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POWDER METALLURGY TITANIUM 6Al-4V PLATE

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ABSTRACT

A powder metallurgical approach has been combined with controlled mill processing to produce a highly uniform plate material suitable for structural applications. Prealloyed ELI Titanium 6Al-4V powder produced by the rotating electrode process was consolidated into billet by hot isostatic pressing. The resulting billet of uniform composition and random texture was then hot cross-rolled to 3cm thick plate. Following rolling, the plate was given a beta annealing heat treatment to maximize damage tolerance. The plate was characterized with respect to metallurgical structure, composition, texture, and room temperature mechanical properties. The results of the study show that a powder metallurgy titanium mill product possessing uniform macro- and microstructure is technically feasible and exhibits tensile and fatigue properties equivalent to those of conventionally produced ingot-source wrought plate. Fracture toughness is, however, lower than expected. The lower toughness is shown to be associated with residual porosity in the particular plate selected for evaluation.

INTRODUCTION

Titanium mill products have historically shown wide variability in mechanical properties including dynamic properties such as toughness and fatigue. Properties are known to vary not only between materials from different heats but also between plates from the same heat or within the same plate. Such variability is due in part to compositional inhomogeniety within the starting ingot that persists through subsequent processing. Other factors contributing to variability in properties include non-uniform temperature/deformation during ingot breakdown and rolling as well as inconsistent mill rolling schedules from plate to plate. Changes in mill processing have been shown by Sommer and Creager (1) and others to exert a considerable effect on resulting crystallographic texture and microstructure and hence mechanical properties.

In view of the progress made recently in controlling the mill processing and chemistry of titanium plate and sheet by Boyer and Bajoraitis (2) for damage tolerant applications, it was considered timely to combine the powder metallurgical route with controlled mill processing with the expectation and

objective of producing a highly uniform plate mill product to demonstrate feasibility. This study was therefore conducted to demonstrate the feasibility and show the potential of the powder metallurgical route to mill products.

The approach of this study was to consolidate state-of-the-art prealloyed ELI grade Ti-6Al-4V rotating electrode powder by hot isostatic pressing into a 13.5 Kg billet of uniform composition and random texture. The billet was then uniformily hot cross-rolled to 3 cm thick plate using a laboratory mill and subsequently given a beta annealing heat treatment aimed at maximizing damage tolerance. The resulting plate was characterized with respect to metallurgical structure, composition, texture, and mechanical properties including tensile, toughness, smooth fatigue, and fatigue crack growth characteristics. The results of this study were then compared to existing data for conventionally produced plate given the same heat treatment. A single plate, produced using fixed parameters during consolidation, rolling, and heat treatment was evaluated since funding limitations precluded a more extensive investigation. However, this study, albeit limited in scope, is considered adequate to show the high potential of the powder metallurgical approach to titanium mill products.

EXPERIMENTAL

For this study, wrought bar stock 2.5 inches in diameter purchased to MIL-T-9047 E, Composition 7 (ELI Ti-6Al-4V) specifications was converted to spherical powder by the well known rotating electrode process (REP). The chemical composition of the starting material and the resulting powder, before and after consolidation, is given in Table I along with appropriate comparative specifications. The processing steps used to produce plate material from powder are shown schematically in Figure 1. The minus 325 mesh powder was placed in a container fabricated by GTA welding of .060-in. low carbon steel sheet. The container also had a seamless steel tube welded to the top portion for filling and evacuation. Vibratory loading was used to achieve a tap density of about 66 percent of theoretical. Evacuation and sealing was accomplished in an electron beam welding chamber. The sealed container was leak checked and then hot isostatically pressed at 1700^{o}F for 4 hours at 15 ksi pressure. The consolidated billet, with the steel container material intact, was then hot rolled from 1730^{o}F using a 2-high, 14-inch laboratory mill. Some 35 passes and 3 intermediate reheats to 1730^{o}F with uniform cross-rolling were conducted as shown in the rolling schedule of Table II. Following rolling, the 1.25-inch thick plate was given a beta annealing heat treatment of 1900^{o}F for 20 minutes followed by air cooling to room temperature. After heat treatment, the protective steel container material was stripped from the titanium plate by a combination of mechanical peeling and dissolution in a nitric acid bath.

