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The erratic behavior of the room temperature yield strength of high purity commercial tantalum sheet was studied. Critical factors in the production process were postulated and then investigated to determine their effects on strength properties. Chemical composition was found to be the most important factor contributing to the strength; thermomechanical factors which affected texture or grain size were also prominent. Recommendations are offered for better controlling the yield strength of tantalum.

The Effect of Typical Production Variables on the Strength of High Purity Commercial Tantalum Sheet

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THE EFFECT OF TYPICAL PRODUCTION VARIABLES ON THE STRENGTH OF HIGH PURITY COMMERCIAL TANTALUM SHEET

I. INTRODUCTION

Tantalum is a soft, high density, low-strength metal. Its many uses as a pure metal do not require great strength, but do depend on stable mechanical properties. During the past ten years the strength of tantalum has experienced a gradual decline. Whereas the yield strength commonly specified for tantalum is 22,000 psi, now this property ranges down to 20,000 psi or lower. This paper is an investigation of the reasons for the lower, varying strength with a goal toward finding means of improving it.

The strength of metals is influenced during production by two basic groups of factors: (a) melting practice which includes the resultant chemical composition and grain structure, and (b) fabrication practice including the effects of size-reducing operations and annealing treatments. The fabrication-related factors are known as thermo-mechanical variables.

Chemical composition is acknowledged as the key strength determinant in pure metals (5). The major property-determining impurities present in tantalum today are the interstitial elements (carbon, oxygen, nitrogen, hydrogen) and the substitutional element tungsten. Other elements are either present in insignificant quantities

or are known to be ineffective strengtheners. At the present time interstitials in commercial tantalum may account for no more than 50 parts per million (ppm) in aggregate, and tungsten content may be as low as 10-20 ppm. A survey of the data for the earlier, higher strength tantalum showed interstitial content to be high with oxygen up to 200 ppm, carbon 50-100 ppm, and nitrogen 50-100 ppm. The tungsten content of the same material was seldom listed, but was likely higher than generally found today.

The interstitial elements carbon, oxygen, nitrogen, and hydrogen provide the greatest strengthening per atom percent present of all the impurity elements (1). Seigle (18) found that the effect of oxygen, nitrogen, and carbon was roughly ten times that of tungsten. Szcopiak (21) recently determined for columbium that among the interstitials, nitrogen and oxygen are the most effective strengtheners, nitrogen being twice as effective as oxygen. The same should be true for tantalum since it is very similar to columbium. Puhr-Westerheide and Elssner (16) investigated the hardening of tantalum by nitrogen and oxygen. It was found that the change in hardness was linear with respect to atomic percent oxygen or nitrogen impurities present, at concentrations above approximately 60 ppm by weight. Calculations from the data indicated hardness increasing in this linear region 100 psi/weight ppm for nitrogen. Below 60 ppm, hardening per ppm present increased as the concentration of interstitial impurities

diminished. Gazza (13) investigated hydrogenation of tantalum sheet and found that 20-30 ppm hydrogen raised the yield strength from 22,000 to 26,000 psi with no appreciable effect on ductility at room temperature. The strength of tantalum is not likely affected by hydrogen content of less than 5 ppm.

The effect of tungsten in providing solid solution hardening to tantalum is more a function of group number in periodic table than of atom size difference (18). The group number represents a valence, or the number of electrons available for bonding. Tungsten with six d-shell electrons and tantalum with five form a strong bond which interacts with and slows down dislocation movement in its vicinity, causing work hardening. The presence of tungsten also limits initial grain size and slows down grain growth (10).

The composition of commercial tantalum is principally established by electron beam melting, the most common method of consolidation. Purity has been continually increasing over the past few years as input material (compressed powder) refined to a higher degree has been melted by improved electron beam techniques.

The electron beam process was developed specifically for tantalum and other metals which require the highest temperature for melting. In the process, electrons are emitted by hot tungsten cathodes. They are then gathered and focused by a combination of electric and magnetic fields, and directed onto a positively charged

electrode of pressed powder or welded scrap. The electrode is rotated slowly and lowered as symmetrically-arranged electron beams impinge on its base and cause melting to occur. The molten tantalum drops into a copper crucible where a liquid pool is maintained by additional electron beams. The ingot is withdrawn from the crucible as it cools.

Tantalum melts at temperatures higher than the vaporization temperature of most of the elements found as impurities in it. During electron beam melting most impurities are removed in combination with gaseous elements (pulled out by the high vacuum) or migrate to the cold walls of the crucible to become part of the slag-like exterior of the as-cast ingot. Each ingot is melted several times and each successive melting increases purity. The outer surface is conditioned by machining, and the finished ingot then possesses an inherent high purity. Fabricability is improved through the melting purification process, but most of the metallurgical strengtheners are removed. Tungsten and columbium, two other refractory metals, are not removed by electron beam melting. They are controlled in the input powder so that tungsten concentration is on the order of 100-300 ppm or less and columbium 300-700 ppm. Columbium, the sister element of tantalum, is the one refractory metal which is not a good solid solution strengthener for tantalum; although it is the impurity present in greatest quantity in high purity tantalum, it does not affect strength

or other properties significantly.

Another feature of the electron beam melted ingot which affects the strength of subsequent fabricated product is as-cast grain structure. Large, radially-oriented grains are formed during the relatively slow solidification process. Grain size refinement by fabrication, an acknowledged strengthening device, is hindered by this large, irregular initial grain size. Ingots that are the starting material for the fabrication process thus have a character of weakness which carries over into the finished product.

The continued commercial usefulness of tantalum requires that the deterioration in strength described above be checked. Electron beam melting is desirable for general purification and economy. Additional remelting cycles, by other methods such as the consumable vacuum arc, are costly in terms of material yield loss and equipment time. In any case, commercial material specifications allow little latitude for adjustment of chemical composition above the presently obtained low values.

Under the above limitations, the fabrication process must provide non-chemical strengthening, if strengthening is possible. Work toward such a goal has been investigated for other metals as the result of the same basic problem. The non-chemical approach is considered under the area of thermomechanical variables. These variables in the fabrication process fall into two categories: those

associated with the mechanical reduction in size of the material and those associated with the accompanying annealing cycles. Examples of each are listed below:

1. Size reduction variables: reduction rates, reduction schedules, type of equipment used, and temperature of primary and secondary size reduction.
2. Annealing variables: size, frequency, time, and temperature of intermediate anneals; vacuum capabilities and other vacuum furnace parameters; time and temperature of final anneals.

The standard fabrication sequence for tantalum sheet was reviewed to determine the thermomechanical variables. Fabrication consisted of initial forging of the ingot followed by subsequent rolling operations to reduce the thickness of the material and obtain the desired properties in the product. Mechanical sizing operations were interrupted by intermediate anneals as required for further processing, followed by a final anneal.

The fabrication process used in this investigation is discussed in the following paragraphs in chronological sequence. The sequence described represents only one of many possible sequences.

Tantalum was received for fabrication in the form of electron beam melted ingot, machine conditioned to a uniform seven-inch diameter. The initial forging operation converted the round ingot to rectangular sheetbar of approximately three-inch x seven-inch

configuration. Forging was accomplished on a 2000-ton Birdsboro press forge which sized the material by a slow, pressing action. (A quick-acting hammer forge might be expected to have imparted different properties to the material, but this was not investigated inasmuch as the equipment was not available.) The forging temperature was 800°F, which increased ductility in the ingot for ease in forging. A temperature of 1800-2200°F would be required to perform "hot-working" on tantalum where the structure of the metal would remain in the recrystallized, strain-free condition after forging. The effect of the low intermediate processing temperature on final size tensile properties was not investigated owing to the non-metallurgical nature of the temperature.

The next step in fabrication was to saw the forging into appropriate lengths for cross forging. Cross forging was done to obtain pieces of optimum width and weight for future rolling operations. A side benefit of this operation was the working of the material 90° to the last forging direction which should further break up the large-grained, as-cast structure. The second forging operation was again performed at 800°F on the Birdsboro press, and resulting size was nominally one inch thick. The one-inch forgings were ground to clean the surface of forging laps and then pickled, or chemically etched, in 1:1:1 HNO_3 - HF - H_2O acid mixture, to remove grinding smears. Pickled slabs were then ready for the first intermediate anneal.

The time and temperature of the intermediate anneal at one inch thick was chosen as one hour at 2200°F. This temperature provided full recrystallization with some grain growth. No variation in this temperature was investigated, and should be considered in future work.

The annealed sheetbars were rolled on a 4-high Waterbury-Farrell cold strip mill consisting of eight inch diameter work rolls and 30 inch diameter backup rolls. The initial reduction was approximately four percent per pass. Oil was used as a lubricant. The sheetbars were cold when rolling was initiated but quickly heated up to 400-600°F during rolling. The rolling operation was terminated as the material reached .300 inch thick. It was flattened by roller leveling at room temperature, pickled, and vacuum annealed.

The temperature chosen for anneal at .300 inch thick was one hour at 2000°F for the same reasons as the one inch anneal. Variation of this temperature might have had an effect on final size properties, but this was not investigated.

Tantalum was normally processed through the above sequence of operations in 1000-2000 lb. lots. The material was stocked at .300 inch and then used over a period of time for custom rolling to lower gage to meet requirements of individual orders. Orders were produced by cutting a sufficient quantity of material at .300 inch to be rolled and cross rolled as necessary for specified thickness, width and length. For example, an order requiring .060 inch x 16 inches x

length sheet was rolled from .300 inch x 12 inches x 8 inches to 12 inches x 16-1/2 inches (in the direction of former rolling) and then cross rolled to .060 inch x 16-1/2 inches x length. It was suspected that the combination of long rolling and cross rolling affected final size tensile properties, and this was investigated.

