THE EFFECT OF INTERSTITIAL ELEMENTS ON THE MECHANICAL PROPERTIES OF TANTALUM AT LOW TEMPERATURES

by

VICTOR BRUCE LAWSON

A THESIS SUBMITTED IN PARTIAL FULFILMENT
OF THE REQUIREMENTS FOR THE DEGREE OF
MASTER OF APPLIED SCIENCE
in the Department of
MINING AND METALLURGY

We accept this thesis as conforming to the standard required from candidates for the degree of MASTER OF APPLIED SCIENCE

Members of the Department of
Mining and Metallurgy

THE UNIVERSITY OF BRITISH COLUMBIA

April, 1961

In presenting this thesis in partial fulfilment of the requirements for an advanced degree at the University of British Columbia, I agree that the Library shall make it freely available for reference and study. I further agree that permission for extensive copying of this thesis for scholarly purposes may be granted by the Head of my Department or by his representatives. It is understood that copying or publication of this thesis for financial gain shall not be allowed without my written permission.

Department of Mining and Metallurgy

The University of British Columbia,
Vancouver 8, Canada.

Date March 28/61

ABSTRACT

An investigation of the low temperature tensile properties of tantalum and of tantalum containing added interstitials was carried out.

Added nitrogen, oxygen, and hydrogen were found to increase the level of the yield point stress relative to that of the starting material. The elongation of material with added nitrogen and oxygen exhibited less elongation at room temperature than did the pure material, but at —196°C it showed more elongation. Hydrogen embrittlement was observed in the material with added hydrogen.

It was demonstrated that the tensile properties of tantalum were sensitive to strain rate. A transition from a ductile-to-less ductile type of behaviour was found on decreasing the temperature. The transition temperature also was found to be strain rate sensitive.

ACKNOWLEDGEMENTS

The author gratefully acknowledges the supervision and encouragement of Dr. J.A. Lund and Professor W.M. Armstrong and wishes to thank Mr. R.G. Butters, Mr. Wm. Cross, and Mr. R. Richter for their technical advice and assistance during this investigation.

The author is also indebted to Dr. E. Teghtsoonian and fellow graduate students for helpful discussions.

The work was financed by Research Grant No. 7510-36 provided by the Defence Research Board of Canada.

TABLE OF CONTENTS

FIGURES

FIGURES

TABLES

TABLES

THE EFFECT OF INTERSTITIAL ELEMENTS ON THE MECHANICAL PROPERTIES OF TANTALUM AT LOW TEMPERATURES

I INTRODUCTION

A. GENERAL

The increasing demand for materials having special high-temperature properties has led to considerable interest in refractory metals and to intensive research on their mechanical properties. This loosely defined group, the refractory metals, is comprised of those metals with high melting points in Groups IV, V, and VI of the Periodic Table; specifically the following:

Metal	Melting Point
Niobium	2415°C
Molybdenum	2650°C
Tantalum	3000°C
Tungsten	3400°C

Since these metals are all body-centered-cubic transition metals, their mechanical properties exhibit those basic characteristics which distinguish them from the face-centered-cubic metals. With decreasing temperature, for example, the tensile strength of the body-centered-cubic metals increases rapidly while the ductility exhibits a transition from a ductile-to-brittle, or from ductile-to-less ductile type of behaviour. It appears that molybdenum and tungsten exhibit the transition from ductile-to-brittle behaviour, but that vanadium and niobium exhibit a ductile-to-less ductile transition. Another characteristic of this refractory group, which face-

centered-cubic metals do not exhibit, is the strong influence of interstitially dissolved elements upon their mechanical properties. This influence is shown in the increase of tensile strength and in the decrease of ductility with increasing interstitial content.

Before these refractory metals may be used efficiently it is necessary to examine and to understand fully their tensile behaviour, but to do this, much more data is required. In this research project the results of a series of tensile tests were used to examine the mechanical properties of tantalum over a temperature range of 100°C to —196°C. Particular attention was given to the effect of nitrogen, oxygen, and hydrogen on these properties.

Although it has the advantages of a high melting point, a low vapour pressure, excellent ductility, and good high-temperature strength, tantalum, like the other refractory metals, has the disadvantage of being extremely reactive at high temperatures. It will combine readily with oxygen, hydrogen, nitrogen, and the halogens in both the solid and the molten states. Annealing must be carried out, therefore, in either a high vacuum or an atmosphere of a purified inert gas.

B. THEORY

The phenomena of strong temperature dependence of the tensile properties, the strain rate sensitivity of the tensile properties, and the transition from a ductile-to-brittle, or from ductile-to-less ductile type of fracture in body-centered-cubic metals have become an active area for research in themselves.

Wessel[1] has given an excellent summary of the thinking up to 1956, on the mechanisms presently used to explain these phenomena. A 1959 publication edited by Overbach, Felbeck, Hahn, and Thomas[2] covers the field in more detail with a theoretical treatment.

1. Ductile-to-Brittle Transition

The generally recognized concept of the ductile-to-brittle transition states that brittle fractures will occur when the yield-point stress exceeds the cleavage stress. This condition appears to be reached by lowering the temperature, or by increasing the strain rate, or by introducing a triaxial stress condition with the use of a notch. Notches cause brittle failure because they increase the ratio of normal to shear stress. The normal stress is increased to a value greater than the cohesive strength of the material, resulting in a brittle fracture.

Wessel's[1] description of the ductile-to-brittle transition is summarized herewith: The increased resistance to initial plastic flow with decreasing temperature, exhibited by the strong temperature dependence of yield point stress, has been explained in terms of the anchoring of dislocations by atmospheres of impurities. This theory was developed by Cottrell and Bilby[3] and later elaborated by Fisher[4]. The Cottrell-Bilby Theory, as it is known, is premised on the migration of solute atoms to the stress field associated with a dislocation, where they relieve some of the stress, thereby lowering the energy of the system. The solute atoms form an atmosphere around the dislocations and exert an anchoring force proportional to the energy decrease of the system, which occurred upon migration of the solute atoms to the

stress field.

Cottrell and Bilby[3] postulated that minor thermal fluctuations add sufficient energy to the applied stress to effect the breaking away of a small dislocation loop, which then drags the remaining part of the dislocation from its atmosphere. The minimum amount of energy required for this process of nucleation was estimated by Cottrell and Bilby[3] and found to be within reasonable limits.

Cottrell[3,5] originally explained the breaking away of the first dislocation from its atmosphere as the beginning of the abrupt yield phenomenon. During subsequent nucleation and extension of one or more loops, other dislocations break away resulting in a catastrophic yielding. Since thermal fluctuations decrease with decreasing temperature, a greater stress is required to tear the dislocation from its atmosphere at low temperatures than at high temperatures; hence, the temperature dependence of yield point stress. This theory requires no pre-yield strain except that which results from the first few dislocation loops' breaking away from their atmospheres; but Clark[6] and his associates have given experimental evidence indicating the existence of considerable pre-yield strain.

Clark's[6] observations can be accounted for if there are a large number of dislocations moving through the lattice prior to abrupt yielding. An estimate of Cottrell's[5] indicates that approximately 1000 piled-up dislocations per grain are required in the case of mild steel tested at $-196^{\circ}C$. He describes the dislocations as being "piled up" because he thinks that, after release, the dislocations move through the lattice until they pile up at barriers which are in the form of grain boundaries,

sub-grain boundaries, inclusions, etc. It is probable that some dislocations start to break away at a stress in the vicinity of the elastic limit, and as the stress increases the number of dislocations breaking away increases, reaching a maximum at the yield point stress.

As a result of the piling up of dislocations at barriers and the increasing stress level, high stress concentrations are developed in regions in the vicinity of the pile-ups. These stresses activate dislocation sources in the neighbourhood of the pile-ups. As a result the stress in the region of the original pile-ups is relaxed. Reaching the critical stress necessary to activate nearby dislocation sources and relaxing the stress is termed the "breaking through" or "breaking away" of the piled-up dislocation group from its barrier. These phenomena cause higher microstresses in neighbouring pile-ups, triggering a cataclysmic yielding and an associated drop in load.

At low temperatures, where brittle fracture is observed to occur during yielding, the mechanism is thought to be different. Here the stress necessary to move a dislocation or to activate a dislocation source is greatly increased, creating higher localized microstresses. When these stresses exceed the material's cohesive strength, microcracks may form if the material is not favourably oriented for slip. The formation of microcracks and plastic flow may, therefore, occur simultaneously. The initiation of one or more microcracks, together with some plastic flow, is assumed to trigger the abrupt yielding. During the associated drop in load the microcracks grow in number and in size to propogate and terminate in brittle fracture. Griffith[7] in his criterion states that they must reach a critical size

before they propagate to cause brittle failure.

2. Temperature Dependence of Yield Stress.

Researchers have expended much time and effort in attempts to derive a theory to express the temperature dependence of yield strength of body-centered-cubic metals. Zener and Holloman[8] developed an emperical relationship to fit their results on steel which Bechtold[9] used to fit his data on tungsten. It states that yield stress, which is taken as an offset stress, is proportional to the exponential (1/T). Plotting the log of the yield stress versus the reciprocal of absolute temperature should result in a straight line. Zener and Holloman have associated with this equation an activation energy, which may be determined from the plot. To what process this activation energy refers is of much contention. In their work Zener and Holloman arrived at a value for steel of 90 to 120 calories per gram mole. It should be noted that this relationship was meant to apply only to offset yield stresses, not to upper yield point stresses. There are, however, some cases[3,5] in which upper yield point data does appear to support the relationship.

More recently Fisher[4] derived a relationship, by elaborating on the Cottrell-Bilby Theory[3]. He suggests that the product of yield point stress and the absolute temperature is a constant; i.e. on plotting yield stress versus the reciprocal of the absolute temperature, a straight line should result if the theory is valid.

This relationship was derived by expressing the rate of nucleation of dislocation loops (described in the Cottrell-Bilby Theory[3]) as being proportional to exponent $(-W^*/kT)$; where W^* is

the minimum work required to form a dislocation loop of critical size for break-away; k is Boltzman's constant; and T is the absolute temperature. The rate is essentially zero until the stress is slightly below the yield point stress, and then increases rapidly as it approaches this point. It is assumed that, with a constant rate of straining at any given temperature, the rate of nucleation will be constant at the yield point stress. Therefore, exponent $(-W^*/kT)$, the rate of nucleation, is a constant. By substituting for W^* and simplifying, Fisher[4] arrived at the equation, ST/G^2 = constant; where S is the yield point stress, T, the absolute temperature; and G, the shear modulus. The validity of this theory has been established to some extent by data on iron[4], molybdenum[4], and vanadium[10]. The relationship does not appear to hold below approximately 140°K. It is thought that below this temperature nucleation of the dislocation loop is no longer required for yielding, as at these temperatures the stress has reached a level high enough to tear the dislocation away from its atmosphere.

II PREVIOUS WORK ON TANTALUM

Investigations by Bechtold[11], Pugh[12], Schussler[13], and Ingram[14] on the mechanical properties of tantalum in the low temperature range of 100°C to —250°C revealed no ductile-to-brittle transition. The yield point stress and ultimate stress were found to increase continuously over the temperature range 100°C to —196°C, and the ductility did not appear to go through any sort of abrupt transition to less ductility on decreasing the temperature. Barrett[15] reports that Basinski found that even at 4.2°K tantalum still exhibits some ductility. Schmidt[18] reports that Magnusson and Baldwin observed a slight embrittlement of annealed tantalum at low temperatures. However, a true transition was not apparent and the embrittlement was attributed to hydrogen.

The room temperature data for annealed tantalum is given in Table I. Exceptional room temperature properties are evident from the reduction in area and per cent elongation values given. The variation in tensile values appears to be due to variations in purity of the material. Table II, page 10, shows the analyses of the materials used by some of the investigators. Because of the variance in the interstitial content and other variables such as grain size, a comparison of the properties is very difficult to make.

The sensitivity of tensile properties and ductility to increases in strain rate appears to be a matter of contention. Kattus'[16] results show a significant decrease in ductility for a corresponding change of 2000:1 in strain rate, while Michael's[17] data indicates little if any effect.

TABLE I

ROOM TEMPERATURE DATA FOR ANNEALED TANTALUM

Investigation	Strain Rate in/in/min.	Yield Stress .2% Offset	Proportional Limit psi.	Ult. Stress x 10^3	Percent Elongation	Per Cent Reduction in Area	Grain Diameter mm.
Bechtold[11]	.018	-	39,300	49,800	45	86	.047
Pugh[12]	5.4	57,350	-	67,140	25.3	-	.088
Kattus[16]	.003	32,700	-	44,600	46.3	-	-
	5.4	54,100	-	61,800	34.2	-	-
Michael[17]	.018	-	42,000	58,000	38.0	84	-
	1.2	-	57,000	67,000	38.0	82	-
Schussler[13]							
Arc-	.005	-	37,000	53,800	-	80	.025
Melted	.005	-	27,800	51,100	-	92	.10
Electron-	.005	-	11,900	30,200	-	95	.2
Beam Melted	.005	-	9,000	28,800	-	99	.5
Gebhardt[19]	-	-	-	27,500	38.0	89	-
Schmidt[20]	-	26,300	-	29,400	36.0	-	-
Yancey[21]	-	30-40,000	-	40-50,000	30-40	-	-
Fansteel[22]	-	-	-	50,000	40.0	-	-
Fansteel[23]	-	-	-	100,000	11.0	-	-
Ingram[14]	-	-	-	35,000	-	95	-
Myres[24]	-	-	-	33,500	50.0	-	-

TABLE II

CHEMICAL ANALYSES OF TANTALUM

Concentration parts per million

Element	Schussler[13] Arc Melted	Schussler[13] Electron - beam Melted	Bechtold[11] Fansteel Vacuum Sintered	Pugh[12] Fansteel Vacuum Sintered
C	150	50	100	200
O_2	280	60	-	56
H_2	10	1	-	-
N_2	60	60	100	130
B^2	1	1	-	-
Cb	100	100	-	1000
Cr	100	100	-	-
Cu	10	10	-	-
Fe	100	100	-	150
Ni	100	100	-	-
Si	50	50	-	-
Ti	100	100	-	-
W	56	56	-	100
V	30	30	-	-
Zr	30	30	-	-

Heat Treatment of above Materials

Annealing Temp.	1304°C*	1350°C*	1190°C†	1304°C†	1700°C✱	- *
Time	1 hour	1 hour	1 hour	1 hour	1 hour	1 hour
Vacuum	-	-	-	-	5×10^{-5} mm. of Hg	-
Grain Size	.025 mm.	.10 mm.	.2 mm.	.5 mm.	.047 mm.	.088 mm.
ASTM	8	4	2	0	5-6	6-7
Material	swaged bar	swaged bar	swaged bar	swaged bar	swaged bar	sheet
Texture	-	-	-	-	-	111 112

* Schussler[13] Arc-Melted Material

† Schussler[13] Electron-beam Melted Material

✱ Bechtold[11] Fansteel Vacuum-Sintered Material

* Pugh[12] Fansteel Vacuum-Sintered Material

Some data on the temperature dependence of the modulus of elasticity have been gathered, but they do not extend below room temperature.

The effects of temperature on the ultimate tensile strength, yield point stress, and ductility are shown in Figures 1 and 2. The results of Bechtold[11], Pugh[12], Kattus[16], and the results for Schussler's[13] arc-melted material agree quite well. The curve for Bechtold's data lies slightly below that of Pugh, but Pugh's data is for 0.2 per cent offset stress, whereas Bechtold's data is for the proportional limit. Under these curves lie the curves for both the arc-melted and the electron-beam melted material, the latter being considerably more pure. The only apparent difference between these materials is their relative interstitial contents, which appear to govern the relative positions of the different materials on the yield point stress axis.

Above 100°C an anomalous increase in ultimate stress appeared in the data of Pugh[12], Schussler[13], and Bechtold[11]. Pugh attributed the increase to strain aging effects.

The percent elongation data shown in Figure 1 gives more evidence of the effect of interstitial content on mechanical properties, the purest material showing the highest room temperature elongation, and the least pure exhibiting the lowest. There are two peaks in Bechtold's[11] data which also appear in Pugh's[12], but at a higher temperature. Pugh ascribes the peaks to interactions between dislocations and different species of interstitials.

The reduction-in-area data given in Figure 2 indicates very strongly that a ductile-to-brittle transition does not exist