The resulting titanium plate was evaluated using the specimen layout scheme shown in Figure 2. Tensile specimens with a nominal gage length of 1 inch and a diameter of .25 inches were tested per ASTM E-8 procedures. The strain rate was .005 in./in./min through 0.2% yield and .10 in/min head speed thereafter to failure. Smooth fatigue testing was conducted with a constant cross-section specimen of .25-in. diameter. Specimens were machined using special low stress grinding procedures developed by Metcut Research Associates Inc. primarily under Air Force sponsorship. Testing per ASTM E466-76 was accomplished in the axial load control mode, an R ratio of 0.1, a frequency of 30 Hz, and a sinusoidal wave form using a closed loop servo controlled hydraulic system of 20 ksi capacity. Fracture toughness testing was accomplished per ASTM E 399-78 using the compact tension type specimen with a thickness, B, of 1.21 inches. The test environment for toughness testing (and all other mechanical property testing) was ambient laboratory air at 78°F and 53% relative humidity. A calibrated 60 kip hydraulic tensile machine in conjunction with a load cell, clip gage, and x-y plotter was used for toughness testing. Fatigue crack growth rate testing was conducted per ASTM E 647-78T specifications using a calibrated rotating mass type Sonntag unit fitted with a 5:1 load multiplier and a calibrated load cell. A constant force sinusoidal waveform of 30 Hz frequency with R= min./max.=.053 was used for test loading. Crack length measurements were made using a 14X traveling microscope. Raw crack length versus number of cycles data were reduced using the 7-point incremental polynomial method as outlined in the cited ASTM specification. Confirmatory K_Q values were also obtained from the crack growth rate specimens the dimensions of which were 3.0 in. in depth, 0.75 in. in thickness, 3.60 in. in height, and 3.75 in. in length.

DISCUSSION OF RESULTS

Mechanical Properties

Tensile and toughness properties of the PM HIP Ti-6Al-4V plate material of this study are presented in Figure 3. The as-rolled plate tensile properties are those of an earlier plate processed almost identically to the plate more extensively evaluated; however, the as-rolled plate was fully dense, that is, no residual porosity was shown. The reduction in area was low in one of the two specimens evaluated. The reason for the anomalously low ductility will be discussed under metallurgical properties later in this paper. Tensile properties of the beta annealed plate compare favorably to those of the reference wrought plate, exhibit high uniformity, and show little directionality except ductility. Fracture toughness was disappointingly low for a beta annealed material and also exhibited directionality. Since rolling was accomplished in a uniform manner, that is, the plate was rotated 90° after each pass, directionality effects were unexpected. Smooth fatigue data are shown in Figure 4. The PM plate material of this study compares quite favorably in fatigue to that of conventional wrought plate, particularly in the high cycle region. Fatigue crack growth rates in the PM plate are considerably lower than the wrought plate counterpart as shown in Figure 5. The two materials crack growth rates tend to converge at lower values of delta K, however.

Metallurgical Properties

Before discussing the metallurgical aspects of the PM plate of this study, a notable observation related to consolidation of the powder should be mentioned. Virtually no distortion of the container during consolidation was evident, only a reduction in dimensions uniformly scaled to the original dimensions of the container was observed. Figure 6 shows the as-consolidated billet which is merely a reduced version of the original container. Such uniform volume reduction during the HIP cycle is believed due to several aspects of the canning techniques, namely, the selection of low carbon steel material (low yield strength), the care in which the vibratory loading was conducted, and the lack of excess stiffening material due to the simple design of the container.

The macrostructure of the PM plate for which full-range mechanical properties were obtained is shown in the isometric view of Figure 7. Residual porosity is evident in both the transverse and longitudinal sections of the plate. Even more significant is the observation that the porosity is elongated in the longitudinal reference direction of the plate, particularly since uniform cross-rolling was used during plate fabrication. Microstructures of the as-rolled plate and after beta annealing are shown in Figure 8, parts C and D, respectively. Porosity in the as-rolled microstructure is difficult to observe and appears extremely fine but somewhat aligned; however, after the high temperature heat treatment, expansion and/or coalescence of the voids occurred. Note the uniformity of structure and the fineness of the prior beta grains shown in Figure 7 and Figure 8, A and B. The scanning electron micrographs of Figure 9 show the pronounced effect residual porosity (less than 2% determined gravimetrically) has on toughness. Micrographs A and B show that the residual porosity is directional and has a strong effect on fracture surface appearance, that is flatness of fracture and hence the extent of energy absorbtion. Micrograph A representing the TL oriented toughness specimen would be expected to exhibit lower toughness due to the aforementioned lesser roughness of the surface. Note, however, that in the precracked regions the voids appear to play a relatively unimportant role in fatigue, again this observation is consistent with the good fatigue and fatigue crack growth properties exhibited. Indeed, in fatigue the voids may well have served as crack arresters. However, in the case of toughness and tensile ductility any geometrical discontinuity such as a void increases the true stress near the void surfaces, linkup is easier (see the micrographs of C and D, Figure 9), and the effective cross-sectional area is reduced. The anomalously low ductility shown by a single tensile specimen from the as-rolled plate is explained by an internal tungsten inclusion as exhibited in Figure 10. The source of the tungsten is presumably the electrode used in the powder making process.