Another variable in the final rolling process was the combination of cold mills used. It was considered likely that the differences in type of strain and strain rate characteristic of the different mills affected final tensile properties. The large 4-high cold strip mill on which most of the sheet was rolled had a relatively low maximum rate of reduction or strain rate of .001 inch - .002 inch per pass at .040 inch thickness. The Sendzimir mill (Z-mill) reduced .040 inch tantalum .003-.004 inch per pass. These differences in reduction rate were directly attributable to differences in roll size diameter: 3/8 inch for the Z-mill and eight inches for the 4-high mill, and to the use of tension rolling on the Z-mill. Information was therefore gathered to correlate tensile properties with the rolling mill employed.

Material rolled to final size was acid-cleaned and vacuum annealed. The production annealing furnace was of cold wall design with refractory metal internal parts including shielding and heating elements. The working size was 22 inches diameter by 72 inches high. Operating characteristics included 3000°F maximum temperature capability, 2 to 5 x 10^{-5} mm Hg vacuum at temperature, and

inert gas backfilling capability for enhanced cooling at lower temperatures.

Temperature was measured by thermocouples ten inches and 50 inches down into the center of the cylindrical heat zone, and was recorded on a chart recorder. Vacuum was measured by a cold cathode ionization gauge and was also recorded continuously on a chart recorder.

Annealed sheet was sampled as per order requirements. Additional samples were requested (1) before annealing and (2) after annealing prior to roller leveling as needed. Roller leveling was usually performed on tantalum sheet to provide optimum flatness. As a final operation, the tantalum sheet was sheared to size and inspected.

II. EXPERIMENTAL PROCEDURES

Commercially-produced electron beam melted tantalum sheet was studied to determine the reasons for variation in strength properties. Data was obtained from an actual production operation at Teledyne Wah Chang Albany Corporation in Albany, Oregon. The variables in the process were changed as necessary and the effects of these changes were observed. Special fabrication sequences were designed and carried out to provide more closely controlled rolling and annealing studies where necessary.

The sheet investigated ranged in thickness from .005 inch to .100 inch, and was nominally 99.9% pure with major impurities as listed in Table 1.

Table 1. Chemical impurities for various tantalum heats. *

Heat	W	Cb	C	O	N	H
A	40	402	<30	<50	16	<5
B	420	358	<30	60	9	<5
C	27	434	<30	<50	12	<5
D	44	400	<30	<50	15	6
E	25	480	<30	<50	17	<5
F	12	<50	35	60	12	<5
G	29	<50	<30	<50	20	<5
H	44	374	<30	<50	22	<5

* Readings shown are average of 2-5 readings. All other elements tested were less than minimum detectable values.

All mechanical, metallographic, and chemical testing was performed by laboratory personnel at Teledyne Wah Chang Albany. Tensile samples, both longitudinal and transverse, were machined 1/2 inch wide with a one inch gage length and tested in accordance with ASTM E8 on an Instron universal mechanical testing machine. A strain rate of .005 inch/inch/min was followed manually through the .2% offset point, and .5 inch/inch/min free crosshead movement from that point to failure. The load-strain curve was continuously recorded. Stress values were calculated from it and specimen cross-sectional area. Elongation (EL) was measured on the failed specimen. Calculated stress values included .2% offset yield strength (YS), lower yield point strength (LYP), and ultimate tensile strength (UTS).

Metallographic examination of the tantalum sheet samples was performed in accordance with ASTM E112. Samples were mounted to show the longitudinal size view, then polished and etched in accordance with standard procedures. Percent recrystallization was estimated and ASTM grain size was determined by comparison on the metallograph. Diamond pyramid hardness (DPH) was measured and calculated for an average of three indentations per sample. The standard DPH load was 0.5 kilogram.

The process histories for the data were obtained from the completed "traveller", the processing instruction document which accompanied each order. Additional annealing history information was taken

from the logbook at the annealing furnace.

Ingot chemistry was obtained from company files. Chemical analysis for interstitial content on the product was initially spot checked but discontinued when no significant differences between it and ingot chemistry were noted.

Special studies on closely controlled fabrication processes were conducted on the same production equipment as described previously. Annealing for these special studies was conducted in the laboratory test furnace, a Marshall model with refractory metal interior parts, $2900^{\circ}F$ maximum temperature capability, and operating vacuum of 2 to 5 x 10^{-5}mm Hg. The heat zone was two inches diameter x ten inches long.

III. RESULTS AND DISCUSSION

General

The results of an investigation into the room temperature strength characteristics of commercially produced tantalum sheet are shown in Table 2. The table is arranged in order of increasing thickness of tantalum sheet, by heat number (the unique number assigned to each ingot).

The major data groupings in Table 2 are: (1) the strengths and a strength-associated parameter: lower yield point strength (LYP), ultimate strength (UTS), and elongation (EL); (2) the data resulting from metallographic examination; and (3) data concerning thermo-mechanical variables. The chemical analysis of significant impurities for each heat is presented in Table 1.

Several comments should be made concerning features of Table 2.

1. Lower yield point strength is reported rather than 0.2% offset yield strength. Due to the nature of the stress-strain curve for tantalum (typical example shown in Figure 1), the 0.2% offset determination varied considerably from one test to another; that is, the descending slope of the stress curve beyond the upper yield point varied from sample to sample and appeared to depend on testing technique. It is seen from the typical location of the 0.2% offset strength

KEY TO TABLE 2

(1) Thickness of sample in inches.

(2) Percent reduction is calculated from .300 inch thick starting size, and final thickness.

(3) Roller leveled samples indicated by 1; non-roller leveled samples by 0. Roller leveling done after final anneal.

(4) LYP_T is transverse lower yield point strength.

(5) UTS_T is transverse ultimate tensile strength.

(6) EL_T is transverse elongation.

(7) DPH is diamond pyramid hardness measured with standard 500 gram load.

(8) % R_x is percent recrystallization of annealed sample.

(9) GS is ASTM grain size number.

(10) Anneal furnace: 1 is the production furnace and 2 is the laboratory furnace.

(11) Rolling method is listed by percent long rolling from .300 inch prior to cross rolling to final size.

(12) Rolling mill: 1 indicates the large 4-high cold strip mill; 2 indicates a combination of large 4-high from .300 inch to .040 inch and Sendzimir mill from .040 inch to final size; 3 indicates large 4-high from .300 inch to .080 inch, small 4-high from .080 to .040 inch and Sendzimir cluster mill from .040 inch to final size.

Table 2. Commercial tantalum strength properties as functions of metallographic and thermo-mechanical variables.

Heat A

Sample No.	(1) Gage inches	(2) Percent Reduction	(3) R/L	(4) LYP_T ksi	(5) UTS_T ksi	(6) EL_T %	(7) DPH	(8) $\%R_x$	(9) GS
1	.013	96	--	--	--	--	77	100	9
2	"	"	1	28.3	45.1	40	113	100	8.5
3	"	"	0	23.1	42.0	44	89	100	8.5
4	.015	95	0	26.1	43.8	39	91	100	8
5	"	"	0	24.0	40.9	39	90	100	8.5
6	"	"	1	25.0	39.3	32	--	100	8
7	"	"	1	24.2	37.7	28	90	100	8.5
8	.016	94.5	1	26.4	42.6	41	83	100	8
9	"	"	--	--	--	--	87	100	7
10	"	"	1	25.2	40.8	44	82	100	8.5
11	"	"	0	20.9	36.7	46	82	100	8
12	"	"	0	25.2	44.4	41	96	100	8
13	"	"	0	27.6	42.8	37	86	100	8
14	"	"	1	26.3	41.9	38	95	100	8
18	.060	80	--	--	--	--	79	100	7.5
19	.063	79	1	24.2	39.5	55	80	100	7.5
20	.063	79	0	19.5	37.2	61	85	100	7.5

Table 2. (Cont.)

Sample No.	Sample Thickness inches	(10) Anneal Furnace	Heat A Final Anneal Time hrs	Heat A Final Anneal Temp. °F	(11) Rolling Method (.300 inch to final size)	(12) Rolling Mill
1	.013	1	1.5	1800	0	2
2	"	1	"	"	0	2
3	"	1	"	"	0	2
4	.015	2	1	"	100	2
5	"	1	1.5	"	100	2
6	"	1	"	"	100	2
7	"	1	"	"	100	2
8	.016	1	"	"	65	1
9	"	1	"	"	65	1
10	"	1	"	"	16	1
11	"	1	"	"	16	1
12	"	2	1	"	0	1
13	"	1	1.5	"	0	1
14	"	1	"	"	0	1
18	.060	1	"	"	100	1
19	.063	1	"	"	40	1
20	.063	1	"	"	40	1

Table 2. (Cont.)

			Heat B *						
Sample No.	(1) Gage inches	(2) Percent Reduction	(3) R/L	(4) LYP_T ksi	(5) UTS_T ksi	(6) EL_T %	(7) DPH	(8) $\%R_x$	(9) GS
22	.010	96.5	--	--	--	--	92	100	7.5
23	.015	95	--	--	--	--	99	95	8.5
30	.025	92	--	--	--	--	121	100	9.5
31	"	"	0	29.2	46.1	39	100	90	8
32	"	"	0	31.6	50.3	35	109	85	9.5
33	"	"	0	28.3	46.7	38	88	95	8
35	.040	87	--	--	--	--	76	100	7
37	.060	80	--	--	--	--	85	90	8
38	.072	76	--	--	--	--	93	70	8
39	"	"	--	--	--	--	77	95	7.5
41	.125	58.5	--	--	--	--	--	90	7.5

* See also data in Table 4 .

