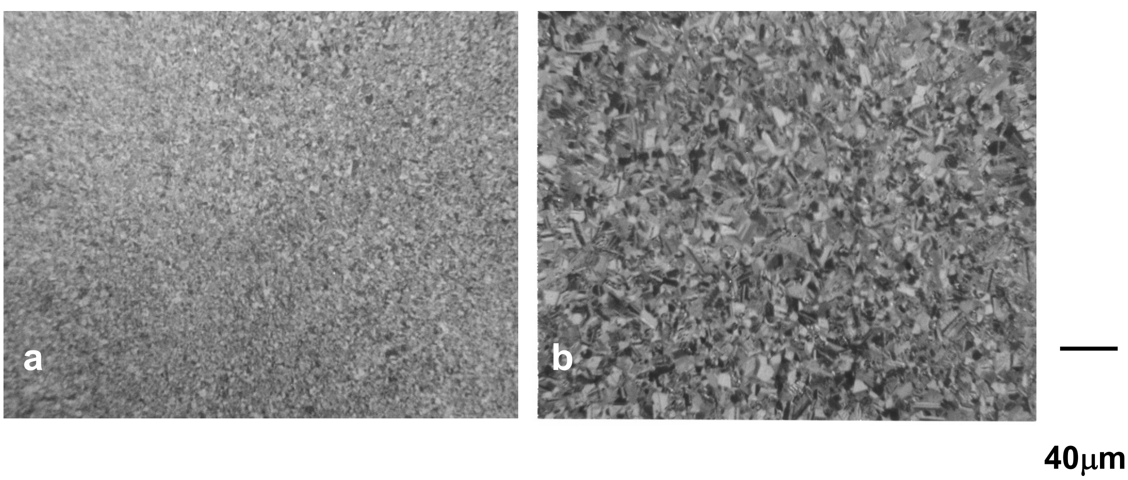
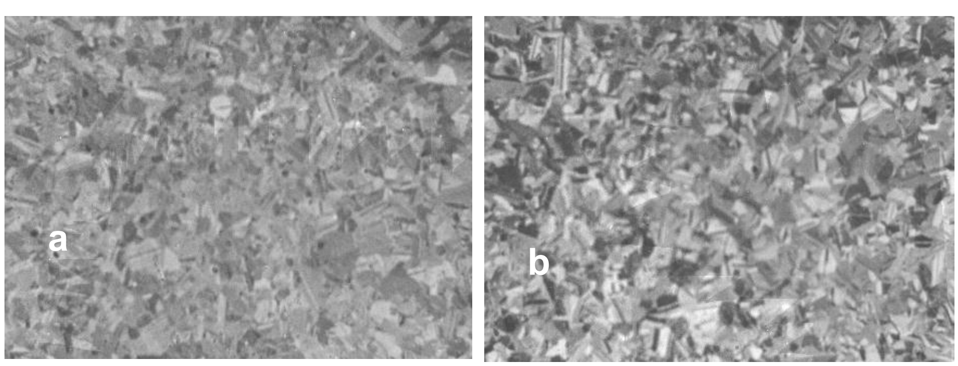


Figure 6. As-HIP microstructures of 395MM alloy. (a) HIP 1000C, (b) HIP 1280C.