CONCLUSIONS

The feasibility of using the powder metallurgical approach combined with controlled mill processing to produce a highly uniform plate product has been demonstrated in this study. Mechanical properties compare quite favorably to those of conventionally produced plate with the exception of toughness which was shown to be directly related to residual porosity in the particular plate selected for evaluation. The metallurgical structure of the PM plate is highly uniform as is the plate chemistry. The potential for further development is considered very good, particularly if the blended elemental route to low cost titanium plate and sheet can be demonstrated and developed. The powder metallurgical approach to mill products offers a number of opportunities for improved and innovative materials such as, dual alloys, graded compositions, laminates, composites, dispersion strengthened high temperature sheet, and new alloy compositions without restriction due to segregation of alloying elements.

REFERENCES

1. A. W. Sommer and M. Creager, Research Toward Developing an Understanding of Crystallographic Texture on Mechanical Properties of Titanium Alloys, AFML-TR-76-222, Air Force Materials Laboratory, Wright-Patterson AFB, Oh, 45433, January, 1977.

2. R. R. Boyer and R. Bajoratis, Standardization of Processing Conditions, AFML-TR-78-131, Air Force Materials Laboratory, Wright-Patterson AFB, Oh, 45433, September, 1978.

ACKNOWLEDGMENT

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CHEMICAL COMPOSITION, WEIGHT PERCENT

Material Condition	O	Al	V	Fe	C	N	H	Y	W
Starting Wrought Bar Stock-Ti-6Al-4V ELI	.121	6.1	4.0	.15	.03	.022	.0052	<.005	--
MIL-T-9047E, Comp 7 Spec.	.13 Max.	5.5/ 6.75	3.5/ 4.5	.15 Max.	.08 Max.	.05 Max.	.0125 Max.		
REP Powder, Lot 4608-A2, Nuclear Metals, Inc. Analysis	.118								
Consolidated Powder Sample, Same Lot, Crucible, Inc. Analysis	.124 .120						.0062		
Consolidated Plate									
Specimen TT-1	.132	5.87	3.87	.21	.019	.015	.0075	<.001	<.02
Specimen TT-3	.138	5.89	3.88	.20	.020	.019	.0068	<.001	<.02
Specimen TT-5	.130	5.91	3.84	.21	.020	.015	.0054	<.001	<.02
Reference Wrought Plate, "Super ELI", AFML-TR-78-131	.102	6.1	4.0	.15	.02	.010	.0048	<.005	--
Proposed Specification, AFML-TR-78-131	.11 Max.	5.7/ 6.2	3.6/ 4.4	.25 Max.	.05 Max.	.03 Max.	.0125 Max.	.005 Max.	

TABLE I

ROLLING SCHEDULE FOR PM HIP TITANIUM PLATE

Pass Number(s)	Mill Gap Reduction Per Pass (inches)	Plate Thickness (inches) Last Pass	Reheat No. & Time (minutes) to 1730°F	Plate Temperature (°F) Just Prior to Reheating
0	--	5.125	Starting Billet	1730
1-12(A)	.125	3.625	(1) 32	1500
13-23	.125	2.25	(2) 30	1400
24-29	.125	1.50	(3) 31	1400
30-33	.060	1.26		
34	.020	1.24		1400
35	0	Flattening Pass		Air Cooled To R.T.

(A) Billet rotated 90° between each pass throughout rolling, e.g., uniform cross rolling.