Table 2. (Cont.)

Heat B						
Sample No.	Sample Thickness inches	(10) Anneal Furnace	Final Anneal Time hrs	Final Anneal Temp. °F	(11) Rolling Method (.300 inch to final size)	(12) Rolling Mill
22	.010	1	2	1900	100	2
23	.015	1	2	1750	"	1
30	.025	1	1.5	1800	"	1
31	"	2	1	"	"	1
32	"	2	1	"	0	1
33	"	1	1.5	"	--	1
35	.040	1	"	"	100	1
37	.060	1	"	"	--	1
38	.072	1	"	"	0	1
39	"	2	2.5	1850	0	1
41	.125	1	1	1850	40	1
42	.260	1	2	1900	100	1

Table 2. (Cont.)

Heat C

Primary Proc. Hist. No.	Sample No.	(1) Gage, inches	(2) Percent Reduction	(3) R/L	(4) LYP_T ksi	(5) UTS_T ksi	(6) EL_T %	(7) DPH	(8) $\%R_x$	(9) GS
I	45	.010	96.5	0	26.3	43.3	33	74	100	7.5
	46	"	"	0	25.0	43.1	34	87	100	7.5
	47	"	"	--	--	--	--	84	100	8
	48	.017	94.5	1	24.4	36.4	46	93	100	8
	49	.020	93	1	23.3	37.3	49	93	100	7
	50	"	"	0	27.3	42.4	48	88	100	7.5
	51	"	"	1	24.5	38.4	50	111	100	7.5
	52	.022	92.5	1	26.2	38.5	41	91	100	7.5
	53	.030	90	--	--	--	--	--	100	7.5
	56	.050	83.5	--	--	--	--	77	100	7
II	62	.030	90	--	--	--	--	82	100	7
	63	"	"	--	--	--	--	98	100	7
	64	"	"	0	22.8	38.6	42	87	100	8
	65	"	"	1	25.3	38.4	50	97	100	7.5
III	66	.020	93	1	27.6	42.5	41	96	100	7.5
	68	.060	80	--	--	--	--	92	100	7.5
IV	70	.010	96.5	--	--	--	--	91	100	7.5
	71	"	"	--	--	--	--	94	100	7.5
	72	"	"	0	24.5	42.6	42	90	100	8
	73	"	"	1	27.5	41.6	42	--	100	8.5
	74	.015	95	--	--	--	--	90	100	7.5
	75	"	"	--	--	--	--	89	100	7.5
	76	"	"	1	24.8	37.5	44	--	100	7.5
	78	.060	80	--	--	--	--	--	100	6

Table 2. (Cont.)

Heat C

Primary Proc. Hist. No.	Sample No.	Gage inches	(10) Anneal Furnace	Final Anneal Time hrs	Final Anneal Temp. °F	(11) Rolling Method (.300 inch to final size)	(12) Rolling Mill
I	45	.010	2	1	1800	100	1
	46	"	1	1.5	"	100	1
	47	"	1	"	"	100	1
	48	.017	2	1	"	0	1
	49	.020	1	1.5	"	67	1
	50	"	1	"	"	5	1
	51	"	1	"	"	5	1
	52	.022	1	2	1750	0	1
	53	.030	1	1.5	1800	65	1
	56	.050	1	1.5	"	60	1
II	62	.030	2	1	"	100	1
	63	"	2	1	"	0	1
	64	"	1	1.5	"	--	1
	65	"	1	"	"	--	1
III	66	.020	1	1	"	70	1
	68	.060	1	1.5	"	--	1
IV	70	.010	2	1	"	100	1
	71	"	2	"	"	0	1
	72	"	1	1.5	"	--	1
	73	"	1	"	"	--	1
	74	.015	2	1	"	100	1
	75	"	2	1	"	0	1
	76	"	1	1.5	"	--	1
	78	.060	1	"	"	4	1

Table 2. (Cont.)

Sample No.	(1) Gage inches	(2) Percent Reduction	(3) R/L	(4) LYP_T ksi	(5) UTS_T ksi	(6) EL_T %	(7) DPH	(8) $\%R_x$	(9) GS
				Heat D					
87	.005	98.3	0	21.3	37.7	34	79	100	7.5
88	.005	"	--	24.6	41.7	36	--	100	7.5
91	.015	95	--	--	--	--	84	100	7
92	.015	95	1	25.9	41.6	29	92	97	8.5
94	.016	94.5	--	18.7	33.9	48	83	100	6.5
95	.020	93	1	23.4	36.2	47	84	100	6.5
96	.020	"	--	--	--	--	80	100	6.5
				Heat E					
101	.010	96.5	--	--	--	--	84	100	8
102	"	"	--	--	--	--	92	100	8.5
103	"	"	--	--	--	--	88	100	8.5
106	.015	95	--	--	--	--	83	100	7.5
109	.030	90	1	25.5	41.5	35	--	100	8
110	.031	89.5	--	--	--	--	89	100	7.5
114	.060	80	--	--	--	--	85	97	6
				Heat F					
122	.015	95	0	20.2	37.0	46	83	100	7
123	"	"	1	20.9	36.8	49	111	100	7
124	"	"	1	23.1	37.9	40	111	100	7
126	.030	90	--	--	--	--	83	70	6.5
127	"	"	--	--	--	--	82	100	6.5
128	"	"	1	23.8	34.4	61	85	100	6.5

Table 2. (Cont.)

Sample No.	Gage inches	(10) Anneal Furnace	Final Anneal Time hrs	Final Anneal Temp. °F	(11) Rolling Method (.300 inch to final size)	(12) Rolling Mill
			Heat D			
87	.005	1	1.5	1800	100	3
88	.005	1	"	"	100	3
91	.015	2	1	"	100	3
92	"	1	1.5	"	100	3
94	.016	1	1.5	"	40	1
95	.020	1	2	1750	100	2
96	"	1	1.5	1800	--	--
			Heat E			
101	.010	--	1	1800	100	1
102	"	1	1.5	"	--	--
103	"	1	"	"	--	--
106	.015	1	"	"	100	1
109	.030	1	"	"	33	1
110	.031	1	"	"	87	1
114	.060	1	"	"	100	1
			Heat F			
122	.015	1	1.5	1800	40	1
123	"	1	"	"	40	1
124	"	1	"	"	40	1
126	.030	2	1	1650	10	1
127	"	2	1	1800	10	1
128	"	1	2	1750	10	1

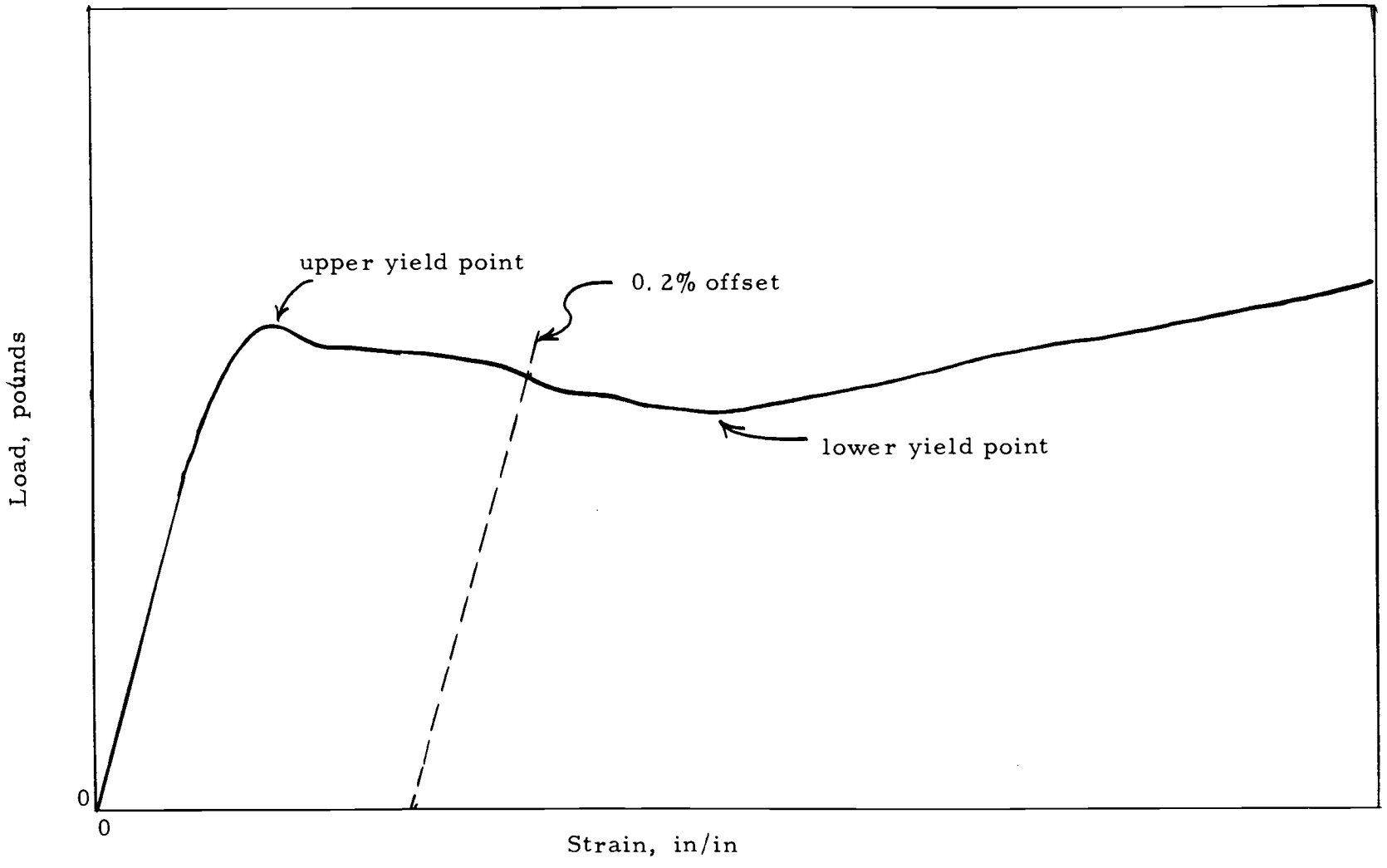

Figure 1. Typical load-strain curve for tantalum sheet.

that this value would be of little design significance. The lower yield point is the minimum strength encountered in plastic deformation.