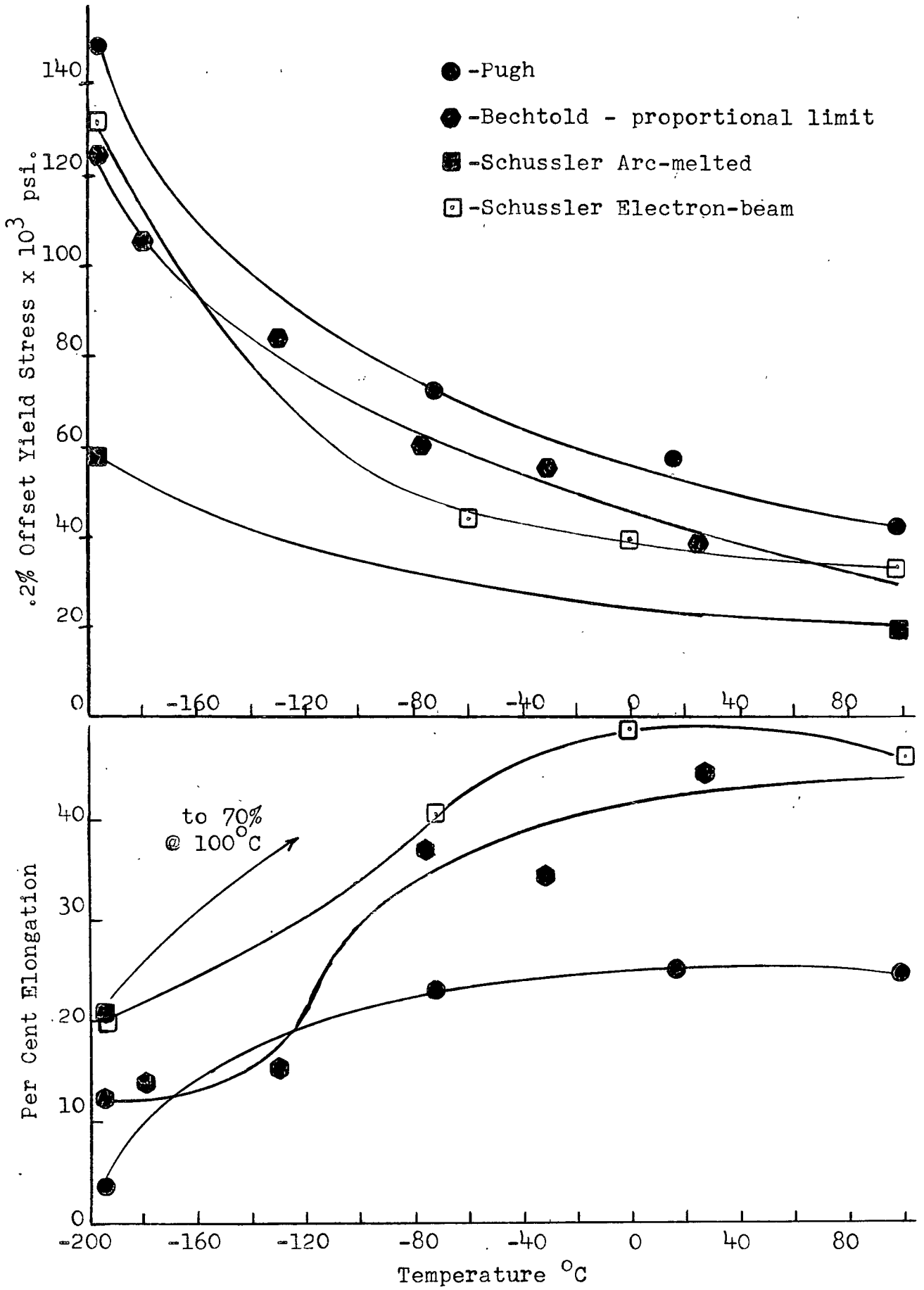

Figure 1. Yield Stress and Elongation of Tantalum

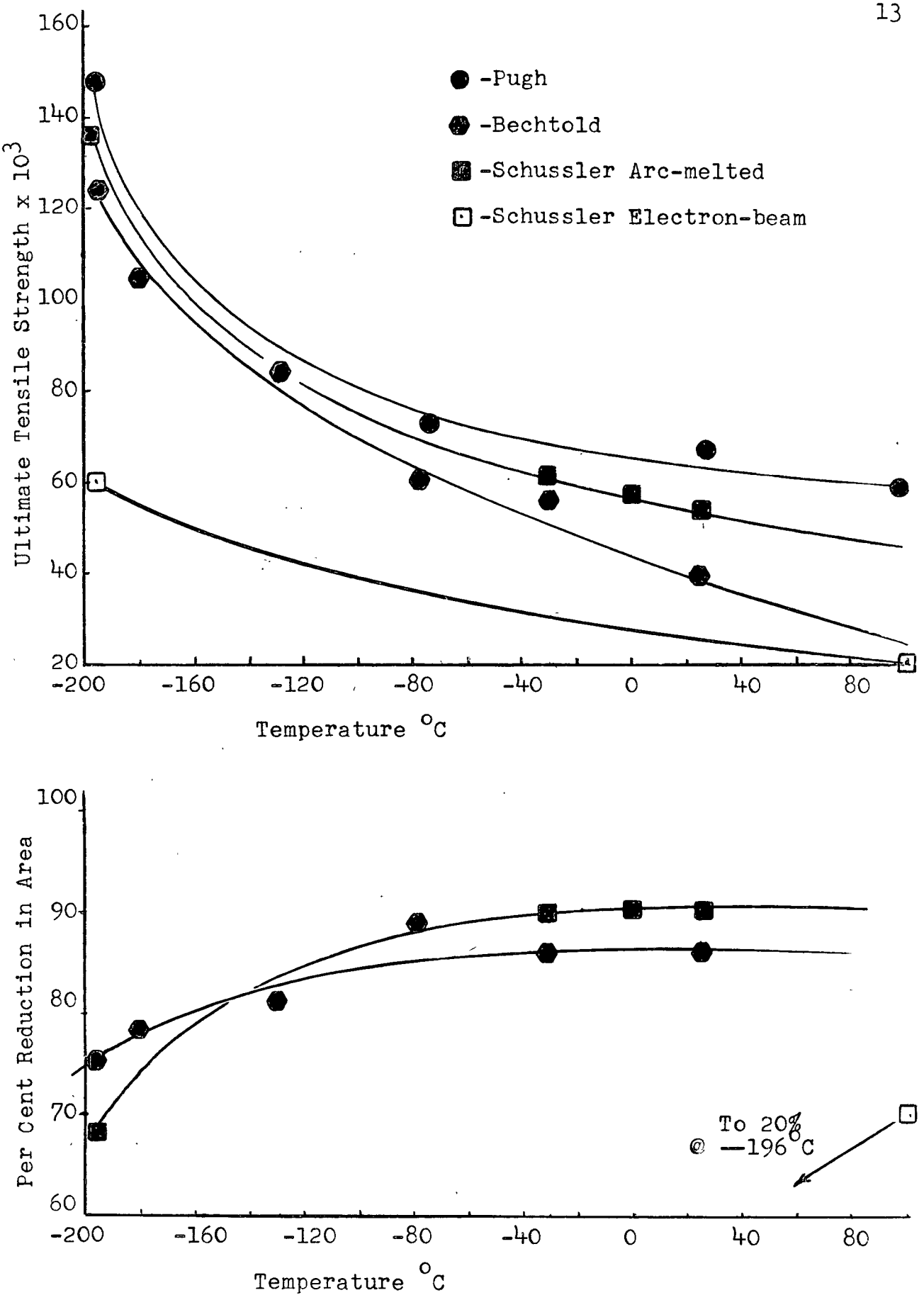

Figure 2. Ultimate Stress and Reduction in Area

in the temperature range covered, and that there is very little effect of strain rate on this property.

There is very little information available regarding the effect of absorbed gases on the mechanical properties of tantalum. It appears that the greatest portion of existing data deals with the variation of hardness with gas content, the most recent data being that of Perkin's[25] group of investigators. Table III[26] shows the effect of various gases on the room temperature elongation of tantalum.

TABLE III

EFFECT OF VARIOUS GASES ON TANTALUM AT ELEVATED TEMPERATURES

Gas	Temp. °C	Duration of Test - Hours	Gain in Weight Percent	Room Temperature Elongation Percent in 1 in.
Hydrogen				31 (original)
	100	2	0.000	31
	200	5	0.000	33
	250	3	0.000	32.8
	300	5	0.000	32.8
	350	5	0.0073	30.6
	400	1	0.011	25.8
	400	2	0.018	-
	400	3	0.031	16.0
				some embrittlement
Nitrogen				31 (original)
	300	5	0.0000	28.2
	400	1	0.0053	22.0
Air				33.2 (original)
	350	5	0.013	27.1
Oxygen				33.2 (original)
	350	5	0.011	26.6

A. THE EFFECT OF HYDROGEN

Miller[27] reports that the hardness of tantalum increases progressively and that the strength and ductility decrease with increasing additions of hydrogen. He reports, however, that the data are not available. Figure 3 (after Perkin's[25]) gives the increase in hardness with increasing hydrogen content. The curve shows a steady rise up to a Diamond Hardness Number of 170 corresponding to a hydrogen content of 16 atomic per cent. Bakish[30] reports that hydrogen in tantalum leads to a brittle type of fracture on bending at room temperature. Seghezzi[31] found that the presence of hydrogen affected the stress-strain curve and mechanical properties in a manner similar to that shown in Figure 4, page 17.

B. THE EFFECT OF NITROGEN

Seghezzi[31] studied the reaction of nitrogen with tantalum. Figure 4, page 17, shows the variation of hardness, elongation, and tensile strength with increasing nitrogen content. Figure 5, page 17, shows the shape of the tensile curves that Seghezzi obtained. He reported finding a very small yield point for degassed material while those of low nitrogen content showed a distinct upper and lower yield point with a well-defined plastic region. The samples with high nitrogen content were found to exhibit a falling-away of the load after yielding, until the specimen broke in a brittle manner.

Perkins[25] also showed that nitrogen increased the hardness of tantalum but at a much faster rate than hydrogen (see Figure 3).

Bakish[30] found that a high nitrogen content embrittled

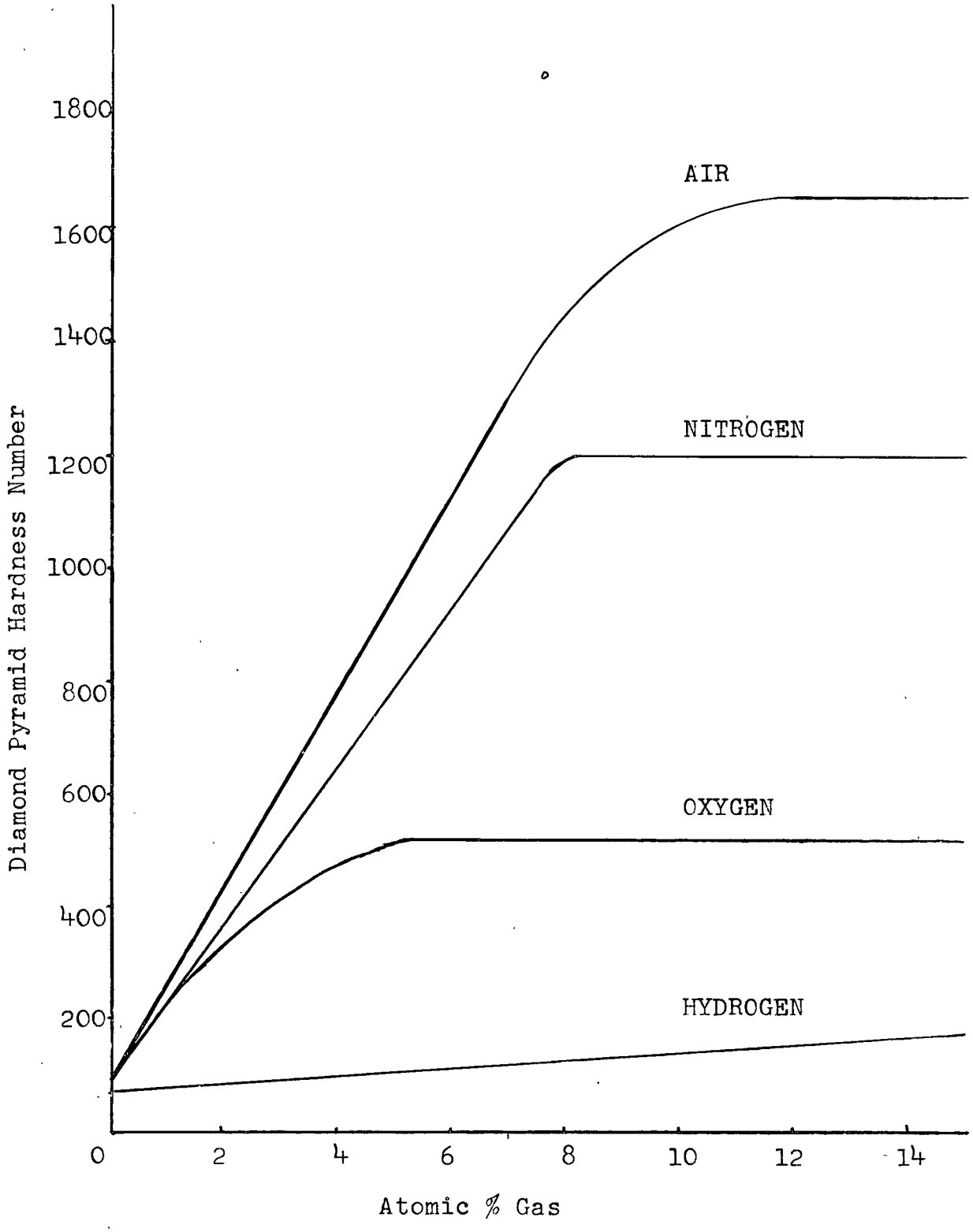

Figure 3. Hardening of Tantalum by Gases.

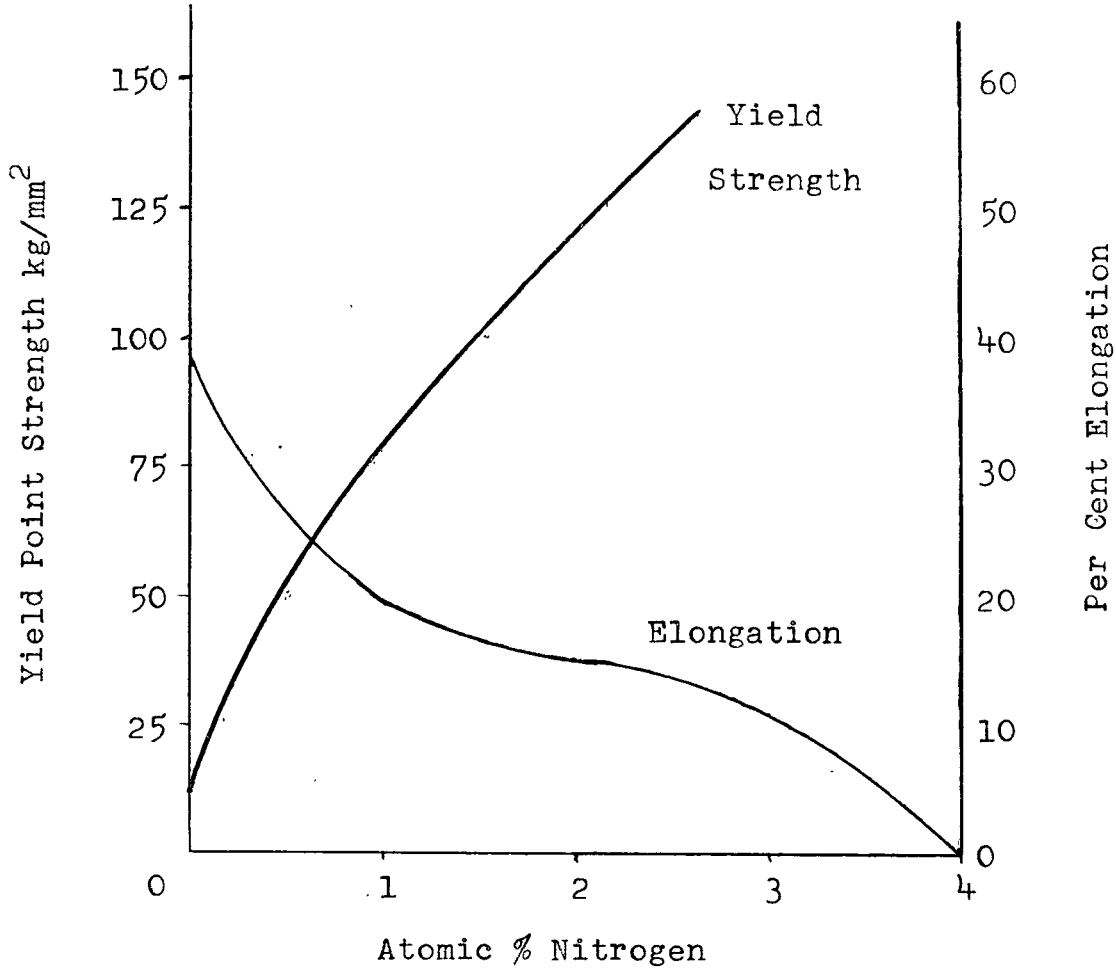

Figure 4. Mechanical Properties of Tantalum containing Nitrogen

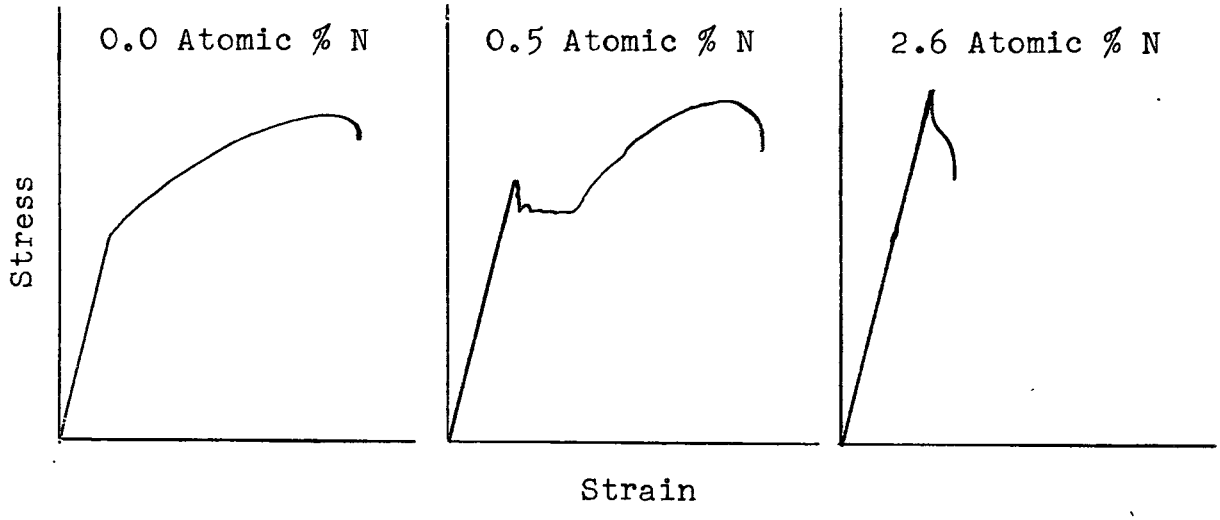

Figure 5. Effect of Nitrogen on the Stress-Strain Curves of Tantalum.

tantalum and that the embrittlement can be identified with the precipitation of a second phase, TaN, on the {100} plane.

C. THE EFFECT OF OXYGEN

Miller[28] reported that tensile strength, elongation, and reduction in area of tantalum all vary with oxygen content as shown in Figure 6 (after Gebhardt and Seghezzi). The modulus of elasticity, shown in Figure 7 was also reported[29] to be affected by oxygen content.

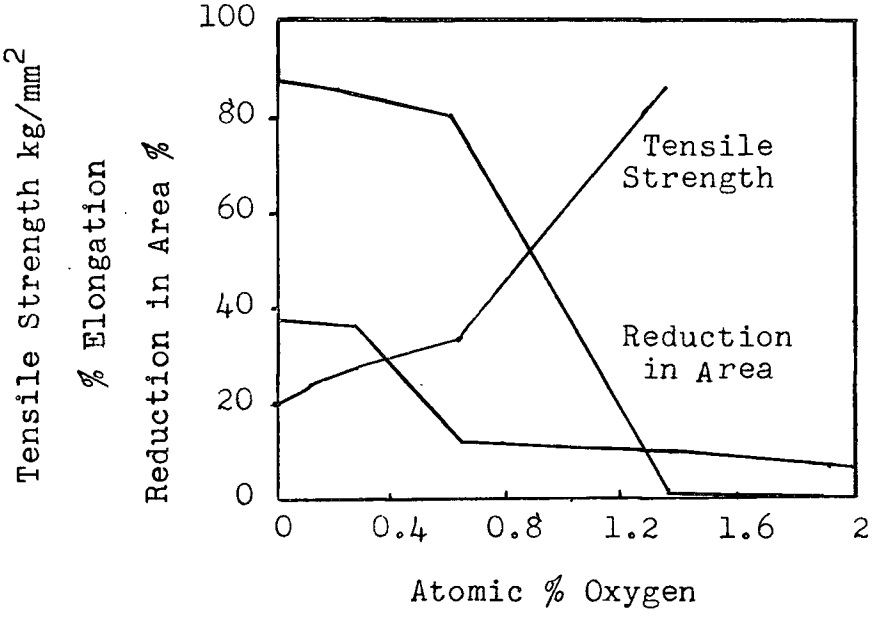

Figure 6. Dependence of Mechanical Properties of Tantalum on the Oxygen Content.

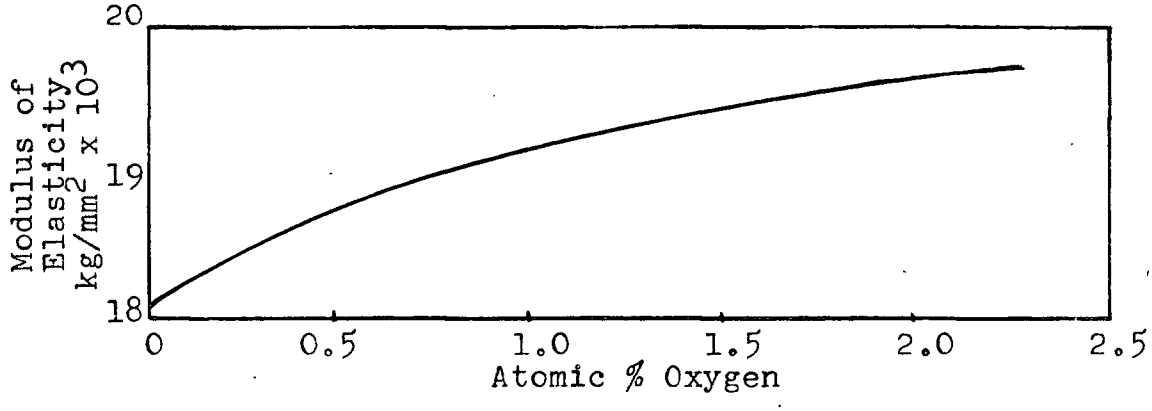

Figure 7. Dependence of the Elastic Modulus on the Oxygen Content.

Gebhardt[32] found that hardness increased with oxygen content. His results agree quite well with those of Perkins[25], whose results are shown in Figure 3, page 16.

Bakish[30] reported that oxygen additions above the solubility limit will cause tantalum to become very brittle.

D. THE EFFECT OF AIR

Perkins[25] observed that the effect of air additions on the hardness was similar to the effects caused by additions of oxygen and nitrogen in an additive manner. This phenomenon is shown in Figure 3, page 16.

III EXPERIMENTAL

A. MATERIALS

The tantalum used in this investigation was supplied by Murex Limited of Rainham, England and by the Wah Chang Corporation of Albany, Oregon, two lots of material being supplied by each company. The analysis of each is shown in Table IV. The material supplied by Murex, designated in this thesis as lot 1M and lot 2M, was vacuum sintered. The electron-beam melted material received from Wah Chang is designated as lot 1WC and lot 2WC.

