NASA CR 72469
NM 9902

PROPERTIES OF POWDERED TITANIUM ALLOYS

by

Gerald Friedman

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Contract NAS 3-10301

WHITTAKER CORPORATION
Nuclear Metals Division

NOTICE

Requests for copies of this report should be referred to

National Aeronautics and Space Administration
Office of Scientific and Technical Information
Attention: AFSS-A
Washington, D. C. 20546

NASA CR 72469
NM 9902

TOPICAL REPORT

PROPERTIES OF POWDERED TITANIUM ALLOYS

by

Gerald Friedman

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

September 1968

CONTRACT NAS 3-1030

Technical Management
NASA Lewis Research Center
Cleveland, Ohio
Large Engine Technology Branch
John M. Kazaroff

WHITTAKER CORPORATION
Nuclear Metals Division
West Concord, Massachusetts

PRECEDING PAGE BLANK NOT FILMED.

PROPERTIES OF POWDERED TITANIUM ALLOYS

by

Gerald Friedman

ABSTRACT

Titanium alloys Ti-6Al-4V and Ti-5Al-2.5Sn were converted to powder by four different techniques. The powders were compared with respect to chemical analysis, particle shape and size distribution, flow rate, and bulk density. Each powder was compacted in evacuated steel cans at three pressures and temperatures. The hot pressed compacts were compared with respect to chemical analysis, density, hardness, and mechanical properties in tension. Additional tensile tests were then performed on heat treated samples of low-oxygen-level compacts.

PROPERTIES OF POWDERED TITANIUM ALLOYS

By Gerald Friedman

Whittaker Corporation, Nuclear Metals Division

SUMMARY

Low oxygen (ELI) Ti-6Al-4V and Ti-5Al-2.5Sn forged bar was converted to powder by the hydride-dehydride process, mechanical attritioning of chips, the rotating electode process, and by a chemical coreduction procedure. Evaluation of the powders showed that only the spherical powders made by the rotating electrode technique retained oxygen contents below 1000 ppm. The irregular powders made by the other techniques had oxygen contents ranging from 1220 (attrited chips) to 8100 ppm (coreduced). The powders were compacted at 1450, 1650, and 1850°F, 50, 75, and 100 tsi. Except for the coreduced powders, all samples were over 99% dense at a pressure/temperature combination of 75 tsi and 1650°F, or higher. Analysis of the various compacts showed that only those made from the rotating electrode process powder had not oxidized further. Excluding the coreduced powder compacts, the oxygen contents in the compacts made by the remaining techniques now ranged from 1570 ppm (hydride-dehydride) to 2800 ppm for one of the attrited powders.

Tensile test results coupled with the oxygen analyses led to the selection of the rotating electrode process powder compacts for further study. Heat treated Ti-6Al-4V samples had these properties: 132-140,000 psi UTS, 124-131,000 YS, 10-11% elongation, and 32-44% RA. For the Ti-5Al-2.5Sn alloy, the comparable values were 122-128,000 psi UTS, 113-118,000 psi YS, 11-19% elongation, and 26-45% RA.

INTRODUCTION

This is a report on the first part of a three-part program performed for the purpose of evaluating the strength and toughness of hot compacted titanium alloy powders.* The program objective is to determine if solid bodies - eventually of large size and complex cross section - formed from high-purity (low oxygen) powders would indeed possess enhanced fracture

---

*The major phases of the program are (1) the comparison of the various titanium powders, (2) determination of optimum hot pressing conditions for the two alloys, and (3) pressing and testing 6 x 6 x 3 inch blocks of each alloy.

toughness expected from the naturally fine grain size of a powder metallurgy product. Additional expected benefits would be the uniformity of grain size and chemical homogeneity that are obtainable by such a powder-based fabrication process.

This paper describes the work done in hot compacting Ti-6Al-4V and Ti-5Al-2.5Sn powders produced by four different techniques. The powders and the compacts were compared with respect to physical, chemical, and mechanical criteria. Heat treatments were developed which would produce optimum mechanical properties in the hot-pressed bodies.

SAMPLE PREPARATION AND TESTING

Ti-6Al-4V and Ti-5Al-2.5Sn ELI grade (Extra-Low Interstitial Content) forged bars were purchased by Nuclear Metals. Sections of each bar were sent to Nuclear Materials and Equipment Corporation, Apollo, Pennsylvania, for conversion to powder by both the hydride-dehydride process (Numec Hyd) and by mechanical attrition (Numec MA). In the former process, powder is prepared by enclosing the titanium bar in a sealed chamber which is then evacuated and back-filled with hydrogen as the chamber temperature is raised. Whereas titanium is capable of dissolving nearly 8 atomic percent of hydrogen at 600°F, the reacted mass of metal can retain only from 0.05 to 0.14 atomic percent hydrogen at temperatures below 250°F. The excess hydrogen is therefore forced out of solution at these lower temperatures and is present as a titanium hydride phase. At levels above 200 ppm, the precipitated hydrogen embrittles the matrix, thus making possible a relatively simple crushing operation. The powder which results from crushing the titanium hydride is then converted back to the metallic state by a subsequent vacuum heat treatment.

In the mechanical attrition process, the forged bar was machined to chips, which were then converted to powder in a hammer mill. The machining and milling operations were performed in a helium atmosphere.

Dominion Magnesium, Ltd., Toronto, Canada (Dom), prepared ten pounds of Ti-6Al-4V powder by the coreduction of titanium, aluminum, and vanadium compounds.

Ten pounds of Ti-5Al-2.5Sn powder were procured from the Penn-Nuclear Corporation, Penn, Pennsylvania (Penn), which used a combination gas impingement and mechanical attrition technique to produce powder from their own starting stock.

Ten pounds of each alloy powder were prepared at Nuclear Metals by their rotating electrode process (REP). Portions of the same forged bars sent to Numec were extruded, cut into 10-inch lengths, and finish machined to 1-1/4-inch diameter bars. A description of the rotating electrode process follows:

Appropriate metal rods are loaded into an 8-foot diameter tank which is subsequently evacuated and back-filled with helium. By means of a glove-port in the tank wall, each bar is positioned in turn in the chuck of a high-speed spindle inside the tank. When the electrode has attained the desired rotational speed, an arc is struck between the face of the titanium bar and a non-rotating tungsten electrode. The combined action of the arc and the rotating electrode results in the formation of spherical droplets which fly off from the electrode face. The droplets freeze in flight into spherical powder particles and are completely solid before they drop to the floor of the tank.

Powder evaluation began by transferring each powder in turn to the inert-gas glove box. Each ten pound powder lot was then passed through a sample splitter so that representative samples could be taken for the various tests. The samples and the balance of the powder from each vendor were then removed from the glove box in sealed containers. Shadowgraphs of the various powder types are shown in Figure 1.

Sealed vials of powder were sent to an independent analytical laboratory* for chemical analysis. Comparing the results (Table I) of the chemical analysis with the appropriate specification,** we see that: (1) the Dominion Ti-6Al-4V powder is grossly out of specification with respect to oxygen (8100 ppm), carbon (940 ppm), hydrogen (2960 ppm), and vanadium (2.7%). The high interstitial content of this powder explains its poor performance in the hot pressing tests (see below); (2) only the Nuclear Metals Rotating Electrode powders meet ELI specification for titanium. The Numec powders were made from the same low-oxygen forged bars that Nuclear Metals used, but the oxygen level in Numec's four powders ranges from 1220 to 1560 ppm.

The Penn Nuclear powder, containing 2800 ppm oxygen, exceeds the limit for standard grade Ti-5Al-2.5Sn.

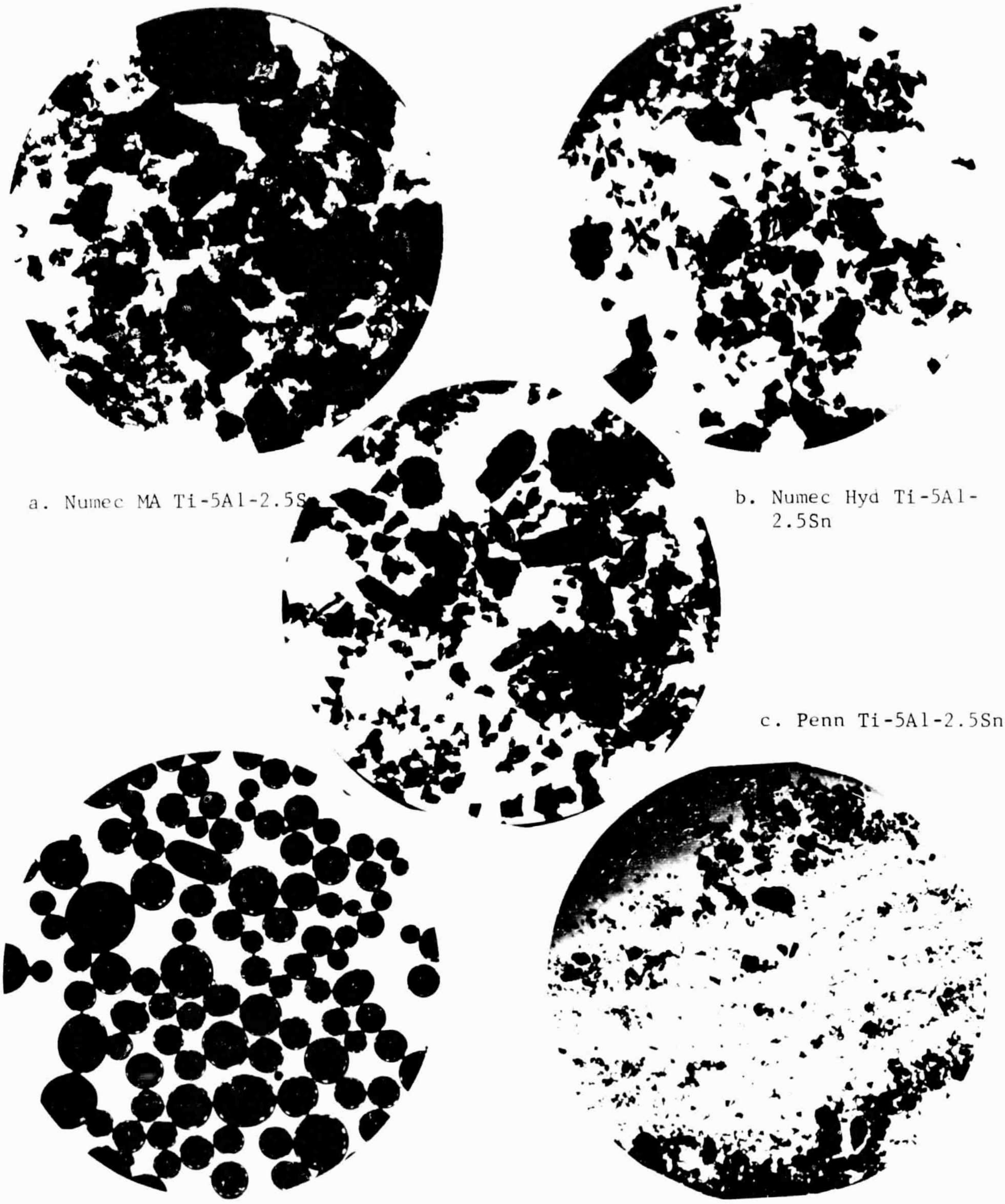
Based on these data, the powders can be placed in three categories: In the first group are the ELI powders, with very low oxygen contents. In the second group are powders with approximately twice the oxygen in the first group; these powders are still within specification for normal titanium alloys. Powders in the third group have very high oxygen contents.

