HOT ISOSTATIC PRESSING OF TITANIUM 6Al-4V

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Hot isostatic pressing (HIP), a relatively new powder consolidation process of perhaps greater potential than conventional pressing and sintering, has been investigated as a method of consolidating prealloyed titanium 6Al-4V powder.

The results show that fully dense Ti-6Al-4V billets consolidated from spherical powder with tensile and fracture toughness properties equivalent to wrought material can be produced by HIP at 1675°F and 10,000 psi for 3 hours. Heat treatment response of HIP spherical powder in both the as-HIP and forged condition is equivalent to that of wrought material. Limited data indicate that fatigue properties of HIP Ti-6Al-4V tubing are lower than those of the wrought counterpart. Ingot source dehydride powder can be fully compacted by HIP at 1775°F and 10,000 psi for 1-1/2 hours and exhibits a uniform microstructure of equiaxed alpha and intergranular beta, however, scrap-source hydride Ti-6Al-4V powder evaluated exhibits a non-uniform microstructure with alpha stabilized regions. HIP spherical powder, in the rectangular billet shape evaluated, has been shown to be essentially isotropic with respect to both crystallographic orientation and mechanical properties.

Introduction

Until recently, attempts to fabricate titanium components by powder metallurgical techniques have been only partially successful. Witt et al. (1) in a recent investigation has shown that fully dense, high-strength, ductile forgings can be produced by isostatic pressing and vacuum sintering Ti-6Al-4V elemental powder preforms using isothermal forging followed by a 1300°F, 2-hour anneal. In another study, Geotzel (2) has demonstrated that good tensile properties can be attained by upset forging spark-sintered prealloyed Ti-6Al-4V powder preforms. Peebles (3) has shown in a major titanium powder metallurgy evaluation program that the fatigue life of a jet engine component (inlet guide vane) forged from a prealloyed Ti-6Al-4V preform pressed and sintered to 85-92% theoretical density is equivalent to that of conventionally produced (wrought barstock) components.

However, hot isostatic pressing (HIP), a relatively new powder consolidation process of perhaps greater potential than conventional pressing and sintering, has been developed into a viable method for fabricating a number of high-quality components, some of complex shape, from beryllium, tool steel, and carbide starting materials.

The present study was initiated to evaluate the potential of the HIP process in consolidating prealloyed Ti-6Al-4V powder free of such problems as porosity, low ductility, and contamination often attendant to conventional pressing and sintering of titanium. An additional benefit of the HIP process is that pressing, sintering, and for some applications, forging as well, can be combined into a single operation at a temperature below the beta transus (as compared to sintering at 2250°F). The HIP process is also an attractive potential method of fabricating highly alloyed titanium products heretofore difficult or impossible to make with homogeneous chemical composition and properties by conventional melting and forging practice.

Processing variables studied include prealloyed powder shape (spherical and irregular), HIP time-temperature-pressure parameters, and post-HIP forging and/or heat treatments. Mechanical property data, including tensile, fracture toughness, and limited fatigue and impact data, have been generated using prealloyed spherical powder; whereas, irregular powders from two sources, namely, hydrided powder (scrap source) and dehydrided powder (ingot source), were processed and evaluated microstructurely only. In addition, the degree of isotropy of HIP spherical powder has been determined by X-ray diffraction and von Mises yield surface techniques.

Experimental

Powder Characterization

Spherical powder was produced by the rotating electrode process (REP) in which a wrought alloy bar is rotated at high speed and arc-melted in a helium filled chamber by the use of a non-consumable tungsten electrode. The molten metal is spun off as small droplets which solidify in flight through the chamber.

Irregular powder received in the dehydrided condition was produced by hydriding an ingot of Ti-6Al-4V at approximately 850°F and subsequently comminuting the embrittled hydrided titanium product at room temperature via a disc pulverizer under argon atmosphere (3). The resulting powder was sieved, magnetically separated, and dehydrided at 1300°F under dynamic vacuum.

Irregular powder received in the hydrided condition was produced from a mixture of cast and wrought Ti-6Al-4V revert scrap. After hydriding at elevated temperature the material was jaw-crushed to minus 6-mesh and further comminuted to minus 40-mesh using a titanium rod mill and then magnetically separated.

The particle size distribution as determined by screen analysis of the starting powders is given in Table I.

TABLE I PARTICLE SIZE DISTRIBUTION OF TITANIUM ALLOY POWDERS

Sieve, U.S. Series	Weight Percent Retained on Screen		
	Spherical	Irregular (Hydrided)	Irregular (Dehydrided)
-35 + 45	22.1		
+ 40			0.87
-40 + 50		16.92	15.17
-45 + 60	43.9		
-50 + 60		26.88	15.20
-60 + 80	22.0	28.32	19.95
-80 +100		11.04	9.95
-80 +120	8.4		
-100 +140			9.80
-120 +170	2.8		
-100 +200		14.80	
-140 +200			7.79
-170 +230	0.4		
-200 +230		1.20	
-200 +270			4.90
-230 +325	0.2	0.48	
-270 +325			3.50
-325 +400			4.06
-325	0.2	0.36	
-400			8.81

The REP powder is the coarsest of the three powder types evaluated with 88% of the particles greater than 177 microns (+80 mesh) in diameter; whereas, the irregular powder received in the dehydrided condition contains a significant fraction of fines