B. SPECIMEN PREPARATION

1. Lot 1M

Lot 1M was supplied in the form of an annealed strip 1 inch x 12 inches x .060 inches in size. This strip was cold rolled in a number of steps to a thickness of .015 inches then cut into strips 0.5 inches x 2.25 inches. The strip was rolled in the direction of the 12 inch measurement and the 2.25 inch dimension was taken in the direction of rolling. The strips were then chemically polished for approximately five seconds in a solution of five parts sulphuric acid, two parts nitric acid, and two parts hydrofluoric acid. The procedure removed all scratches, the oxide layer, and any dirt that might have caused contamination during the annealing operation that followed. A series of hardness measurements were taken before and after the chemical polishing to see if there was any hydrogen absorption. It was found that none took place, because the hardness measurements remained consistent. A subsequent analysis supported this fact. All hardness measurements taken in this investigation were made

TABLE IV

CHEMICAL ANALYSIS OF TANTALUM

Concentrations(parts per million)

Element	Lot 1M*	Lot 1M✱	Lot 2M*	Original Ingot of Lot 1WC✱ Lot 2WC	Lot 1WC†	Lot 1WC✱	Lot 2WC†	Lot 2WC✱
C	50	31	177	<30	47	110	35	30
O_2	20	282	487	<50	120	< 50	170	110
N_2	100	162	84	20	17	65	30	75
H_2	2	11	3.8	2	1.6	2.6	2.2	3.0
Al				<20				
B				< 1				
Cb				<500				
Cd	<50			< 5				
Cr				<20				
Cu			20*	<40				
Fe	350		150*	<100				
K	<20							
Mg				<20				
Mn				<20				
Mo	1800		100*	<20				
Na	<20							
Ni	<100		50*	<20				
Pb				<20				
Sn				<20				
Si			100*	<100				
Ti	100		50*	<150				
V				<20				
W	700		<100*	<300				
Zn				<20				
Zr				<500				

* - Analysis done by Murex Limited

✱ - Analysis done by The Wah Chang Corporation

† - Analysis done by Ledoux and Company

with a Tukon Microhardness Tester using a 136° diamond indentor.

These strips were then annealed in a zone refiner described elsewhere[33]. For this operation a tantalum specimen holder was designed to hold twenty-five to thirty specimens within the loop of a long filament as shown in Figure 8. Molybdenum wire was used to make extensions for the filament so as to place it adjacent only to the holder. A platinum, 10% rhodium/platinum, thermocouple was inserted into the stack of specimens to allow for temperature measurements. This thermocouple was brought out through soldered connections in the top of the zone refiner and millivolts were read on a Pye potentiometer.

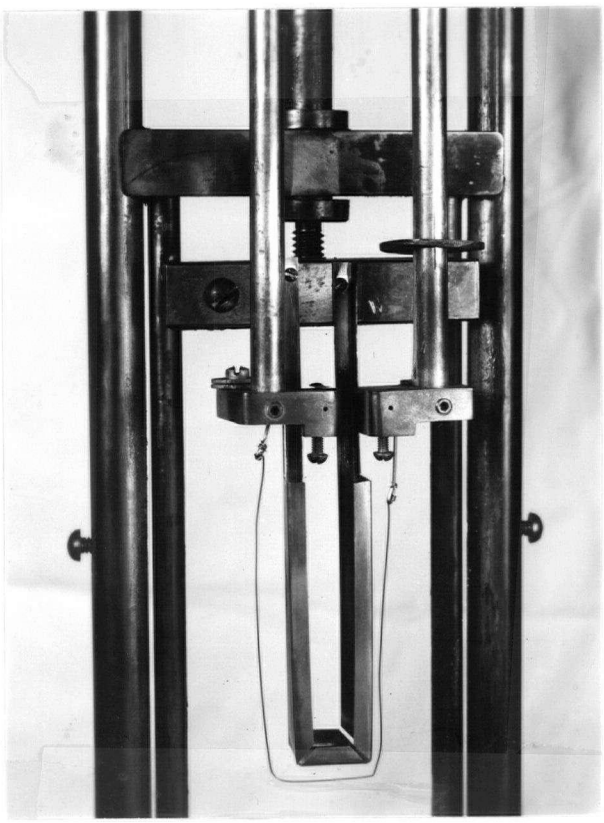

Figure 8 Specimen Holder and Filament in Zone Refiner.

Annealing was conducted in seven different batches at approximately 1350°C in a vacuum of less than 5 x 10^{-5} mm. of Hg for one hour. It was very difficult to maintain a constant temperature for this length of time and to reproduce the same conditions for each of the batches. The average recrystallized grain size that resulted varied from 30.4 to 49.3 grains per mm. (ASTM 7 to 8), and the average Diamond Pyramid Hardness Number varied from 133.3 to 144.5. The range of grain size was 26 to 56 grains per mm. (ASTM 7 to 9), and the range of hardness was 121 to 158. This large range is thought to be due to a heat gradient between the center and the outside of the stack of specimens in the holder, and, to the close stacking of the strips, which may have decreased the efficiency of degassing.

A number of strips from this lot were then cold rolled further to give reductions in area of 10%, 25%, 40%, and 50%. These strips were then cut to specimen shape, the dimensions of which are shown in Figure 9, and a hardened steel jig was used to shape the specimens. The specimens were then polished mechanically with various grades of emery paper down to 000 grade. This procedure was followed by chemical polishing as described earlier.

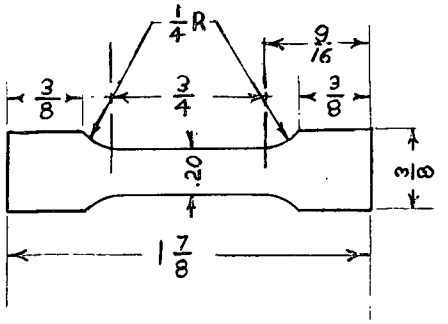

Figure 9. Dimensions of the Tensile Specimen

Aging was then conducted at temperatures of 700°C, 400°C, and 300°C for one hour in a vacuum-annealing furnace described elsewhere[34].

The remaining annealed strips were shaped to specimen size by first milling to approximate size, then by filing in the hardened steel jig, and finally by polishing as described earlier. A hardness traverse was taken to check that all effects of shaping the specimens were removed by finishing and polishing. All such effects had been removed.

2. Lot 2M

Lot 2M came in the same form as lot 1M and was also rolled down to .015 inches thick and cut into strips as before. For this lot a punch and die (see Figure 10) was employed to obtain a uniform specimen. The specimens were chemically polished as before.

As lot 2M was more pure than lot 1M (see Table IV, page 21), the vacuum-annealing furnace mentioned earlier was employed. Three specimens were laid in a molybdenum boat and inserted in the furnace tube, and annealing was conducted at 1100°C in a vacuum of less than 1×10^{-5} mm. of Hg for twenty-four hours. When a hardness traverse was made it was found that annealing had taken place only on the side away from the molybdenum boat, while the side facing the boat was still in the work hardened condition. It was thought that annealing failed to take place throughout the sample because a temperature gradient existed from one side of the specimen to the other.

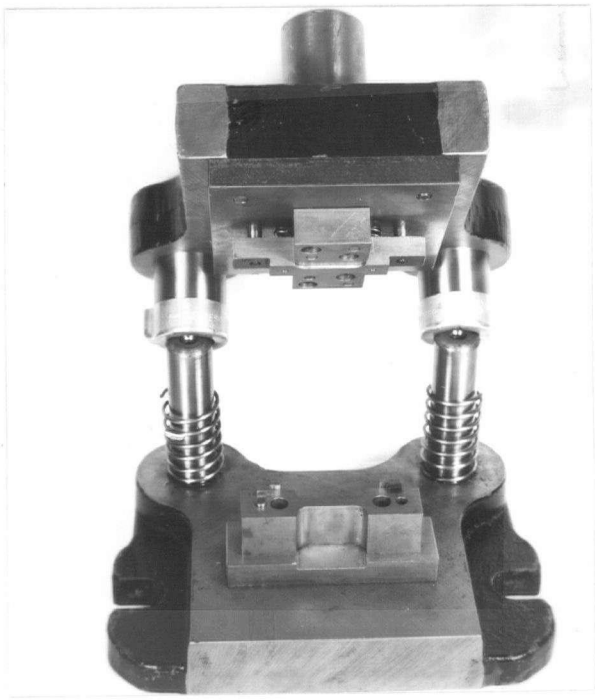

Figure 10. The Specimen Punch and Die

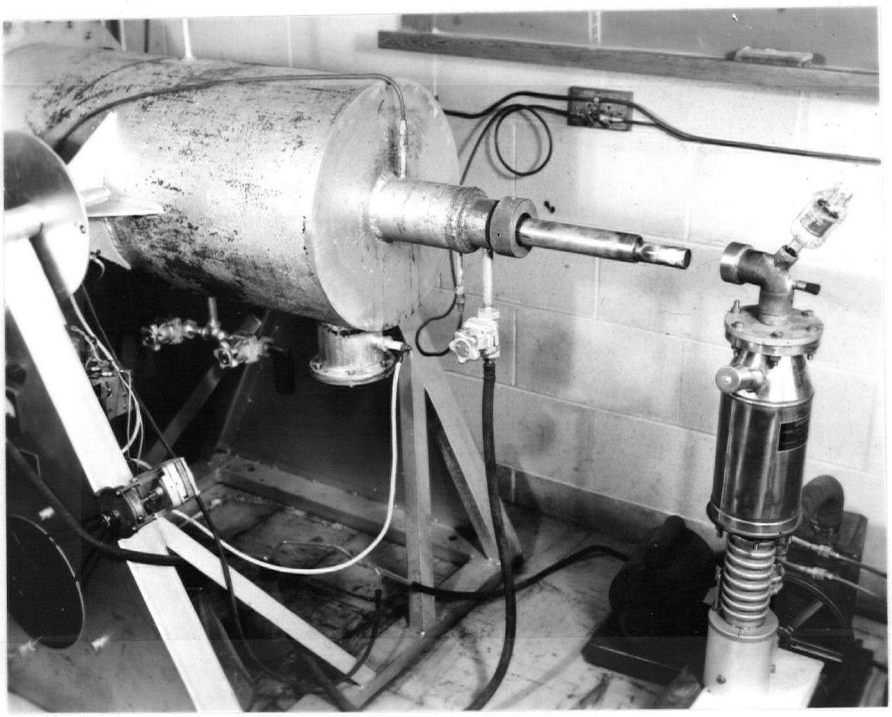

Figure 11. Modified Sintering Furnace showing Vacuum System

Because of this effect and the small capacity of the furnace, this method of annealing was abandoned. Instead a sintering furnace manufactured by Pereny Equipment Company was modified as shown in Figure 11. A four foot piece of 1 5/8 inch O. D. 316 stainless steel water pipe with one end welded closed was inserted into the tube of the furnace with the open end protruding through a rubber bung in the opening of the furnace. To the open end was fitted an Edward's mercury diffusion pump. The seal between the pipe and the elbow was made by rubber "O" rings. This end of the pipe was kept cool by a water jacket. The temperature was measured by a tungsten, 26% rhenium/tungsten, thermocouple and the voltage was read on a Pye potentiometer. The molybdenum windings of the furnace were protected by a continuous flow of hydrogen. Because, above 1000°C, it was found that the hydrogen diffused rapidly through the stainless steel pipe, the atmosphere in the furnace was changed to argon. With argon, a vacuum of less than 5×10^{-4} was obtained above 1000°C, whereas previously at that temperature a vacuum of only approximately 9×10^{-3} was possible.

A trial run in this modified annealing furnace revealed that the vapour pressure of the stainless steel at 1300°C was too high, resulting in a coating (containing considerable amounts of nickel, chromium, and iron) on the tantalum specimens. In an effort to combat this problem a vitreosil tube was sealed at one end and placed in the stainless steel tube, so that the open end of the tube was situated in the cooled section of the furnace. A trial run using this arrangement appeared to be successful, even though annealing had not taken place. This failure to anneal was thought to be due to insufficient temperature. The

entire batch was then put in molybdenum boats, stacking them so as to leave a space between the gauge length of each individual specimen. The boats were put into tubes in the furnace and annealing was carried out at 1350°C for forty hours in a vacuum of 4×10^{-4} mm. of Hg. During this time the specimens became contaminated. The material had recrystallized, but the hardness was high, the average Diamond Pyramid Hardness Number being 220. The average number of grains per mm. was 55.5 (ASTM 9). A spectographic analysis conducted in Vancouver by the firm, Coast Testing, did not detect the presence of silicon. On examining thermochemical data the reactions $2SiO_2 \rightarrow 2SiO + O_2$ and $2SiO \rightarrow 2Si + O_2$ were found to be possible. An analysis done by Ledoux and Company of Teaneck, New Jersey revealed a high oxygen content. (see Table V, page 31)

The contaminated specimens were chemically polished to remove the layer of oxide. Then a hardness traverse was done on the cross section of a mounted specimen to check for any gradients of oxygen. There was no evidence of a gradient.

3. Lots 1WC and 2WC

The material from the Wah Chang Corporation came in the form of a sheet 10 inches x 29 inches x .015 inches. From this sheet, 100 specimens were prepared by using the punch and die as was done for lot 2M. This material was ordered as an annealed sheet, but it was soon discovered that it was still in the work hardened condition, so the cut specimens and the remaining part of sheet were returned for annealing.

It was found that after annealing, the cut specimens had a lower Diamond Pyramid Hardness Number (82) than the

remaining part of the sheet (104.5). The differences in grain size account to some extent for the differences in hardness as the cut specimens had on average of 20 grains per mm. (ASTM 6), while the sheet had an average of 16 grains per mm. (ASTM 5). The difference in interstitial content of the two also accounts for the differences in hardness. (see Table V, page 31).

C. GAS ADDITIONS

The vacuum-annealing furnace previously mentioned was modified, as shown in Figure 12, by the addition of a standard volume with a mercury manometer and a metering volume of 9.975 c.c. They were added in the position shown in Figure 12 to allow the diffusion pump and cold trap to be isolated from the furnace tube and the volume, to allow evacuation of the system before the introduction of the desired gas, and to aid in a speedy introduction and removal of the gas.

The procedure followed was to place the specimens in a molybdenum boat, which almost enclosed them. The specimens were stacked so that their ends would overlap, thereby leaving a gap between the individual gauge lengths of the specimens. The boat containing the specimens was inserted into the furnace tube and the system was evacuated to less than 5×10^{-5} mm. of Hg. The diffusion pump and cold trap were then isolated and the gas permitted to pass through a liquid nitrogen trap into the furnace and the 4-liter volume. The gas was introduced until the pressure in the volume reached slightly less than atmospheric pressure, approximately 687 mm. of mercury. At this pressure the valves to the standard volume were closed and the system was once again evacuated to less than 1×10^{-5} mm. of Hg. When this

vacuum was attained the furnace was slowly heated to the desired temperature. The rate of heating to temperature was governed so as to keep the vacuum less than 1×10^{-4} mm. of Hg at all times. At temperature, with the vacuum less than 5×10^{-5} mm. of Hg, the diffusion pump and cold trap were once again isolated and the gas allowed to enter the furnace tube.

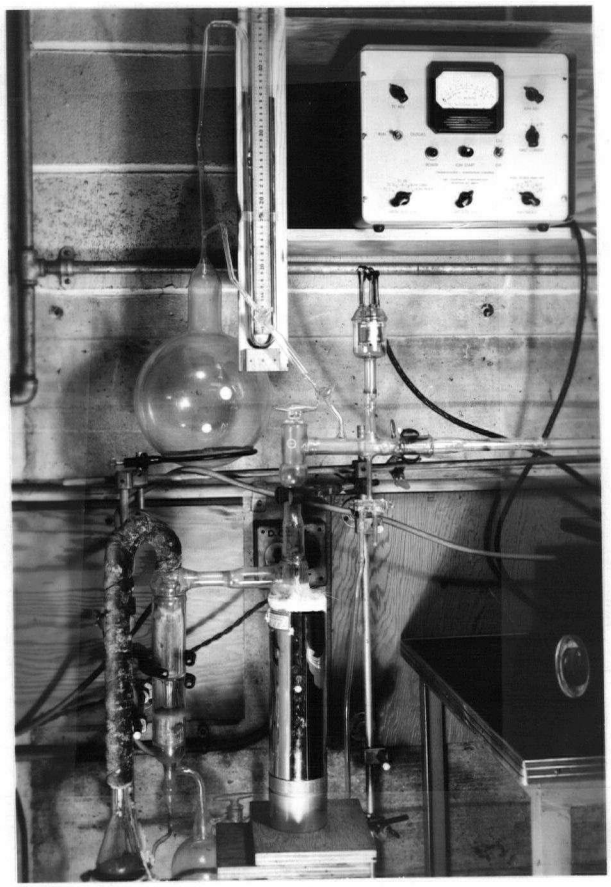

Figure 12. Modified Vacuum Annealing Furnace, showing Manometer and the 4-liter Volume.

After various trial runs it was found that it was advantageous to allow the pressure between the 4-liter volume and the furnace tube to equalize rather than to introduce only a set amount of gas. Using this modified procedure it was found that

the amount absorbed could be controlled by the temperature, holding the time constant at one hour. Nitrogen additions were carried out using temperatures of 800°C, 850°C, 900°C, and 1000°C. Oxygen and hydrogen were added at 500°C. After permitting the specimens to soak in the controlled atmosphere at this temperature for one hour, the system was again evacuated. When the vacuum became less than 1×10^{-5} mm. of Hg, the temperature was raised to 1100°C for a homogenization treatment of eighteen hours. During this time the vacuum increased until, at the end of the treatment, it was less than 2×10^{-6} mm. of Hg. The hydrogen addition was an exception to this procedure, as the specimens were soaked in the hydrogen for three hours at 500°C, and then the system was evacuated and immediately cooled to guard against any degassing of the specimens, since tantalum may be degassed of hydrogen in a vacuum at 1000°C[35].

The specimens were cooled quite rapidly and then removed for hardness testing. A traverse of hardness was done to ensure that the absorption of gas had been uniform; very good results were obtained on sample specimens from each batch. The results of analyses done by Ledoux and Company and by the Wah Chang Corporation of the specimens which contained the various gas additions are shown in Tables V and VI, respectively. The average Diamond Pyramid Hardness Numbers are also shown in Table V.

TABLE V

ANALYSIS OF TANTALUM BY LEDOUX AND COMPANY

Lot	Batch	Treatment Temp.	Treatment Hours	Concentration (parts per million) C	O	N	H	Average D.P.H. No.
1WC	1	1000°C	1	42	130	487	3.1	241
2WC	2	1000°C	1/2	30	190	488	3.4	249
2WC	3	800°C	1/2	-	200	80	-	132
2WC	4	800°C	1/2	-	-	90	-	128
2WC	5	900°C	1/2	-	-	350	-	225
2WC	6	800°C	1	-	-	130	-	148
2WC	7	850°C	1	-	-	190	-	165
2WC	8	500°C	1	70	1090	20	-	448
2WC	9	500°C	3	-	-	-	299	126
2M	-	contaminated		177	487	84	3.8	220

TABLE VI

ANALYSIS OF TANTALUM BY WAH CHANG CORPORATION

Lot	Batch	Concentration (parts per million) C	O	N	H	
1WC	1	-	-	NA	-	
2WC	2	-	-	470	-	
2WC	3	-	-	NA	-	
2WC	4	-	-	75	-	
2WC	5	-	-	330	-	
2WC	6	-	-	NA	-	
2WC	7	-	-	230	-	
2WC	8	< 30	840	-	-	
2WC	9	-	-	-	100) 79) 150)	ave. 110

D. TESTING PROCEDURE

All specimens were tested in tension using an Instron Tensile Tester which autographically recorded the load-elongation curve of each specimen. The crosshead speeds used for tests on Lot 1M were .05 and 20 inches per minute; those on lots 2M and 1WC, .02 and 2 inches per minute; those on lot 2WC, .02 inches per minute.