The Nuclear Metals (NM) powders fall into the first category, the Numec powders into the second, and the Penn and Dominion powders constitute the third group.

---

*Ledoux and Company, Teaneck, New Jersey.

**MIL-T-9047D, "Titanium Alloy Bars, Forgings, and Forging Stock", 9 June 1967.



a. Numec MA Ti-5Al-2.5Sn

b. Numec Hyd Ti-5Al-2.5Sn

c. Penn Ti-5Al-2.5Sn

d. NM (REF) Ti-5Al-2.5Sn

e. Dom Ti-6Al-4V

Figure 1. Titanium alloy powders. 25X

TABLE I.- CHEMICAL ANALYSES FOR EIGHT TITANIUM ALLOY POWDERS

A. Ti-6Al-4V	Al (%)	V (%)	Fe (%)	O (ppm)	C (ppm)	N (ppm)	H (ppm)
Sample							
NUMEC MA[a]	5.84	4.19	0.16	1560	130	189	98
NM[a]	5.67	4.30	0.15	750	67	146	58
Dominion	5.72	2.70	0.07	8100	940	187	2960
NUMEC Hyd[a].	5.94	4.32	0.21	1300	61	155	213
6 inch forged bar	6.31	4.27	0.11	700	270	70	16
MIL Spec., Ti-6Al-4V[b]	5.50-6.75	3.50-4.50	0.30 (max)	2000 (max)	1000 (max)	500 (max)	150 (max)
MIL Spec., Ti-6Al-4V, ELI . . .	5.50-6.75	3.50-4.50	0.25 (max)	1300 (max)	800 (max)	500 (max)	125 (max)

B. Ti-5Al-2.5Sn	Al (%)	Sn (%)	Fe (%)	O (ppm)	C (ppm)	N (ppm)	H (ppm)
Sample							
NM[c]	5.25	2.49	0.20	750	27	106	39
Penn	5.16	2.45	0.20	2800	96	523	187
NUMEC Hyd[d].	4.93	2.41	0.04	1380	22	149	56
NUMEC MA[d]	4.98	2.40	0.07	1220	42	191	90
6-1/2 inch forged bar	5.46	2.80	0.02	740	60	140	6
1-1/4 inch ground bar	5.50	2.40	0.25	950	100	100	60
MIL Spec., Ti-5Al-2.5Sn	4.0-6.0	2.0-3.0	0.50 (max)	2000 (max)	1500 (max)	700 (max)	200 (max)
MIL Spec., Ti-5Al-2.5Sn, ELI. .	4.25-5.75	2.0-3.0	0.25 (max)	1200 (max)	800 (max)	700 (max)	125 (max)

[a]These three powders were made from the 6 inch forged bar.

[b]MIL-T-9047, "Titanium Alloy Bars, Forgings, and Forging Stock", 9 June 1967.

[c]Made from 1-1/4 inch ground bar to replace accidentally contaminated batch of powder made from the 6-1/2 inch bar.

[d]These two powders made from the 6-1/2 inch forged bar.

Additional samples obtained from the sample splitter* were used to determine flow rate, apparent density, and particle size distribution. Flow rate and apparent density determinations were carried out in accordance with the appropriate ASTM specifications:

	Flow Rate (seconds/50g)	Apparent Density (g/cc)	Apparent Density (% of theo.)
1. Ti-6Al-4V			
NM	24	2.72	61.5
Numec MA	No flow	1.13	25.6
Numec Hyd	44.5	1.65	37.2
Dominion	No flow	1.29	29.2
2. Ti-5Al-2.5Sn			
NM	22	2.83	63.5
Numec MA	No flow	1.52	34.2
Numec Hyd	51	1.51	34.0
Penn	44	1.79	40.2

These two tests illustrate a major difference between the irregular and the spherical powders. The angular-blocky Dominion and Numec powders are much more "fluffy" than the Nuclear Metals powder, and in fact occupy twice the volume for equal weights. The blocky-particle powders have a strong tendency to bridge over, which accounts for their poor flow characteristics in the Hall Flowmeter. The Nuclear Metals spherical powders flow very easily.

Sieve analyses were determined by slightly modifying the ASTM technique. Instead of obtaining an exact 100 g sample of each powder, the split closest to 100 g was used. In this way, a truly representative sample of powder was obtained from each lot, and the risk of analyzing a non-representative sample was avoided. The sieve analyses and size distribution curves for the four powders are presented in the attached data sheets, Figures 2-5.

---

*A sample splitter, or riffler, reduces a large powder sample to a quantity suitable for testing purposes, while still retaining in the small "split" sample the same proportion of fine and coarse powder. The splitter divides the sample into halves, and by repeating the operation the sample can then be split into quarters, eighths, sixteenths, etc., as desired.

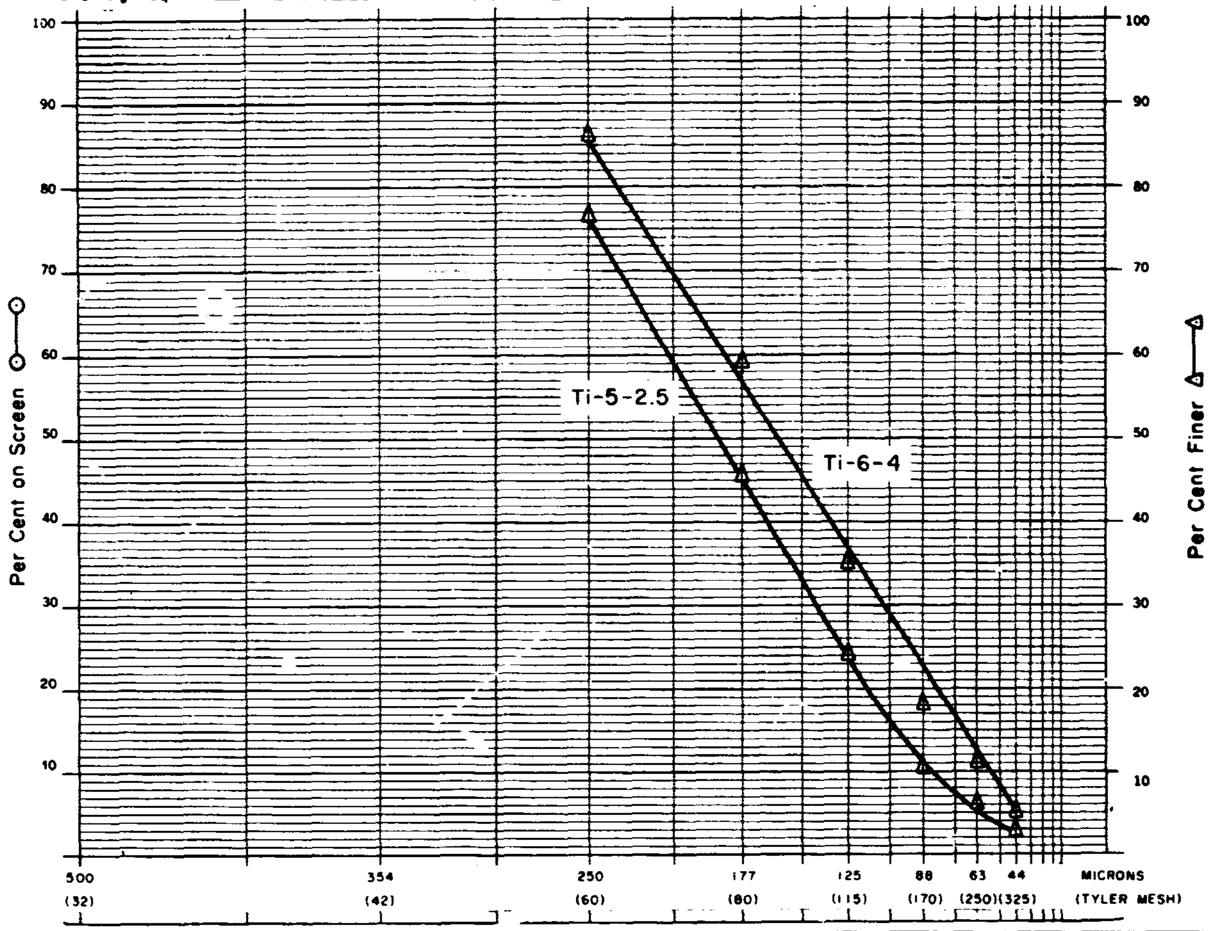
Standard Screen Scale

Job No 321-0002 Electrode Dia. (in.) —

Date 11-14-67 Speed (rpm) —

Name NUMEC MECHANICAL ATTRITED POWDER

Shotting Req. No ______ Material Ti-6Al-4V & Ti-5Al-2.5Sn



	Printed Screen Scale Ratio 1.414				Ti-6Al-4V			Ti-5Al-2.5Sn		
	Openings		Mesh							
	Microns	Milli-meters	Tyler	U.S.	Sample Weights	Per Cent	Per Cent Finer	Sample Weights	Per Cent	Per Cent Finer
	500		32	35						
					INITIAL WT. = 81.50g			INITIAL WT. = 77.15g		
	354		42	45						
ON	250		60	60	11.28	13.84	86.16	18.31	23.61	76.38
ON	177		80	80	22.14	27.17	58.99	23.88	30.79	45.59
ON	125		115	120	3.73	24.20	34.79	16.94	21.85	23.74
ON	88		170	170	14.02	17.20	17.59	10.22	13.18	10.56
ON	63		250	230	5.67	6.95	10.64	3.37	4.34	6.22
ON	44		325	325	4.54	5.57	5.07	2.48	3.19	3.03
ON	PAN				4.13 (1)	5.07	—	2.35 (1)	3.03	—
Pass										
				Totals,	81.50	100			99.99	

(1) INCLUDES 0.14g LOST (Ti-6Al-4V)

(1) INCLUDES 0.05g LOST (Ti-5Al-2.5Sn)

DATA

Total Shot Weight ______

Split Sample Yes ______ No ______

Sample Weight ______ gms.

Remarks

Figure 2. Sieve analysis for Numec MA Ti-6Al-4V and Ti-5Al-2.5Sn.