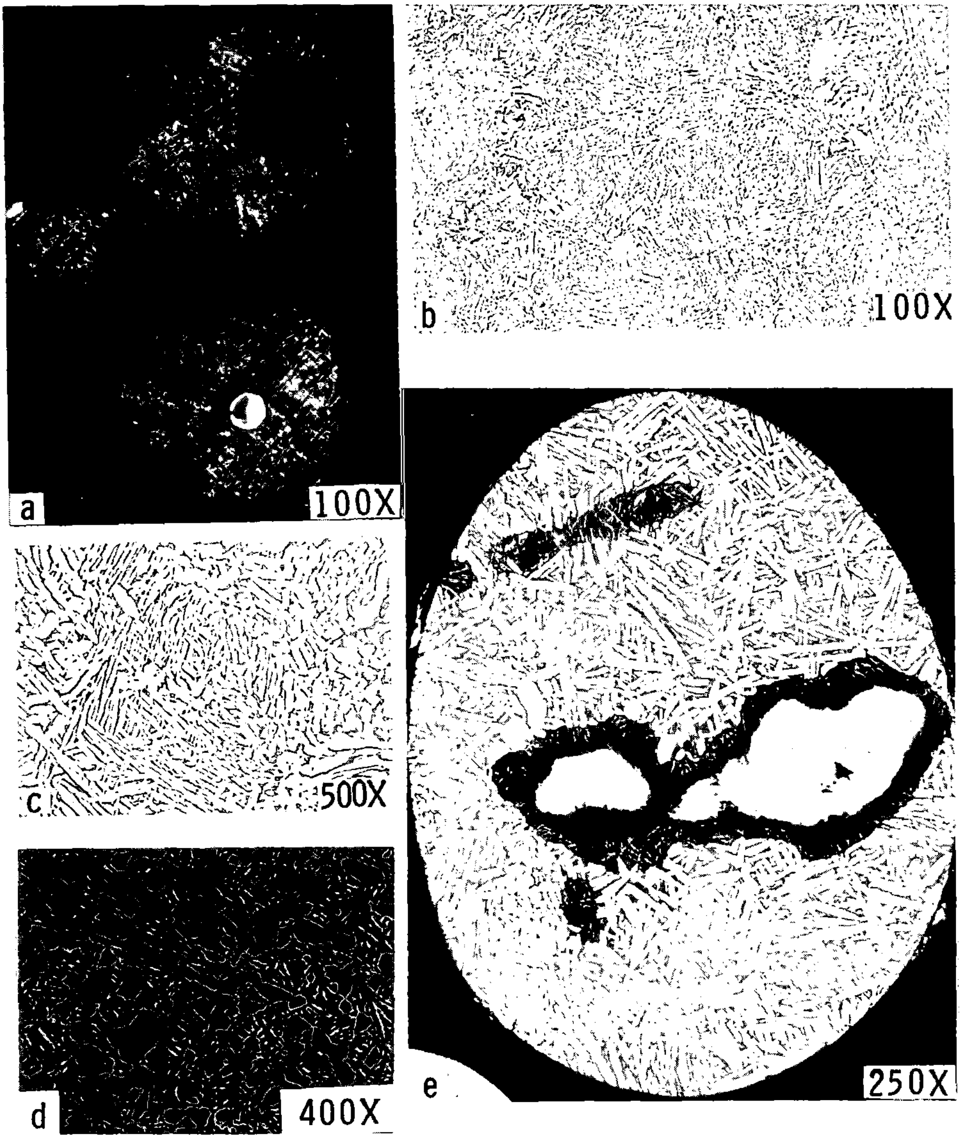

Figure 1 Microstructures of spherical Ti-6Al-4V powder; (a) typical as-received powder particles (note the void in the lower particle), (b) and (c) as-HIP, (d) HIP plus STA (1775°F, 2 hours, W.Q. plus 1100°F, 4 hours, AC), and (e) a powder particle (after 1750°F, 1 hour, FC) containing a tungsten inclusion with a beta-stabilized zone surrounding it.

(nearly 13% of the particles pass through a minus 325-mesh screen (44 microns)).

The results of vendor and AFML chemical analyses of the starting powders are listed in Table II. Both the spherical and dehydride powders were within current AMS specifications; however, the hydride powder produced from scrap material contained excessive oxygen.

TABLE II. CHEMICAL ANALYSIS OF PREALLOYED TITANIUM POWDERS (WEIGHT PERCENT)

Element	Spherical Powder As Received		Spherical Powder	Irregular Hydride Powder (Scrap Source) As Received		Irregular Hydride Powder (Scrap Source)	Irregular Dehydride Powder As Received		Irregular Dehydride Powder
	Vendor Analysis	AFML Analysis	As HIP	Vendor Analysis	AFML Analysis	As HIP	Vendor Analysis	AFML Analysis	As HIP
O	0.1560	0.1470	0.1530	0.250-0.280	0.302(1)	(2)	0.148	0.147	0.220
H	0.0028	0.0070	0.0049	--	~0.1600	(2)	0.0068	--	0.008
N	0.0215	0.017	0.020	0.017	0.022	0.034	0.129	--	0.016
C	0.0084	--	0.026	--	0.030	0.040	0.0154	--	0.077
Fe	--	--	0.160	0.160	0.180	0.190	0.210	--	0.150
Cu	--	--	0.020	0.004	<0.005	<0.005	--	--	<0.005
Mn	--	--	0.050	0.003	<0.005	<0.005	--	--	<0.005
Mg	--	--	--	<0.010	0.005	0.010	0.005	--	<0.005
Na	--	--	0.050	--	--	--	0.002	--	--
W	--	--	0.010	--	--	--	--	--	--
Ca	--	--	--	--	<0.020	<0.020	--	--	<0.020
Mo	--	--	--	0.027	<0.005	<0.005	--	--	<0.005
Ni	--	--	--	<0.020	<0.005	<0.005	--	--	--
Sn	--	--	--	0.029	<0.020	<0.020	--	--	<0.020
V	4.02	--	3.900	3.620	3.800	3.800	4.030	--	3.700
Al	6.30	--	5.980	6.300	6.400	6.400	6.090	--	5.900
Ti + other trace elements	Balance	--	Balance	Balance	Balance	Balance	Balance		Balance

-- Indicates Not Determined
(1) Oxygen analysis of questionable validity at high level of Hydrogen.
(2) Oxygen 0.283 - 0.661 (Average of 8 samples 0.540); Hydrogen 0.001 - 0.071 (Average of 8 samples 0.034).