TABLE II

SCHEMATIC OF THE POWDER METALLURGICAL ROUTE TO PLATE FABRICATION

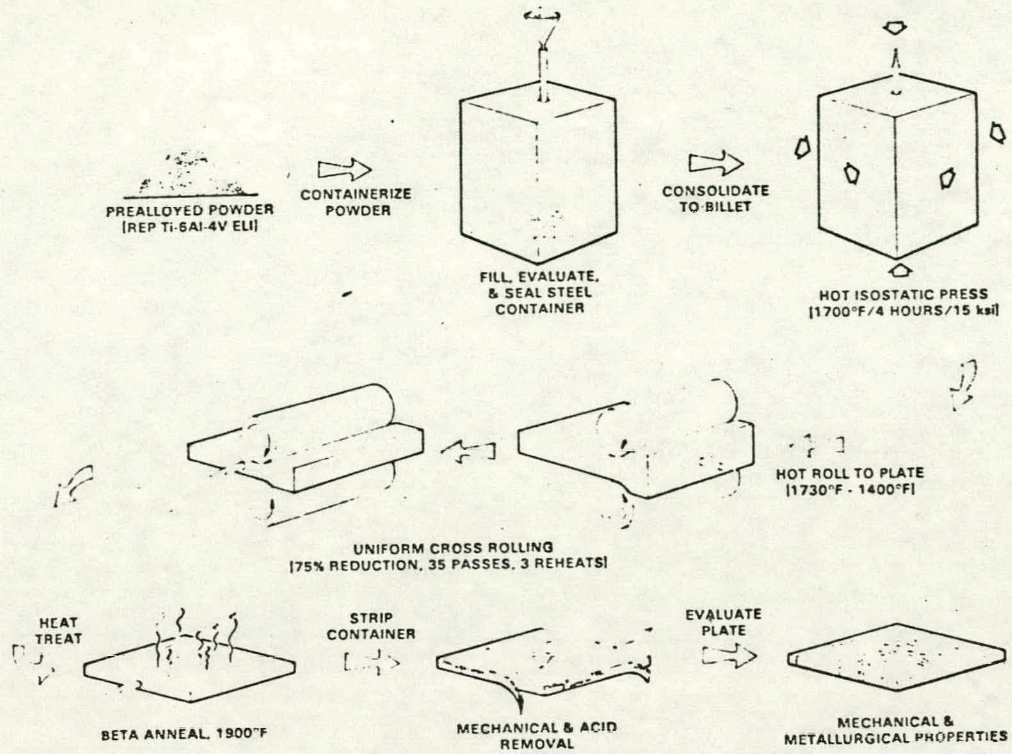


FIG 1

SPECIMEN LAYOUT FOR PM HIP TITANIUM PLATE

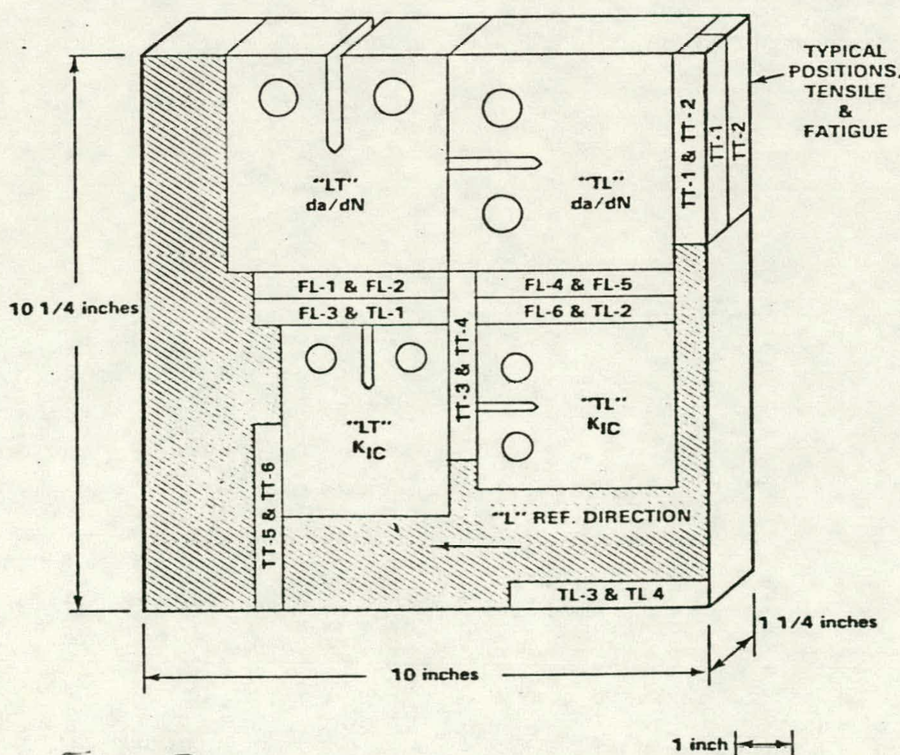


FIG 2.

TENSILE AND TOUGHNESS PROPERTIES OF PM HIP Ti-6Al-4V PLATE

Condition/ Orientation	Number of Tests	UTS(A) (ksi)	.2% YS (ksi)	% Elong in 1 inch	% RA	E (x10³ ksi)	KIC (ksi √in.)
As-rolled PM Plate							
Transverse	2	144(B)	139	12	35 (46. 23)	18	Not Determined
Beta Annealed PM Plate							
Longitudinal (LT)	5	130±.8	119±1.1	13	31	17	66.4(C)
Transverse (TL)	5	130±.8	118±1.1	11	23	17	55.3
Reference Wrought "Super ELI" Beta Annealed Plate (AFML-TR-78-131)							
Longitudinal	3	132±1.5	122±3.1	11	20		96
Transverse	3	133±1.0	122±1.2	11	23		89
Reference Wrought Mill-Annealed Plates From 2 Vendors (AFML-TR-78-158)	3	145±3.7	138±3.4	17	27	18	66
Proposed Specification Minimums		125	112	8			85

(A) Mean room temperature values ± one standard deviation where indicated.
(B) To convert from ksi to MPa, multiply by 6.89.
(C) To convert from ksi ·√in. to MPa ·√m, multiply by 1.1.