For lots 1M and 2M, elongation of the gauge length was measured directly from the gauge marks on each specimen; yield stress values were calculated by using the load values taken from the autographic recorder and the measured area. Per cent elongation data for lots 1WC and 2WC were calculated from the load-elongation curve plotted by the autographic recorder.

A modification of the Instron Tensile Tester[33] employing the special file-faced grips[34] was used for these tests. The arrangement of grips and universal joints used in the modification is shown in Figure 13.

The low temperature tests were conducted in a petroleum ether (30-60 technical grade) constant-temperature bath contained in a wide-mouth dewar flask, which fitted over the assembly shown in Figure 13. The bath was cooled by means of a small externally-fed liquid nitrogen container (see Figure 14) and stirred continuously by an electric-driven stirrer. The use of the liquid-nitrogen cooled petroleum ether bath covered the range of temperature between 0°C and —140°C inclusive. The tests at —196°C were conducted by immersing the assembly in liquid nitrogen; those at —183°C, in liquid oxygen. The temperature of —175°C was obtained by first cooling the apparatus and specimen to -196°C and then by allowing them to heat up to —175°C.

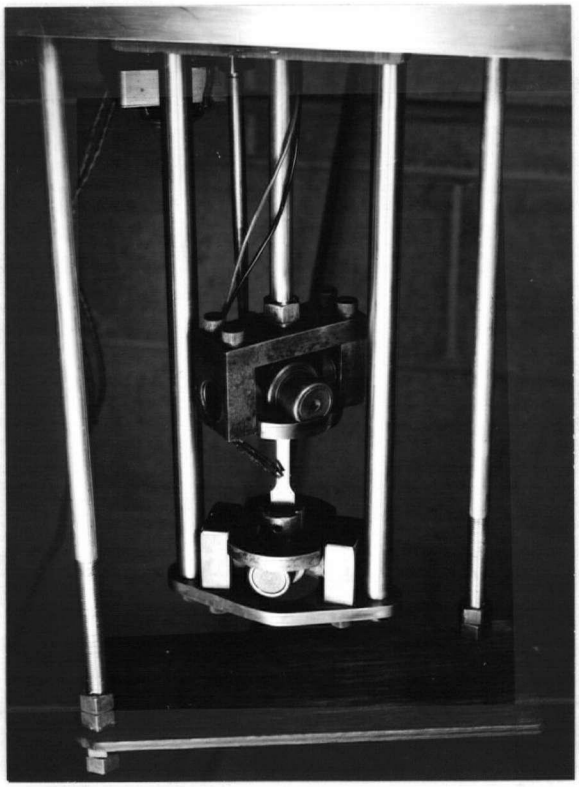

Figure 13. The Modification of Instron Tensile Tester

Figure 14. Dewar Flask with Liquid Nitrogen Container

The tests above room temperature were carried out in a constant-temperature silicone oil bath heated by an immersion heater and stirred continuously. It was found that the bath took a very short time to reach a state of equilibrium and that the temperature remained essentially constant for the duration of the test. The temperature was measured by means of a copper, constantan thermocouple hooked around the specimen's edge. The e.m.f. of the thermocouple was measured on a Pye potentiometer of 10 microvolts sensitivity.

IV EXPERIMENTAL RESULTS AND OBSERVATIONS

The results obtained from tensile testing the different lots of tantalum at temperatures ranging from 100°C to —196°C are tabulated in Appendices I to VIII. This data includes the upper yield point stress, the ultimate stress based on the original cross-sectional area, and the per cent elongation based on a uniformly elongated gauge length of 0.75 inches. All results demonstrated that mechanical properties depend strongly on temperature and on the amount of interstitial solute elements.

Typical load-elongation curves for the various lots of tantalum are shown in Figure 15. The curve forms vary as shown: some have a particularly well defined upper and lower yield point, while others have only a very small yield point phenomenon, and some have none at all.

A. RESULTS OF INITIAL LOTS OF TANTALUM

1. Lot 1M (annealed Murex Tantalum)

The results are given in Figure 16 and tabulated in Appendix A. Tests were conducted at temperatures ranging from 22°C to —196°C at strain rates of 0.067 and 26.7 in./in./min. Only the per cent elongation values are shown for the test done at the higher strain rate, as it is impossible for the autographic recorder to operate at this level. The elongation values for both strain rates were calculated by using a direct measurement of the elongated gauge length.

The yield point stress is shown to increase from 47,000 psi. at room temperature to 132,000 psi. at —196°C. The plot of elongation versus temperature, shown in Figure 16,

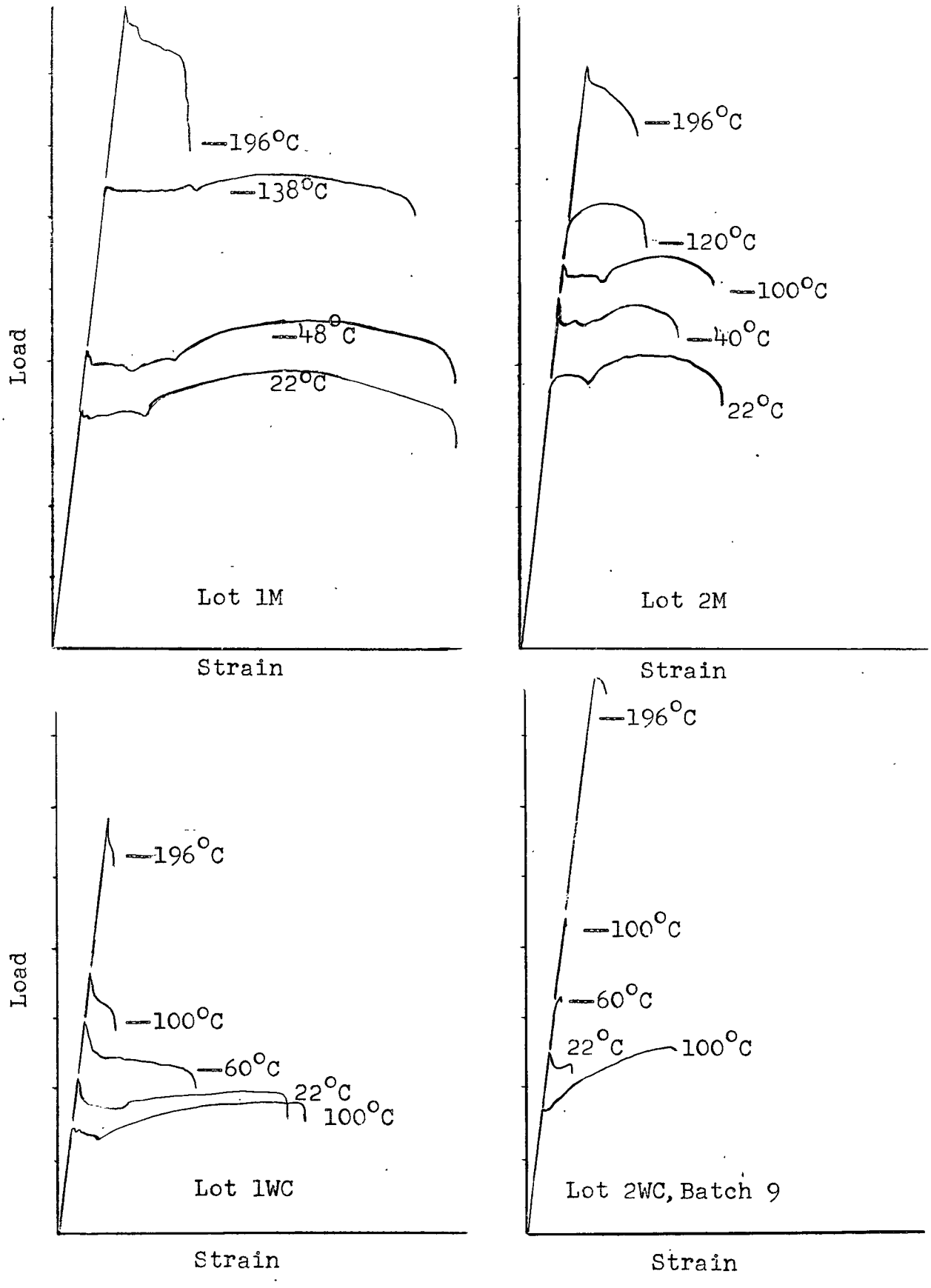

Figure 15. Typical Load-Elongation Curves.

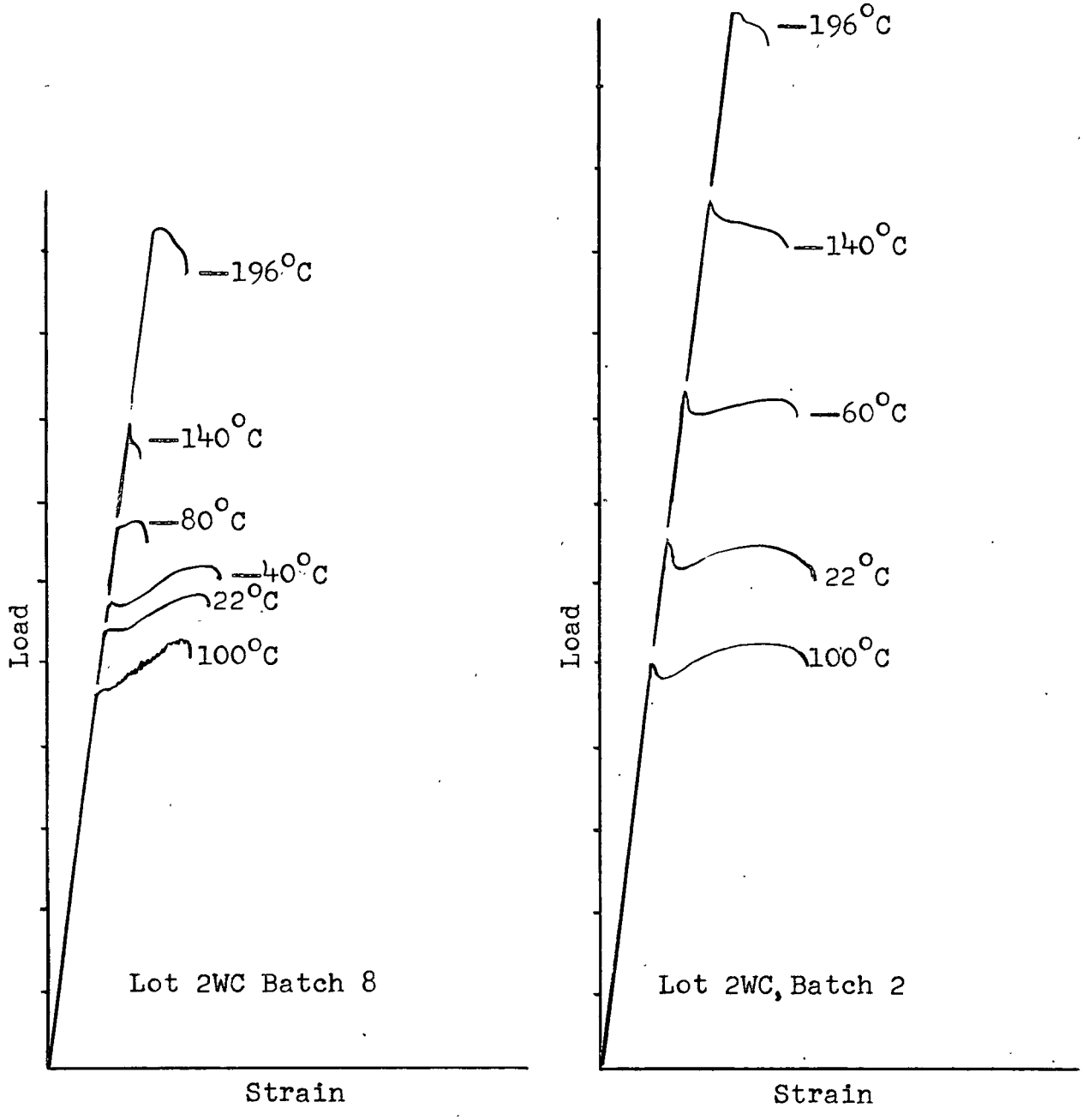

Figure 15. (continued) Typical Load-Elongation Curves.

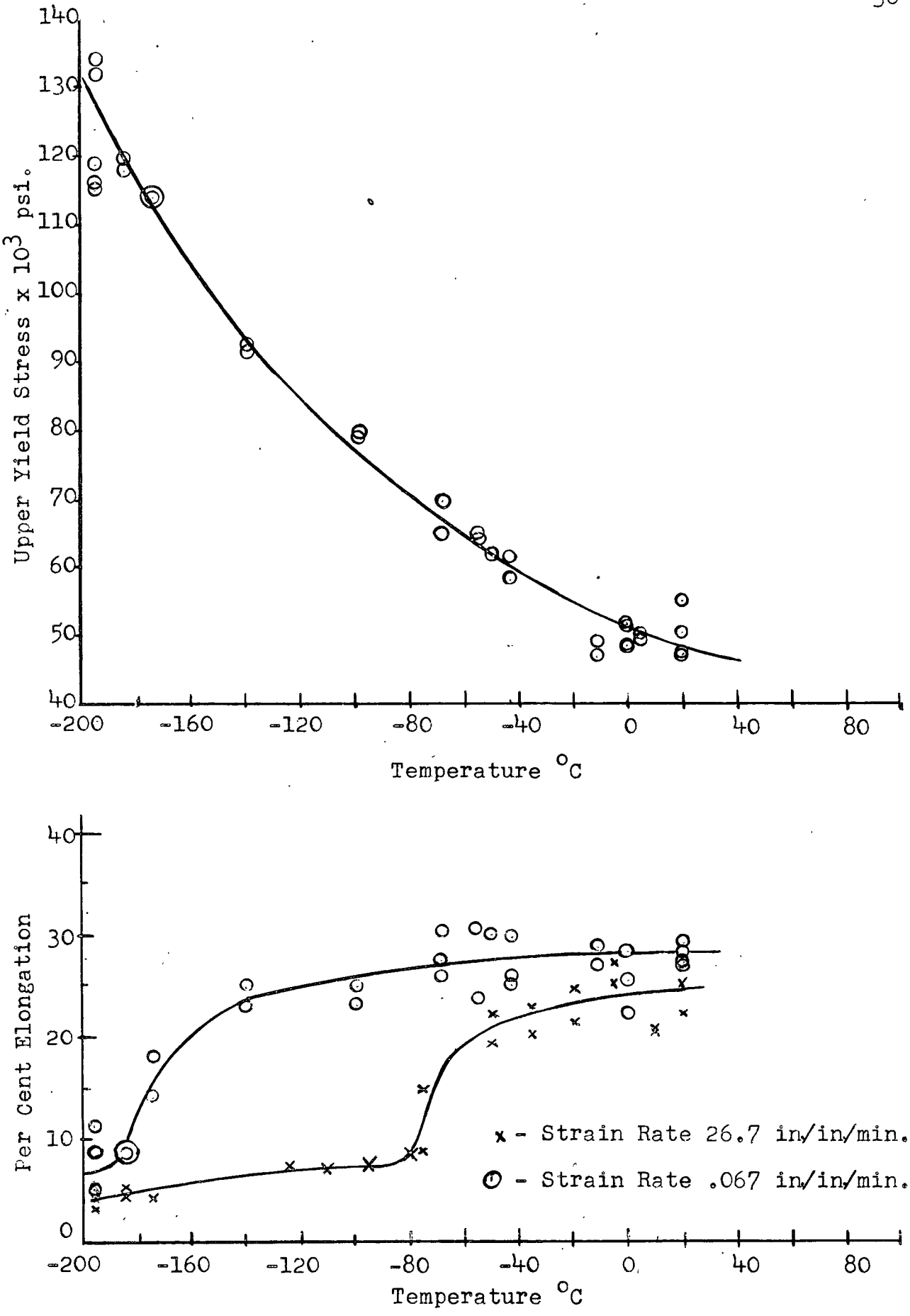

Figure 16. Properties versus Temperature for Tantalum Lot 1M

exhibits the presence of a ductile-to-less-ductile transition occurring in the range of —140°C to —180°C for the lower strain rate. The increase in strain rate raises the transition temperature significantly, by 90°C, and accentuates this transition, making it more abrupt.

The typical load-elongation curves, given in Figure 15, indicate that a large discontinuous yield elongation takes place. On loading a specimen at room temperature to the upper yield point, it was observed that a Lüder's band appeared at each end of the gauge length at an angle of 45° to the length of the specimen. During the discontinuous elongation these bands enlarged, moving down the gauge length towards each other. On meeting there was a slight drop in the load followed by an increase due to work hardening. At low temperatures, yielding occurred within one of these Lüder's bands at either end of the gauge length. An example of this can be seen in Figure 17.

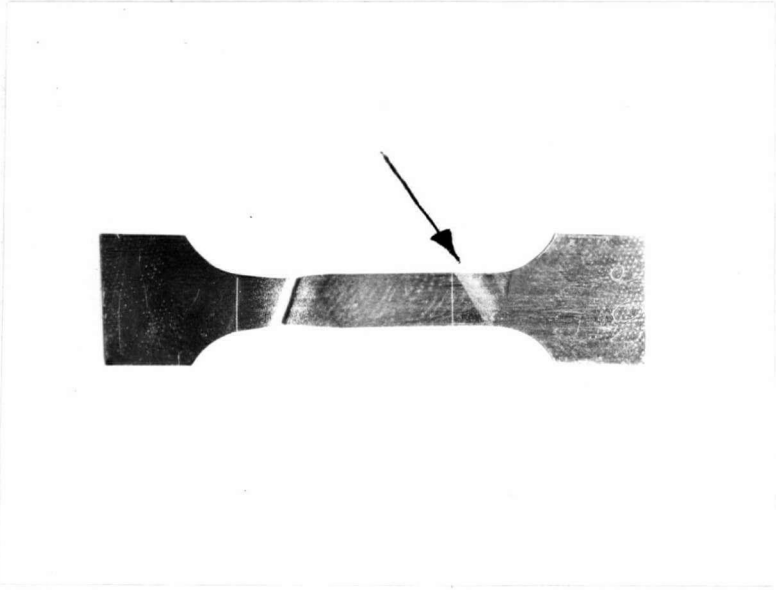

Figure 17. Specimen showing Lüder's Band

Only a limited number of work-hardening and aging experiments were carried out. Since it was decided that they did not really pertain to the problem, they were abandoned. For the sake of completeness, the results are tabulated in Appendix B.

2. Lot 2M (Contaminated Murex Tantalum)

Plots of yield point stress and of per cent elongation versus temperature for this lot of tantalum are given in Figure 18 and the results are tabulated in Appendix C. The plot of yield point stress shows an increase from 70,000 to 155,000 psi., for the lower strain rate, and the higher strain rate gives an increase from 80,000 to 160,000 psi. The scatter of results for this lot makes it difficult to isolate the effect of strain rate, but there does appear to be a very definite increase in yield point stress with the increase in strain rate.

The plot of per cent elongation versus temperature shows the transition from ductile-to-less-ductile behaviour for both strain rates. This transition appears to occur in the region between $-100^{\circ}C$ and $-140^{\circ}C$ for the lower strain rate and between $-100^{\circ}C$ and $-120^{\circ}C$ for the higher strain rate.

Typical load-elongation curves in Figure 15, page 36, exhibit a variance in form, and the results for yield point stress showed a large scatter, thought to be due to non-uniform material resulting from contamination in the annealing furnace. Lüder band behaviour similar to that in Lot 1M was also observed.