The microstructures of each starting powder are shown in Figures 1a, 2a, and 3a for spherical, dehydride, and hydride powders, respectively. Note the acicular structure of the spherical particles shown in Figure 1a and that particles consist of several grains. The angular hydride particle surfaces are highly irregular as would be expected from crushing an embrittled material. The microstructures of both spherical and dehydride powder particles are quite uniform from particle to particle while the hydride powder particles reflect widely varying microstructures. Both cast and wrought microstructures are evident and hence are indicative of a variety of scrap source materials.

Powder Consolidation

Prior to HIP the spherical powders were loaded into stainless steel cans previously cleaned in a 10% HNO_3-2% HF aqueous solution, rinsed successively in water and ethyl alcohol and then air dried. The powders were vibratory loaded to a tap density of

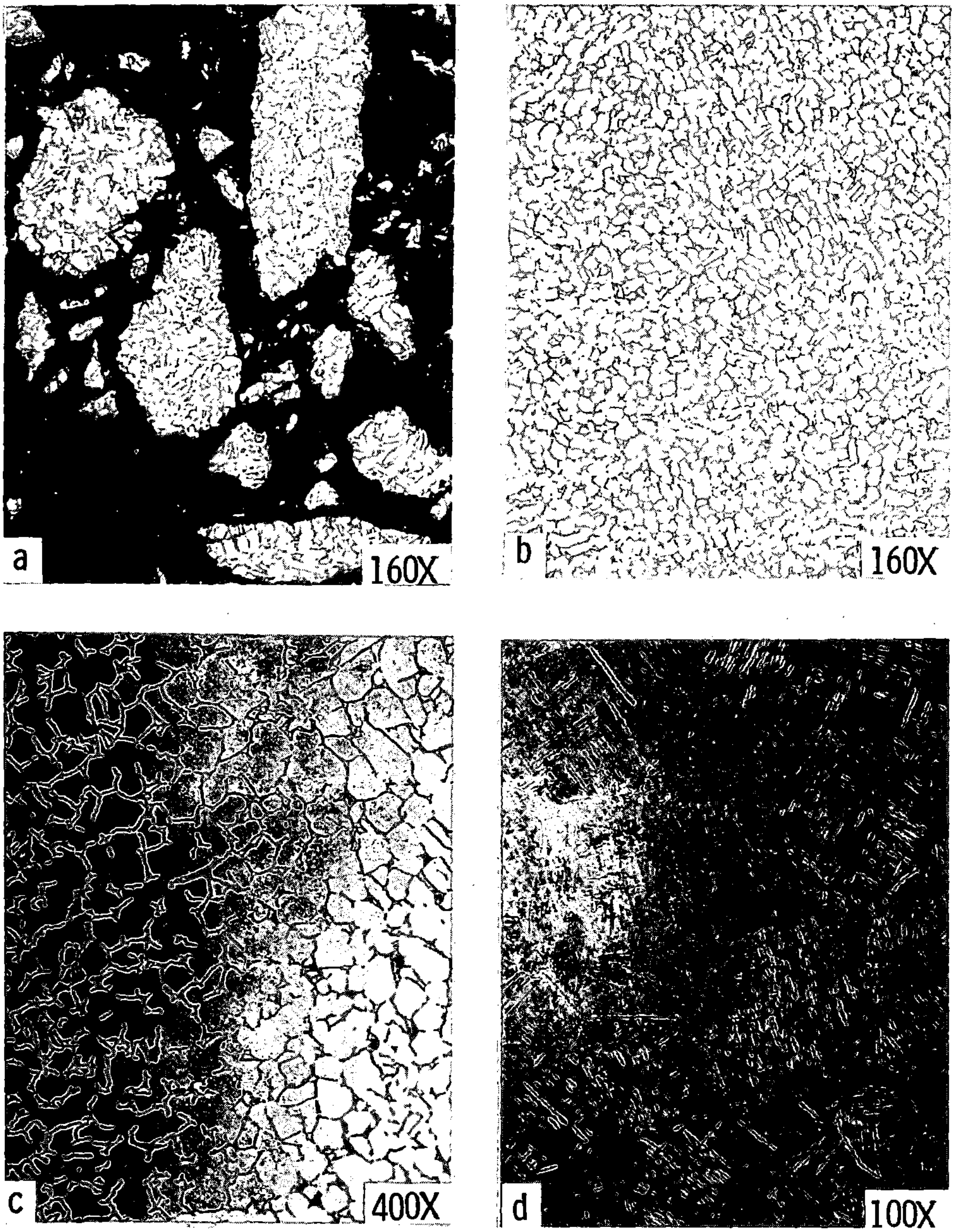

Figure 2 Microstructures of irregular dehydride Ti-6Al-4V powder; (a) as-received microstructure, (b) and (c) as-HIP (1775°F, 1-1/2 hours, 10,000 psi), and (d) after a 2100°F, 1 hour post-HIP exposure illustrating no evidence of porosity.