FIG. 3

SMOOTH FATIGUE DATA FOR BETA ANNEALED ELI Ti-6Al-4V PLATE

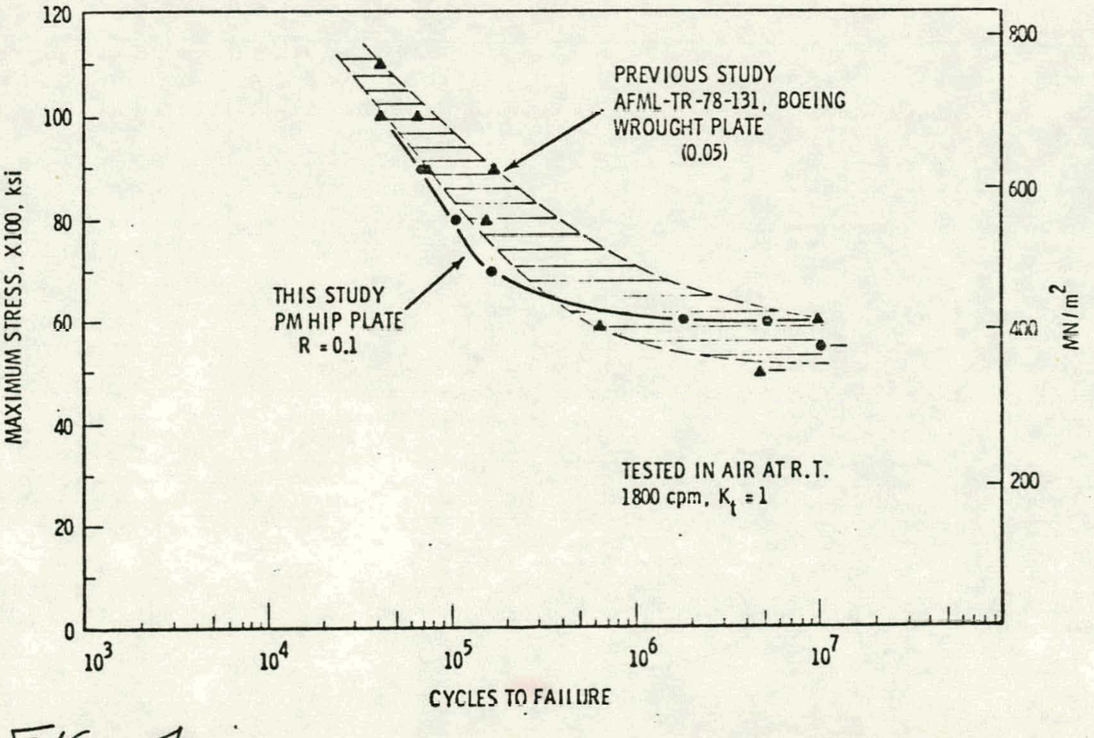


FIG. 4

FATIGUE CRACK GROWTH RATE VERSUS ΔK FOR BETA ANNEALED Ti-6Al-4V PLATE

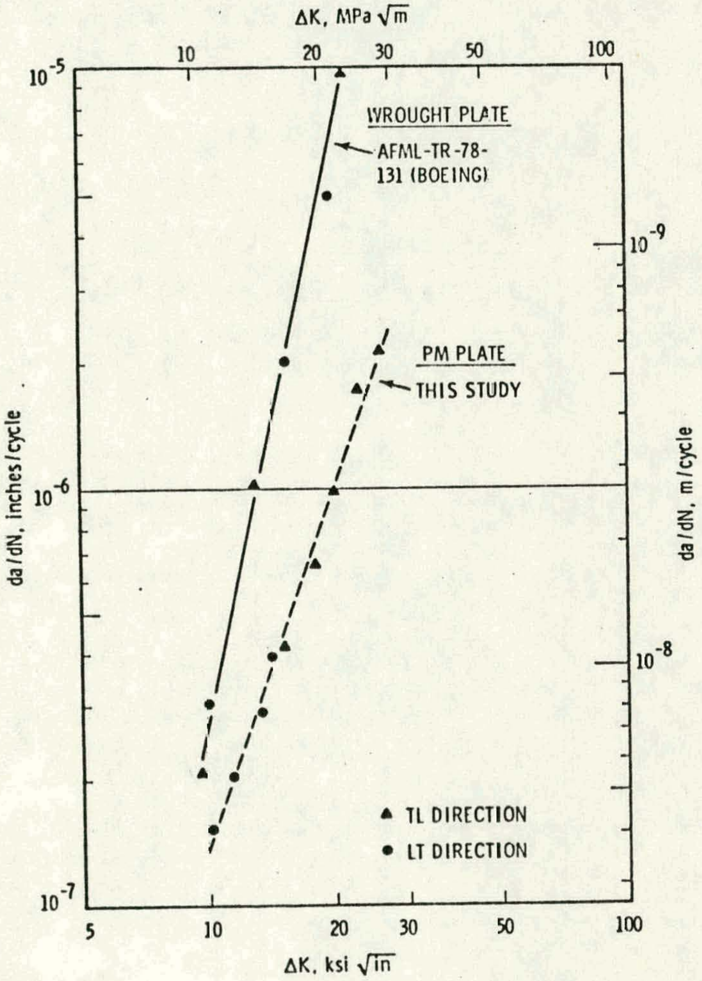


FIG 5.