Standard Screen Scale

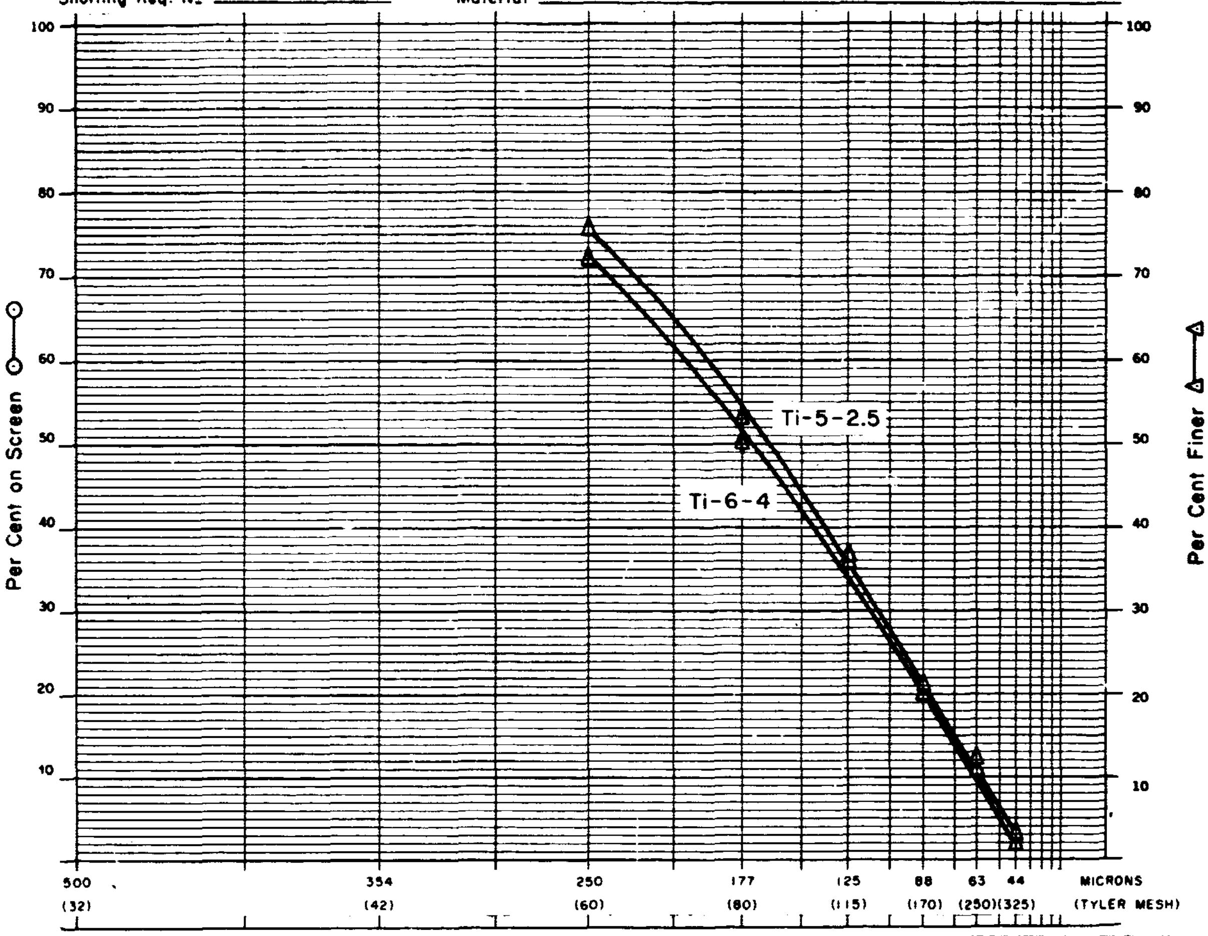
Job Nº 321-0002 Electrode Dia. (in.) —

Date 12-5-67 Speed (rpm) —

Name NUMEC HYDRIDE-DEHYDRIDE POWDER

Shotting Req. Nº

Material Ti-6Al-4V & Ti-5Al-2.5Sn



	Printed Screen Scale Ratio 1.414				Ti-6Al-4V			Ti-5Al-2.5 Sn		
	Openings		Mesh							
	Microns	Millimeters	Tyler	U.S.	Sample Weights	Per Cent	Per Cent Finer	Sample Weights	Per Cent	Per Cent Finer
	500		32	35						
					INITIAL WT. = 134.157g			INITIAL WT. = 59.055g		
	354		42	45						
ON	250		60	60	37.60	28.02	71.98	14.50	24.56	75.44
ON	177		80	80	28.98	21.60	50.38	13.02	22.05	53.39
ON	125		115	120	19.26	14.36	36.02	10.40	17.61	35.78
ON	88		170	170	20.97	15.63	20.39	9.00	15.24	20.54
ON	63		250	230	11.32	8.44	11.95	4.84	8.20	12.34
ON	44		325	325	11.92	8.89	3.06	5.26	8.91	3.43
ON	PAN				4.11(+)	3.06	—	2.03	3.43	—
				Totals,	134.15		99.99	59.05		99.98

DATA

Total Shot Weight ______

Split Sample Yes ______ No ______

Sample Weight ______ gms.

Remarks

Figure 3. Sieve analysis for Numec Hyd Ti-6Al-4V and Ti-5Al-2.5Sn.

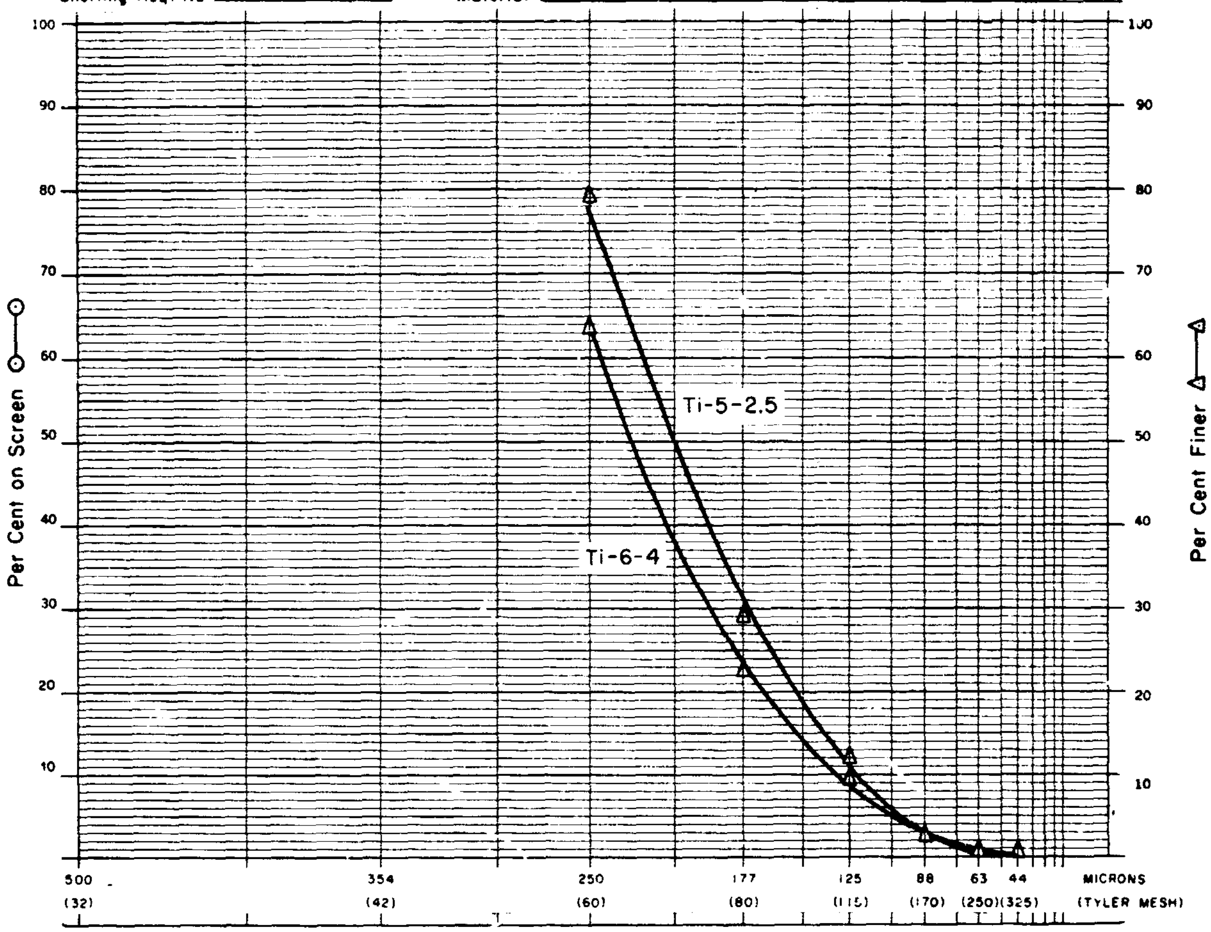
Standard Screen Scale

Job No 321-0002 Electrode Dia. (in.) 1 & 1 1/8" DIA.

Date 11-14-67 Speed (rpm) 15-20,000

Name NUCLEAR METALS ROTATING ELECTRODE POWDER

Shotting Req. No ____ Material Ti-6Al-4V & Ti-5Al-2.5Sn



	Printed Screen Scale Ratio 1.414				Ti-6Al-4V			Ti-5Al-2.5Sn		
	Openings		Mesh		Sample Weights	Per Cent	Per Cent Finer	Sample Weights	Per Cent	Per Cent Finer
	Microns	Millimeters	Tyler	U.S.						
	500		32	35						
					INITIAL WT. = 125.89g			INITIAL WT. = 160.79g		
	354		42	45						
ON	250		60	60	45.65	36.26	63.74	33.77	21.01	78.99
ON	177		80	80	51.52	40.93	22.81	79.70	49.50	29.42
ON	125		115	120	16.56	13.15	9.66	28.09	17.47	11.95
ON	88		170	170	9.27	7.36	2.30	15.01	9.33	2.62
ON	63		250	230	1.91	1.52	0.78	2.80	1.74	0.88
ON	44		325	325	.54 (1)	0.43	0.35	0.79	0.49 (1)	0.39
ON	PAN				.45	0.35	—	0.63	0.39	—
Pass										
				Totals,	125.89	100.00		160..9	100	

(1) INCLUDES LOSS OF 0.36g

(1) INCLUDES LOSS OF 0.42g

DATA

Total Shot Weight ____

Split Sample Yes ____ No ____

Sample Weight ____ gms.

Remarks

Figure 4. Sieve analysis for Nuclear Metals REP Ti-6Al-4V and Ti-5Al-2.5Sn.