2. The annealing temperature for 90% of the data was 1800°F, with time at temperature being 1.5 hours in the production furnace or one hour in the laboratory furnace. The two differing times were considered to give equivalent results as determined through previous testing. One possible reason for this is the difference in heat-up rates for the two furnaces, with the rate for the laboratory furnace being about 20 times faster. The difference should be kept in mind, as well as an awareness of the presence in Table 2 of annealing temperatures other than 1800°F. Percent recrystallization must also be checked prior to comparing tensile values.

3. Percent reduction was calculated based on .300 inch starting thickness, held constant for all the data listed in Table 2. The singular starting size represents an arbitrary limit and influences the perspective from which this data is viewed.

4. Only transverse strength values are listed in Table 2. Longitudinal values are available in Appendix I and were not included in Table 2, in order to minimize data and use the values usually considered to be lower in value. Many specifications require only transverse tensile data to be reported.

5. Considerable scatter is seen in the strength values in Table 2. The scatter is due in part to the samples having been taken directly

from material which was not as closely controlled as laboratory samples would have been. The measured strength of tantalum is also sensitive to small differences in testing technique.

6. Roller leveling (R/L) is an effective strengthening process as discussed in the section "Effects of Roller Leveling." Comparisons of different samples must take differences of roller leveling into consideration. Paired un-roller leveled and subsequent roller leveled samples in Table 2 are indicated by brackets.

Several general trends and interesting data can be noted from Table 2.

Within each heat, annealed strengths and hardnesses generally increase with increasing amounts of reduction prior to anneal. All of the heats except one, heat B, show considerable similarity. The reasons for the differences in heat B will be discussed in the section "Effect of Chemical Composition." A survey of all of the data indicates that 1 to 1-1/2 hours at 1800°F is a near optimum annealing treatment as most samples were 100% recrystallized with a grain size average of 7-1/2 - 8. Such a grain size is 1-2 ASTM grain sizes finer than that normally expected from electron beam melted sheet product at these sizes. The reason for the smaller grained structure may be the 90% minimum reduction prior to final anneal. It may also be the cross forging operation included in the primary processing, which would help to break up and refine the large-grained, cast structure.

The predominant material size observed in the data is .015-.020 inch, the most commonly asked for tantalum sheet size. Other factors being equal, the average hardness for this size material appears to be 90 DPH. The average strength values (not roller leveled) are: (a) LYP: 20,000-24,000 PSI; (b) UTS: 38,000-42,000 PSI; and (c) EL: 40-50%.

Lower yield strength was selected as the principal dependent variable for this study, and its variation with respect to the other factors listed in Table 2 is discussed in the following sections.

Relationships with Metallographic Variables

Effect of Grain Size

The grain size of annealed tantalum sheet proved to be a most significant factor with regard to yield strength. Higher strengths and hardness were accompanied by small grains. Figures 2, 3, and 4 show this dependency. Early investigators (7) stated that yield strength was not a function of grain size. This conclusion was based on tests of high-interstitial content tantalum and the effects of composition likely overshadowed the effect of grain size.

The work of Petch (14), which related strength inversely to the square root of the grain "diameter", has since been accepted as showing the importance of grain size in determining strength for most metals.

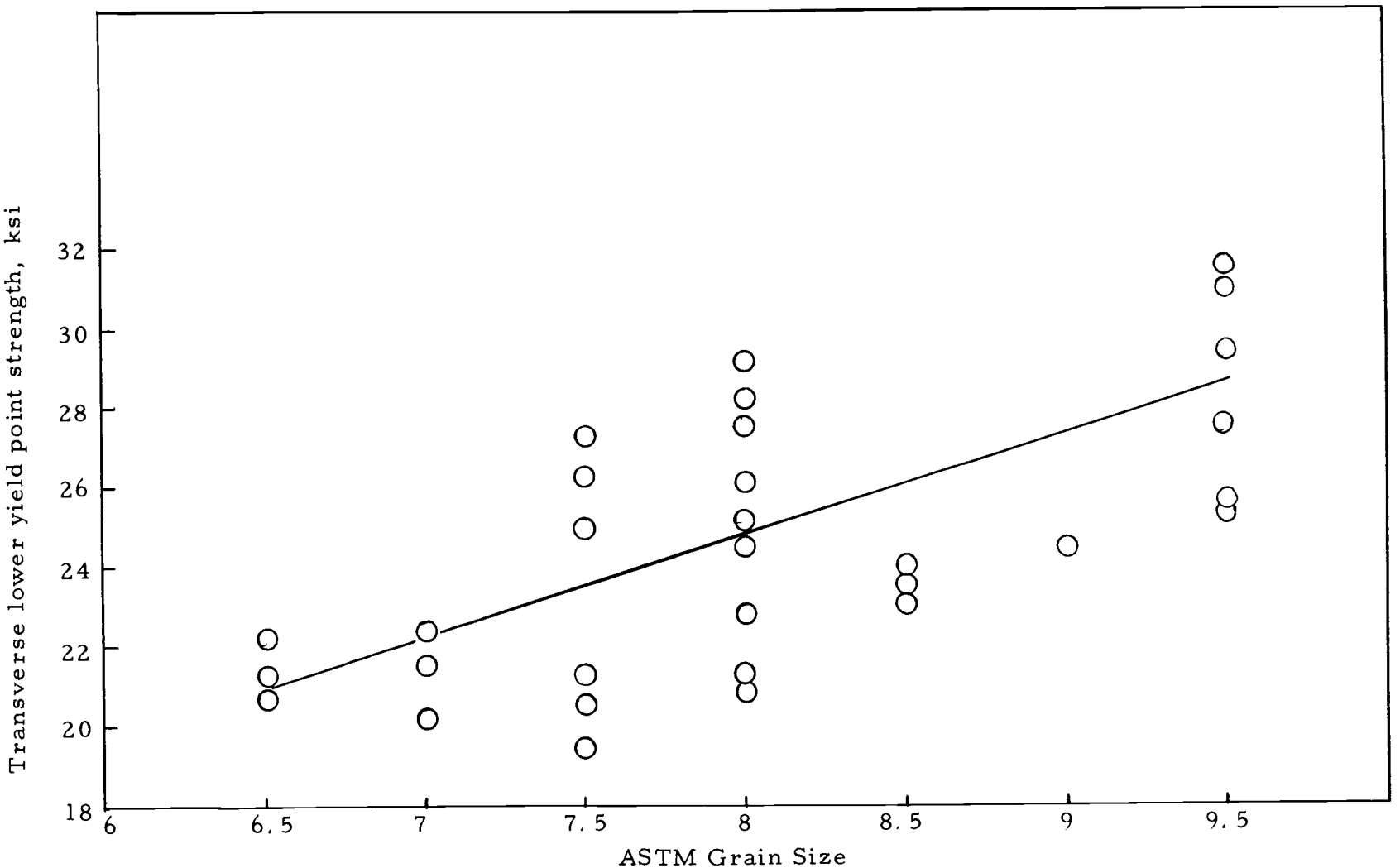

Figure 2. Transverse lower yield strength vs. ASTM grain size (un-roller levelled data only from Tables 2 and 4).