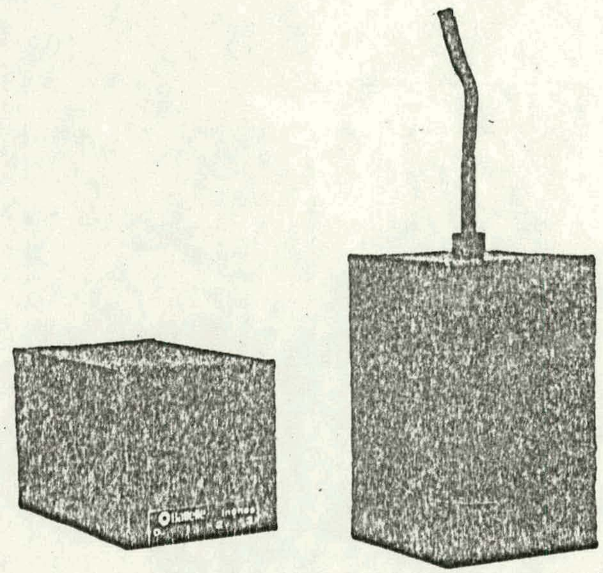


FIGURE 6. AS-CONSOLIDATED (HIP) TITANIUM BILLETS WITH STEEL OUTER JACKETS (CONTAINERS) STILL IN PLACE. NOTE THAT THE BILLET SHAPES ARE NEARLY DISTORTION FREE.

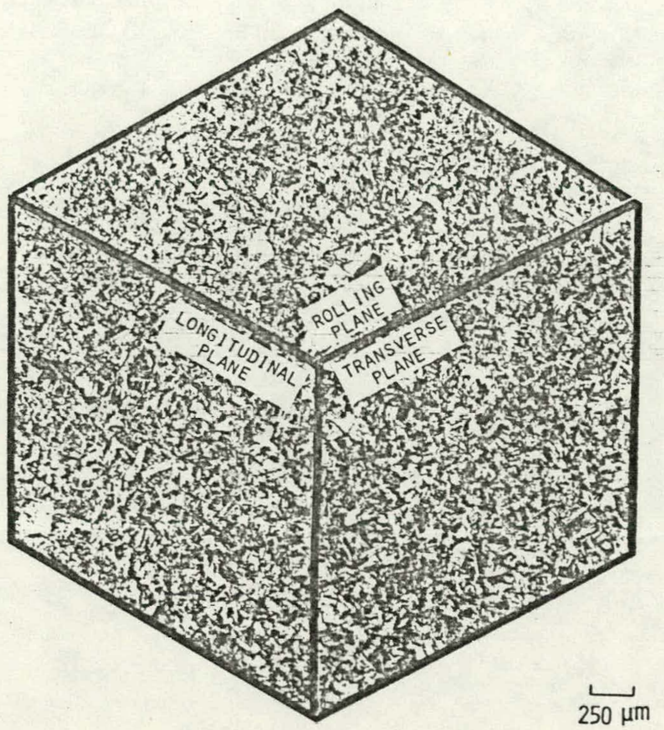


FIGURE 7. ISOMETRIC VIEW OF THE PM HIP TITANIUM PLATE MACROSTRUCTURE SHOWING A HIGHLY UNIFORM AND SMALL PRIOR BETA GRAIN SIZE AND THE ALIGNMENT OF VOIDS IN THE LONGITUDINAL DIRECTION.