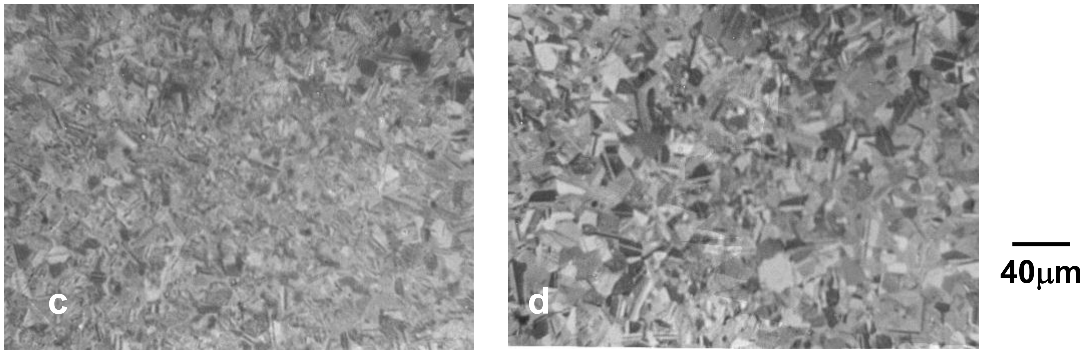


Figure 7. As-HIP microstructures of 318mm diameter by 813mm 395MM compact in different locations in compact a.) top, edge b.) top, center c.) bottom, edge d.) bottom, center

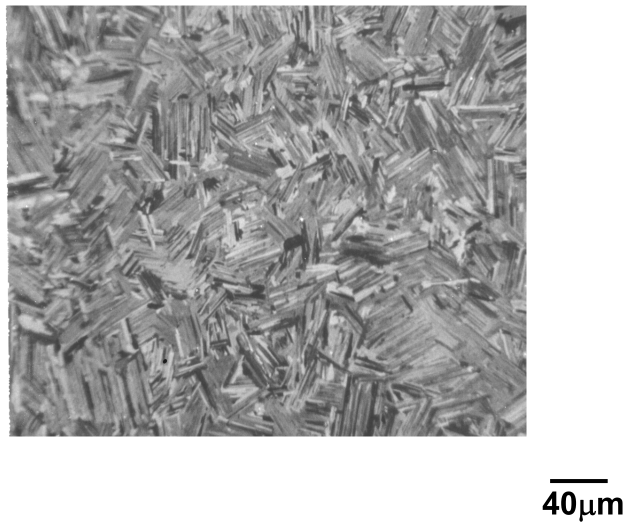


Figure 8. Fully lamellar microstructure in HIP plus heat treated (1360C) 395MM

Mechanical Properties

As described in the previous section, hot isostaticly pressed gas atomized powders are fully dense and have fine uniform microstructures. Table III lists the chemical analysis of two variations of the nominal Ti-47Al-2Nb-2Cr (at%) composition that have been tested. These powders were hot isostatically pressed at 1300C (2372F) and 103MPa (15 ksi) for 3 hours to produce compacts nominally 127mm (5 inch) diameter by 241mm (9.5 inch). As a result of the difference in aluminum content, the higher aluminum composition had a duplex microstructure and the lower aluminum composition had a lamellar microstructure after HIP at 1300C (2372F). The alpha transus temperatures were determined to be 1320C (2408F) and 1295C (2363F) respectively. Following HIP the Ti-47Al-2Nb-2Cr composition was given a duplex heat treatment of 1290C (2354F) 2h + 900C (1652F) 2h. The Ti-46Al-2Nb-2Cr-0.2B composition with the lamellar microstructure was tested in the as-HIP condition. Tensile properties of the two compositions are given in Table IV. The test results show that the tensile properties are uniform and do not vary significantly with location or test direction in either compact. It is not common to see higher strength and tensile ductility in material with a lamellar structure than a duplex structure but the effects of aluminum content, oxygen content, boron content and cooling rate are probably more important considerations in this case.

Table III Chemical Composition of HIP PM Compacts.

Alloy	Al at%	Cr at%	Nb at%	B at%	Fe wppm	O wppm	N wppm	C wppm	H wppm	Ti
Ti-47-2-2	46.73	2.02	2.01	$\leq$ 0.01	300	645	70	245	20	Bal.
Ti-46-2-2+B	45.61	2.02	2.03	0.20	300	735	65	240	24	Bal.