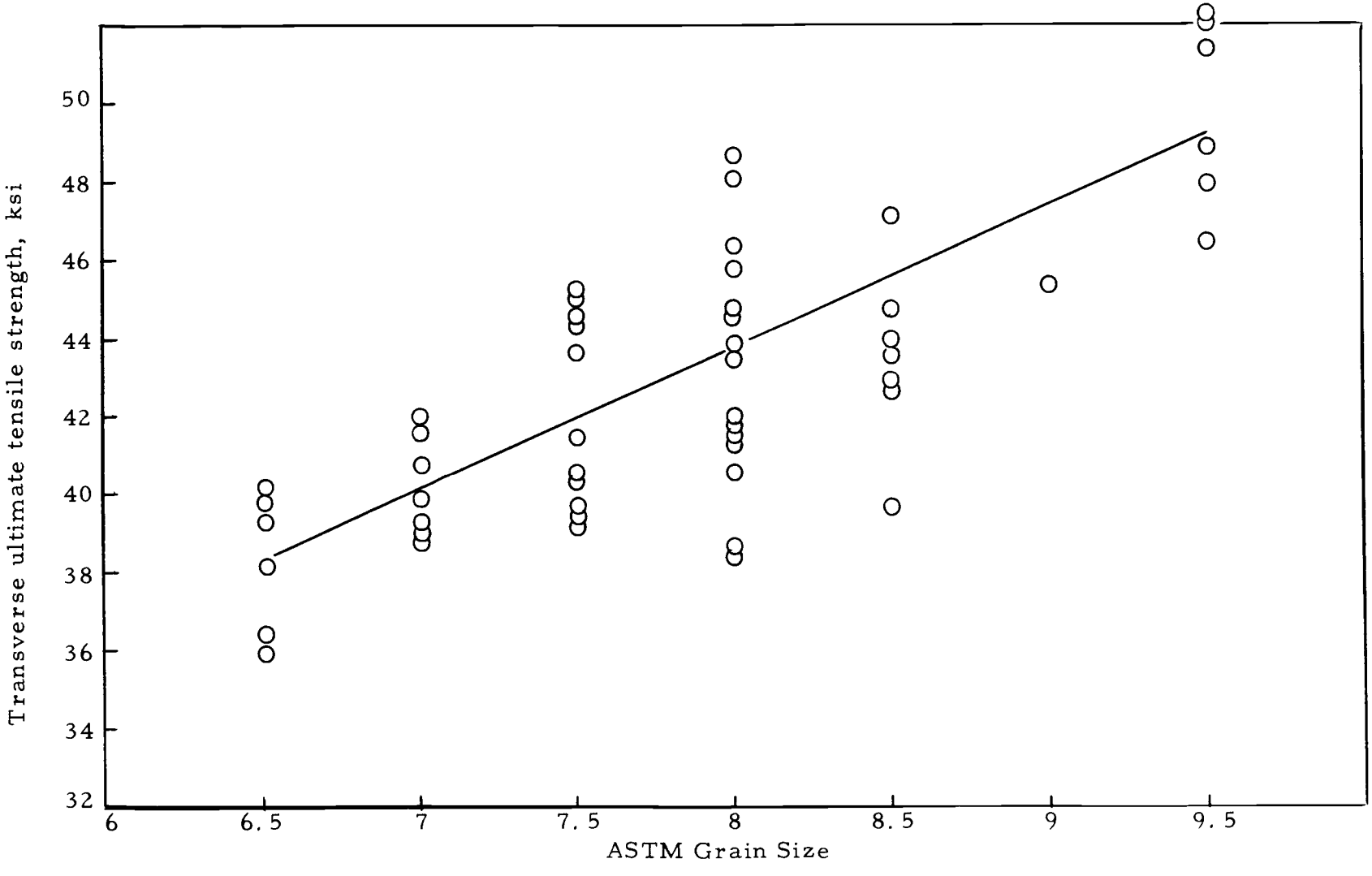

Figure 3. Transverse ultimate tensile strength vs. ASTM grain size (data from Tables 2 and 4).

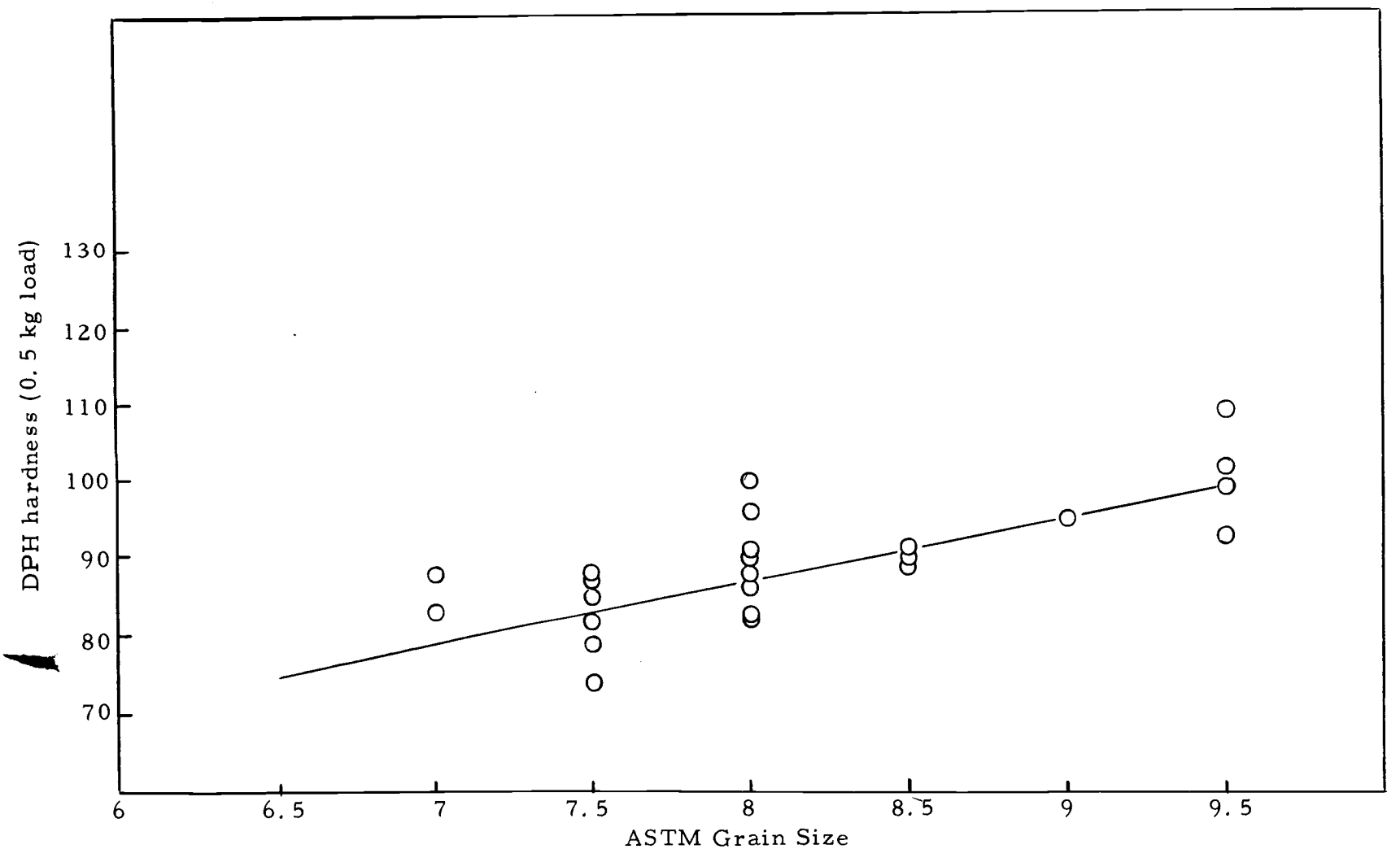

Figure 4. DPH hardness vs. ASTM grain size (un-roller levelled data only from Tables 2 and 4).

Grain size is itself a function of thermomechanical parameters, among them percent reduction, annealing temperature, and prior grain size. Increased percent reduction decreases grain size; higher annealing temperature increases grain size; and small prior grain size begets small final grain size (5). It is also a function of chemical composition (see discussion of "Effects of Chemical Composition"). However obtained, small-grained structure and larger yield strengths go hand-in-hand. A recent review by B. A. Wilcox (22) on "Basic Strengthening Mechanisms in Refractory Metals" cites the following means of strengthening: (1) strengthening by second phase particles, (2) solid solution strengthening, (3) strain hardening and grain size refinement, (4) retaining worked structures at high temperatures, (5) dynamic strengthening, and (6) fiber reinforcement. Second phase particles and fiber reinforcement were not considered in this study of high purity tantalum. Retained worked structures at high temperatures were not applicable to the room temperature properties investigated. Grain size refinement is thus one of the three available means of strengthening tantalum.

The grain size shown for heat B in Table 2 is seen to be considerably finer than for all other heats. The one factor observably different about it is the tungsten content, highest of all heats at 450 ppm average. Tungsten in solid solution is considered to limit grain size by providing more nucleation sites for new grains and then limiting

grain growth by serving as obstacles to grain boundary movement (10).

The average grain size for .010-.030 inch material of each of the heats in Table 1 is listed in Table 3.

Table 3. Average grain sizes for tantalum heats (.010-.030 inch material from Table 2).

Heat	Average Grain Size
F	7
D	7
C	7.5
E	8
A	8
B	9

A comparison of these grain sizes with the chemical composition listed in Table 1 again indicates tungsten as an important factor in grain size. Tungsten content increases as grain size decreases, with the exception of heat D.

Effect of Percent Recrystallization

Partially recrystallized grain structures are characterized by a higher yield strength, ultimate strength, and hardness as all stress has not been removed. An attempt was made in this data to standardize at 100% recrystallization in order to limit the number of variables under consideration and provide data consistent with normal production requirements.

The data in Table 2 shows that the 1 - 1-1/2 hour at 1800°F annealing cycle did completely recrystallize most of the sizes

considered, even with their varying histories. Heat B was an exception and had the greatest occurrence of incomplete recrystallization. The higher tungsten content of this heat appeared to raise the required recrystallization temperature.

Effect of Hardness

Yield strength was observed (Figure 5) to vary directly with hardness. This standard relationship makes it possible to use the hardness test in lieu of the tensile test for rapid metallurgical evaluations. Hardness also increased with increasing percent reduction prior to final anneal, for data in Table 2. Heat B exhibited hardnesses an average of 8-10 DPH greater than the other heats. This appeared to be attributable to the ten-fold increase in tungsten content of this heat as compared to the others.

Relationships with Thermomechanical Variables

Effect of Time and Temperature of Anneal

Time and temperature of anneal were not varied appreciably in the production data shown in Table 2. For this reason, special studies were run to determine the effect of variation of time and temperature on yield strength. The results are shown in Table 4.