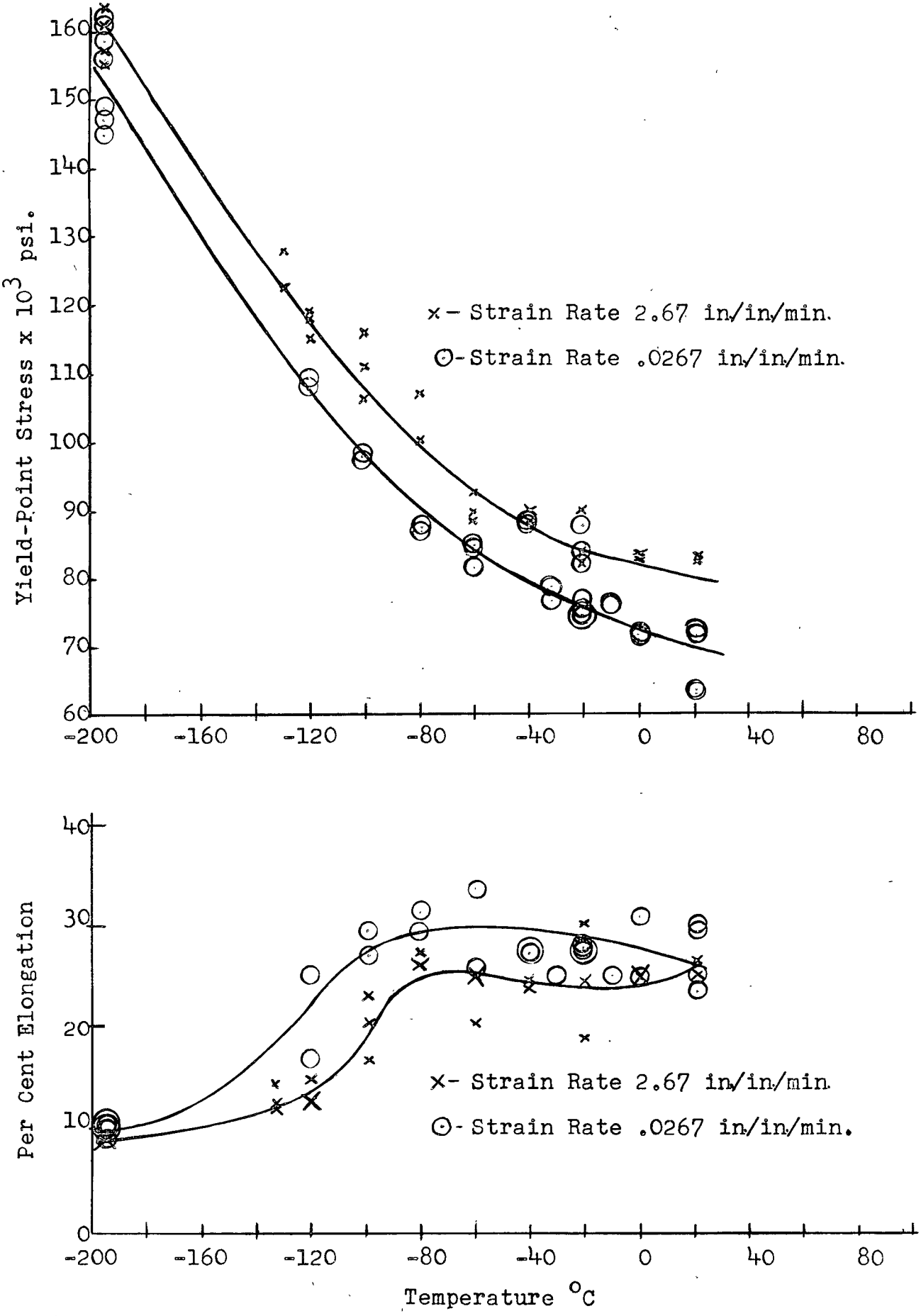

Figure 18. Properties versus Temperature for Tantalum Lot 2M

3. Lot 1WC (Pre-cut Tensile Specimens from Wah Chang Corporation)

The tensile test results obtained for this lot of tantalum at temperatures ranging from 100°C to —196°C are compiled in Appendix D. Two rates of strain were used for these tests: .0267 and 2.67 in./in./min.. Plots of yield point stress and per cent elongation versus temperature are given in Figure 19.

The yield point stress for the lower strain rate increases from 25,000 psi. at 100°C to 111,000 psi. at —196°C. The elongation plot shows that a transition of ductile-to-less-ductile behaviour from 40 per cent elongation to less than 1 per cent occurs at —80°C for the low strain rate, and at —20°C for the high strain rate. The expression "less-ductile behaviour" is used in this case because, even at —196°C, some reduction in area occurred before failure.

Typical load-elongation curves in Figure 15, page 36, show that below about —20°C a very sharp yield point occurs, which is considerably higher than the rest of the curve in most cases. The scatter shown in Figure 19 is thought to be due to differences in accuracy of specimen alignment.

There was no evidence of a prominent Lüder's band at room temperature; rather the elongation seemed to take place uniformly along the whole gauge length, but the specimen warped across the width (see Figure 20(a)). At low temperatures yielding took place in one very narrow Lüder's band as shown in Figure 20(c), page 44.

4. Lot 2WC (Wah Chang Sheet)

The results of tensile tests conducted at a strain rate of .0267 in./in./min. between 100°C and —196°C are contained in

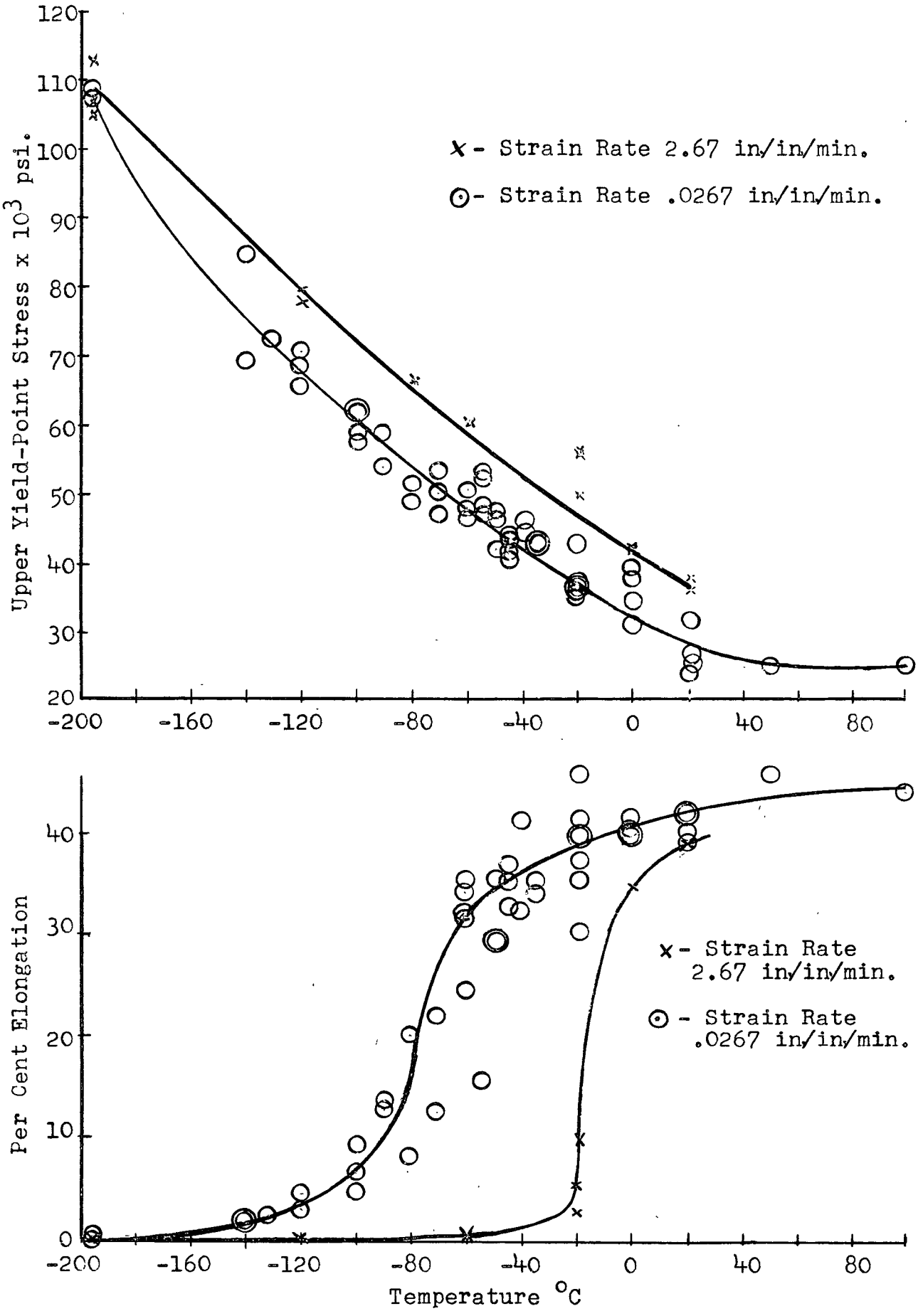

Figure 19. Properties versus Temperature for Tantalum Lot 1WC

Appendix E. Plots of yield point stress and per cent elongation versus temperature are shown in Figure 21. The yield point stress increased from 28,000 psi. at 100°C to 125,000 psi. at —196°C. The elongation plot shows a gradual transition from 30 per cent elongation to less than 1 per cent between —60°C and —196°C.

The load-elongation curves and failure behaviour were similar to those of Lot 1WC.

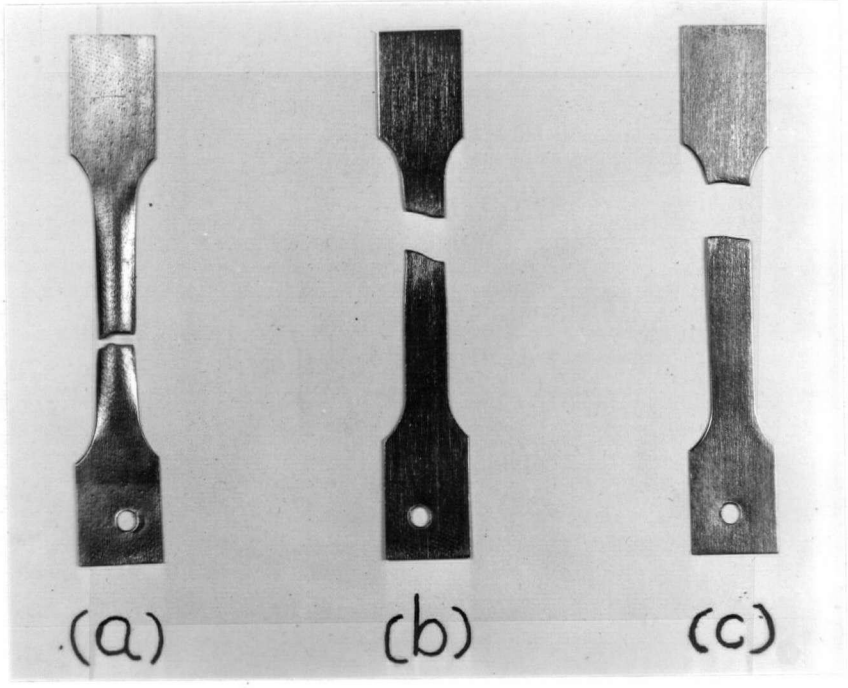

Figure 20. Typical Broken Specimens. Illustrating from left to right: failure at room temperature, failure at —60°C, and failure at —196°C.

B. RESULTS OF INITIAL LOTS OF TANTALUM

1. Nitrogen Additions

The nitrogen contents of the individual batches are shown in Tables V and VI. Results of tests on these batches conducted at a strain rate of .0267 in./in./min. at temperatures

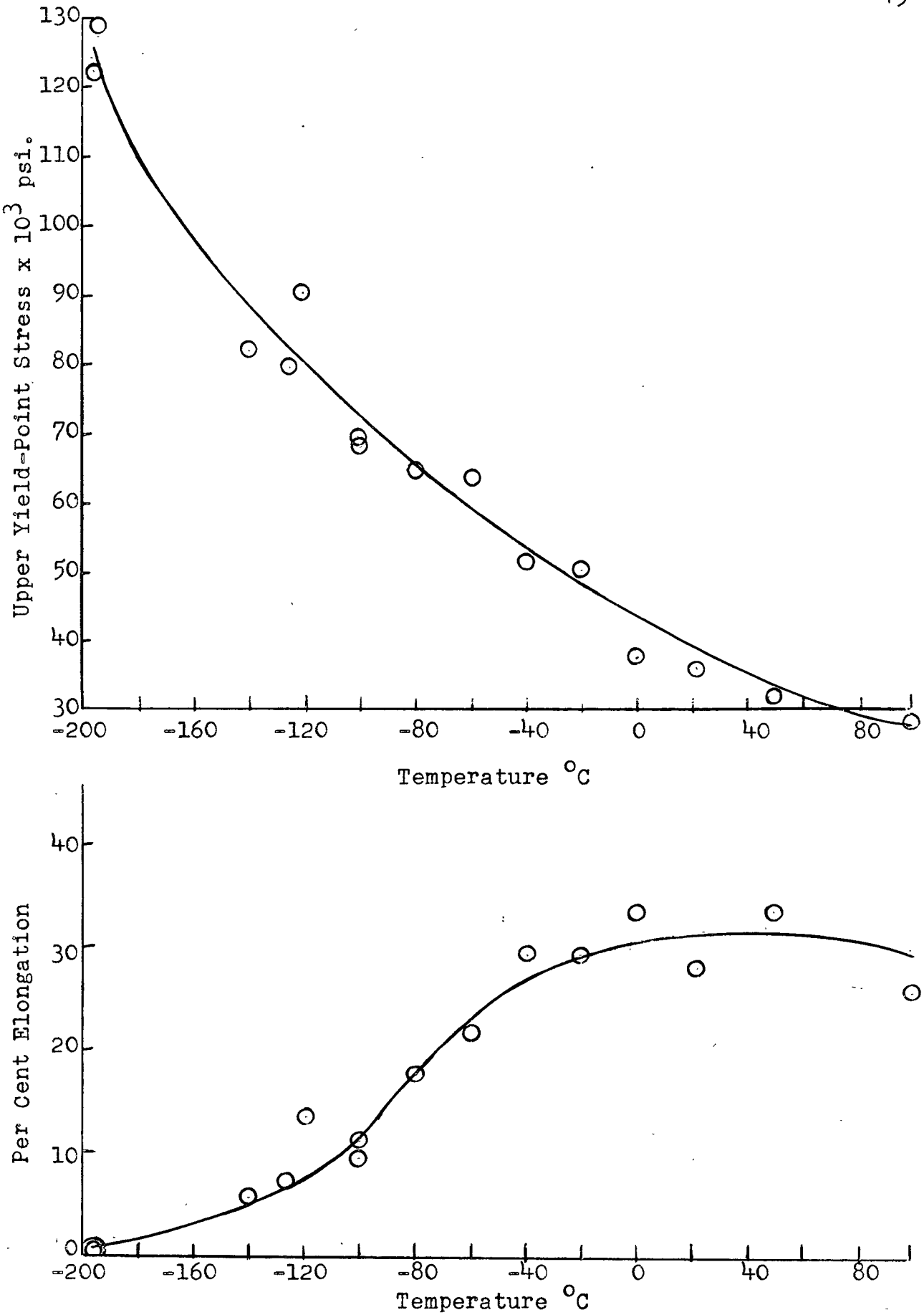

Figure 21. Properties versus Temperature for Tantalum Lot 2WC

between 100°C and —196°C are tabulated in Appendix F. The plot of yield point stress versus temperature is presented in Figure 22; the per cent elongation versus temperature plot, in Figure 23, page 48.

The yield point stress values of Batches 3, 4, and 6, having an average of 100 parts per million nitrogen, all lie on the same curve in Figure 22. They exhibit an increase from 40,000 psi. at 100°C to 130,000 psi. at —196°C. Batch 7, having 190 ppm. nitrogen, shows an increase from 52,000 psi. at 100°C to an estimated 150,000 psi. at —196°C. Batch 5, having 350 ppm. of nitrogen, exhibits an increase from 70,000 psi. at 100°C to 184,000 psi. at —196°C. The results for Batches 1 and 2, having an average nitrogen content of 488ppm., appear to lie on a common curve showing an increase from 80,000 psi. at 100°C to 204,000 psi. at —196°C.

The elongation values for Batches 1 and 2 do not lie on a common curve, however (see Figure 23, page 48), the elongation of Batch 1 is less than that of Batch 2. Both curves indicate measurable ductility at —196°C. Lot 1WC, Batch 1 shows 2.5 per cent; while Lot 2WC, Batch 2 shows 9.5 per cent elongation. Both values represent an increase over the elongation of the starting materials. These differences in elongation are probably due to the differences in starting material.

The elongations for the other batches, which were from Lot 2WC, all exhibited a decrease from approximately 20 per cent at 100°C to 10 per cent for Batch 5; 2.5 per cent for Batches 3, 4, and 6; and an estimated 2.5 per cent for Batch 7, at —196°C. The load-elongation curves and fractures were similar to those of Lot 1WC. No evidence of Lüder's bands at 20°C was observed,