Standard Screen Scale

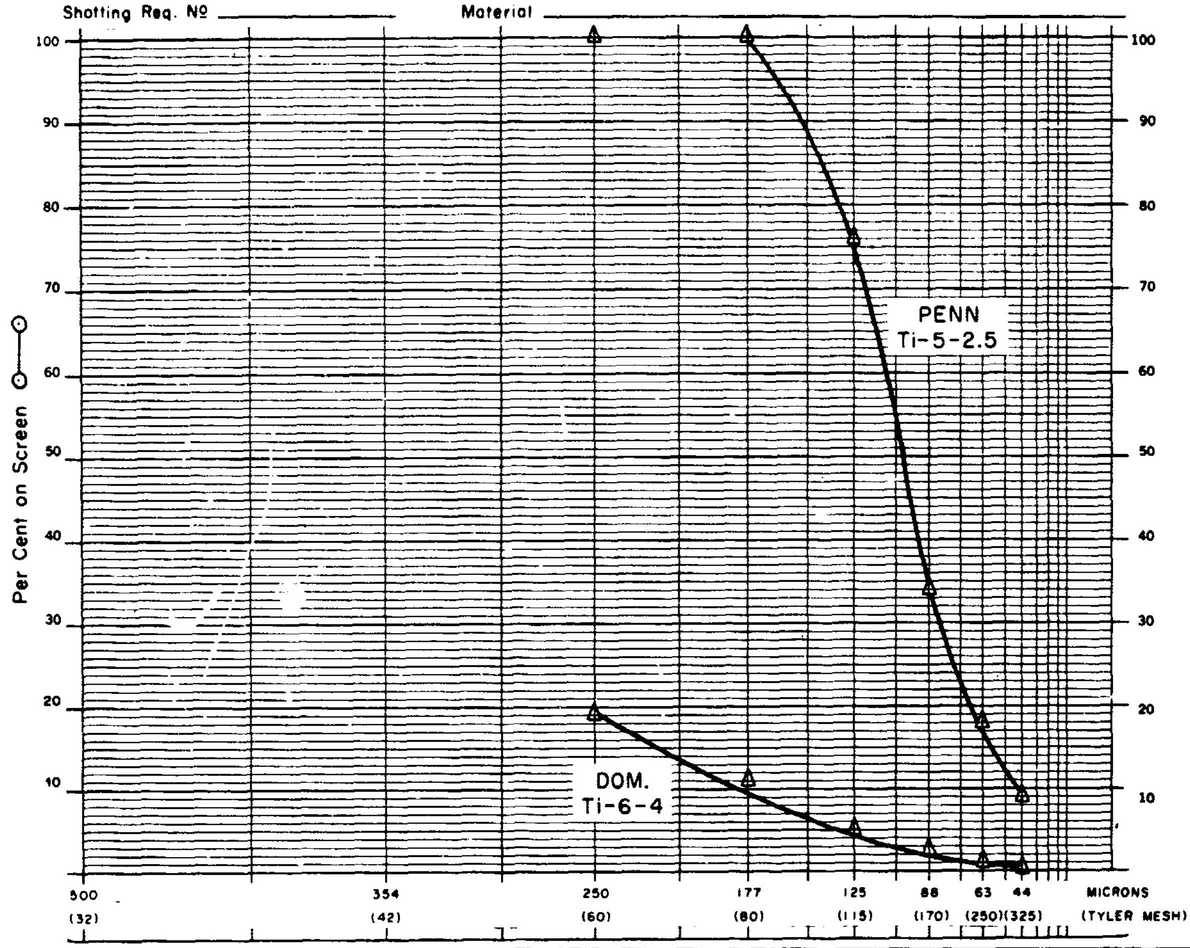
Job Nº 321-0002 Electrode Dia. (in.) —

Date 12-12-67 Speed (rpm) —

Name DOMINION Ti-6Al-4V & PENN NUCLEAR Ti-5Al-2.5Sn

Shotting Req. Nº

Material



	Printed Screen Scale Ratio 1.414				DOM. Ti 6Al-4V			PENN Ti-5Al-2.5Sn		
	Openings		Mesh							
	Microns	Millimeters	Tyler	U.S.	Sample Weights	Per Cent	Per Cent Finer	Sample Weights	Per Cent	Per Cent Finer
	500		32	35						
					INITIAL WT. = 70.50g			INITIAL WT. = 125.644g		
	354		42	45						
ON	250		60	60	59.08	83.81	19.37	.001	0	100
ON	177		80	80	4.98	7.06	12.31	.006	.01	99.99
ON	125		115	120	2.73	3.88	5.25	30.41	24.20	75.79
ON	88		170	170	1.86	2.64	2.61	52.50	41.79	34.00
ON	63		250	230	1.28	1.82	0.79	19.77	15.73	18.27
ON	44		325	325	0.10	0.13	0.66	11.19	8.91	9.36
ON	PAN				0.46 (1)	0.66		11.76 (1)	9.36	
				Totals,	70.50	100		125.64		

(1) INCLUDES 0.07g LOST

(1) INCLUDES LOSS OF 0.13g

DATA

Total Shot Weight

Split Sample Yes

No

Sample Weight gms.

Remarks

Figure 5. Sieve analysis for Dominion Ti-6Al-4V and Penn Ti-5Al-2.5Sn.

Each powder was then cold compacted in an arrangement comprising an inert gas glove box containing a 2-inch ID steel packing die, a 1-1/4-inch diameter punch, and an O-ring-sealed piston driven by an electro-hydraulic unit located beneath the box (Figure 6). Ten carbon steel compaction cans were loaded into the box with each lot of powder. The cans were 2-inch OD x 1-3/4-inch ID x 5 inches long, with an end plate welded across one end. The end plate welds were all leak checked with a helium mass spectrometer before being transferred into the glove box.

The compacts were prepared by filling each can with powder and transferring it to the packing die, where it was pressed for 10 seconds at 1000-1400 psi. More powder was added to the can and pressure was again applied. These steps were repeated until each can contained approximately one pound of powder.

The compacts were then encased in individual rubber bags and were transferred to a welding box where the top lid, with steel evacuation tube attached, was joined to the can body. This weld was also checked for leaks with the helium mass spectrometer. The sound billets were then connected to a vacuum pump and were heated to 800°F to drive off adsorbed gasses. The billets were evacuated overnight and were then sealed off by forge welds on the steel evacuation tubes.

The compacts from each powder vendor were then hot pressed at 1450, 1650, and 1850°F under pressures of 50, 75, and 100 tons per square inch. Each compact was pressed individually, using the arrangement illustrated in Figure 7. The extrusion press container, ram, and the hardened punches were maintained at 900°F, a temperature which minimizes thermal shock to the tooling while maintaining full tool hardness. Transfer time from the billet-heating furnace to the container of the 300 ton press averaged 10-15 seconds; placement of the backer plate and application of ram pressure required approximately 5 additional seconds. Pressure was maintained on the billet for approximately 10 seconds.

An additional set of billets (series 100, 200,* etc.) was prepared from powder which was cold pressed in air, rather than under an inert gas cover. These billets were then evacuated, sealed-off, and hot pressed at 1650°F, 75 tsi, except for No. 200, which was pressed at 100 tsi. The purpose of preparing these compacts was to determine the degree of interstitial contamination that would occur as a result of performing the initial filling and pressing operations in air, rather than in an inert atmosphere.

---

*The first digit in the sample identification scheme refers to the alloy and powder manufacturer, as listed in Tables II and III. For the samples prepared under inert atmosphere, the second digit refers to the temperatures and pressures indicated in the tables. The samples prepared in air were all compacted under the same conditions (except as noted below) and therefore are undifferentiated with respect to the final digit.

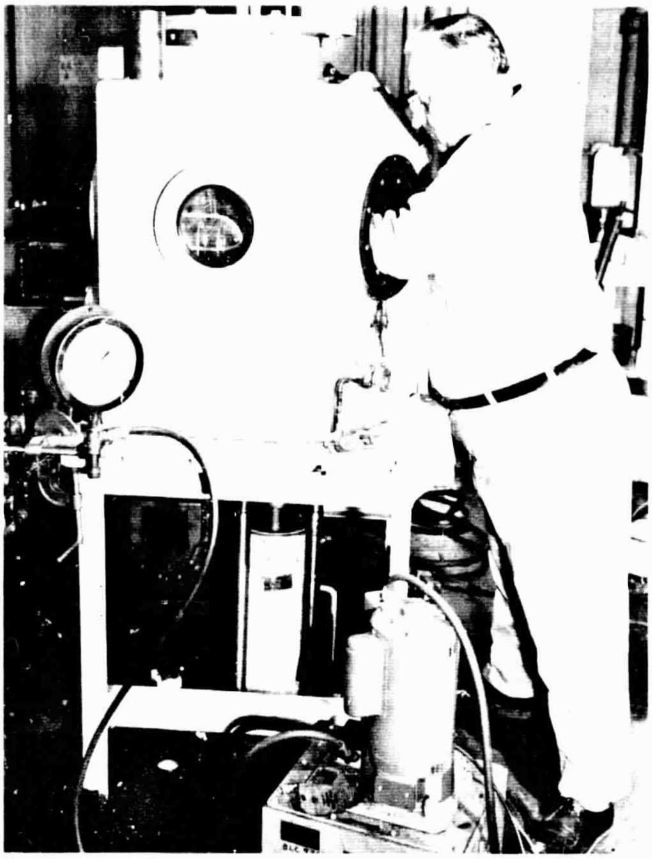


Figure 6. Inert-gas glove box, showing electro-hydraulic oil pump in foreground and compacting piston under box.

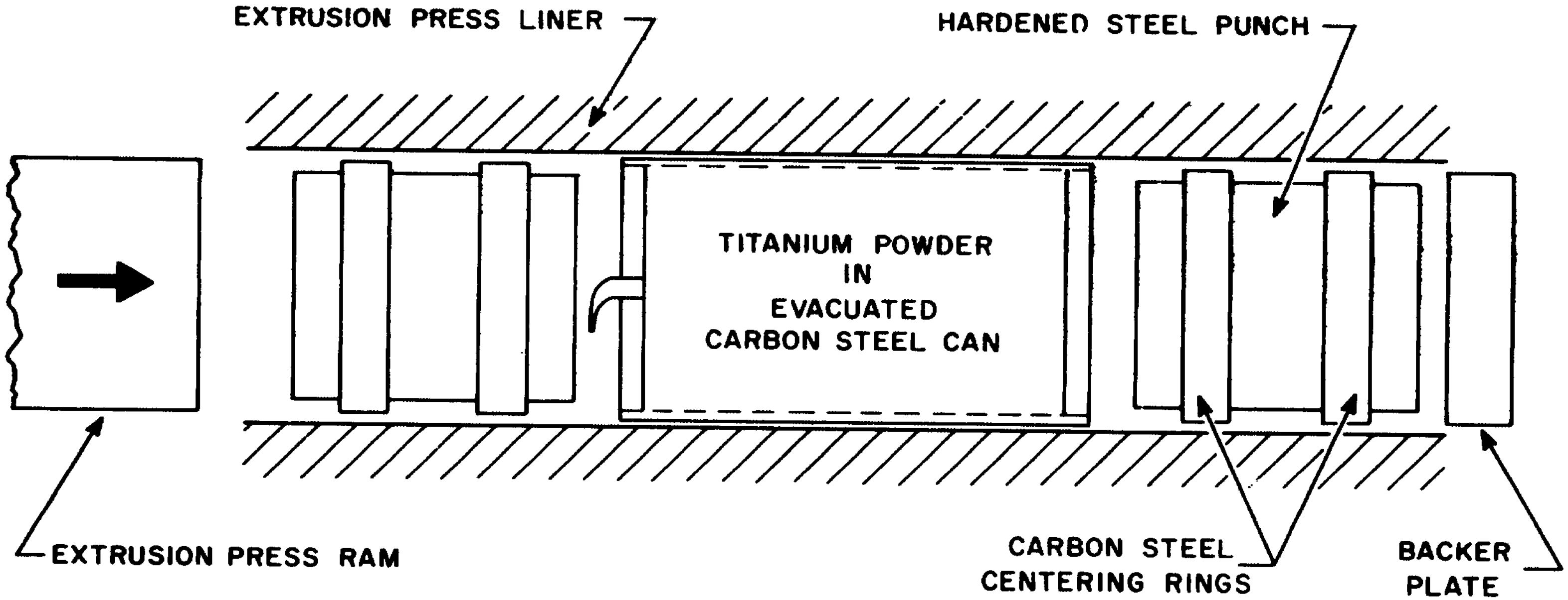


Figure 7. Press setup for hot pressing titanium alloy powder.