The .005 inch foil from Heat D in Table 4 exhibited little variation in strength properties as time and temperature were varied from

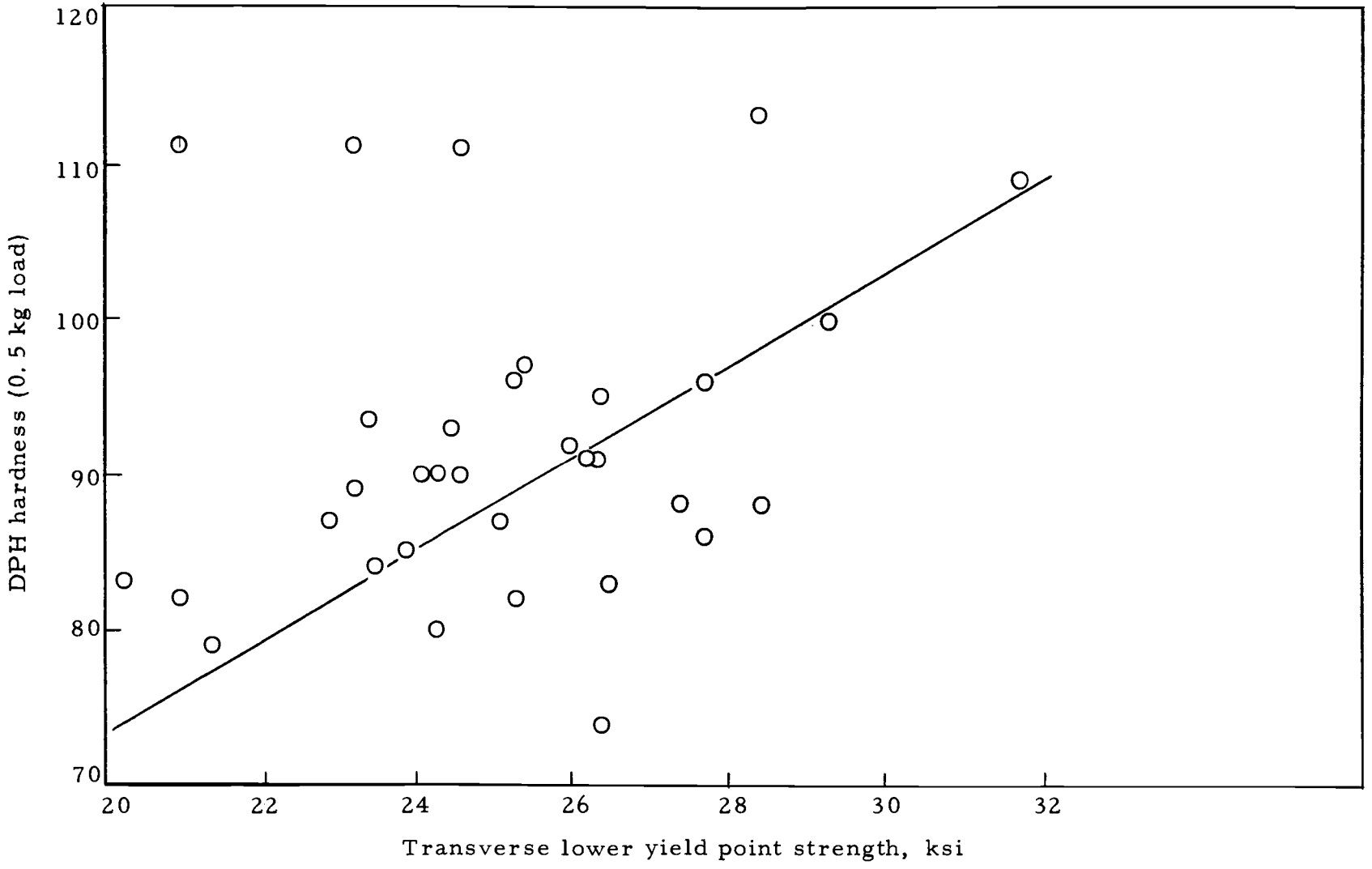

Figure 5. DPH hardness vs. transverse lower yield strength (data from Table 2).

Table 4. Variation of mechanical and metallographic properties with time and temperature of final anneal.

Heat	Anneal Time (hr) Temperature (°F)	LYP_T ksi	LYP_L ksi	UTS_T ksi	UTS_L ksi	EL_T %	EL_L %	DPH	% R_x	GS
a. D (.005) inch thick	3 hr @ 1600°F	22.5	20.9	40.0	39.6	38	35	*109	100	7
	1 hr @ 1700°F	21.7	20.6	37.8	38.4	35	32	*109	100	6.5
	2 hr @ 1700°F	22.3	20.3	39.6	39.3	37	33	*106	100	7
	1 hr @ 1800°F	21.3	21.7	37.3	37.7	37	34	* 99	100	6.5
	2 hr @ 1800°F	22.2	20.6	38.2	38.1	32	33	*112	100	6.5
	1 hr @ 1900°F	21.6	20.0	36.7	36.7	33	27	* 91	100	5.5
b. C (.015) inch thick	1 hr @ 1550°F	23.6	--	42.8	--	35	--	91	95-100	8.5
	1 hr @ 1600°F	22.7	--	40.0	--	35	--	87	95	8
	1 hr @ 1650°F	21.4	--	39.8	--	37	--	83	95-100	8
	1 hr @ 1700°F	21.2	--	39.5	--	37	--	91	100	8
	1 hr @ 1800°F	20.6	--	37.6	--	40	--	82	100	7.5
	1 hr @ 1850°F	21.6	--	38.8	--	40	--	88	100	7
c. B (.020 inch thick)	3 hr @ 1600°F	31.0	32.0	49.4	51.3	36	35	102	90	9.5
	1 hr @ 1700°F	27.6	27.1	46.9	47.7	40	42	93	95	9.5
	2 hr @ 1700°F	29.5	32.1	50.1	51.5	36	38	102	90	9.5
	1 hr @ 1800°F	25.7	24.8	44.5	45.7	50	50	99	95	9.5
	2 hr @ 1800°F	25.3	26.6	46.0	47.1	43	42	93	95	9.5
	1 hr @ 1900°F	24.5	23.6	43.4	44.4	45	49	95	100	9

* .025 kg load

three hours at 1600°F to one hour at 1900°F. These samples also displayed unexpectedly low strengths considering their gage and the percent reduction (98%). In view of this large reduction, the annealing time/temperature combination was probably too high in all cases and grain growth occurred. The large grain size noted reinforced this probability. Imgram (7) states that grain growth is more a function of temperature than of time. Data from Heat D is limited but appears to confirm this. One and two hour heat treatments at both 1700°F and 1800°F indicate no grain growth.

The hardness of the .005 inch foil appeared to be high, but the load used on this thin material was .025 kilograms instead of the .5 kilograms used throughout the balance of this report. Direct comparisons of these hardnesses with others in the report should thus be avoided.

A detectable difference in strength and hardness was noted between the one hour anneals and the two and three hour anneals. Tantalum at this thin gage usually experiences a surface pickup of interstitial contaminants (O_2, N_2, H_2) during anneals, and this was considered to be the main factor in the slight hardness and strength increases. It should also be noted that the hardness decreased with increasing time/temperature combinations more significantly than did either of the strength values. The .005 inch sheet had strength properties near the average of those listed in Table 2.

The . 015 inch sheet from Heat C in Table 4 showed a steady decrease in strengths from the 1550°F to the 1800°F annealing temperature. This trend and the steady grain growth seen were as expected. The 1850°F behavior was possibly the same phenomenon as noted but not explained by others, namely, a slight increase in strength just past the optimum annealing temperature. The data indicated that the one hour recrystallization temperature for Heat C material was between 1600°F and 1700°F. With other factors held constant, this further confirmed the possibility that the . 005 inch foil from Heat D, Table 4, was overannealed. The greater percent reduction in the . 005 inch material as compared to the . 015 inch material would have supplied greater driving force for recrystallization.

The study of the . 015 inch sheet indicated the difficulty in improving yield strength toward the acceptable 22,000 psi value solely by varying the time and temperature of anneal. Both the . 015 and . 005 inch material data reveal the hazards of over-annealing. The . 015-inch sheet, as with the . 005-inch foil, had tensile values average with respect to the general results of Table 2.

The . 020 inch sheet from Heat B in Table 4 displayed the unusual properties of this heat. The higher-than-usual strengths were consistent throughout the data shown. Hardnesses were correspondingly high. Grain sizes were uniformly small.

A small component of the high strengths in the Heat B data was the lack of full recrystallization. Thermomechanical factors in the production of the Heat B were similar to those for the low strength heats. Therefore, chemical composition was indicated as the principal reason for the higher strength of Heat B.

A comparison of the above three studies again reveals the difficulty in obtaining a minimum yield strength of 22,000 psi along with acceptable ultimate strength and elongation values. Varying the time and temperature of anneal accomplished only minor adjustments in strength. Raising the strength through incomplete recrystallization is not an acceptable practice.

Effect of Annealing Furnace

The production and laboratory furnaces had similar operating characteristics despite considerable size differences. The laboratory furnace was in effect a scale model of the production furnace. There was no noticeable difference in their effects on tensile strengths, in spite of the difference in time at temperature. A more accurate method of grain size determination than the ASTM method might have been better differentiated between the two treatments.