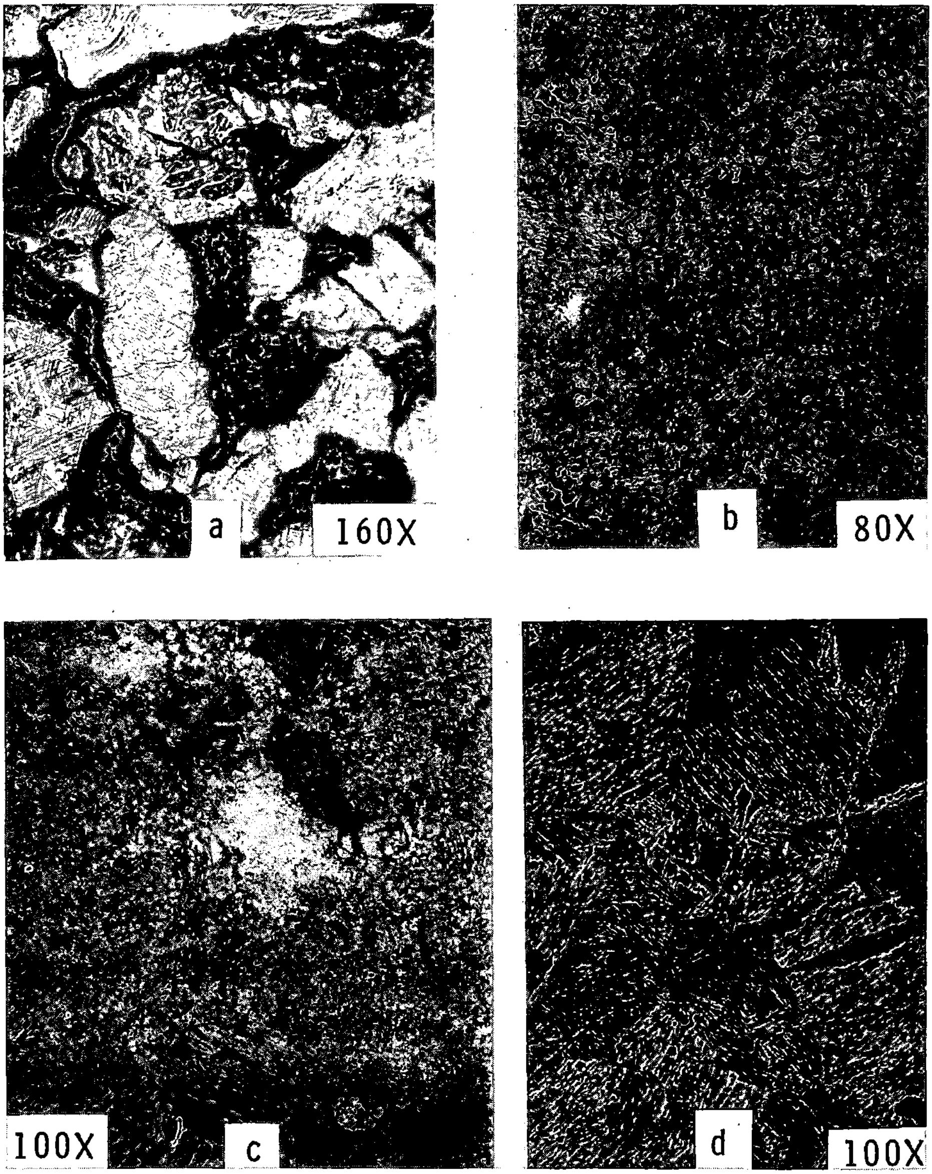

Figure 3 Microstructures of irregular hydride (scrap-source) Ti-6Al-4V powder; (a) as-received microstructure showing non-uniformity between particles, (b) as-HIP (1675°F, 3 hours, 10,000 psi) showing alpha-stabilized and included areas, (c) after HIP plus a 2100°F, 1 hour treatment, and (d) after HIP plus a 2500°F, 1 hour treatment.

about 65% of theoretical density. The cans were then sealed by evacuating and electron-beam welding in a chamber evacuated to 10^{-5} torr.

The as-received dehydrided powder was processed under argon by vibratory packing to approximately 55% theoretical density into a mild steel can followed by evacuating at room temperature. Outgassing was subsequently accomplished at 250°F for 4 hours to 10^{-5} torr. The evacuation stem was then sealed by forging and welding.

However, the as-received hydride powder (scrap source) was first hydropressed at 30,000 psi to 67% theoretical density. The green pressed powder compacts of about 3 pounds each were then canned in mild steel containers with 1/4-inch diameter steel evacuation stems attached. Canning was performed in an argon welding chamber to prevent contamination of the powder. The dehydriding cycle consisted of the following steps; (1) a 12-hour evacuation to 10^{-5} torr at room temperature, (2) a 3-hour heat-up to 1100°F with a 24-hour hold at temperature, (3) a 6-hour heat-up to 1400-1500°F with an 18-hour hold at temperature using a mechanical pump initially to accommodate the rapid evolution of hydrogen and then a 24-hour period at temperature using a diffusion pump ($\sim 10^{-5}$ torr). The evacuation stems were then forged and welded closed and the cans air cooled to room temperature.

The evacuated and sealed cans of titanium powders were hot isostatically pressed using an apparatus fully described elsewhere (4, 5). The basic components of the HIP apparatus are a pressurization system, heating system and a cold-wall pressure vessel. Pressurization was achieved using a multi-stage compressor and helium gas; whereas, heating was accomplished via resistance-type internal heating elements. A typical HIP cycle is shown in Figure 4.

Forging

Two HIP blocks, one of six cubic inches in volume and one of 23 cubic inches in volume (shown in Figure 5), were forged using a 500-ton hydraulic press in conjunction with 1/8-inch preheated (to forging temperature) Inconel face plates. The forging blocks (or preforms) were wrapped in glass cloth and coated with a graphite-base lubricant (Fiske 604D). Blocks were held one-hour at the forging temperature (1700°F or 1750°F), upset forged 40% and water quenched off the press. The total cycle time was less than 30 seconds for each block.

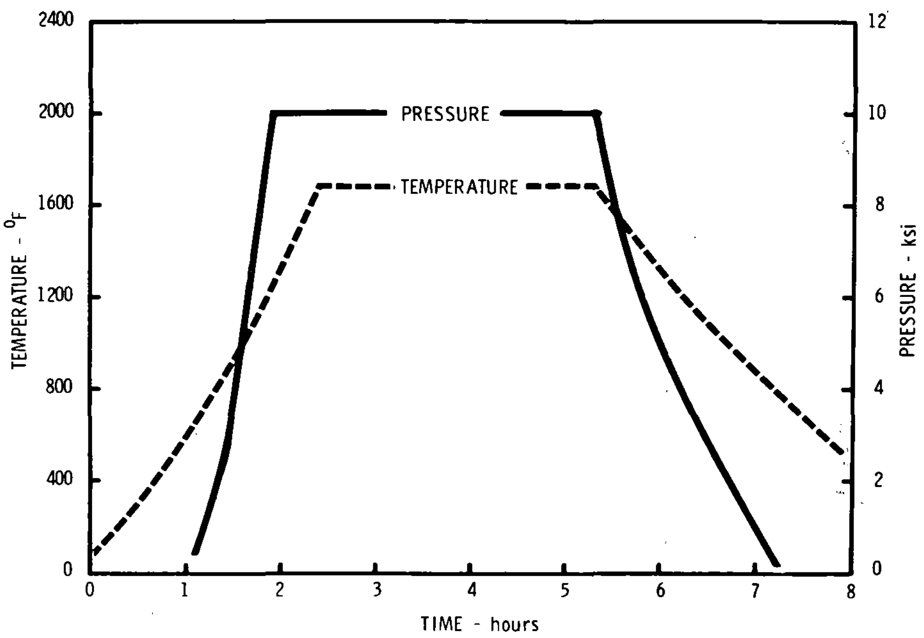

Figure 4 Schematic of a typical hot isostatic pressing cycle.