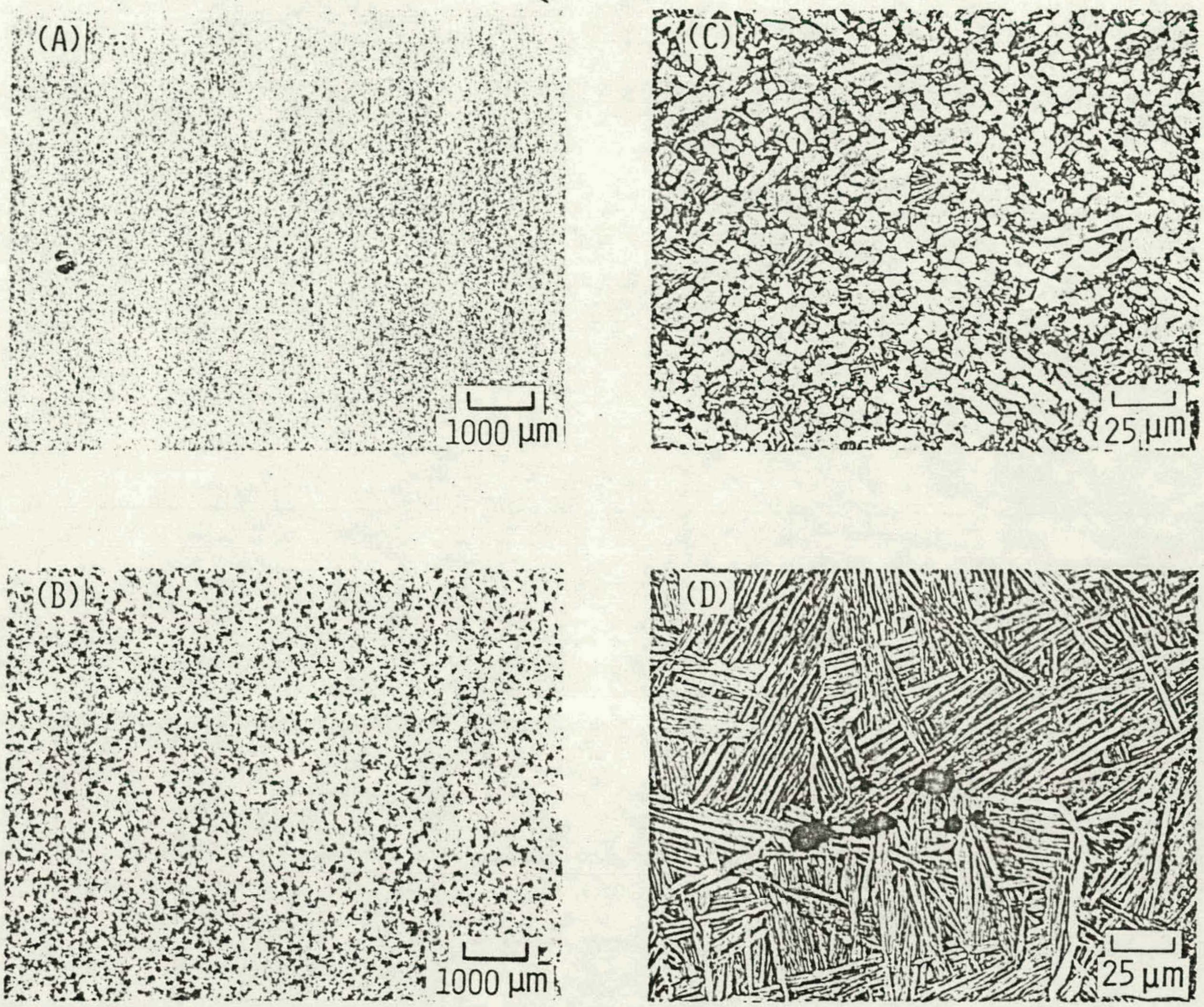


Figure 8. PM HIP Titanium Plate Macrostructures (A) As-Rolled and (B) After Beta Annealing and, Microstructures (C) As-Rolled and (D) After Beta Annealing. Although Some Residual Porosity is Shown in the As-Rolled Microstructure (C), the Porosity is More Pronounced After a High Temperature Heat Treatment as Shown in (D).