Furnace parameters were not investigated in depth. Some of these parameters that should be given future study include heat up rate, cooling rate, and vacuum conditions. Harris (4) reported that faster

heating rates cause greater absorption of gaseous interstitials by the material being annealed. Impurities are provided by the heated internal furnace parts and are gettered by the annealed material where the vacuum system cannot keep pace with the rapid heat-up. On cool down, slower cooling rates allow more interstitial segregation and its accompanying softening effect (18). Inert gas backfilling is not generally accepted for rapid cooling of final-size refractory metals because of the possibility of contamination by the impurities in the inert gas. Other quenching techniques have proven difficult to adapt to vacuum equipment. Owen et al. (12) reported that a vacuum of 5×10^{-6} mm Hg or better was found to yield more uniform tensile test results and lower yield stresses than for material annealed at 10^{-4} mm Hg. A survey of data in the literature from ten years ago showed most yield strengths to be 24,000-30,000 psi. Although lower purity was perhaps the major factor causing this, the possibility of a lower vacuum heat treatment at that time should also be considered. Most of the past data found did not specify the vacuum conditions of anneal. Owen et al. (12) postulated that changes in annealing temperatures and vacuums caused changes in the substructure of refractory metals and that substructure, especially in high purity metals, principally determines strength.

Effect of Percent Reduction

The data in Table 2 shows relatively little difference within each heat for the strength of material having thickness .010-.030 inch (the majority of the data). These thicknesses correspond to 90-96% reduction since the last anneal, and it is likely that the effect of cold working has slowed down appreciably in this range. Strength and microstructure are most affected at reductions just beyond the critical strain (5), which has been established at 18% reduction for material of similar chemical composition (10). Data scatter inherent in the production data may be another factor in the lack of a trend in the effect of reduction on tensile properties. In order to better investigate the effect of percent reduction on yield strength, a special study was performed. Annealed .300 inch thick tantalum plate was long rolled to .150, .100, .060, .045, and .030 inch, and annealed. Samples were then long rolled to .015 inch and annealed in the laboratory furnace one hour at 1800°F. Transverse tensiles and microstructure samples were taken at final size and the results are listed in Table 5.

Final reductions in thickness of 50, 67, 75, 85, and 90% on Heat G imparted little change in any of the strength parameters. Accepted theory states that strength (even after anneal) should increase with increasing percent reduction prior to anneal (5, 7). The data for Heat G shows a slight opposite trend as strength decreases from 50

Table 5. Variation of mechanical and metallographic properties with percent reduction prior to final anneal (.015 inch tantalum sheet).

Heat	% Reduction Prior to Final Anneal	LYP_T ksi	UTS_T ksi	EL_T %	DPH	% R_x	GS
G	50	21.3	38.8	50	79	100	6
	67	21.1	38.5	51	80	100	6
	75	20.9	37.8	56	76	100	6
	85	20.7	37.8	53	77	100	6.5
	90	20.8	38.0	50	78	100	6.5
H	50	24.6	42.9	40	73	100	6
	67	23.3	41.8	46	84	100	7
	75	22.0	40.9	47	87	100	7.5
	85	22.8	41.7	48	79	100	8

through 85% reduction, and then appears to rise slightly at 90%. The microstructure is large-grained.

Heat H exhibits a more pronounced downward trend in strength with increasing percent reduction, with a slight upturn at 85% reduction. The grain size for Heat H also decreased faster than that for Heat G, with increasing percent reduction. Thus the grain size follows the predicted pattern by decreasing with increased percent reduction (5) but the strength proceeds opposite to theory. Both heats were similar in chemical composition and in thermomechanical processing. A tentative explanation for this phenomena is that impurities are an implicit requirement in work hardening tantalum and they are present in such small quantities in this high purity tantalum that work hardening is kept to a minimum.

Another possible view of the observed decrease in annealed strength with increasing percent reduction prior to anneal concerns the interaction of texture and chemical impurities. Greater percent reduction prior to final anneal strengthens the rolling texture. It also increases the stored energy available to change the texture through annealing. The evidence shown in Table 5 indicates that the annealing texture for these two heats of long-rolled tantalum sheet was a low-strength texture, and that the effects of the low strength texture evidently superseded the grain boundary strength component. If grain boundary strengthening is partially dependent on impurities

which are located initially in the grain boundary region or move there in an energy-lowering maneuver during the anneal, then low impurity content would aid in decreasing the effects of grain boundaries relative to the effect of texture.

A further effect of increased percent reduction was observed in the shape of the grains as annealed. Micrographic examination indicated that increased percent reduction resulted in flatter grains. Elongated grains have more of their grain boundary area parallel to the rolling direction, oriented for yielding through shear (weaker) rather than tension. Both the micromechanism, texture, and the macro-mechanism, grain flattening, may have contributed to the lower strengths noted with increasing percent reduction.

Effect of Cross Rolling

The data in Table 2 appeared to show considerable strength variation for different combinations of long rolling and cross rolling from .300 inch to final size. Many of the low yield strength samples had 10-70% long rolling prior to cross rolling. Cross rolling would be expected to cause differences in strength by changing the texture of the sheet. To further investigate the effect of cross rolling, a special study was conducted on two heats, Heat B and Heat C, to determine the effect of gradually increased amounts of long rolling. Samples were rolled from .300 inch 20, 40, 60, and 80% in the long direction

prior to cross roll to .015 inch. One sample (100) was also long rolled directly to .015 inch without cross roll, and another (0) was cross rolled from .300 inch to final. The results are shown in Table 6. Comparison of the two heats reveals that each heat responded differently to the variation in rolling direction.

Heat B had its maximum yield strength with 100% long rolled, 100% cross rolled, and 80% long rolled followed by cross rolling. Percent recrystallization was affected by mixed rolling directions, more so in this heat than in the other heat. Grain size was not observed to vary in spite of a 25% variation in strength. Clearly cross rolling with its "texturizing" effect is a definite, but, at present, unpredictable strengthening device. Heat C has its high strengths at 20% and 40% long roll where Heat B had its low strengths. Heat C was most similar in chemical composition to the other heats shown in Table 2 (low W content), and those other heats generally showed weakening in the lower percent long roll categories.

Heat C shows little variation in percent recrystallization or grain size, remarkable again in the face of a 35-40% variation in tensile properties. Hardnesses and ultimate strengths for both heats generally follow the trend of the yield strength.

Little work has been done by investigators in correlating cross rolling with tensile or metallographic properties. The rolling texture of tantalum is considered to be similar to that of other body

Table 6. Variation of mechanical and metallographic properties with percent long roll prior to cross roll to final size (.015 inch tantalum sheet).

Heat	Percent Long Roll Prior to Cross Roll	LYP_T ksi	LYP_L ksi	UTS_T ksi	UTS_L ksi	EL_T %	EL_L %	DPH	% R_x	GS
B	0	--	27.0	--	48.2	--	40	87	90	9
	20	22.7	22.0	41.8	42.7	49	45	84	100	9
	40	24.1	21.9	43.5	43.9	44	48	82	95	9
	60	24.5	22.9	44.5	43.6	34	53	81	95	9
	80	32.5	32.0	51.5	51.3	29	31	100	82	9
	100	28.5	27.1	49.4	48.5	36	40	82	97	9
C	0	21.2	22.6	40.0	42.2	41	50	83	100	7.5
	20	25.1	24.7	43.8	43.6	45	45	84	97	7.5
	40	26.4	24.7	43.5	43.1	45	50	81	100	8
	60	18.0	17.8	36.1	37.3	46	48	76	100	7.5
	80	20.3	20.4	39.6	39.5	41	51	78	100	7.5
	100	20.5	18.3	37.6	37.7	40	54	80	100	7.5

centered cubic metals. Pugh and Hibbard (15) determined the as-rolled texture to be predominantly (112) [011] and the annealed texture to be predominantly (111) [112]. Tantalum rolled in one direction only, and annealed, would thus be characterized by orientation of the (111) planes in the rolling plane and the [112] directions in the rolling direction. Imgram (7) states that mixed long rolling and cross rolling should minimize the directionality characteristics of the product. As stated previously, the data in Tables 2 and 6 does not confirm this.

Effect of Roller Leveling

Roller leveling is a mechanical process for producing flat sheet. Final size sheet is passed through a machine consisting of small diameter opposing but offset rollers which bend the sheet in a washboard effect and then flatten it. By varying the pressure at different points across the width of the sheet, small variations in flatness may be removed and a more usable sheet obtained. The operation does not reduce the thickness of the material, but does impart work due to bending. The average effect of roller leveling as shown in Table 2 was to raise the yield strength 2, 000 to 4, 000 psi. There was no noticeable effect on the grain structure. DPH hardness also appeared to increase slightly with roller leveling.

The data generally shows that roller leveling effects yield strength more than it effects ultimate strength, as indicated by the bracketed pairs of matched roller leveled and un-roller leveled data.

It should be noted that the roller leveling done on the material listed in Table 2 was not closely controlled as to number of passes through the roller leveler or severity of working per pass.

Effect of Rolling Mill

The tantalum sheet in the present investigation was rolled from .300 inch to final size either (1) completely on the large 4-high cold mill, or (2) with a combination of rolling mills: large 4-high to .080 inch, small 4-high to .040 inch, and Z-mill to final size. The data revealed no significant difference in these methods.

It was assumed that since the Z-mill took .003-.004 inch reduction per pass in the .040 range compared with .001 inch per pass for the large 4-high that a difference due to strain rate would be noted. (The Z-mill capability was due to both its small diameter rolls and the use of tension in rolling.) The effect of the Z-mill, if any, was probably small in the region of 85-95% work. The major influence of strain rate likely occurred in the earlier stages of working. A study of the effects of the Z-mill compared to the large cold mill starting with .040 inch annealed material with all rolling being done on either mill would be useful.