TABLE II.— DENSITY OF HOT COMPACTED TITANIUM ALLOY POWDER BILLETS, g/cc

Temperature	1450°F			1650°F			1850°F			1650°F
Pressure (tsi)	50	75	100	50	75	100	50	75	100	75
Reference No.	1	2	3	4	5	6	7	8	9	100[a]
1. NUMEC MA 6-4. .	4.256	4.429	4.429	4.435	4.449	4.444	4.448	4.460	4.443	4.451
2. NM 5-2.5. . . .	--	4.405	4.445	4.299	4.438	4.444	4.446	4.447	4.450	4.441[b]
3. NM 6-4	4.286	4.393	4.397	4.397	4.419	4.418	4.411	4.418	4.417	4.411
4. DOM 6-4	4.038	4.211	4.238	4.180	4.244	4.259	4.256	4.275	4.272	4.237
5. PENN 5-2.5. . .	4.276	4.442	4.442	4.386	4.438	4.463	4.453	4.452	4.452	4.430
6. NUMEC HYD 6-4 .	4.265	4.392	4.409[c]	4.393	4.411	4.415	4.416	4.456	4.416	4.425
7. NUMEC HYD 5-2.5	4.236	4.422	4.440	4.306	4.433	4.443	4.459	4.440	4.444	4.414
8. NUMEC MA 5-2.5	4.261	4.434	4.450	4.340	4.418	4.432	4.452	4.448	4.413	4.448

[a]The samples were handled in air, rather than inert gas, before evacuation and sealing.
[b]Compact 200 - pressed at 100 tsi instead of 75 tsi.
[c]Compact 63 pressed at 1850°F, 100 tsi (duplicating 69).

Legend:

NUMEC MA - NUMEC Mechanically Attrited Powder
NM - Nuclear Metals Rotating Electrode Powder
DOM - Dominion Magnesium Co-reduced Powder
PENN - Penn Nuclear Gas Attrited Powder
NUMEC HYD - NUMEC Hydride-Dehydride Powder

TABLE III.– PERCENT THEORETICAL DENSITY OF HOT COMPACTED TITANIUM ALLOY POWDER BILLETS[a]

Temperature	1450°F			1650°F			1850°F			1650°F
Pressure (tsi)	50	75	100	50	75	100	50	75	100	75
Reference No.	1	2	3	4	5	6	7	8	9	100[b]
1. NUMEC MA 6-4 . . .	96.2	100.1	100.1	100.2	100.6	100.4	100.5	100.8	100.4	100.6
2. NM 5-2.5	--	99.0	99.9	96.6	99.7	99.9	99.9	99.9	100.0	99.8[c]
3. NM 6-4	96.9	99.3	99.4	99.4	99.9	99.9	99.7	99.9	99.8	99.7
4. DOM 6-4.	91.3	95.2	95.8	94.5	95.9	96.3	96.2	96.6	96.6	95.8
5. PENN 5-2.5	96.1	99.8	99.8	98.6	99.7	100.3	100.1	100.0	100.0	99.6
6. NUMEC HYD 6-4. . .	96.4	99.3	99.7[d]	99.3	99.7	99.8	99.8	100.7	99.8	100.0
7. NUMEC HYD 5-2.5. .	95.2	99.4	99.8	96.3	99.6	99.8	100.2	99.8	99.9	99.2
8. NUMEC MA 5-2.5 . .	95.8	99.6	100.0	97.5	99.3	99.6	100.0	100.0	99.2	100.0
Compacts over 98% T.D.:	0	6	6	4	6	6	6	6	6	6

[a]Based on a density of 4.424 g/cc for Ti-6Al-4V and 4.450 g/cc for Ti-5Al-2.5Sn.
[b]These samples were handled in air, rather than inert gas, before evacuation and sealing.
[c]Compact 200 pressed at 100 tsi instead of 75 tsi.
[d]Compact 63 pressed at 1850°F, 100 tsi (duplicating 69).

<u>Legend:</u>

NUMEC MA - NUMEC Mechanically Attrited Powder
NM - Nuclear Metals Rotating Electrode Powder
DOM - Dominion Magnesium Co-reduced Powder
PENN - Penn Nuclear Gas Attrited Powder
NUMEC HYD - NUMEC Hydride-Dehydride Powder

When the billets had cooled to room temperature, they were machined on all surfaces to a depth of 0.025 inch below the titanium-steel interface. The density of each compact was then determined from measurements of weight and physical dimensions (Tables II, III). These data show:

1. None of the Dominion Ti-6Al-4V powder compacts achieved the 98% cutoff density established before the start of the program. This situation is explainable in view of this powder's very high interstitial content, which would increase the difficulty of plastically deforming the powder particles.

2. No powder could be compacted to 98% density at 50 tsi, 1450°F. Pressures of 75 and 100 tsi were sufficient to produce compacts of over 99% density at 1450, 1650, and 1850°F.

It is assumed that the density fractions greater than unity, e.g., 100.5% for sample 17 (Numec MA, 1850°F, 50 tsi) and 100.3% for sample 56 are due merely to differences in specific chemistry for the powders from which these compacts were made.

A series of hardness determinations were then made along the side, i.e., parallel to the pressing direction, and across the face of each cylinder. The average of these values for each compact over 98% dense is listed in Table IV. It can be seen that the softest Ti-5Al-2.5Sn compacts are those made by Nuclear Metals (NM). The Numec hydride-dehydride process (Numec Hyd) and the Nuclear Metals Rotating Electrode process produced Ti-6Al-4V powder compacts of approximately equal softness. The hardest compacts are those made from the Penn Nuclear Ti-5Al-2.5Sn powder.

All of the 1850°F/100 tsi compacts were then sectioned as indicated in Figure 8, providing samples for chemistry, heat treating, metallography, and tensile testing. The choice of this set of samples, rather than a set compacted at a lower temperature or pressure, was made on the theory that properties obtained from these samples would be equal to or better than those in any other set. Samples from other sets were subsequently used in the heat treatment study described below.

Tables V and VI contain the results of chemical analyses performed on the 1850°F/100 tsi compacts. Only the Nuclear Metals samples have low enough oxygen contents to be considered in the ELI category. In all other cases, the oxygen contents are higher than the approximately 1200 ppm established as the ELI limit. For all powders, oxygen content increased during further processing. Table VI charts the progression of oxygen contamination during the conversion of these samples from bar to powder to pressed compact. The oxygen content for the NM powders and most of the Numec powders is low enough to be included in the commercial range for ELI (NM) or standard grade (Numec) titanium. Therefore, the lower-than-standard values for tensile

TABLE IV.— HARDNESS OF HOT PRESSED TITANIUM ALLOY COMPACTS (R_C)[a]

Pressing Temperature (°F):	1450		1650			1850			1650
Pressure (tsi):	75	100	50	75	100	50	75	100	75
Sample No.:	2	3	4	5	6	7	8	9	100, 200, 300, etc.[b]
Longitudinal									
Ti-6Al-4V									
Numec MA	31.5	31.5	32	36	38	38	40.5	37	38
NM	29.5	29.5	27	34.5	33	35.5	36	36	33.5[c]
Numec Hyd	28	36[d]	24	36.5	37	35.5	34	37.5	29.5
Ti-5Al-2.5Sn									
NM	19.5	30.5	--[e]	23.5	28.5	21	32	32.5	21.5
Penn	31.5	34.5	24.5	32	39.5	36	40.5	44	37.5
Numec Hyd	25	27.5	--	27	29.5	31.5	30	39	24
Numec MA	23	30	--	27	28	31.5	38	39	28.5
Transverse									
Ti-6Al-4V									
Numec MA	36.5	38	36.5	36	35.5	37	36.5	37	37
NM	36	35	33	34	34.5	39	35	35.5	34.5
Numec Hyd	35.5	3	38.5	37	37.5	42	35	36.5	38.5
Ti-5Al-2.5Sn									
NM	33	33	--	32	32.5	32.5	33	33	32.5
Penn	43.5	42	33.5	41	42	42.5	47	41	42
Numec Hyd	32.5	32.5	--	33	31.5	31.5	32.5	31.5	33.5
Numec MA	35.5	34	--	34	36	34	37	38.5	34

[a]Converted from R_A.
[b]Powder handled in air prior to evacuation and sealing.
[c]Pressed at 100 tsi.
[d]Pressed at 1850°F, 100 tsi.
[e]Hardness readings omitted for samples less than 98% dense.

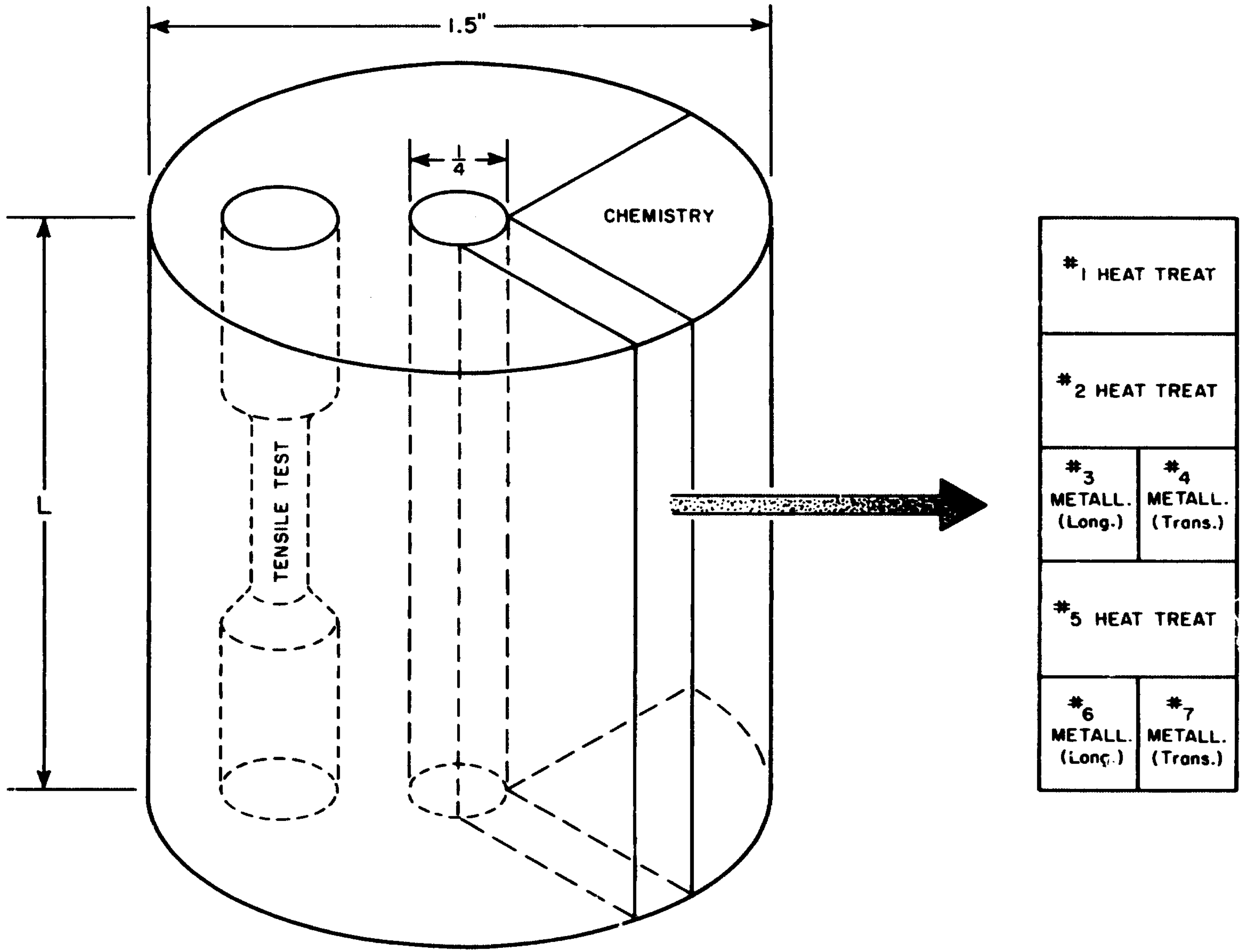


Figure 8. Sectioning of hot pressed billet for test samples.