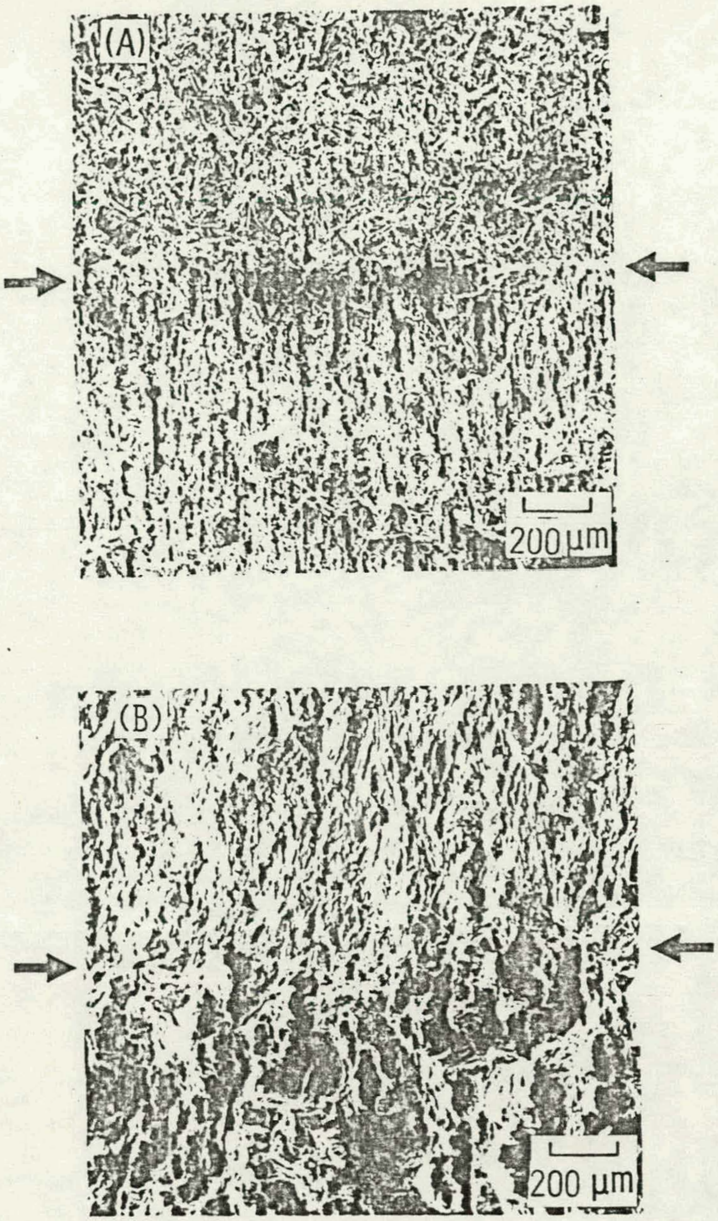
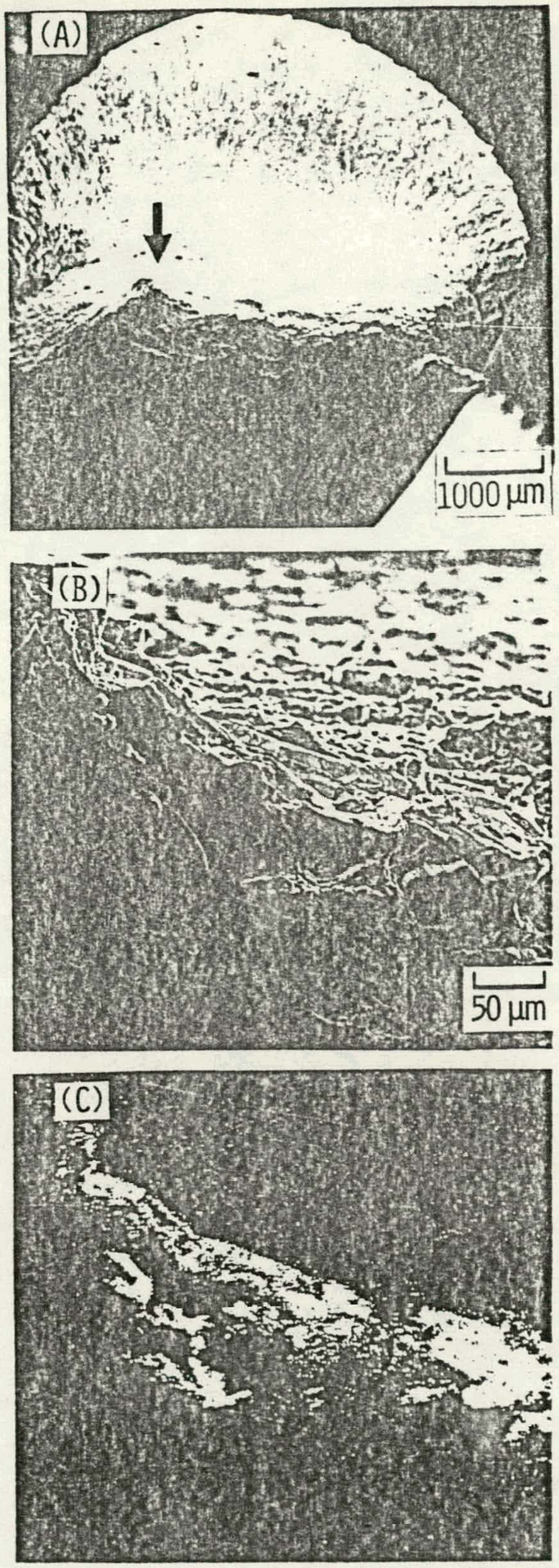


FIGURE 10. SCANNING ELECTRON MICROGRAPHS OF THE FRACTURE SURFACES OF FRACTURE TOUGHNESS SPECIMENS: (A) AND (B) SHOWS THE TRANSITION REGION FROM FATIGUE PRECRACKING TO RAPID FRACTURE (INDICATED BY ARROWS) FOR K_{IC}-TL AND K_{IC}-LT, RESPECTIVELY; (C) A HIGHER MAGNIFICATION OF (A) ABOUT 5MM BELOW THE TRANSITION LINE SHOWING VOID LINKING AND SOME EVIDENCE OF CRACKING BETWEEN VOIDS; (D) A FAST FRACTURE REGION OF SPECIMEN K_{IC}-TL RELATING THE UNDERLYING MICROSTRUCTURE TO THE FRACTURE SURFACE.



X-RAY DETECTOR

FIGURE 9. FRACTURE SURFACE EVALUATION OF TENSILE SPECIMEN TT-7 EXHIBITING LOW DUCTILITY: (A) LOW MAGNIFICATION SEM OF THE FRACTURE SURFACE WITH THE FRACTURE ORIGIN SHOWN BY THE ARROW, (B) HIGHER MAGNIFICATION OF THE FRACTURE ORIGIN, (C) CHARACTERISTIC X-RAY (TUNGSTEN M LINES) AREA SCAN OF (B).