Effect of Variation in Primary Fabrication

To investigate the effects of varying the primary fabrication sequence of forging, annealing, and rolling another study was conducted.

Material from Heat C was fabricated according to the following processes:

I. (a) Long forge 7-inch diameter ingot to approximately 2 inches x 6 inches x L

(b) Saw for cross forge

(c) Cross forge to 1 inch x W x L

(d) Condition

(e) Vacuum anneal

(f) Roll on cold mill to .300 inch x W x L

(g) Vacuum anneal

II. Same as I except step (e) was omitted.

III. (a) Long forge to 1 inch x 6 inches x L

(b) Condition

(c) Roll on cold mill to .300 inch x 6 inches x L

(d) Vacuum anneal

IV. Same as III except that vacuum anneal was added between steps (b) and (c).

No significant results were obtained from these variations, as seen for Heat C in Table 2. Houck (5) reinforces this finding by

stating that mechanical properties are most affected by the fabrication sequence since the last recrystallization anneal.

Relationships with Chemical Composition

The significant chemical impurities of the various heats of tantalum investigated in this report are listed in Table 1. A comparison of the results shown in Table 2 with these chemistries follows.

The interstitial content of all heats is quite uniform and low. Most carbon, hydrogen and oxygen values were below the available limits of chemical analysis. As mentioned in the introduction, the values shown are approximately 50% less than those reported in the literature six to ten years ago. Most researchers on tantalum report that interstitials are the most influential property determinants, on an equal parts basis (18). As previously mentioned, the effect of interstitials has been determined as ten times that of tungsten.

The columbium content in this tantalum is several hundred parts. Columbium, however, does not provide measurable solid solution strengthening to tantalum. The tungsten content of the seven heats varies from 12 ppm to 420 ppm, with an average at about 40 ppm.

The strength properties of Heat B, with 420 ppm tungsten, are seen to be approximately 25% higher than those heats having 40 ppm tungsten and less. Thermomechanical processing of Heat B was similar to those of the other heats. Thus, tungsten content is

designated as a major factor in the strength of this high purity tantalum. The effect of tungsten is indirectly manifested in the grain size, which is controlled as to nucleation and growth by this element (10). Hence, the higher time/temperature combination required for full recrystallization, and the smaller resulting grain size.

The strengthening effect of tungsten comes about through grain size limitation and also by solid solution strengthening. Data from an investigation by Schmidt (17) confirms the effect of tungsten as on the order of 1,000 psi increase in strength per 100 ppm tungsten present.

The present study was restricted by the relatively high limits of analysis. Oxygen values were usually reported as less than 50 ppm, and it would have been useful to know if the actual value was 10 parts or 40 parts. Unmeasurable variation in interstitial content was likely a factor in the tensile strength data scatter.

The chemical analyses shown in Table 1 were taken from the ingot, prior to fabrication. Sample product chemical analysis for interstitials was performed early in the study. The results showed product chemistries to be nearly identical to the ingot analysis. Samples taken before and after final anneal were in complete agreement within the limits of analysis.

Data Analysis by Computer

A statistical study of the data in Table 2 was performed to confirm the tabular and graphical results discussed above, and to find other possible trends. The Oregon State University Statistics Instruction Programming System was employed to determine correlation coefficients between all the data categories. The correlation coefficients obtained were interpreted as a relative indication of interdependency of the two variables simultaneously compared. A sample size of approximately 50, as supplied by the Table 2 data, requires only a minimum correlation coefficient of approximately 0.3 to establish statistical significance with a high level of confidence. In this investigation only coefficients greater than 0.5 were noted, and only those with values greater than 0.7 were considered to provide good assurance of dependence.

Table 7 shows the results of the first computation of correlation coefficients. The data for this test consisted of all the data from Table 2, data for heats A and B from Table 4, and the chemical composition as listed in Table 1. The dominance of tungsten content and grain size among all factors is readily seen. Ultimate strength was more prominent than yield strength as a result of the all-inclusive nature of this data. Both roller leveled and non-roller leveled data were included and ultimate strength was more stable than yield strength

with variations in roller leveling.

Table 7. Correlation coefficients for data from Tables 1, 2, and 4 (no limitations).

Correlation Coefficient	Variables
.813	GS vs Tungsten content
.781	UTS_L vs GS
.773	UTS_T vs GS
.756	UTS_L vs Tungsten content
.734	UTS_T vs Tungsten content
.548	LYP_T vs GS
.522	LYP_L vs GS

In the second computer analysis only un-roller leveled data was considered, with the results shown in Table 8. Comparing Tables 7 and 8 it is seen that grain size versus tungsten content again is the most prominent association. The longitudinal strengths versus hardness relationships rank second. Third is ultimate strength versus grain size for which the correlation factor is very similar between the two tables, further illustrating the non-dependency of ultimate strength on roller leveling. Yield strength versus grain size became more significant in Table 8 as compared to Table 7 as the effect of limiting roller leveling is seen.

Table 8. Correlation coefficients for data from Tables 1, 2, and 4 (non-roller leveled only).

Correlation Coefficient	Variables
.921	GS vs Tungsten content
.822	LYP_L vs DPH
.821	UTS_L vs DPH
.781	UTS_T vs GS
.776	UTS_L vs GS
.719	UTS_L vs Tungsten content
.719	UTS_L vs Nitrogen content
.715	UTS_T vs Tungsten content
.700	LYP_T vs DPH
.680	UTS_L vs DPH
.667	GS vs Nitrogen content
.660	LYP_L vs GS
.627	DPH vs GS

IV. CONCLUSIONS AND RECOMMENDATIONS

The strength properties of high purity tantalum sheet have been demonstrated to be primarily dependent on chemical composition. Variations in individual thermomechanical factors investigated here did not significantly increase the strengths in an acceptable manner, acceptable meaning without undesirable changes in other properties. Nonetheless, certain principles of fabrication have been reconfirmed which, when simultaneously considered, should give the optimum strength allowed by the chemical composition of the material. These principles are contained in the following list of recommendations for maintaining optimum strength for tantalum sheet. (1) Maintain smallest possible grain size through presence of maximum allowable effective solid solution strengthening impurities; through use of large percentage reductions prior to final size; and by determining lowest possible annealing time/temperature combination for recrystallization. (2) Roller level annealed sheet, where allowable, for maximum mechanical strengthening with minimum effect on microstructure. (3) Where possible, determine optimum rolling and cross rolling combinations to take advantage of unique textures. (4) Where allowable, use longer time anneals for interstitial strengthening.

The occasional failure of tantalum to meet a 22,000 minimum yield strength must be ascribed to non-uniformity of structure (and

thus of strength) as much as to overall low strength. Data from Table 2 showed the wide variation in properties for material having very similar chemical composition and process history. It was concluded that the variations were in part attributable to the following factors: (a) small variations in the low interstitial content which affected strength significantly and which, in the case of oxygen and carbon, were not specifically known due to the high limits of analysis; (b) uncontrollable differences in thermomechanical processing; (c) variations in the structure of the sheet as carried over from the original electron beam structure; and (d) variations in tensile testing technique.

Additional work should be done to determine specific textures for various combinations of rolling and cross rolling. The reasons for variations in textures for duplicate rolling schedules on similar heats should be identified.

Alteration of chemical composition by adding back strengthening-type impurities to allowed levels should be investigated. Determination of true levels of interstitial impurities should also be undertaken.

Conditions of final annealing, particularly the effects of vacuum, should be investigated in an attempt to reduce scatter in the data.

The possibilities and economics of melting tantalum by vacuum arc rather than by electron beam should also be studied. Arc melting is known to provide a finer, more uniform grain size with some increase in interstitial impurities.

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APPENDIX

APPENDIX I

Heat A

Sample No.	LYP_L	UTS_L	EL_L
1	--	--	--
2	27.5	45.7	43
3	--	--	--
4	--	--	--
5	--	--	--
6	24.3	41.8	50
7	21.3	38.6	57
8	23.5	42.1	55
9	--	--	--
10	24.0	41.3	52
11	--	--	--
12	--	--	--
13	--	--	--
14	25.6	43.2	48
18	--	--	--
19	--	--	--
20	--	--	--

Heat C

Process History	Sample No.	LYP_L	UTS_L	EL_L
I	45	--	--	--
	46	--	--	--
	47	--	--	--
	48	23.6	37.0	38
	49	22.3	37.1	44
	50	--	--	--
	51	25.0	40.9	47
	52	25.4	39.3	46
	53	--	--	--
	56	--	--	--
II	62	--	--	--
	63	--	--	--
	64	--	--	--
	65	--	--	--
III	66	26.8	44.7	39
	68	--	--	--
IV	70	--	--	--
	71	--	--	--
	72	--	--	--
	73	24.8	40.9	44
	74	--	--	--
	75	--	--	--
	76	24.7	39.4	54
	78	--	--	--

Heat D

Sample No.	LYP_L	UTS_L	EL_L
87	--	--	--
88	--	--	--
91	--	--	--
92	24.0	41.3	51
94	20.9	34.9	44
95	22.8	36.1	63
96	--	--	--
Heat E			
101	--	--	--
102	--	--	--
103	--	--	--
106	--	--	--
109	26.3	42.8	54
110	--	--	--
114	--	--	--
Heat F			
122	--	--	--
123	20.9	37.2	49
124	23.1	37.3	40
126	--	--	--
127	--	--	--
128	25.2	35.6	50