TABLE V.– CHEMICAL ANALYSIS OF HOT PRESSED TITANIUM ALLOY COMPACTS

Element: Sample	C (ppm)	O (ppm)	H (ppm)	N (ppm)	Al (%)	V (%)	Sn (%)	Fe (%)
Ti-6Al-4V								
Numec MA	650	1710	144	239	5.99	4.17	--	0.19
NM	200	900	90	234	6.04	4.30	--	0.13
Numec Hyd	210	1570	210	148	5.75	4.31	--	0.14
Numec MA, loaded in air	--	1890	--	158	--	--	--	--
NM, " " "	--	750	--	110	--	--	--	--
Numec Hyd, " " "	--	3430	--	89	--	--	--	--
Ti-5Al-2.5Sn								
NM	255	980	78	189	5.06	--	2.38	0.21
Penn	640	3530	183	591	5.44	--	2.31	0.31
Numec Hyd	150	3620	52	208	5.03	--	2.38	0.06
Numec MA	500	1640	258	155	6.33	--	a	0.14
NM, loaded in air . . .	--	845	--	67	--	--	--	--
Penn, " " " . . .	--	b	--	520	--	--	--	--
Numec Hyd, loaded in air	--	2090	--	340	--	--	--	--
Numec MA, " " "	--	1755	--	380	--	--	--	--

[a] Incorrect analysis.
[b] 1400-4700.

TABLE VI.– OXYGEN PICKUP DURING FABRICATION OF HOT PRESSED TITANIUM COMPACTS

Sample	Oxygen Content (ppm)		
	Starting Bar	Powder	Hot Pressed Compact
Ti-6Al-4V			
Numec MA	700	1560	1710
NM	700	750	900
Dominion	---	8100	---
Numec Hyd.	700	1300	1570
Ti-5Al-2.5Sn			
NM	950	750	980
Penn	---	2800	3530
Numec Hyd.	740	1380	3620
Numec MA	740	1220	1640

ductility cannot be entirely attributable to oxygen content, but are also related to the poorer forming characteristics of the higher interstitial content, hence harder, powders.

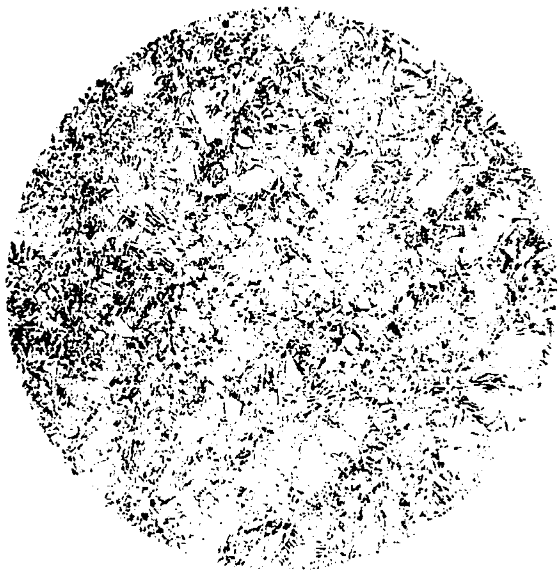
High oxygen powders are difficult to form because the oxygen in them is not uniformly distributed but is instead present in a heavy concentration on the surface of the powder particles. Surface oxygen levels can therefore be considerably higher than the nominal oxygen content for the powder. The high-oxygen surface layer is quite hard and impedes plastic deformation and surface welding of the powder particles. The direct consequence of the hardened particle surface layer is a lowering of bond strength between powder particles, and hence lower mechanical properties for compacts in the as-pressed condition. High temperature annealing treatments can offset this condition somewhat by diffusing oxygen away from the particle surface.

Photomicrographs of the as-pressed compacts are presented in Figure 9. Prior particle boundaries are visible in some of the samples. The structure of the Ti-6Al-4V samples is predominently that of the acicular alpha phase in a framework of prior beta grain boundaries. The Ti-5Al-2.5Sn alloy is single-phase, and the structures observed are those of the alpha phase.

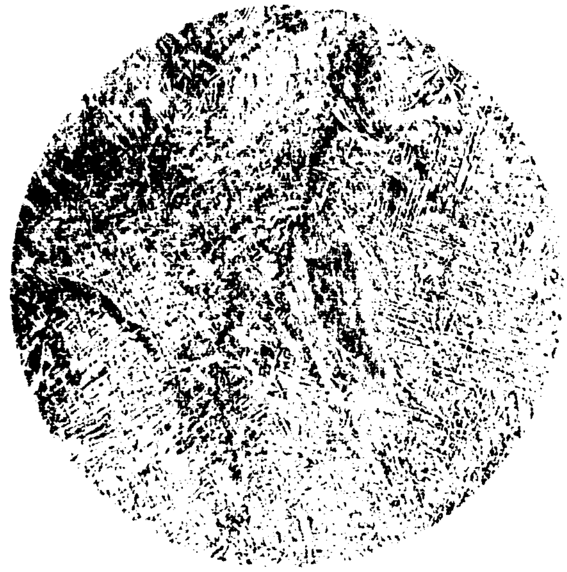
In Table VII are presented the results of a series of annealing treatments performed on 1850°F/100 tsi samples to separate the contributions of sample chemistry and pressing stresses on sample hardness. The heat treatments seem not to have affected the hardness of any sample to a noticeable degree. This may be due to the fact that all heat treatments (which were based on published recommended annealing cycles) took place below the temperature at which the samples were pressed. A more marked response, with a greater difference in hardness between samples, might have been obtained if we had tested samples which had been pressed at 1450°F or 1650°F.

Tensile properties for the as-pressed compacts are listed in Table VIII. It should be noted that in both groups of compacts - those in which powder-filling was performed in helium and those filled in air - the billets were evacuated before being sealed and heated for pressing. From these data we can note a rough correlation between the hardness data in Table VII and the elongation values presented here. It can be seen that the compacts made from the Penn powder have high hardness and a correspondingly low level of tensile ductility. The Nuclear Metals and the Numec hydride powders produced softer compacts which yielded higher values of tensile elongation.

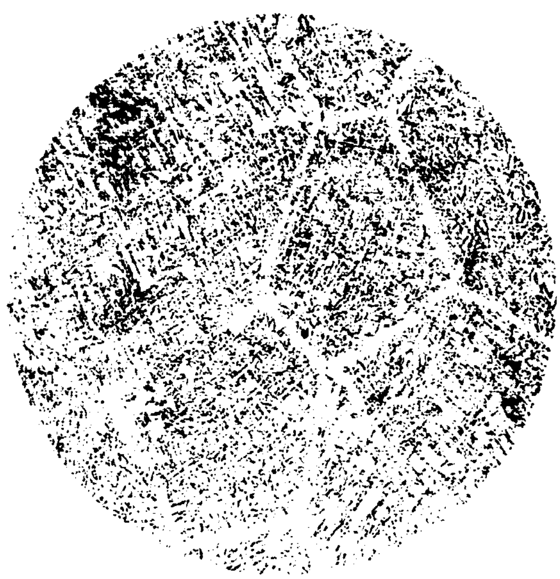
The compacts originally filled in air rather than in helium seem in general to reflect a decrease in ductility as a result of this procedure. In the Ti-5Al-2.5Sn alloy, both the NM and the Numec Hyd specimens show this effect, as does the NM Ti-6Al-4V specimen. It appears that the higher purity powders are the most vulnerable to oxygen contamination when processed in air.



Ti-6Al-4V Numec MA

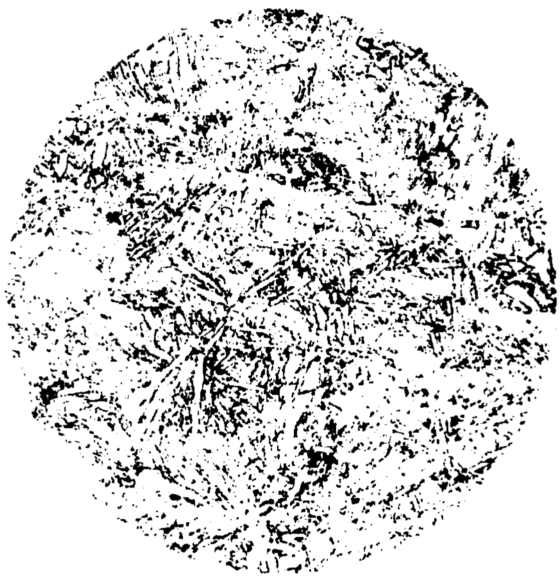


Ti-6Al-4V Numec Hyd

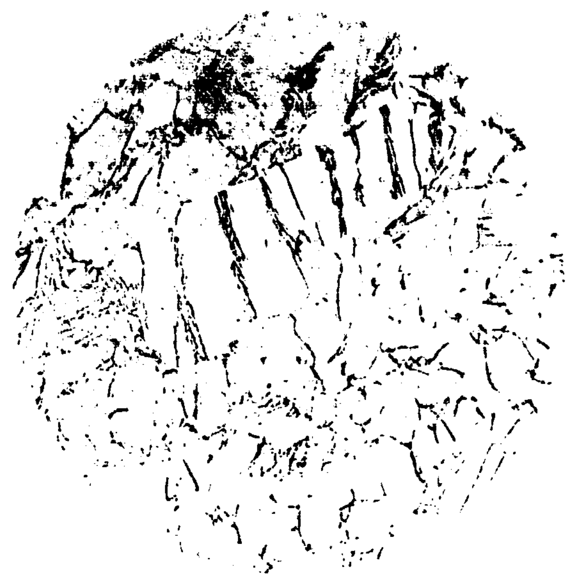


Ti-6Al-4V NM

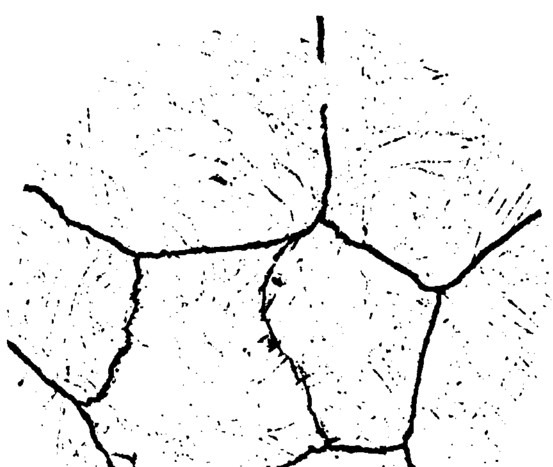
Figure 9a. As-pressed compacts, 250X, etched.