Table IV. Room Temperature Tensile Properties of Ti-47Al-2Nb-2Cr and Ti-46Al-2Nb-2Cr-0.2B

Alloy	Test Location	Orientation	Condition	0.2%YS MPa	UTS MPa	Elong. (%)
Ti-47-2-2	Top	Transverse	1290C+900C	425	555	1.3
	Mid-height	Transverse	1290C+900C	430	540	1.5
	Mid-height	Longitudinal	1290C+900C	440	570	1.5
	Bottom	Transverse	1290C+900C	440	565	1.7
Ti-46-2-2+B	Top	Transverse	As-HIP	465	615	2.5
	Mid-height	Transverse	As-HIP	445	620	3.0
	Mid-height	Transverse	As-HIP	470	655	3.5
	Bottom	Longitudinal	As-HIP	460	645	3.0

A limited amount of testing has been conducted on the 395MM alloy (Ti-46.2Al-4(Cr,Nb,Mo,B,C) (at%). This alloy has been tested in the HIP and HIP plus forged condition. For this work argon atomized powder was screened to –100 mesh (< 150µm) and loaded into a cylindrical 51mm (2.0 inch) x 203mm (8.0 inch) titanium can. After outgassing and sealing the compact was HIPed at 1260C (2300F) and 170MPa (25ksi) for 4 hours. The as-HIP compact was cut in half and one half was machined to a cylinder 41 mm (1.6 inch) diameter x 81mm (3.2 inch). The machined cylinder was then placed in an insulated steel can, preheated to 1260C (2300F) and upset forged on a hydraulic press in one pass. The 395MM cylinder was upset nominally 85% to a thickness of 11mm (0.45 inch) The as-forged pancake is shown in Figure 9. Duplicate tensile tests were conducted on this material in the as-HIP, HIP plus heat treated, as-forged and forged plus heat treated conditions. The heat treatment used for the HIPed and forged conditions was 1370C (2500F) 1h AC + 950C (1740F) 8h which produced a fully lamellar microstructure. The four microstructures are shown in Figure 10.

Room temperature tensile properties for the four conditions are given in Table V. In the HIP and HIP plus forge conditions, strength is very high with very little ductility. The similarity in room temperature tensile properties for both conditions can be attributed to the similar microstructures.

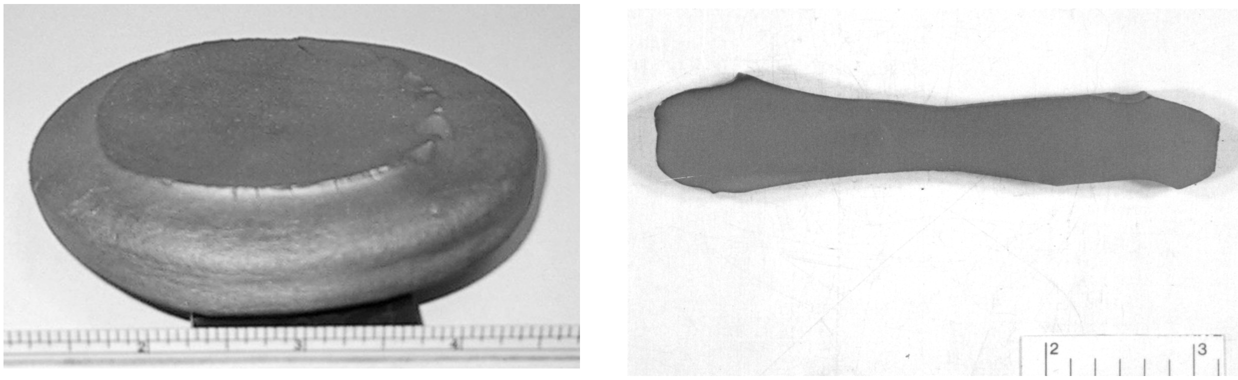


Figure 9. As-forged 395MM pancake and cross-section.