Testing

Tensile tests were conducted in air at room temperature on subsize specimens, with a reduced section 1-inch long and 0.160-inch in diameter, using an Instron machine of 10,000 pound capacity at a constant crosshead speed of 0.01 inches per minute.

Fracture toughness tests were performed using both Charpy and compact-tension specimens. Standard notched Charpy specimens, 0.394-inch square by 2.165 inches long, were precracked in fatigue using a ManLabs Model FCM-300B machine to introduce a crack at the notch root nominally 0.060-inch in depth. Three-point slow bend tests were performed on a ManLabs Model SB-750 slow bend machine using a crosshead speed of 0.1-inch per minute. Fracture toughness numbers were obtained using methods based on ASTM recommendations for three-point specimens (6). Compact-tension specimens were prepared, precracked in fatigue, and tested in air at room temperature in accordance with ASTM recommendations (6). Tensile, Charpy and compact tension specimen blank locations and relative orientations for a typical HIP billet are shown in Figure 5.

Axial load fatigue testing was accomplished in air at room temperature using a 5 kip Krouse machine at a stress ratio (minimum stress/maximum stress) of $R = 0.1$ at 60 Hz. The fatigue test specimen configuration is shown in Figure 6. Fatigue specimens removed from HIP and wrought tubing were tested as-sectioned from the tubing, i.e., specimens were not flattened.

Dilatometric analysis of an as-HIP spherical powder specimen was made using a Daytronic linear variable displacement type transducer in conjunction with a fused silica measuring rod. The specimen was of 0.25-inch square cross-section and 1.5670 inches in original length. Temperature was measured to ±2°F; whereas, the accuracy of length measurement was 1×10^{-6}-inch. Thermal expansion test runs were conducted, under a positive pressure of filtered argon, at a linear heating rate of about 2°F per minute. Both temperature and dilatation of the specimen were continuously recorded.

Results and Discussion

Tensile and fracture toughness test results of Ti-6Al-4V spherical powder consolidated by HIP are tabulated in Table III.

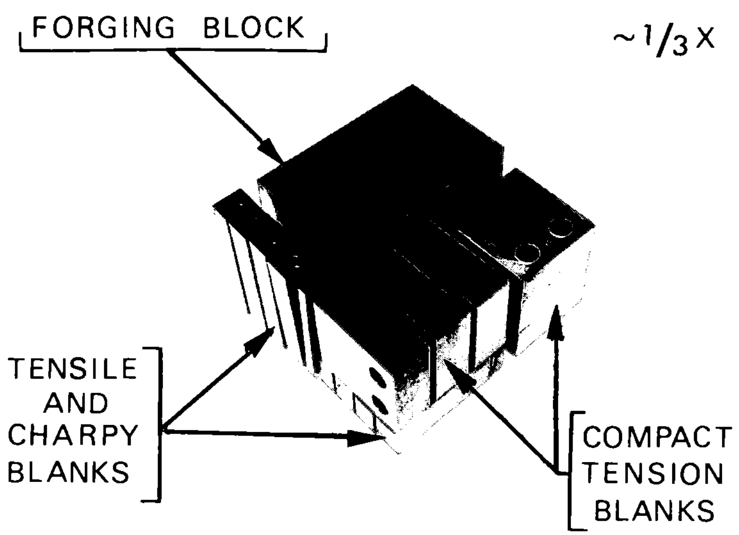

Figure 5 Isometric view of HIP billet showing specimen blank and forging block locations.

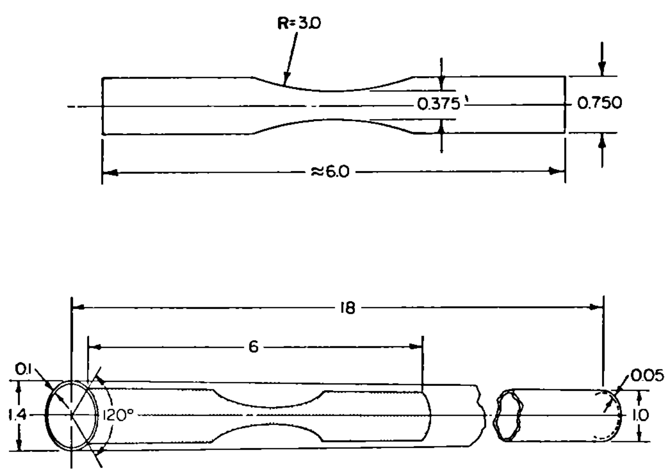

Figure 6 Illustration of fatigue test specimen configuration and its location in the HIP Ti-6Al-4V tapered tube.