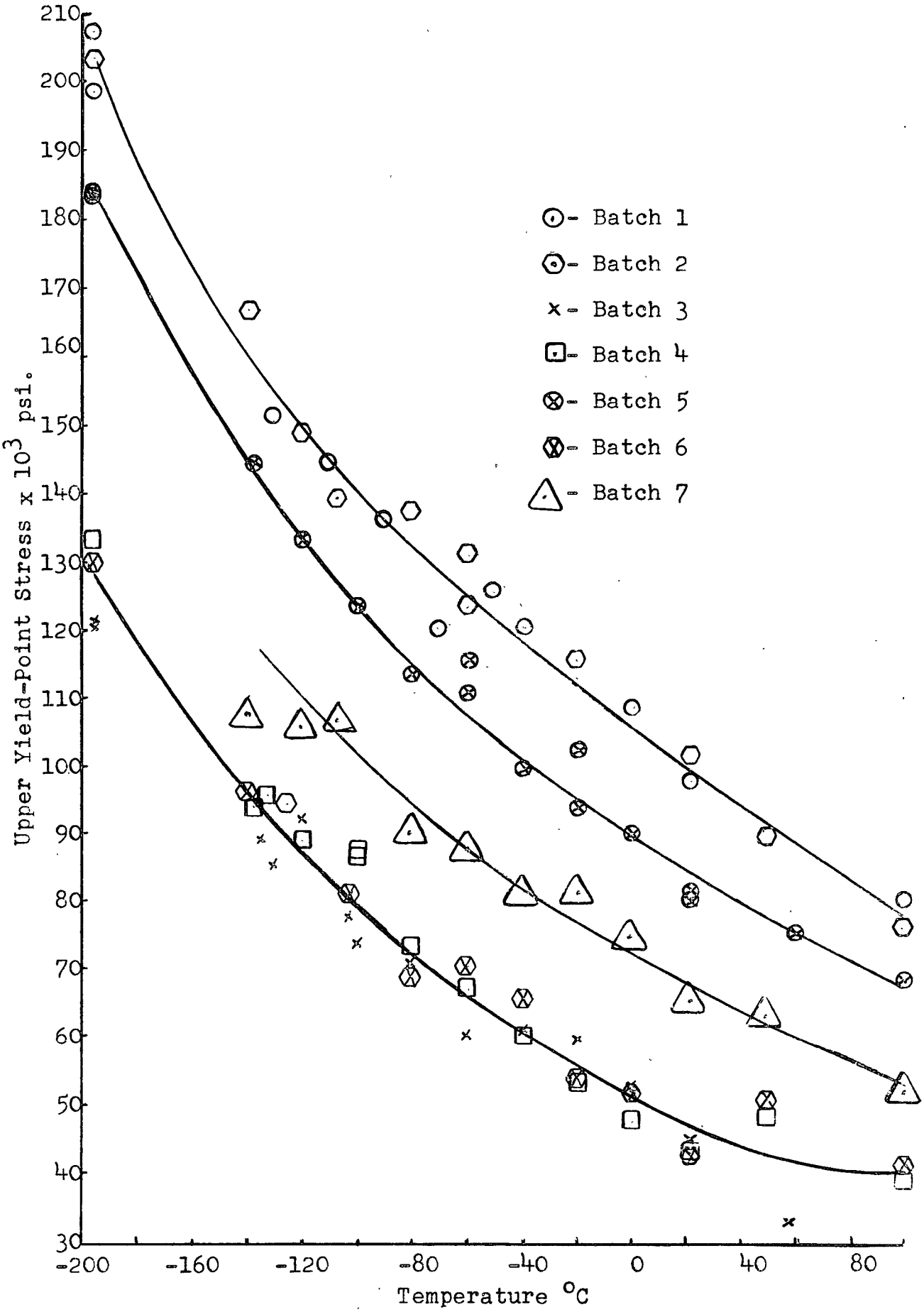

Figure 22. Yield-Point Stress versus Temperature for Tantalum plus Nitrogen

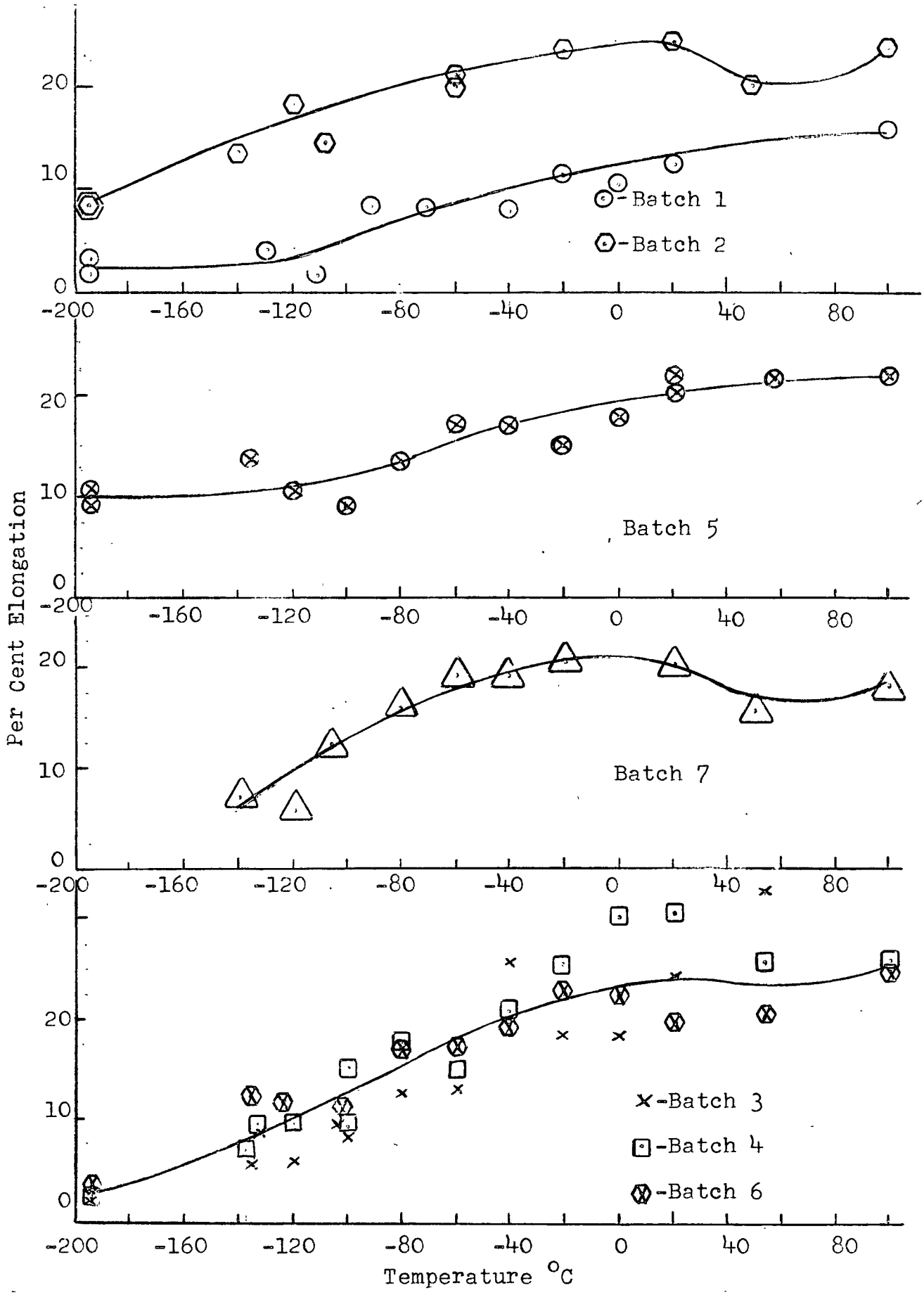

Figure 23. Elongation versus Temperature for Tantalum plus Nitrogen.

but the fracture, at low temperatures, occurred within a narrow Lüder's Band.

2. The Oxygen Addition

The material with an oxygen content of 1090 ppm. was tested at temperatures between 100°C and —196°C and at a strain rate of .0267 in./in./min. The results are tabulated in Appendix G; the plots of yield point stress, and elongation versus temperature are presented in Figure 24.

The yield point stress increases from 85,000 psi. at 100°C to 200,000 psi. at —196°C. The elongation exhibits a transition from 16 per cent to 5 per cent in the region between —40°C and —90°C. The ductility at —196°C was greater than that of Lot 2WC, the starting material. Some typical load-elongation curves for this material with a high oxygen content are shown in Figure 15, page 36. Many curves showed no yield point.

3. The Hydrogen Addition

Results of tests conducted at a strain rate of .0267 in./in./min on material contaminated with 200 ppm. hydrogen are tabulated in Appendix H and plotted in Figure 25, page 51.

The yield point stress increased from 28,000 psi. at 100°C to 134,000 psi. at —196°C. The elongation decreased from 23 per cent at 100°C to 0 per cent (i.e. brittle failure) at —60°C, which prevailed to —107°C and then increased to 3 per cent at —196°C.

The typical load-elongation curves are shown in Figure 15, page 36. The failures at —60°C, —80°C, and —107°C are brittle, but that at —196°C is definitely ductile.

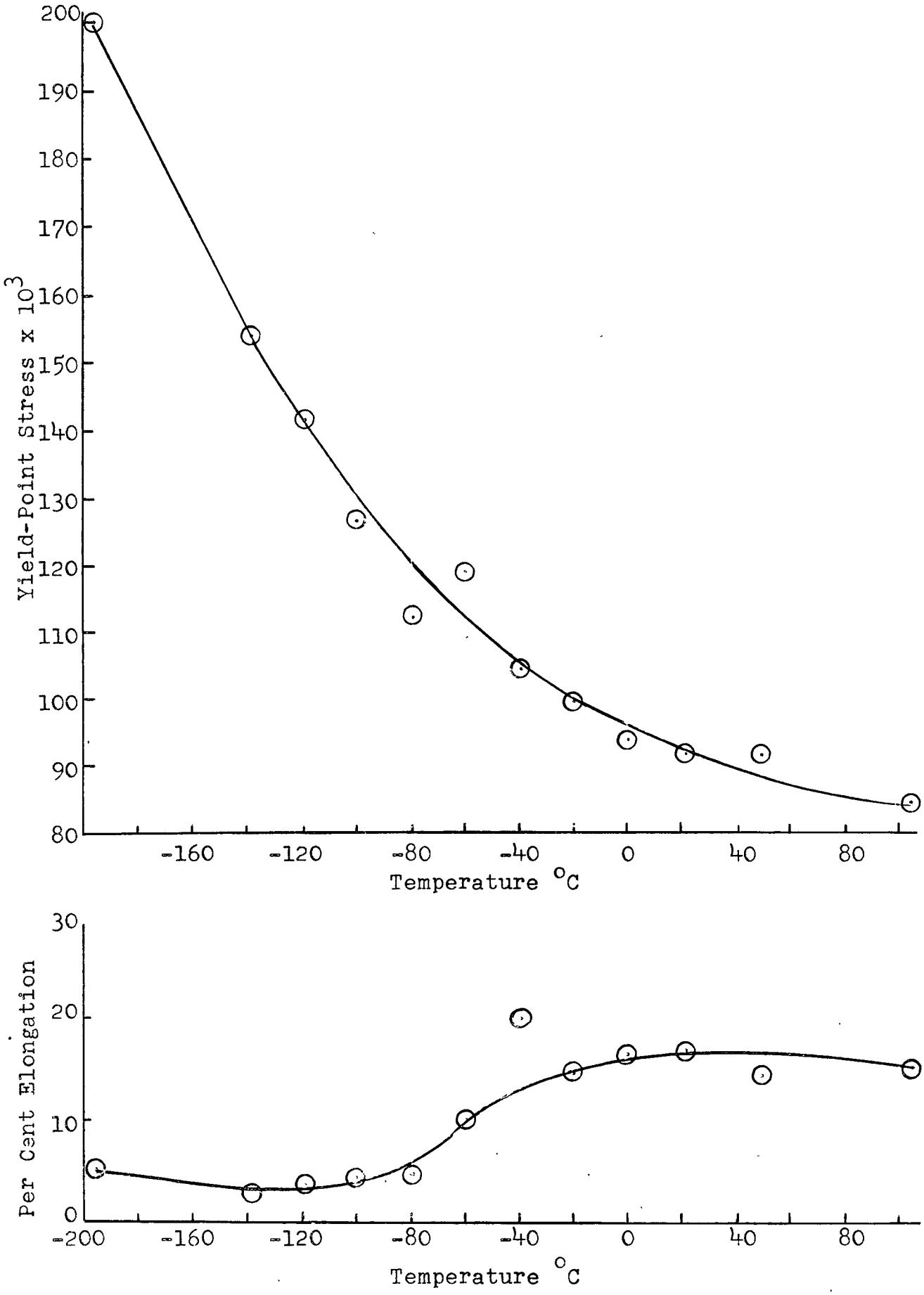

Figure 24. Yield Point versus Temperature for Tantalum plus 1090 ppm. Oxygen.

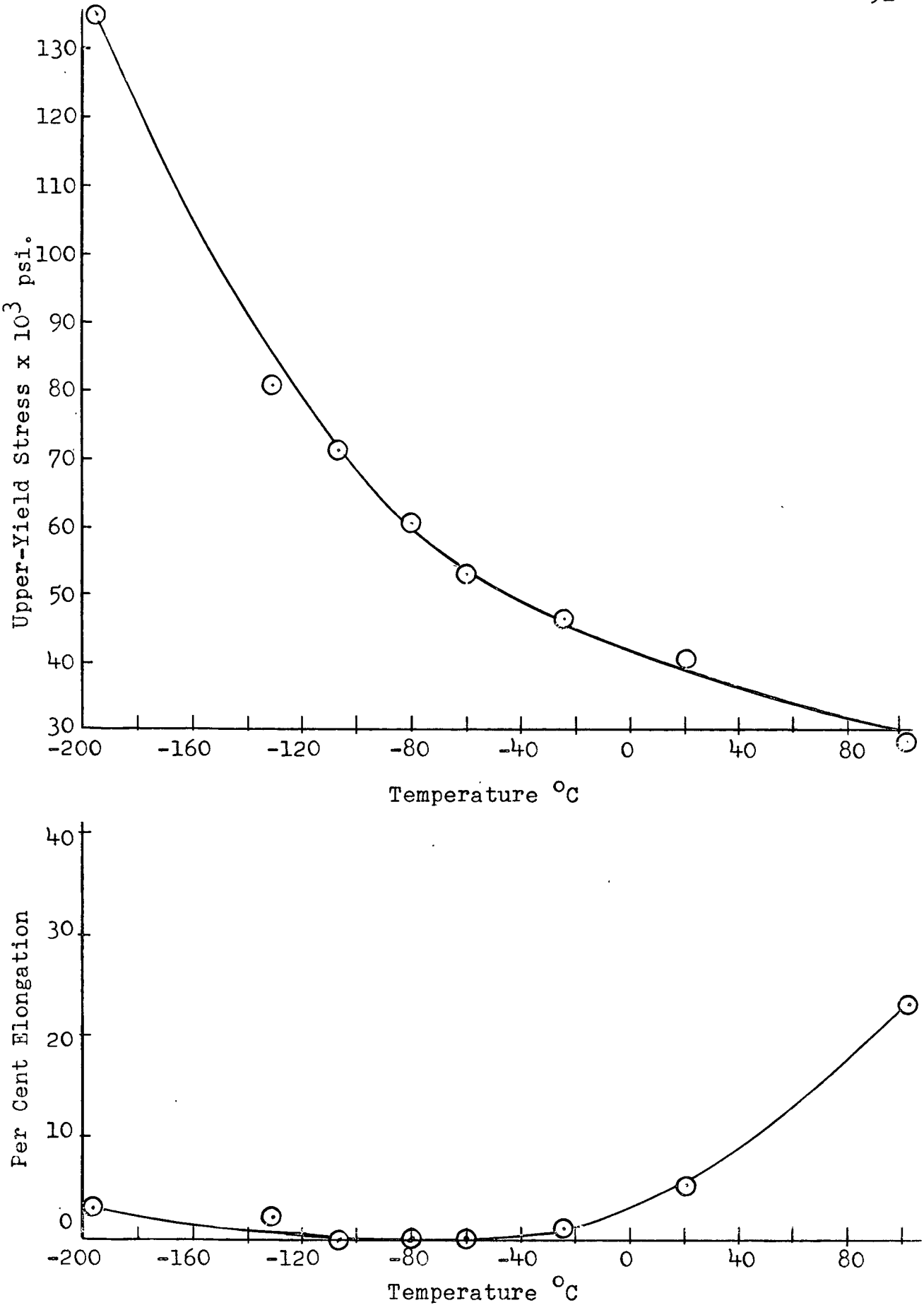

Figure 25. Yield-Point Stress and Elongation versus Temperature plus 299 ppm. Hydrogen.

C. THE ACCURACY OF RESULTS

The error in results is due to uncertainties in measured values. The total possible error is an accumulation of the uncertainties in each measured quantity. These individual uncertainties are discussed below.

1. The width of the gauge section was measured with a micrometer capable of reading to .0001 inches, but the thickness of the specimens made measuring very difficult. The error in this measurement is estimated to be ± 0.0005 or ± 0.25 per cent for a gauge length width of 0.200 inches. The thickness was measured with the same micrometer, the error in measurement being estimated to be 0.00005 or ± 0.033 per cent for a specimen thickness of 0.015 inches. The resulting error in cross-sectional area is ± .283 per cent.

2. The load from the autographic recorded load-elongation curve may be read with an accuracy of ± 1/2 pound on the 200-pound full scale, and with an accuracy of ± 1.5 pounds on the 500-pound full scale. For a 150-pound load at the upper yield point, the error would be ± 0.333 per cent, and for a 300-pound yield point load the accuracy would be ± 0.5 per cent.

The accuracy of the autographic recording unit is stated by the manufacturer[36] as being less than 1 per cent of full scale, if the time required for the pen to travel the full scale is more than five seconds. A recorded point on a curve will also be within this accuracy if it lies on a curve whose slope at this point is such that more than five seconds would be required for the pen to travel the full scale at this slope.

The error using the 200-pound full scale, therefore, would be less than ± 2 pounds, and would be less than ± 5 pounds in the case of the 500-pound full scale. In the cases cited above, the respective errors would be less than ± 1.33 and ± 1.66.

The accuracy in calculating upper yield point stress is calculated to be, therefore, ± 1.95 per cent for the 150-pound load and ± 2.44 per cent for the 300-pound load.

3. To determine the per cent elongation for Lots 1M and 2M, a 0.75 inch gauge length was marked on the specimen. After testing, the broken pieces were fitted together and a measurement of the elongated gauge length was made. The accuracy of both measurements is estimated to be ± 0.01 inches, and the error introduced on fitting the two pieces together is estimated to be 0.01 inches. When considering, for example, a 0.75 inch gauge length that has elongated to 0.95 inches, the accuracy is ± 1.33 per cent for the first measurement and ± 2.17 per cent for the second, resulting in an overall error of ± 3.50 per cent. In this example the elongation was calculated as being 26.7 per cent ± 3.50 per cent. The per cent elongation may, therefore, be expressed as 26.7 ± 1.31 per cent.

For the per cent elongation values of the remaining materials, the results of Lots 1M and 2M were used in conjunction with their associated load-elongation curves to plot a graph of recorder-chart divisions from the upper yield point to failure versus per cent elongation. The error in these values is the original error plus the error in reading the divisions on the recorder chart. The accuracy of this reading is estimated to be ± .2 of a division. Using the slope of this plot, which is 1.2

divisions for each per cent elongation, the accuracy is ±0.167 per cent. Elongation may be expressed, in this example, as 26.7 ± 1.48 per cent.

A calibration of the thermocouple used to measure the temperature of the constant temperature both showed that, at 100°C it read 98.8°C; at 0°C it read 0.03°C; and at —196°C it read —212.7 °C. The voltage was taken on the same Pye potentiometer used during experimentation.

The preceding estimates of errors do not include those unknown errors which are incurred by dislocation density, presence of microstress raisers, and other such factors. The yield point stress is known to be a very inconsistent value. In order to analyze completely the error of yield point stress, a statistical approach is necessary.

D. METALLOGRAPHY

Samples from the different lots were mounted in bakelite and polished by using various grades of emery paper down to 0000 grade and by using a chemical polish previously described. Etching was done by immersion or swabbing using the chemical polish for periods greater than thirty seconds. Swabbing required less time and resulted in a three-dimensional effect. In some cases the chemical polish was followed by a different etch which was done by immersion for 90 seconds in a solution of three parts nitric acid and one part hydrofluoric acid. This procedure gave a quick grain boundary etch.

Fracture areas were examined for evidence of twins, but none were found. Instead, short wavy lines parallel to the fracture surface were observed before polishing and etching. These lines were also found in Lüder's bands, such as those shown

in Figure 26. This figure also shows these lines at a high magnification. In this case the lines were found to parallel to the boundaries of Lüder's bands. It was thought that they might be microcracks, but on etching they disappeared; and, furthermore, no evidence of microcracks was found in any of the polished and etched specimens.

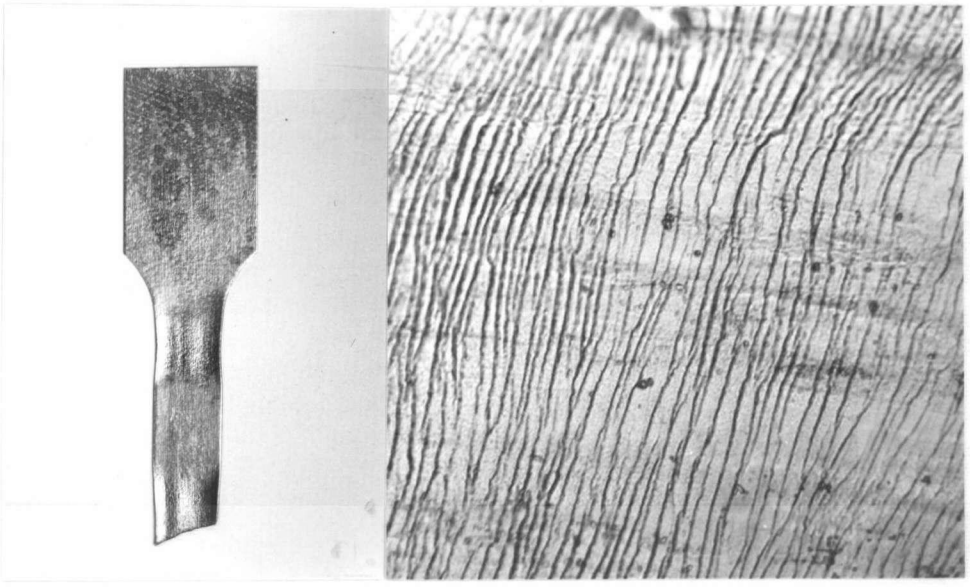

Figure 26. Photograph of Lines found in Lüder's Bands.

Microstructures of the various lots of tantalum are shown in Figure 27. No grain boundary precipitates or inclusions were observed in any of the material used in this investigation. The microstructures of the material containing oxygen and the largest amount of nitrogen showed no evidence of any grain-boundary precipitate (see Figure 27 (e), (f)). Etch pits are seen in most of these microstructures.

The difference in grain size is clearly evident in these microstructures. It is also evident that the grain size within these individual specimens is irregular and not equiaxed.