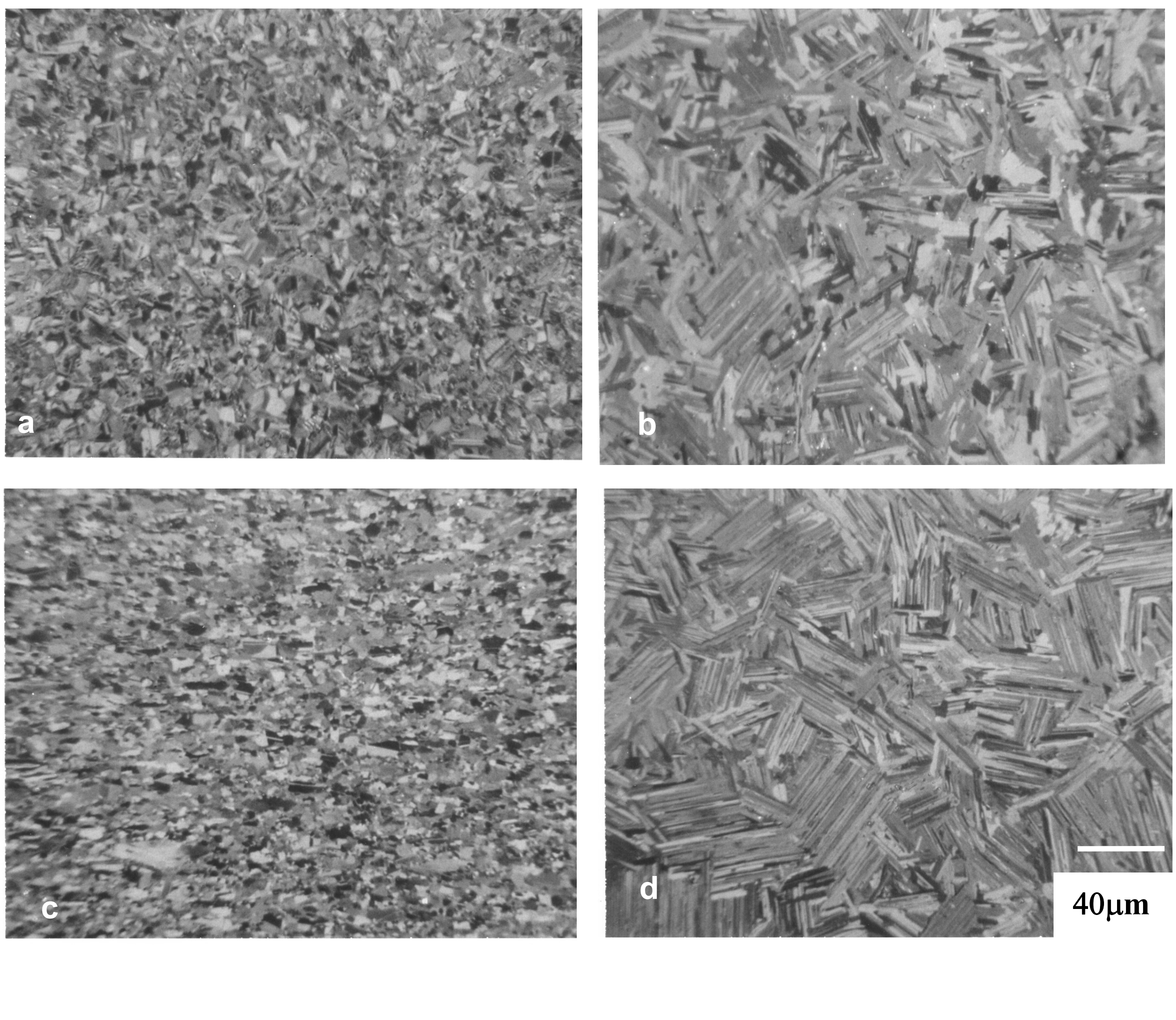


Figure 10. Microstructures of HIPed and forged 395MM compact. a.) As-HIP 1260C (2300F) b.) HIP + 1370C (2500F) 1h AC + 950C (1740F) 8h c.) As-forged 1260C (2300F) d.) Forge + 1370C (2500F) 1h AC + 950C (1740F) 8h

Both have very fine near gamma microstructures which would be expected to result in high strengths and low ductilities. After heat treatment to produce fully lamellar microstructures tensile properties are significantly improved. Microstructures are very similar for both conditions as are tensile properties. In the heat treated condition yield strength is approximately 580 MPa, ultimate tensile strength is approximately 645 MPa and elongation is 1%.