TABLE III MECHANICAL PROPERTIES OF Ti-6Al-4V CONSOLIDATED BY HOT ISOSTATIC PRESSING [1]

Condition	Specimen Identity	Ultimate Strength ksi	Yield Strength 0.2% offset, ksi	Elongation in 4D, %	Reduction in Area, %	Fracture Toughness K_Q[2]	K_{IC}[3]
Annealed							
Typical Wrought Properties		130-150	120-135	10-20	20-30		
As-HIP	1-4	134.6	124.8	18	41	65(ave. of 2)	
	2-10	139.3	126.4	12	22	70	79[4]
	2-11	137.3	127.9	9	16	67	78[4]
	3-1[5]	137	125	13	21		
	3-2[6]	144	137	23	32.		
As-HIP + Vacuum Anneal 1400°F/2 hrs/FC	3-3[5]	136	123	15	25		
Solution Treated And Aged							
Typical Wrought Properties		155-172	146-162	7-11	25-30		
HIP + 1750°F/1 hr. WQ + 900°F/8 hrs. AC	1-7	155	146	18	30	56(ave. of 2)	
HIP + 1750°F/1 hr. WQ +1100°F/4 hrs. AC	2-9	170.3	160.3	11	24		56
	2-13	171.1	161.2	14	34		56
HIP + 1775°F/2 hrs. WQ + 1100°F/4 hrs. AC	2-7	169.1	157.8	14	38		55
	2-14	168.4	157.8	12	35		55
Forged							
HIP + 40% Upset From 1700°F + 1750°F/1 hr. WQ + 1100°F/4 hrs. AC	1-13	150	141	22	44	48	
HIP + Same Except Forged From 1750°F	2-F5	167	158	7	15	33	43
	2-F11	168	159	10	26	--	43
HIP + 40% Upset From 1750°F + 1300°F/2 hrs. AC	2-F7	142.1	132.7	.9	16	54	--
	2-F9	143.5	133.5	13	38	54	58

(1) 1675°F, 3 hours, 10,000 psi except as noted (see (5) and (6) below).
(2) K_Q values determined from pre-cracked Charpy specimens tested in slow bend.
(3) K_{IC} values determined from Compact Tension specimens per ASTM requirements.
(4) Not valid K_{IC} - specimen width is .75 inch; minimum width for valid test is .966 - and .941-in respectively.
(5) HIP Cycle was 1775°F, 4 hours, 2500 psi.
(6) HIP Cycle was 1750°F, 3 hours, 10,000 psi.

Tensile properties and fracture toughness of HIP material in all conditions evaluated compare favorably to those of typical wrought Ti-6Al-4V. Room temperature Charpy V-notch impact fracture energy of as-HIP spherical powder specimens (1675°F, 3 hours, 10,000 psi), averaged 19 ft-lbs (3 specimens); typical annealed wrought Ti-6Al-4V fracture energy values range from 14 to 21 ft-lbs. As-HIP properties are equivalent to wrought material in the annealed condition. Heat treatment response of HIP titanium with or without an intermediate forging step is quite good.

Forging from 1750°F plus an STA treatment yields somewhat lower mechanical properties, particularly fracture toughness, than the STA treatment alone (compare specimens 2-F5 and 2-F11 with 2-9 and 2-13). However, forging from 1700°F plus an STA treatment essentially doubles the tensile ductility with a sacrifice of about 20 ksi in both ultimate and yield strength. Also note that fracture toughness K_Q values from precracked Charpy specimens are, in each case where comparative data have been obtained, lower than K_{IC} values determined from compact tension specimens and are hence conservative values. Ronald, et al. (7) has presented an analysis of the validity of Charpy slow-bend specimens in determinations of fracture toughness.

Room temperature axial-load fatigue data for HIP tapered tubing (8) and billet materials are compared to wrought tubing in Figure 7. The endurance limit of HIP tubing material evaluated is about 8 ksi lower than that of wrought tubing similarly processed.

The thermal expansion of as-HIP Ti-6Al-4V spherical powder is shown in Figure 8. Dilatation was essentially linear from 400°F to the beta transus of 1840°F (the beta transus was determined by optical metallography to be 1840-1850°F). No irregularities in specimen dilatation that could be attributed to entrapped gas porosity was observed.

Electron-beam welding has been shown by Peebles (3) to be a sensitive test for porosity in consolidated titanium powders. The results of concurrently EB-welded HIP and wrought titanium specimens of identical size are shown in Figure 9. No evidence of porosity was observed in the fusion or heat affected zones. Note the similarity of microstructures in the two zones even though the base metal microstructures are different.

The microstructure of as-received spherical powder is shown in Figure 1 a, while the as-HIP microstructure consisting of plate-like alpha is shown in Figure 1 b and c. However, after 40% upset forging followed by an STA treatment (1775°F, 2 hours, W.Q., plus 1100°F, 4 hours, AC) the microstructure is equiaxed primary alpha in a matrix of acicular alpha plus beta (Figure 1 d). The microstructure of as-received dehydride powder is shown in Figure 2 a. Note the uniformity in microstructure from particle to particle and also the significant fraction of fines present.

The microstructure of as-HIP dehydride powder is shown in Figure 2 b and c, and is uniform equiaxed alpha and intergranular beta. Figure 2 d shows the microstructure of alpha prime plus beta and prior beta grain boundaries typical of Ti-6Al-4V water quenched from the beta phase field (2100°F, 1 hour). No evidence of "thermally induced porosity", a phenomenon that plagued a previous titanium powder investigation (3), was observed.

The microstructures of scrap-source hydrided powders are shown in Figure 3 a. Note the gross differences in the

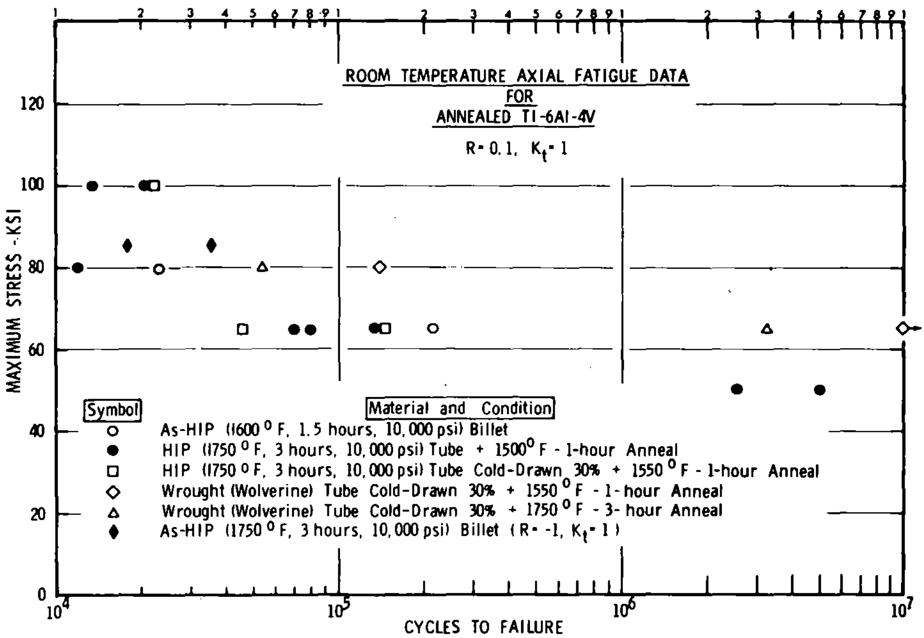

Figure 7 S/N graph of axial-load fatigue data for HIP and wrought Ti-6Al-4V.