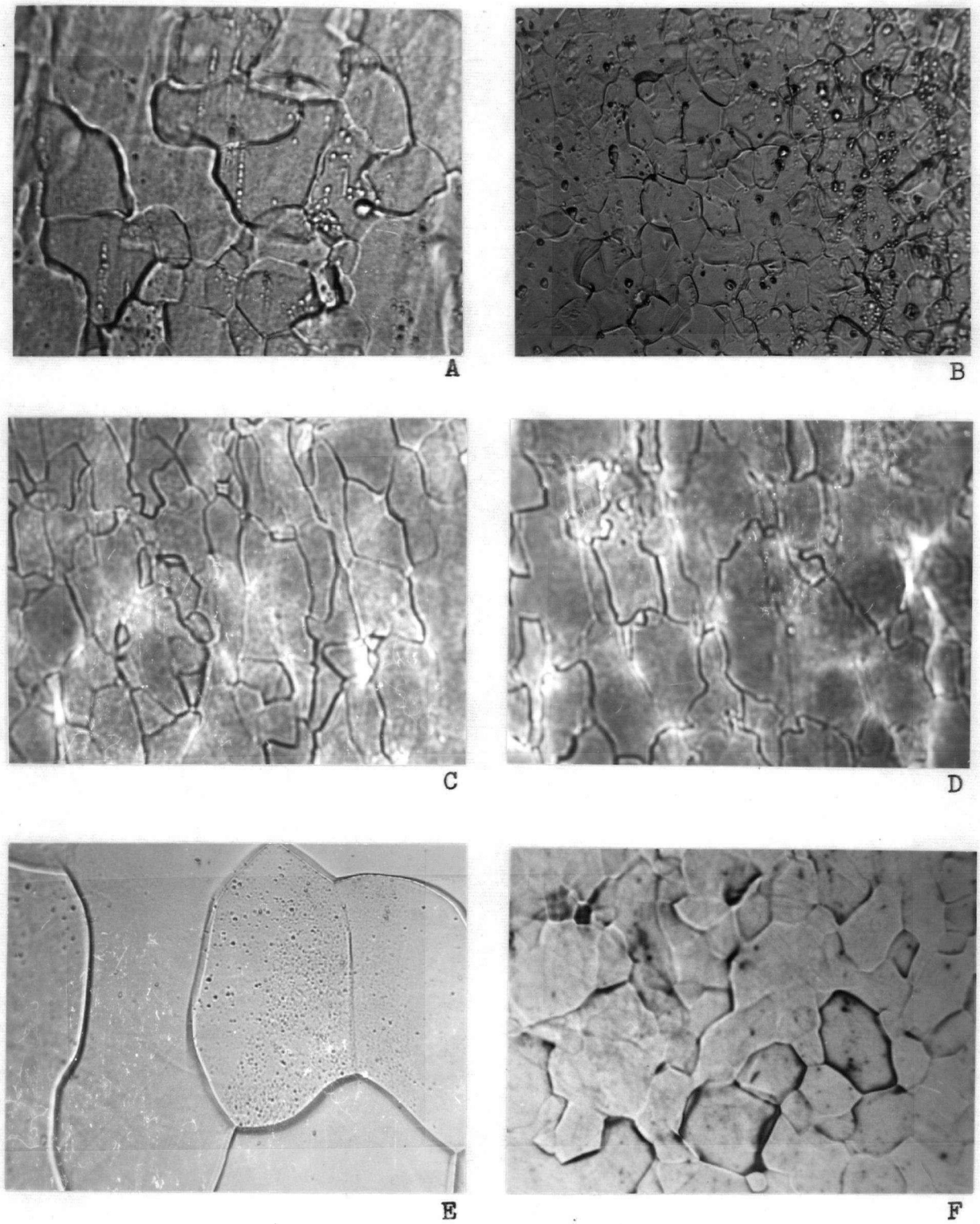

Figure 27. Typical Microstructures

A - Lot 1M
B - Lot 2M
C - Lot 1WC
D - Lot 2WC
E - Lot 2WC - Batch 1
F - Lot 2WC - Batch 8

V DISCUSSION

This investigation has shown that tantalum follows the pattern exhibited by the other body-centered-cubic metals in that a very strong dependence of yield-point stress on temperature exists. On a decrease in temperature or an increase in strain rate, the per cent elongation values go through a transition from ductile-to-less ductile behaviour as do the other body-centered-cubic metals of Group V of the periodic table.

A. YIELD POINT RESULTS

The temperature dependence of yield point stress is presented in Figures 16, 18, 19, 21, 22, 24, and 25. They show that as the temperature decreases the yield-point stress rises continuously in the temperature range investigated.

An attempt was made to fit the results of these experiments to the theoretical relationship derived by Fisher[4], as described previously in this thesis; but a plot of upper yield point stress versus the reciprocal of absolute temperature did not yield a straight line.

The empirical relationship of Zener and Holloman[8] was also tried but rejected, as a plot of the logarithim of yield-point stress versus the reciprocal of absolute temperature did not yield a continuous straight line. This plot was made up of a series of straight lines indicating that the relationship was valid in certain ranges of temperature, but that there was no consistent pattern. Therefore, no conclusions can be drawn from these results. The validity of this relationship in only certain

ranges of temperature may be due to the effect of the various interstitial species as suggested by Snowball[33]. He postulated that within a certain temperature range, interstitial species react with dislocations thus anchoring them, while in a different temperature range the interacting species are different and a different relationship with yield point stress exists. The results from this investigation are not complete or conclusive enough to support Snowball's[33] hypothesis.

The effects of interstitials on the yield point stress of tantalum are quite large, as can be seen in Figures 22, 24, and 25, pages 47, 50, and 51 respectively. There is a suggestion that the same temperature dependence of yield point stress exists even after the addition of large amounts of interstitials. The only difference appears to be their relative levels on the stress axis.

Figure 28 shows a plot of yield point stress versus nitrogen content for several temperatures. This plot indicates that the yield point stress is a linear or near-linear function of nitrogen content in the range of temperature and range of nitrogen content investigated. This same Figure shows the effect of oxygen on the yield point stress. Lots 1M and 2M were included in this plot as their analyses showed relatively high oxygen content. These points did not lie in a straight line, probably because Lots 1M and 2M had a higher nitrogen, hydrogen, and carbon content than Lots 1WC and 2WC, and because a grain size difference is present in these materials. The slopes of these lines increase with decreasing temperature, indicating that the effect of interstitials increases with decreasing temperature.

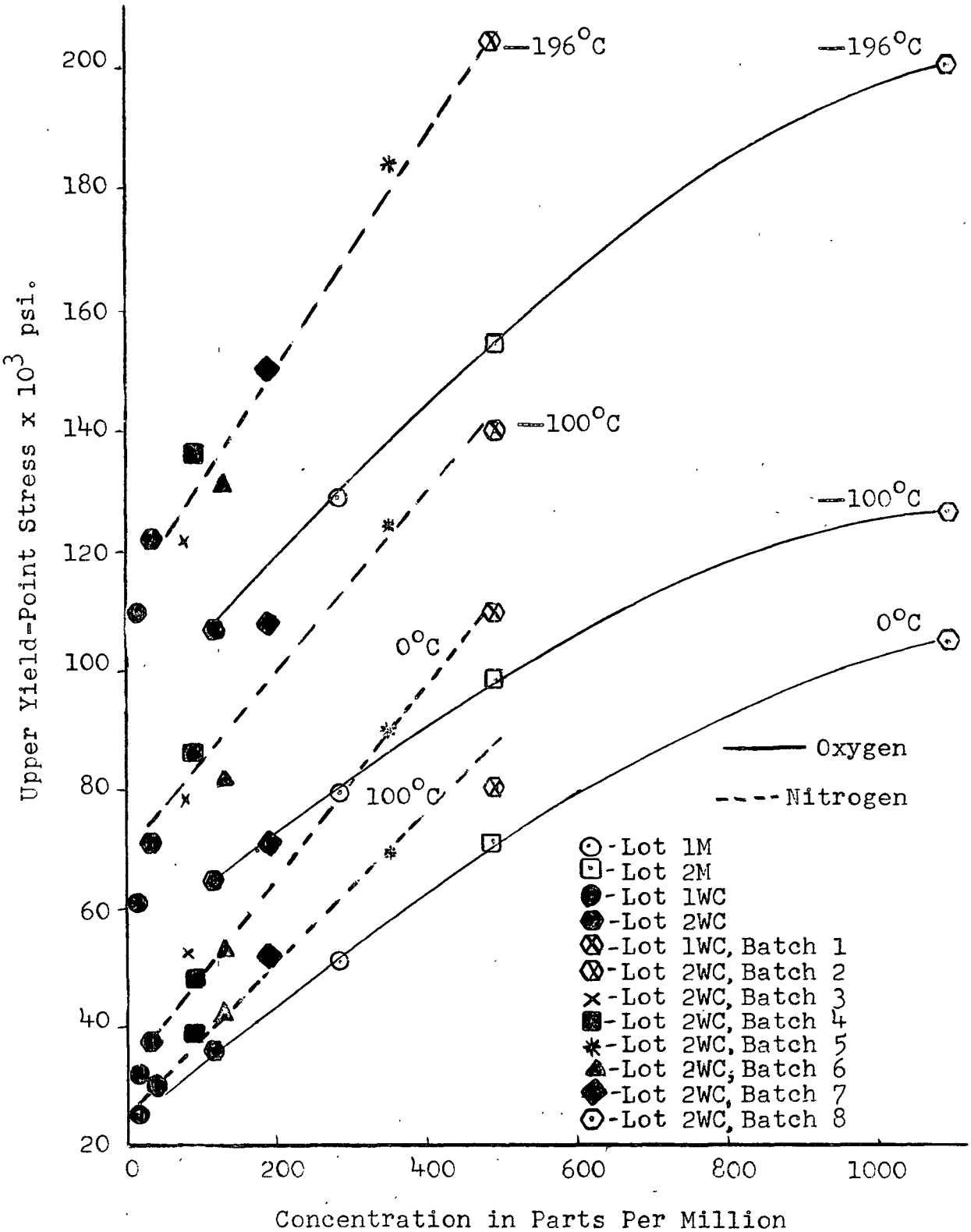

Figure 28. Yield-Point Stress versus Nitrogen and Oxygen Contents.

Figure 29 shows a plot of the yield point stress versus the total interstitial content. The points for material with the added nitrogen lie in a straight line as expected from the previous plot. When the points corresponding to the material with added oxygen were joined to those for the initial material (Lot 2WC), it was found that the points for Lots 1M and 2M fell in the same lines. The points corresponding to the hydrogenated material were then joined to those of Lot 2WC, the starting material.

The relative effects of increasing interstitial content at a constant temperature may be assessed from this plot. It is evident that the effects are linear, that nitrogen has a greater effect than either oxygen or hydrogen, and that hydrogen has the least effect. As shown in Figure 30, the slopes of these three sets of lines increase with decreasing temperature. These conclusions are supported by the data of Perkins[25] who found that nitrogen affected the hardness much more than either oxygen or hydrogen.

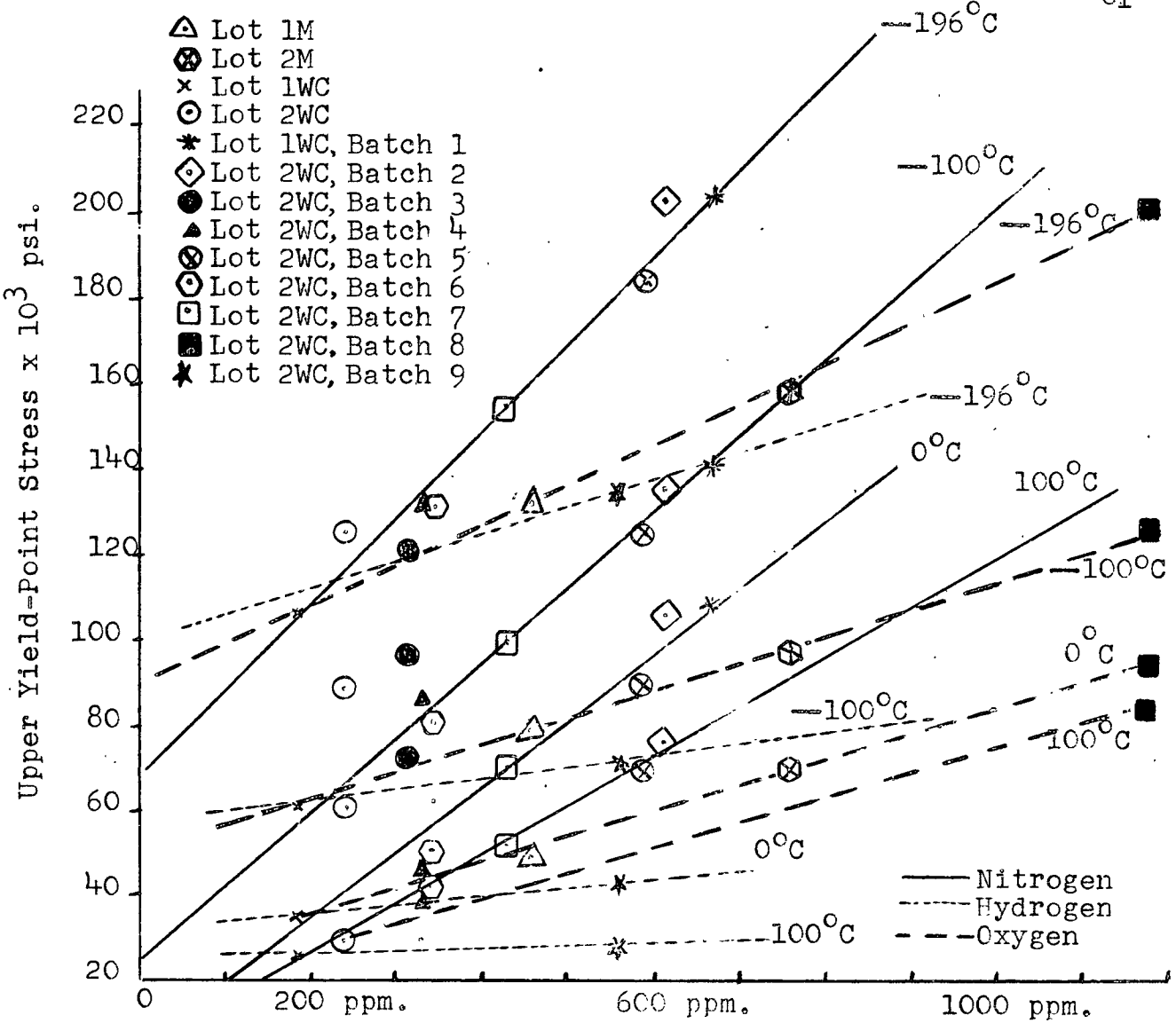

Figure 29. Yield-Point Stress versus Total Interstitial Content.

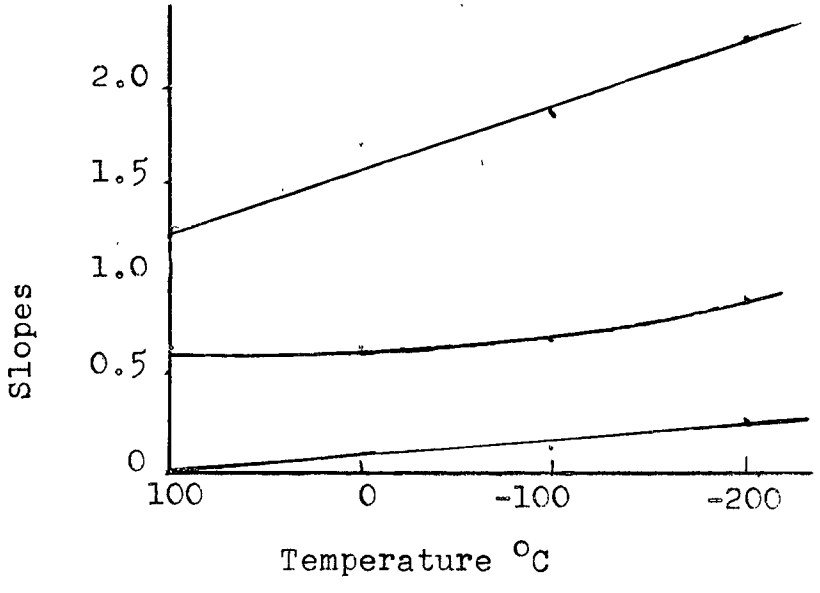

Figure 30. Slopes of Yield-Point Stress versus Total Interstitial Content Lines versus Temperature.

The effect of interstitial content on yield point stress appears to agree with the Cottrell-Bilby Theory[3] of dislocation locking by interstitially dissolved elements. Experimental results indicate that the locking effect of the interstitials varies linearly with their concentration and that a saturation limit within the range of interstitial contents investigated is not present.

The yield point stress is markedly increased by an increase in strain rate as exhibited by Lots 2M and 1WC shown in Figures 18, page 41 and 19, page 43. Kattus[16] observed an increase in yield point stress when testing at temperatures above room temperature.

The effects of strain rate on mechanical properties can be explained by considering the frequency at which thermal fluctuations occur. If the rate of strain is high, a higher stress level is attained before thermal fluctuations add to the stress level to break away dislocation loops, thus initiating yielding.

B. DUCTILITY RESULTS

The per cent elongation of pure tantalum decreases with decreasing temperature in the same manner as for single crystals of vanadium[33], polycrystalline vanadium[34], and niobium[37]. The transition from a ductile-to-brittle behaviour is what previous investigators[11, 12, 13, 38] have expected, but it has never been demonstrated. The absence of a ductile-to-less ductile transition can be explained by the fact that investigators took too few values between $0^{\circ}C$ and $-196^{\circ}C$. Another reason is that a brittle fracture was expected at low

temperatures instead of one showing a small amount of elongation.

The transition from a ductile-to-less ductile behaviour observed in this investigation is quite definite in the electron-beam melted material (Lots 1WC and 2WC). Lots 1M and 2M, which are vacuum-sintered material, also exhibit this transition but to a higher final elongation.

The materials with added amounts of nitrogen and oxygen do not show a distinct transition but rather a gradual decrease in per cent elongation as the temperature decreases. It is interesting to note that the per cent elongation at $-196^{\circ}C$ is greater for the materials with additions than for the pure material.

Lot 2WC shows less room temperature elongation than does Lot 1WC, but both exhibit the same elongation at $-196^{\circ}C$. This variance is attributed to the different interstitial content and the different grain size of the two materials.

The low per cent elongation of the purer material, i.e. Lots 1WC and 2WC, is contrary to observations on other very pure metals[37, 39] which, upon purification, exhibit greater ductility at low temperatures. This unusual behaviour may be understood in terms of the following discussion.

Examination of the fracture surfaces of specimens from Lots 1WC and 2WC, tested at temperatures near $-196^{\circ}C$, revealed that deformation took place in a narrow Luder's band and that a reduction in area of 80 to 90 per cent had taken place prior to failure; i.e. the elongation was not uniform. At room temperature the elongation took place uniformly, but in the transition temperature range the elongation took place in only a small part of the gauge length. The size of the deformed region decreased

as the temperature decreased. To explain this low temperature behaviour it is suggested that upon yielding, a large reduction in area takes place. This effect is much greater than the work hardening which takes place on deformation. The result of this effect is that the shear stress necessary for fracture is reached in this narrow band and failure occurs.

The fact that material with added interstitial content shows a relatively large elongation at low temperature is explained by considering the effect of the interstitials on the work hardening ability of the material. Since it is probable that the work hardening ability is increased with interstitial content, the behaviour can be explained by the following: On yielding, the greater work hardening ability of tantalum with added interstitials does not allow such a large initial reduction in area to take place on formation of the Lüder's band; some work hardening occurs to cause more elongation before failure occurs.

The shapes of load-elongation curves associated with tests of Lots 1WC and 2WC, some of which are shown in Figure 15, page 36, show very high sharp yield points, then a continued falling off of the load to failure. The curves for tests below $-20^{\circ}C$ show that the high sharp yield point is followed by a very flat, and sometimes decreasing, work hardening portion. The load never recovered to the value achieved at the yield point. The shape of these curves suggests that tantalum does not work harden; but since tantalum does work harden[40], the curves indicate that a large reduction in area takes place on yielding.

The shapes of load-elongation curves for low temperature tests on materials with added interstitial elements show

that the sharpness of the yield point and the associated load drop decreased with increasing interstitial content. The curves for tests conducted at temperatures near —20°C show that the load recovers to a value equal to or greater than the yield point load. These curves indicate that the interstitial content does increase the work hardening ability of the material as has been suggested. They also indicate that the reduction in area, which occurs on the formation of the Lüder's band, is not as large as that which occurs in the pure material.