Table V. Room Temperature Tensile Properties of PM 395MM (Ti-46.2Al-4(Cr,Nb,Mo)–xB-yC at%)

Condition	0.2%YS MPa	UTS MPa	Elong. %	Modulus GPa
As-HIP 1260C	-	720	0.1	185
HIP + Heat Treat	585	650	1.1	160
As-Forge 1260C	-	720	0.1	175
Forge + Heat Treat	570	640	1.1	170

Thermally Induced Porosity

Thermally induced porosity (TIP) is a phenomenon that can occur in any inert gas atomized powder [6]. This generally occurs when as-HIP material is heated above the original HIP temperature. The pores are very small and normally can only be detected metallographically. The volume change is so small that it cannot be detected by standard density measurements. It is generally accepted that the pores originate from small amounts of entrapped argon. Figure 11 shows the relationship between powder particle size and argon content for two gamma alloys. As the graph indicates, the argon content increases with increasing particle size to a maximum of about 1 wppm. The pores have not been found to be detrimental to room or elevated temperature tensile properties. Their effect on second tier properties is currently being investigated.

To evaluate the effect of HIP temperature and post-HIP heat treatment on the occurrence of pores in the 395MM alloy, the evaluation outlined in Table VI was conducted. Initially a compact of –100 mesh 395MM powder was HIPed at 1000C (1832F) and 172 MPa (25 ksi) for 4h. Nominal dimensions of the as-HIP compact were 40mm (1.6in.) diameter x 220mm (8.7in.). The compact was then cut into six equal sections and five of the sections were re-HIPed at 1200C (2192F) to 1400C (2552F) in 50C intervals. Three 3mm (0.12 in.) disks were cut from each of the six compact sections. One set of disks was examined in the as-HIP condition, one set was heat treated at 1360C (2480F) for 0.5h and one set was heat treated at the same temperature for 2h. Each of the disks was radiographed and examined metallographically in the as-polished condition for the presence of pores.

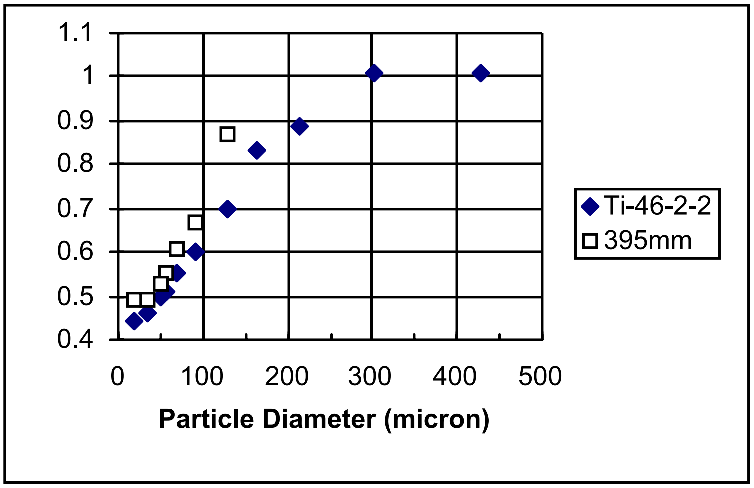


Figure 11. Argon content of gas atomized titanium aluminide powder vs particle size.