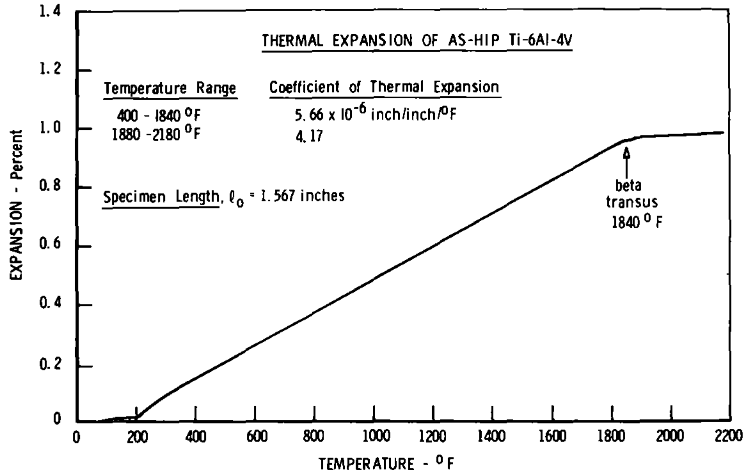

Figure 8 Thermal expansion versus temperature for as-HIP Ti-6Al-4V spherical powder.

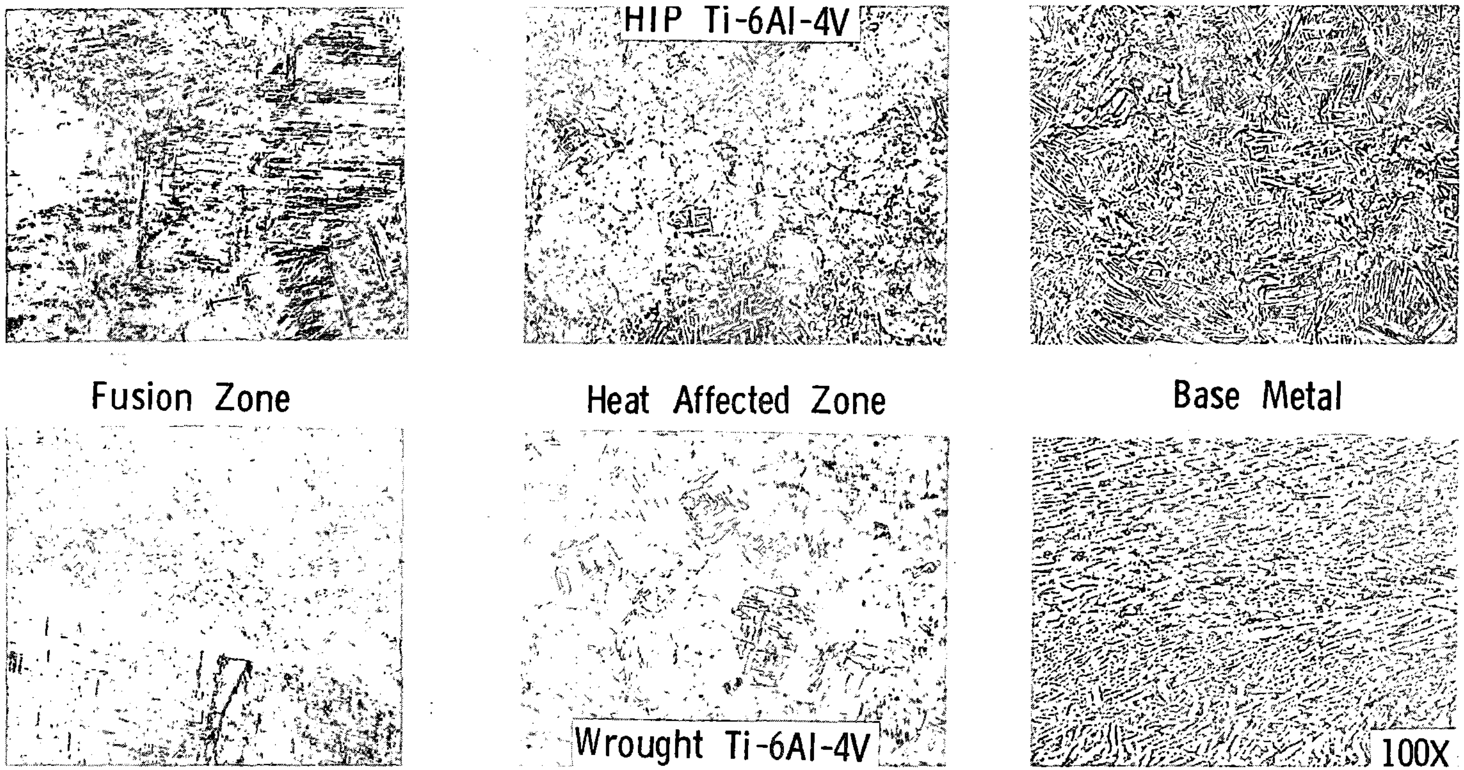

Figure 9 Comparative micrographs of electron-beam weldments of HIP (upper series) and wrought (lower series) Ti-6Al-4V showing similar microstructural behavior and freedom from porosity.

microstructures of different particles. Both cast and wrought microstructures are observable. The non-uniformity of microstructure in as-HIP material is shown in Figure 3b. Alpha-stabilized and included regions are readily visible and are indicative of gross chemical inhomogeneities in the starting powder. The interstitially stabilized alpha regions persisted even after a 2000°F, 1 hour vacuum anneal as shown in Figure 3c. Microhardness surveys showed a variation in average VHN readings (100 g load) of 380 and 620 for transformed and stabilized alpha regions, respectively. After a 2500°F, 1 hour vacuum treatment, the structure was fully transformed but some porosity remained as shown in Figure 3d.