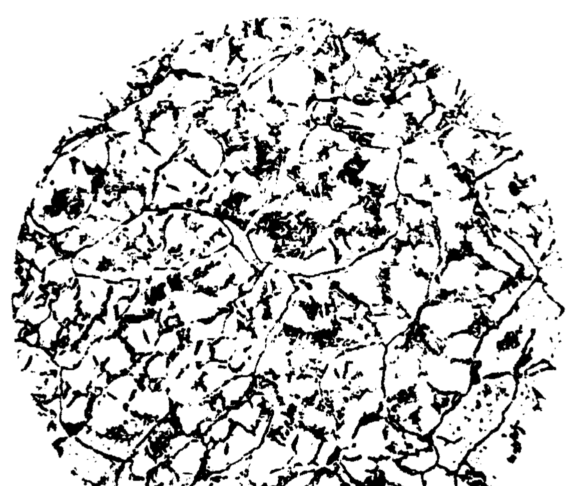
Ti-5Al-2.5Sn Numec MA



Ti-5Al-2.5Sn Numec Hyd



Ti-5Al-2.5Sn NM



Ti-5Al-2.5Sn Penn

Figure 9b. As-pressed compacts, 250X, etched.

TABLE VII.— SAMPLE HARDNESS (R_C) AS A FUNCTION OF HEAT TREATMENT

Ti-6Al-4V Sample:	Numec MA			NM			Numec Hyd					
	1	2	5	1	2	5	1	2	5			
As hot pressed[a]	33.6	35.3	35.1	30.1	29.6	31.1	33.3	34.0	34.5			
1300°F/4 hr/WQ[b]	34.3	--	--	30.4	--	--	34.5	--	--			
1425°F/3 hr/WQ	--	35.6	--	--	32.0	--	--	33.2	--			
1550°F/2 hr/WQ	--	--	36.3	--	--	31.3	--	--	34.3			
Ti-5Al-2.5Sn Sample:	NM			Penn			Numec Hyd			Numec MA		
	1	2	5	1	2	5	1	2	5	1	2	5
As hot pressed	29.2	29.5	29.0	38.7	40.8	41.8	28.7	28.8	29.3	34.0	34.6	34.1
1325°F/2 hr/AC[c]	31.5	--	--	39.0	--	--	29.6	--	--	36.3	--	--
1440°F/1 hr/AC	--	29.2	--	--	41.2	--	--	27.8	--	--	34.0	--
1550°F/1/2 hr/AC	--	--	29.3	--	--	46.3	--	--	28.2	--	--	33.5

[a]All samples pressed at 1850°F/100 tsi.
[b]Water quenched.
[c]Air cooled.

TABLE VIII.— TENSILE PROPERTIES OF TITANIUM ALLOY POWDER COMPACTS

Sample	Billet Cans Filled in Helium Hot Pressed at 1850°F/100 tsi			Billet Cans Filled in Air Hot Pressed at 1650°F/75 tsi		
	UTS (ksi)	0.2% YS (ksi)	Elong. (%)	UTS (ksi)	0.2% YS (ksi)	Elong. (%)
<u>Ti-6Al-4V</u>						
Numec MA	144.8	135.9	1.5	147.5	142.4	1.25
NM	145.1	134.1	7.5	145.6	136.4	2.0
Numec Hyd	149.3	141.3	2.0	153.8	145.8	2.5
MIL Spec.[a], Ti-6Al-4V, Ann.	130	120	10	---	---	---
" " Ti-6Al-4V, ELI, Ann. . .	120	110	10	---	---	---
<u>Ti-5Al-2.5Sn</u>						
NM	130.7	130.7	4.0	44.5	---	0
Penn	129.9	---	0	85.4	---	0
Numec Hyd	126.3	120.3	5.5	21.2	---	0
Numec MA	92.8	---	0	131.7	129.7	2
MIL Spec., Ti-5Al-2.5Sn, Ann. . . .	115	110	10	---	---	---
" " Ti-5Al-2.5Sn, ELI, Ann. .	105	100	10	---	---	---

[a]MIL-T-9047D.

A comparison between the mechanical property data presented in Table VIII and MIL-T-9047 shows that none of the samples exhibited the minimum tensile elongation required by the specification. A series of heat treatments was therefore performed on the compacts made from the Nuclear Metals REP powder. This family of compacts was chosen because it possessed the highest purity and therefore offered the greatest potential in achieving high values of ductility and fracture toughness.

The results of these heat treatments are tabulated in Tables IX and X. Figures 11 and 12 illustrate microstructures associated with the heat treated samples.

Examination of the tensile results for the Ti-6Al-4V alloy shows that tensile ductility, as measured by elongation and reduction in area, increases with an increase in compacting and heat treatment temperature and time. The effect of the heat treatments would therefore appear to be that of enhancing the disappearance, by diffusion, of the prior particle boundaries. This effect is shown very clearly in Figures 11a and 11b, in which samples compacted at 1650°F/75 tsi were annealed for two and four hours at 1650°F.

As the annealing temperatures are increased, further evidence of particle-boundary elimination is offered by the increase in grain size which occurs. While the samples annealed at 2200°F possess adequate ductility, we see that tensile strength has begun to fall off (as a result of greatly increased grain size) as compared to the as-pressed condition. There is a large difference in grain size between sample 11 (Figure 11c), annealed at 2200°F, and sample 25 (Figure 11d), which was treated at 1775°F for a similar period of time. The microstructure shown in Figure 11c is one of acicular alpha and prior beta grain boundaries, obtained by heating entirely in the beta phase field (the bounary between the alpha-plus-beta and the beta phase fields lies at 1820°F for Ti-6Al-4V). Sample 25, annealed at 1775°F, was therefore heated at the high end of the alpha-plus-beta field. The structure illustrated is that of primary alpha (white regions) and transformed beta (acicular alpha). The properties obtained by heating at this temperature offer a good balance between tensile strength and ductility.

Higher strenths can be realized by subjecting the sample to a solution treatment, as was done for samples 6-10. The aging treatment, which was omitted for these samples, would have raised strengths even further, with a probable further loss in ductility. However, this procedure would be undesirable for several reasons:

1. Mechanical properties based upon water quenching small samples would be difficult to reproduce in objects of large size, such as large forged valve and pump bodies, which are of ultimate interest here.

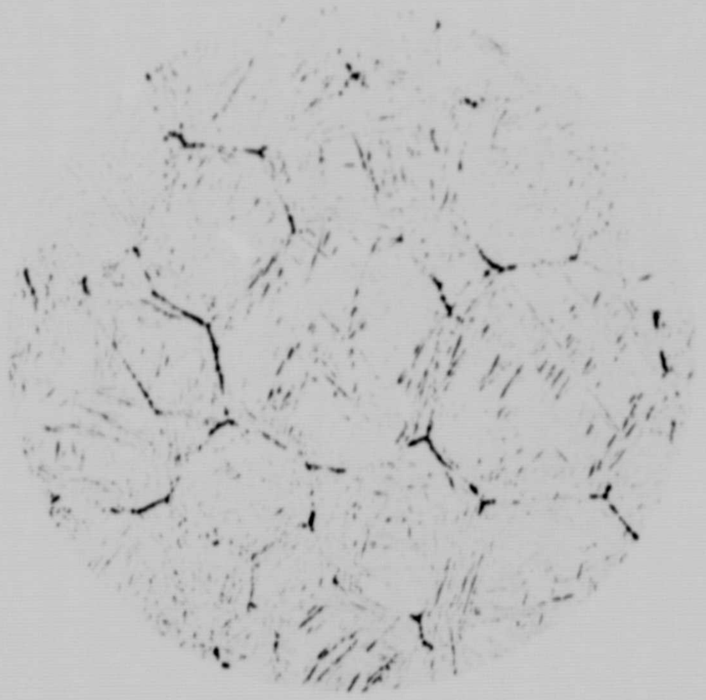
2. The solution treatment sacrifices ductility for strength, whereas it would be most desirable to maximize ductility.

TABLE IX.– MECHANICAL PROPERTIES OF REP TI-6Al-4V, ELI TITANIUM ALLOY COMPACTS AFTER VARIOUS HEAT TREATMENTS

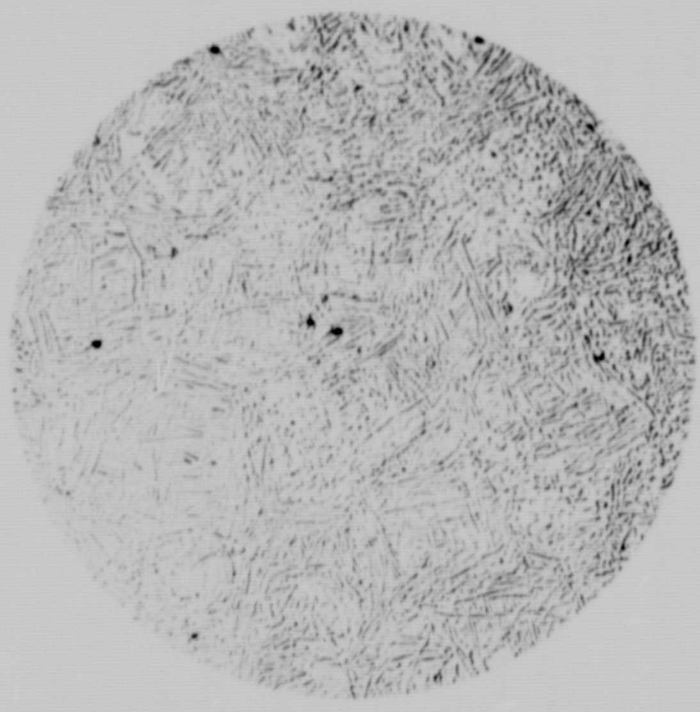
No.	Compacting Conditions Pressure (tsi)	Compacting Conditions Temp. (°F)	Heat Treatment	U.T.S. (ksi)	0.2 Y.S. (ksi)	% Elong. 1" G.L.	% R.A.	True Fracture Stress (ksi)
1	100	1650	1500/2/WQ	131.5	112.3	1.7	8.1	143.0
2	100	1650	1500/2/WQ	144.0	112.8	6.8	12.2	164.0
3	100	1650	1500/2/WQ	143.1	110.9	6.1	14.4	167.2
4	100	1650	1500/4/WQ	149.1	112.3	7.2	12.9	171.3
5	100	1650	1550/2/AC	143.2	130.7	5.9	12.9	163.8
6	75	1650	1650/2/WQ	157.6	138.7	6.3	18.9	186.9
7	75	1650	1650/2/WQ	157.4	138.4	8.4	33.9	200.6
8	75	1650	1650/4/WQ	158.4	138.7	8.2	37.6	210.1
9	75	1650	1650/4/WQ	160.2	158.8	7.2	20.4	191.5
10	100	1650	1650/4/WQ	158.4	138.5	8.1	37.3	215.2
11	75	1650	2200/4/AC	133.2	121.1	9.3	38.1	175.7
12	75	1650	2200/4/AC	126.4	114.1	10.6	46.2	188.1
13	100	1850	1775/4/SC+ 1300/2/AC	132.4	123.6	10.8	43.8	189.6
14	100	1850	"	132.8	123.6	10.9	43.8	189.6
17	75	1850	1650/4/AC	139.7	124.3	12.0	25.3	203.4
18	75	1850	"	136.9	123.6	10.2	27.5	196.5
19	75	1850	1650/4/AC+ 1300/2/AC	133.5	122.1	13.0	24.2	201.2
20	75	1850	"	132.3	121.2	9.8	27.0	195.9
21	50	1850	2200/4/AC	140.6	131.5	3.6	19.2	188.8
22	50	1850	"	137.9	127.8	4.1	21.7	198.6
23	50	1850	1870/4/FC+ 1300/2/AC	140.1	134.8	5.6	16.2	160.5
24	50	1850	"	140.0	131.9	7.0	15.6	159.5
25	75	1850	1775/4/FC+ 1300/2/AC	140.0	131.5	10.16	32.1	209.5
26	75	1850	"	139.5	131.5	11.8	42.8	191.0