Table VI. HIP and Heat Treatment Conditions For TIP Evaluation of PM 395MM

HIP Conditions	As HIP	1360C 0.5h	1360C 2.0h
1000C/170MPa/4h	A0	B0	C0
1200C/203MPa/3h	A1	B1	C1
1250C/203MPa/3h	A2	B2	C2
1300C/203MPa/3h	A3	B3	C3
1350C/203MPa/3h	A4	B4	C4
1400C/203MPa/3h	A5	B5	C5

A radiograph of the as-HIP disks is shown in Figure 12. The resolution of indications in the radiograph is 1% of the thickness therefore anything larger than 30μm should be detected in the radiograph. No indications were found in any of the disks other than film defects. The same was true for the disks heat treated for 0.5h and 2h at 1360C(2480F). All were free of indications. A radiograph of the disks heat treated for 2h at 1360C (2480F) is shown in Figure 13.

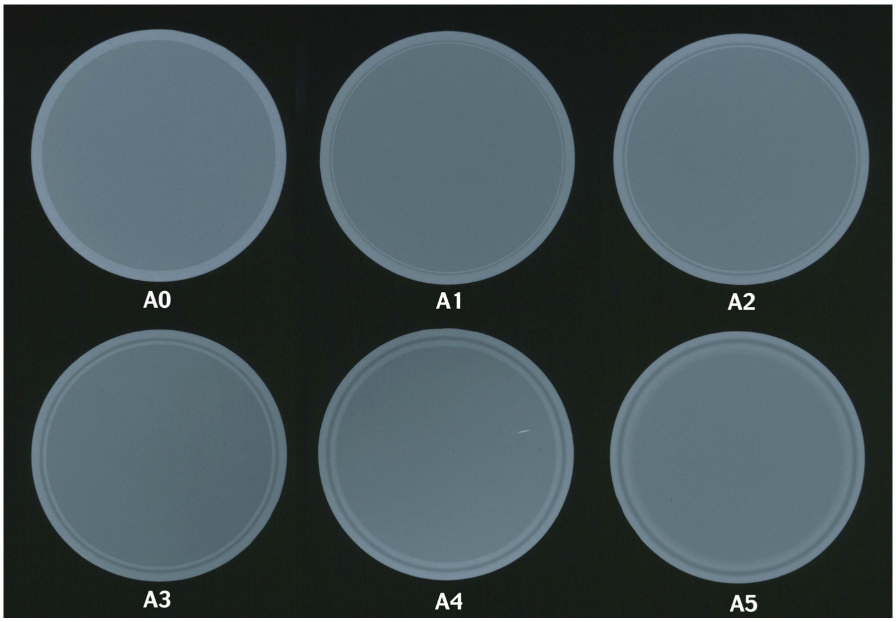


Figure 12. Radiographs of 395MM 3mm thick disks HIPed at the temperatures indicated in Table VI.

Metallographic examination of samples from the as-HIP disks did not reveal any pores in the structure nor were any found in the disks heat treated for 0.5h at 1360C (2480F). Pores were found, however, in the disks heat treated for 2h at 1360C (2480F). The largest pores were 25