Tungsten contamination of a single REP powder particle is shown in Figure 1e. Tungsten was identified by electron microprobe (X-ray spectral) scans.

The degree of isotropy of HIP Ti-6Al-4V spherical powder was determined by two methods; the Knoop hardness technique of Ireland (9), and X-ray diffraction techniques.

The experimental yield surface for as-HIP spherical powder, determined from hardness data, compared to that of the von Mises yield surface for an isotropic material is shown in Figure 10, and indicates excellent agreement.

Relative integrated intensities theoretically expected from a random sample were compared with those measured experimentally. Theoretical intensities were calculated from the equation (10):

$$I \sim |F|^2 p \left(\frac{1 + \cos^2 2\Theta}{\sin^2 \Theta \cos \Theta} \right)$$

where F is the structure factor; p the multiplicity factor, and Θ the Bragg angle and are presented in Table IV. It should be noted that the calculated intensities are based on the pure titanium lattice. Any ordering of solute elements in the α phase could affect the calculated values. However, the relative intensities are not expected to be affected by the alloying elements.

The intensities for the $\{10\bar{1}0\}$ and (0002) diffraction peaks were measured on a Norelco Diffractometer with CuK_α radiation. The measured intensity ratio is reasonably close to the theoretical value. In addition, the randomness of the HIP specimen was checked by determining the diffracted intensities from basal (0002) and first order prism planes $\{10\bar{1}0\}$ tilted up to 50 degrees to the sample surface on a Norelco Pole Figure Device. The ratio of

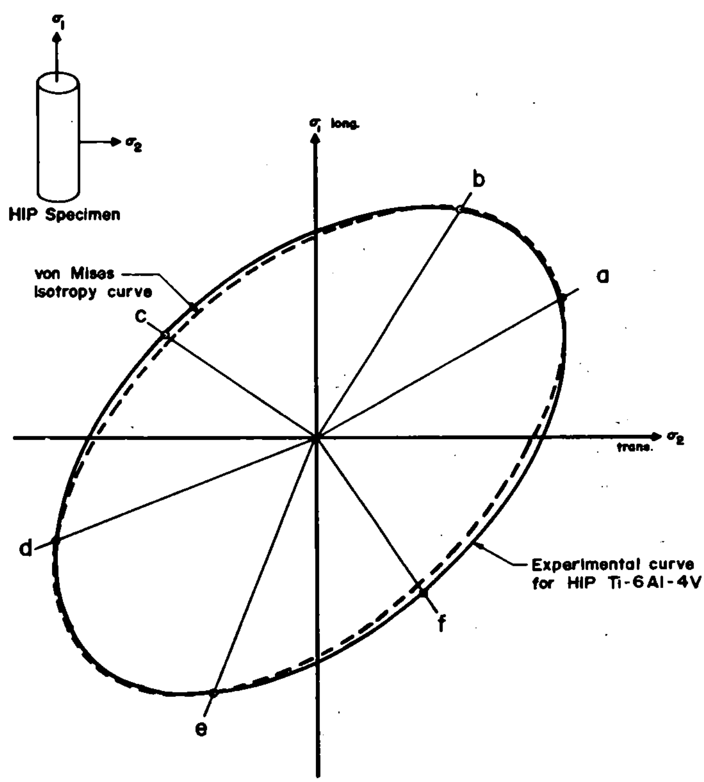

Figure 10 Experimental yield surface for as-HIP Ti-6Al-4V spherical powder showing degree of isotropy.

Table IV. Calculated Intensities From a Random Sample Compared to Measured Intensities of HIP Spherical Powder

hkiℓ	I_{rel}
$10\bar{1}0$	26.32
0002	25.94
$10\bar{1}1$	100.

$I_{(10\bar{1}0)}/I_{(0002)} = 1.01$ (calculated)

$I_{\{10\bar{1}0\}}/I_{(0002)} = 1.09$ (measured)

intensities remained constant at all angles investigated and, therefore, it is concluded that the as-HIP specimen is essentially random.

Conclusions

(1) Tensile properties and fracture toughness of Ti-6Al-4V spherical powder in as-HIP and solution treated and aged conditions are equivalent to those of wrought material.

(2) Unnotched axial-load fatigue properties are lower for HIP-Ti-6Al-4V tapered tubing material investigated than those of similarly processed wrought tubing.

(3) Ti-6Al-4V spherical powder consolidated by HIP can be subsequently hot worked by forging followed by standard annealing or STA-type heat treatments and still maintain good levels of strength, ductility and toughness.

(4) No evidence of porosity was apparent in either the dehydride or spherical HIP powders, as tested by a 2100°F/1-hour exposure of HIP dehydride powder, and electron-beam welding and dilatometric analysis of HIP spherical powder.

(5) As-HIP spherical powder, in the rectangular billet shape investigated, has been shown to be essentially isotropic with respect to both crystallographic texture and mechanical properties.

(6) This study has demonstrated that Ti-6Al-4V powder can be consolidated by hot isostatic pressing without serious increases in contamination provided careful handling and processing techniques are used.

(7) The as-HIP microstructure of Ti-6Al-4V is strongly dependent upon the microstructure of the start powder.

(8) Several problem areas of the starting prealloyed Ti-6Al-4V powders have been identified. Tungsten contamination has been observed in REP spherical powders; whereas, microstructural non-uniformity and excessive oxygen have been identified as major problem areas in the production of hydride powder from scrap-source Ti-6Al-4V material.

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