TABLE X.– MECHANICAL PROPERTIES OF REP Ti-5Al-2.5Sn, ELI TITANIUM ALLOY COMPACTS AFTER VARIOUS HEAT TREATMENTS

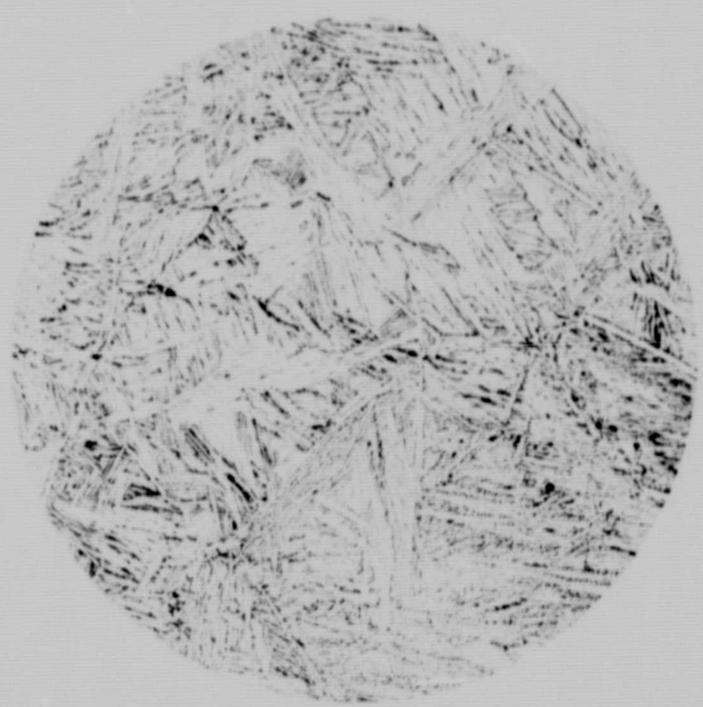
No.	Compacting Conditions Pressure (tsi)	Compacting Conditions Temp. (°F)	Heat Treatment	U.T.S. (ksi)	0.2 Y.S. (ksi)	% Elong. 1" G.L.	% R.A.	True Fracture Stress (ksi)
1	100	1650	1550/4/AC	120.7	114.5	6.5	16.8	135.5
2	100	1650	1550/4/AC	119.2	112.6	9.0	22.0	136.9
3	100	1650	2200/4/AC	100.2	96.5	3.8	29.0	112.3
4	100	1650	2200/4/AC	102.4	96.6	5.1	30.6	107.7
5	100	1850	1850/4/AC	126.6	116.4	13.2	23.4	158.7
6	100	1850	1850/4/AC	127.1	116.4	15.2	42.5	184.3
7	100	1450	1850/4/AC	123.2	113.6	7.7	7.9	139.5
8	100	1450	"	123.8	113.2	10.8	14.2	156.3
9	100	1850	1700/4/AC	122.4	113.0	13.8	26.4	186.1
10	50	1650	1775/4/AC	126.0	113.1	19.1	41.9	180.5
11	75	1650	"	122.5	115.1	11.3	25.8	153.0
12	75	1850	"	128.0	118.5	14.8	44.5	189.5
13	75	1850	"	125.0	116.0	13.7	40.9	177.8
14	75	1850	1850/4/AC	127.0	116.5	13.5	38.8	174.5
15	75	1850	"	127.5	116.9	13.0	36.0	173.4



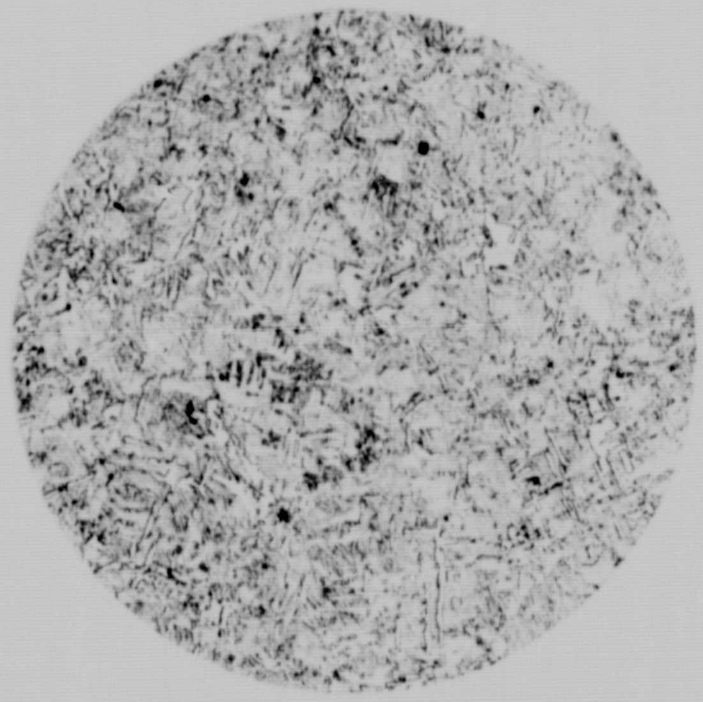
a. Pressed: 1650°F/75 tsi
Heat treated: 1650°F/2/WQ



b. Pressed: 1650°F/75 tsi
Heat treated: 1650°F/4/WQ



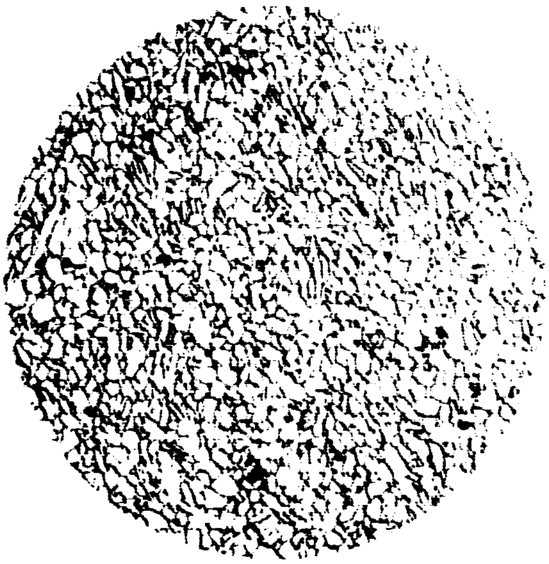
c. Pressed: 1650°F/75/tsi
Heat treated: 2200°F/4/AC



d. Pressed: 1850°F/75 tsi
Heat treated: 1775°F/4/AC +
1300°F/2/AC

Figure 11. REP Ti-6Al-4V, pressed and heat treated as indicated. Transverse sections, 100X, etched.

a. Pressed: 1650°F/100 tsi
Heat treated: 1550°F/4/AC



b. Pressed: 1850°F/100 tsi
Heat treated: 1850°F/4/AC

c. Pressed: 1850°F/75 tsi
Heat treated: 1775°F/4/AC

Figure 12. REP Ti-5Al-2.5Sn, pressed and heat treated as indicated. Transverse sections, 100X, etched.

3. The lower tensile strengths which are produced in samples tested at room temperature by annealing rather than by solution treating will be more than made up in the cryogenic strengthening which will occur naturally when samples are tested at very low temperatures.

Ti-5Al-2.5Sn, being a single-phase alloy, cannot be strengthened by heat treatment. The objective of any heat treatment for this alloy is therefore only to relieve working stresses, or (as in the present case) to diffuse away particle boundaries. Figure 12 illustrates the structure of Ti-5Al-2.5Sn tensile specimens. Sample 16, which was compacted at 1850°F/75 tsi, was subsequently annealed for four hours at 1775°F. The microstructure consists of alpha grains in a plate form.

The result of the heat treatment-tensile test experiments is that a suitable treatment has been developed to produce adequate tensile strength along with good ductility in hot pressed REP titanium alloy powders. Both the hot pressing techniques and the post-pressing heat treatments are applicable to larger bodies with no anticipated loss in properties as section sizes are increased.

CONCLUSIONS

The following conclusions may be drawn from the work performed thus far:

1. Of the five powder-making techniques investigated, only the Rotating Electrode Process (REP) was capable of producing ELI-grade titanium powder. The oxygen content of REP Ti-6Al-4V and Ti-5Al-2.5Sn powder compacts was less than 1000 ppm, whereas compacts made from the hydride-dehydride, mechanical attriting, gas attriting, and chemical reduction processes had oxygen contents ranging from 1600 to over 8000 ppm.

2. All powders, except those made by chemical reduction, could be compacted to full density at a temperature/pressure combinatio of 1650°F/75 tsi. The chemically reduced powder could not be compacted to over 96.6% density at 1850°F/100 tsi, the highest temperature/pressure combination investigated.

3. The as-compacted powder samples possessed only limited ductility, with elongation values ranging from 0 to 7.5%. The REP powder samples developed over 10% elongation and 40% reduction in area after four-hour anneals at 1775°F. The rotating electrode process was chosen for the balance of the program because of its low oxygen content, and therefore greatest potential for high toughness at cryogenic temperatures. This powder is also the simplest to work with, due to its high bulk density and free-flowing characteristics.

As a result of the information gained from this stage of the investigation, it is now possible to prepare samples for cryogenic testing. Rectangular blocks 6 inches x 6 inches x 3 inches will be pressed from REP powder in carbon steel cans. The blocks will then be heat treated and sectioned to provide specimens for testing in tension and under plain strain conditions, in the longitudinal and the transverse directions. The tests will be performed at room temperature, -320°F, and -423°F. Triplicate samples will be used in all tests.

At the conclusion of this work, it should be possible to determine the value to NASA of hot pressing large bodies from titanium alloy powders, as compared to fabricating the same objects from large, forged bar.