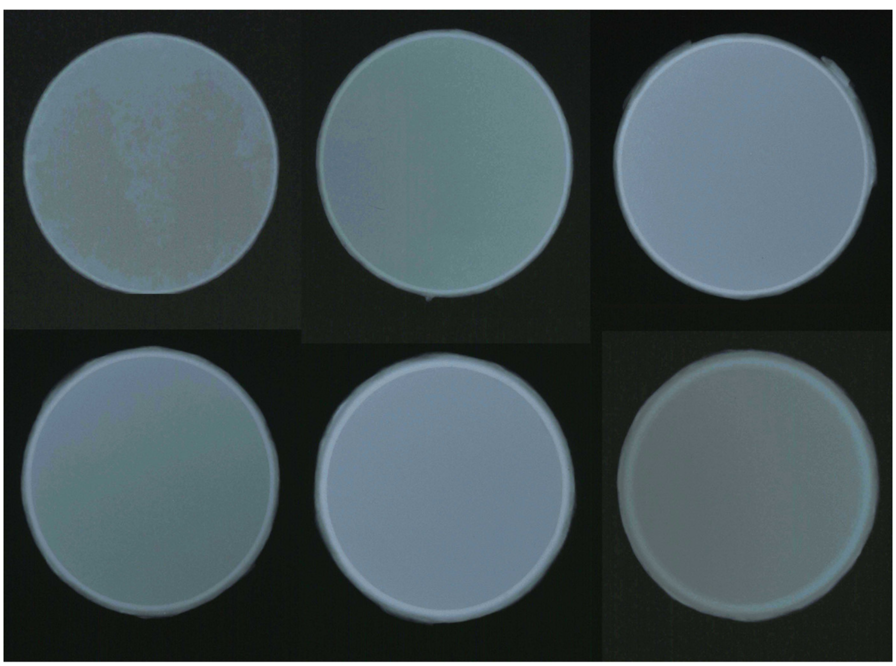


Figure 13. Radiographs of 395MM 3mm thick disks HIPed and heat treated at 1360C (2480F) for 2h.

μm which would not have been detected by radiography. Examples of the pores are shown in Figure 14. There did not appear to be any correlation of HIP temperature with maximum pore size. After the 2h at 1360C (2480F) heat treatment maximum pore size was the same for the disk HIPed at 1000C as it was for the disk HIPed at 1400C.

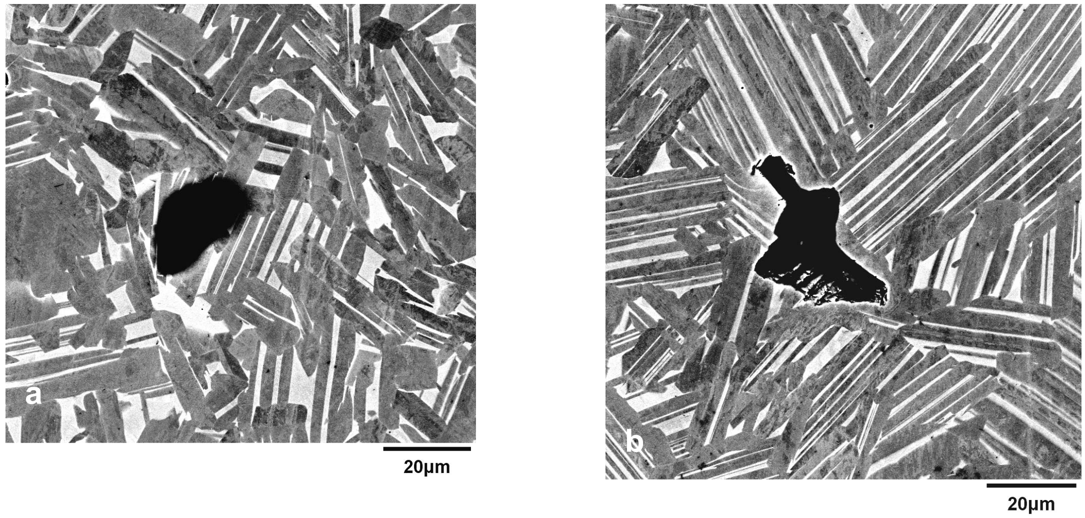


Figure 14. Pores in HIPed plus heat treated disks a) HIPed at 1200C and heat treated for 2h at1360C b) HIPed at 1400C and heat treated for 2h at1360C

Summary

The gas atomization process for making gamma titanium aluminide powder has been presented. The powder can be consolidated to full density by hot isostatic pressing or by other means. This process is a viable method for making fully dense gamma titanium aluminide components. Microstructures of as-HIP materials are uniform independent of the size of the component. Tensile properties are isotropic and show good strength and ductility. As-HIP materials can be further processed or directly heat treated to produce fully lamellar microstructures. Post HIP heat treatment at temperatures high in the alpha plus gamma field regenerates porosity, however, experimental results indicate that size does not exceed 25 μm in diameter.

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