The strain rate effect on elongation is shown in the results of Lots 1M, 2M, and 1WC (see Figures 16, 18, and 19; pages 38, 41, and 43 respectively). The transition becomes more abrupt and occurs at a higher temperature. There is no indication of any strain rate effect on elongation at —196°C. The strain rate effects may be explained in part by the frequency of thermal fluctuations and by the probability that a thermal fluctuation of sufficient energy to release a dislocation will occur in a small time increment related to the rate of strain. As the temperature decreases the probability that a fluctuation of sufficient energy will occur in this small increment of time decreases. Also as the strain rate increases, the time increment decreases. Therefore, on increasing the strain rate or decreasing the temperature, the probability of releasing dislocations decreases. Thus larger stresses in front of dislocation pile-ups are achieved before thermal fluctuations and short range diffusion can take place to relieve them, resulting in the nucleation of microcracks or other mechanisms to cause failure.

The elongation of the material with added hydrogen

exhibits a decrease from 23 per cent elongation at 100°C to a completely brittle failure at —60°C then recovers from —120°C to 5 per cent elongation at —196°C. This behaviour is similar to that of vanadium[41, 42]. The effect which has been termed hydrogen embrittlement. is thought to be due to the effect of hydrogen on the formation of cracks. Hydrogen is thought to diffuse to microcracks and to affect their growth at temperatures where brittle failure is exhibited, but at lower temperatures or high strain rate the hydrogen is unable to diffuse to the cracks and a ductile failure results. A complete coverage of this effect can be found in the report "Hydrogen Embrittlement in terms of Modern Theory of Fracture"[43].

The elongation results for Batches 1 and 2 do not show the agreement that is shown in the results for yield point stress. The elongation of the material with the larger grain size is considerably greater than that of the other. The analysis has shown that essentially they contain the same amount of nitrogen, oxygen, carbon, and hydrogen. Therefore, it is assumed that a different interstitial content is not the reason for the difference in elongation. The only explanation left to explain this difference is that it is a grain size effect. To explain this phenomena completely, more data are needed. Any theory must explain why the large grained material with added nitrogen exhibits greater elongation than fine grained material containing nitrogen, when the reverse is true of the two materials with a low nitrogen content.

VI SUMMARY AND CONCLUSIONS

1. The annealing of tantalum is a very critical operation since many of the metals and refractory materials used in furnace construction have appreciable vapour pressures at the temperatures required.
2. The mechanical properties of tantalum and tantalum with added interstitial elements exhibit a strong temperature dependence.
3. Nitrogen additions increase the yield stress of tantalum in a continuous manner in the temperature range investigated and in the range of nitrogen contents used.
4. Oxygen and hydrogen increase the yield strength of tantalum within the temperature range investigated.
5. The relative effects of the three interstitial elements are different within the temperature range studied.
6. The yield stress of tantalum is strain rate sensitive.
7. The transition from ductile-to-less ductile type of failure is present in the pure tantalum.
8. Tantalum with added interstitial elements exhibits less elongation at room temperature than the pure material, but decreases continuously to a higher elongation than the pure material at —196°C. A sharp transition is not present.
9. The form of the ductile-to-less ductile transition when found is strain rate sensitive.
10. The transition temperature is increased with increasing strain rate.
11. Yield point stress of pure tantalum exhibits a dependence on grain size.

VII RECOMMENDATIONS FOR FUTURE WORK

1. A lack of facilities to anneal in a hard vacuum at temperatures greater than 1100°C caused much difficulty. It is recommended therefore, that a molybdenum tube similar to the stainless steel tube used in this investigation be purchased.
2. The scatter of results indicates a non-uniform specimen. For future work a closer control on preparation should be used, i.e. annealing and shaping. Cylindrical specimens are recommended because more accuracy is achieved in the area measurement and a more uniform gauge length can be obtained.
3. The yield stress versus temperature plot of Lots 1WC and 2WC suggest the presence of a Snowball[33] anomaly. It is suggested that zone refined tantalum be used to determine the presence of an anomaly.
4. The data for material containing added hydrogen and oxygen are not complete. Further work should complete these data.
5. The effect of the addition of carbon was not studied. To complete this study, the effect of carbon should be investigated.
6. The indication of a drastic reduction in area occurring during yielding should be proven by halting the test as soon as yielding has taken place and measuring the cross-sectional area of the samples. Perhaps a different shape of specimen could also help to determine the events taking place to cause the reduction in the section of gauge length undergoing deformation.

7. The effect of grain size on the ductility of the material with nitrogen additions should be determined by the addition of various amounts of nitrogen to materials having various grain size.

VIII BIBLIOGRAPHY

1. Wessel, E.T., Abrupt Yielding and the Ductile-to-Brittle Transition in Body-Centered-Cubic Metals, J. Inst. Metals, 9 No. 7, July 1957, 930.

2. Overbach, B.L., D.K. Felbeck, G.T. Hahn, and D.A. Thomas (eds.), Fracture, Technology Press, New York, 1959.

3. Cottrell, A.H. and B.A. Bilby, Dislocation Theory of Yielding and Strain Ageing in Iron, Proceedings Royal Soc., London, A62, 1949, 49.

4. Fisher, J.C., Application of Cottrell's Theory of Yielding to Delay Yield in Steel, A.S.M. Trans., 47, 1955, 451.

5. Cottrell, A.H., Dislocations and Plastic Flow in Crystals, Oxford Press, Clarendon England, 1953.

6. Vreeland, T. Jr., D.S. Wood, and D.S. Clark, A Study of the Mechanism of the Delayed Yield Phenomenon, A.S.M. Trans., 45, 1953, 620.

7. Griffith, A.A., Theory of Rupture, Philosophical Trans., Royal Soc., London, A 221, 1920, 163.

8. Zener, C., and J.H. Holloman, Effect of Strain Rate upon Plastic Flow of Steel, J. Applied Physics, 15, 1944, 22.

9. Bechtold, J.H., and P.G. Shewmon, Flow and Fracture Characteristics of Annealed Tungsten, A.S.M. Trans, 46, 1954, 397.

10. Clough, W.R., and A.S. Pavlovic, The Flow, Fracture, and Twinning of Commercially Pure Vanadium, A.S.M. Trans., Preprint 52, 1958.

11. Bechtold, J.H., Tensile Properties of Annealed Tantalum at Low Temperatures, Acta Metallurgica, 3, 1944, 249.

12. Pugh, J.W., Temperature Dependence of Annealed Tantalum, A.S.M. Trans. 48, 1956, 677.

13. Schussler, M. and J.S. Brunhouse Jr., Mechanical Properties of Tantalum Consolidated by Melting, A.I.M.E. Trans., 218, October 1960, 893.

14. Ingram, A.G., et.al., Tantalum and Tantalum Alloys, D.M.I.C. Report 133, July 25, 1960, 215.

15. Barrett, C.S., Metallurgy at Low Temperatures, A.S.M. Trans., 49, 1957, 69.

16. Tietz, T.E., B.A. Wilcox, J.W. Wilson, Mechanical Properties and Oxidation Resistance of Certain Refractory Metals, Stanford Research Institute Report, SRI Project SU - 2436, 1958.

17. Miller, G.L., Tantalum and Niobium, Butterworths Scientific Publications, London, 1958, p. 406.

18. Schmidt, F.F., Tantalum and Tantalum Alloys, DMIC Report 133, July 25, 1960, 215.

19. Ibid., p 197.

20. Ibid., p 197.

21. Ibid., p 197.

22. Ibid., p 197.

23. Ibid., p 197.

24. Myers, R.H., Some Properties of Tantalum-Rich Alloys with Wolfram and Molybdenum, Metallurgia 42 (6), 1950, 3.

25. Perkins, R.H., Tantalum Annealing and Degassing and Hardness Effects of Dissolved Gases, Los Alamos Scientific Laboratory Report, LA - 2136.

26. Miller, G.L., Tantalum and Niobium, Butterworths Scientific Publications, London, 1958, p. 446.

27. Ibid., p 456.

28. Ibid., p 497.

29. Ibid., p 498.

30. Bakish, R., Some Observations on the Effect of the Interaction of Tantalum with Oxygen, Nitrogen and Hydrogen, J. of the Electrochemical Society 105 No. 10, October 1958, 574.

31. Seghezzi, H.D., E. Gebhardt and W. Durrschnabel, New Investigations into the Tantalum-Nitrogen System, 3rd Plansee Seminar, Reutte, Austria, 1958, 291.

32. Gebhardt, E., and H.D. Seghezzi, New Investigations into the Tantalum-Oxygen System, 3rd Plansee Seminar, Reutte, Austria, 1958, 280.

33. Snowball, R.F., M.A. Sc. Thesis submitted in the Department of Mining and Metallurgy, University of British Columbia, October 1960.

34. Fraser, R.W., M.A. Sc. Thesis submitted in the Department of Mining and Metallurgy, University of British Columbia, November, 1960.

35. Miller, G.L., Tantalum and Niobium, Butterworths Scientific Publications, London 1958, p. 454.

36. Operating Instructions for the Instron Tensile Tester, Manual No. 155, Instron Engineering Corporation, Canton, Massachusetts, 17.

37. Mincher, A.L. and W.F. Sheely, Effect of Structure and Purity on the Mechanical Properties of Columbium, A.I.M.E. Trans. 221 No. 1, February 1961, 19.

38. Wessel, E.T., Some Exploratory Observations of the Tensile Properties of Metals at Very Low Temperatures, A.S.M. Trans., 49, Preprint No. 3, 1956.

39. Smith, R.L. and J.L. Rutherford, Tensile Properties of Zone Refined Iron in the Temperature Range from 298° to 4.2°K., J. of Metals 9 No. 7, July 1957, 857.

40. Miller, G.L., Tantalum and Niobium, Butterworths Scientific Publications, London 1958, p. 420.

41. Roberts, B.W. and H.C. Rogers, Observations of Hydrogenated Vanadium, A.I.M.E. Trans., 206, October 1956, 1213.

42. Loomis, B.A. and O.N. Carlson, Investigation of the Ductile-to-Brittle Transition in Vanadium, Paper Presented at the Reactive Metals Conference, May 27-29, 1958.

43. Blanchard, P.A., and A.R. Troiano, Hydrogen Embrittlement in Terms of Modern Theory of Fracture, WADC Technical Report 59-444, August 1959.

APPENDIX A

TABLE VII

THE RESULTS OF TENSILE TESTS ON TANTALUM LOT 1M

Test No.	Temp. °C	Ultimate Tensile Stress x 10^3 psi.	Upper Yield Stress x 10^3 psi.	Strain Rate in/in/min.	Percent Elongation
1	21	56.0	47.4	.067	29.2
2	21	58.6	50.4	.067	27.2
3	21	63.2	55.0	.067	28.1
4	21	54.3	47.0	.067	27.0
5	7	58.0	50.0	.067	
6	7	60.0	49.0	.067	-
7	0	59.5	48.7	.067	22.8
8	0	60.0	52.2	.067	25.2
9	0	59.7	48.6	.067	28.0
10	0	58.8	51.8	.067	-
11	-12	59.8	52.2	.067	26.5
12	-12	63.4	53.8	.067	28.4
13	-43	64.2	58.3	.067	29.4
14	-43	64.5	61.6	.067	24.6
15	-50	67.0	61.6	.067	29.8
16	-55	72.8	65.0	.067	30.2
17	-55	70.6	64.0	.067	23.4
18	-68	77.0	64.8	.067	25.6
19	-68	77,8	70.0	.067	30.4
20	-98	80.4	78.8	.067	24.6
21	-98	84.6	79.6	.067	22.8
22	-138	91.7	91.7	.067	23.0
23	-138	96.6	92.5	.067	24.5
24	-195	119.0	119.0	.067	5.0
25	-195	132.0	132.0	.067	8.6
26	-195	134.2	134.2	.067	11.4
27	-175	114.0	114.0	.067	17.8
28	-175	114.0	114.0	.067	14.2
29	-195	116.0	116.0	.067	8.0
30	-195	115.0	115.0	.067	6.8
31	-184	118.2	118.2	.067	8.34
32	-184	120.0	120.0	.067	8.34
33	-195	-	-	26.7	4.18
34	-195	-	-	26.7	3.75
35	-175	-	-	26.7	4.18
36	-175	-	-	26.7	4.18
37	23	-	-	26.7	22.1
38	23	-	-	26.7	25.0
39	11	-	-	26.7	20.4
40	11	-	-	26.7	20.8
41	- 5	-	-	26.7	25.0
42	- 5	-	-	26.7	26.8
43	- 19	-	-	26.7	21.2
44	- 19	-	-	26.7	24.2

Test No.	Temp. °C	Ultimate Tensile Stress x 10^3 psi.	Upper Yield Stress x 10^3 psi.	Strain Rate in./in./min.	Percent Elongation
45	- 35	-	-	26.7	20.0
46	- 35	-	-	26.7	22.9
47	- 50	-	-	26.7	18.8
48	- 50	-	-	26.7	22.0
49	- 74	-	-	26.7	14.6
50	- 74	-	-	26.7	8.75
51	- 80	-	-	26.7	8.34
52	- 80	-	-	26.7	8.34
53	- 95	-	-	26.7	7.5
54	- 95	-	-	26.7	7.5
55	-110	-	-	26.7	7.3
56	-110	-	-	26.7	7.3
57	-125	-	-	26.7	7.5
58	-184	-	-	26.7	6.25
59	-184	-	-	26.7	4.17
60	15	-	-	26.7	25.0
61	15	-	-	26.7	20.8
62	10	-	-	26.7	23.0
63	10	-	-	26.7	25.0
64	5	-	-	26.7	25.0
65	- 60	-	-	26.7	17.3
66	- 60	-	-	26.7	15.0
67	-140	-	-	26.7	5.2
68	-140	-	-	26.7	8.0
		-			

APPENDIX B

TABLE VIII

THE RESULTS OF WORK HARDENING AND AGING TEST

Test No.	Temp. °C	Ultimate Tensile Stress x 10^3 psi.	Upper Yield Stress x 10^3 psi.	Strain Rate in/in/min.	Percent Elongation	Percent Cold Work	Aging Temp. °C for 1 Hour
1	21	67.4	66.0	.067	8.4	10	-
2	21	79.2	77.2	.067	1.4	25	-
3	-195	145.2	137.2	.067	5.2	10	-
4	-195	145.5	138.0	.067	6.2	10	-
5	-195	165.0	153.0	.067	5.6	25	-
6	-195	159.0	159.0	.067	4.4	25	-
7	-195	161.5	149.0	.067	2.4	40	-
8	-195	156.5	144.0	.067	2.6	40	-
9	-195	160.5	149.0	.067	2.0	50	-
10	-195	164.3	156.0	.067	5.2	50	-
11	-195	138.5	138.5	.067	7.2	10	700
12	-195	140.5	140.5	.067	5.6	10	700
13	-195	151.3	149.5	.067	5.0	25	700
14	-195	149.5	149.5	.067	6.3	25	700
15	-195	136.0	136.0	.067	4.4	10	400
16	-195	133.5	133.5	.067	5.4	10	400
17	21	73.0	70.4	.067	4.2	10	400
18	21	93.8	91.6	.067	1.0	25	400
19	-195	145.5	145.5	.067	3.2	25	400
20	-195	142.0	142.0	.067	3.4	25	400
21	23	96.5	96.5	.067	1.2	25	300
22	23	93.5	93.5	.067	1.2	25	300
23	23	78.2	78.2	.067	6.3	10	300
24	23	70.7	70.2	.067	8.3	10	300
25	-195	148.0	148.0	.067	6.3	10	300
26	-195	149.0	149.0	.067	4.1	10	300
27	-195	167.0	167.0	.067	4.1	25	300
28	-195	158.5	158.5	.067	3.4	25	300

APPENDIX C

TABLE IX

THE RESULTS OF TENSILE TESTS ON TANTALUM LOT 2M

Test No.	Temp. °C	Ultimate Tensile Stress x 10^3 psi.	Upper Yield Stress x 10^3 psi.	Strain Rate in./in./min.	Percent Elongation
1	21	68.8	63.2	.0267	29.2
2	21	71.6	62.8	.0267	30.2
3	21	78.8	72.2	.0267	25.0
4	21	79.2	71.7	.0267	23.5
5	0	80.8	71.7	.0267	27.2
6	0	80.0	71.0	.0267	31.2
7	- 20	94.0	87.8	.0267	27.1
8	- 20	95.0	82.2	.0267	27.1
9	- 20	91.6	84.0	.0267	27.1
10	- 40	89.0	88.2	.0267	27.1
11	- 40	81.0	87.7	.0267	27.1
12	- 60	92.2	85.0	.0267	26.0
13	- 60	83.5	81.7	.0267	33.4
14	- 60	93.0	84.7	.0267	33.4
15	- 80	91.8	87.2	.0267	29.2
16	- 80	90.5	87.7	.0267	31.4
17	-100	97.4	97.4	.0267	29.2
18	-100	99.0	98.0	.0267	27.0
19	-120	109.7	109.7	.0267	25.0
20	-120	108.0	107.0	.0267	16.7
21	- 10	86.7	76.0	.0267	25.0
22	- 20	84.0	75.5	.0267	27,8
23	- 20	81.7	74.6	.0267	27.8
24	- 20	84.2	74.6	.0267	25.0
25	- 20	84.2	76.8	.0267	27.8
26	- 30	84.2	76.6	.0267	-
27	- 30	88.2	78.8	.0267	25.0
28	-195	156.0	156.0	.0267	10.0
29	-195	161.8	161.8	.0267	10.4
30	-195	158.0	158.7	.0267	10.0
31	-195	145.0	145.0	.0267	10.4
32	-195	147.2	147.2	.0267	9.4
33	-195	149.0	149.0	.0267	10.4
34	-195	161.0	161.0	.0267	9.4
35	21	88.6	83.0	2.67	26.5
36	21	87.0	82.5	2.67	25.0
37	0	88.4	83.4	2.67	25.0
38	0	88.4	82.4	2.67	25.0
39	- 20	89.6	89.6	2.67	18.8
40	- 20	90.2	82.0	2.67	24.0
41	- 20	88.0	83.6	2.67	31.0
42	- 40	90.8	90.0	2.67	24.0
43	- 40	93.0	88.0	2.67	23.0
44	- 60	88.5	88.5	2.67	20.8

Test No.	Temp. °C	Ultimate Tensile Stress x 10^3 psi	Upper Yield Stress x 10^3 psi.	Strain Rate in./in./min.	Percent Elongation
45	- 60	94.0	92.5	2.67	25.0
46	- 60	93.0	89.5	2.67	25.0
47	- 60	92.0	-	2.67	25.0
48	- 80	107.0	107.0	2.67	27.2
49	- 80	107.5	107.0	2.67	26.1
50	- 80	100.2	100.2	2.67	26.0
51	-100	116.5	116.5	2.67	16.7
52	-100	111.0	111.0	2.67	20.8
53	-100	106.0	106.0	2.67	23.0
54	-100	115.0	115.0	2.67	12.5
55	-120	118.8	118.8	2.67	14.6
56	-120	118.0	118.0	2.67	12.5
57	-130	121.0	-	2.67	12.5
58	-130	122.5	122.5	2.67	12.0
59	-130	128.0	128.0	2.67	14.6
60	-195	155.0	155.0	2.67	9.4
61	-195	161.0	161.0	2.67	9.4
62	-195	157.2	157.2	2.67	9.4
63	-195	163.2	163.2	2.67	9.4

APPENDIX D

TABLE X

THE RESULTS OF TENSILE TESTS ON TANTALUM LOT 1WC

Test No.	Temp. °C	Ultimate Tensile Stress x 10^3 psi	Upper Yield Stress x 10^3 psi.	Strain Rate in./in./min.	Percent Elongation
1	100	30.2	25.4	.0267	44.0
2	50	30.5	24.8	.0267	46.0
3	22	33.7	31.8	.0267	40.5
4	22	32.2	26.5	.0267	39.0
5	22	31.9	25.2	.0267	42.0
6	22	33.9	23.8	.0267	42.0
7	0	35.6	30.8	.0267	41.5
8	0	36.1	34.4	.0267	40.0
9	0	38.1	38.1	.0267	41.0
10	0	39.5	39.5	.0267	40.0
11	- 20	37.1	37.1	10267	36.0
12	- 20	36.9	38.2	.0267	46.0
13	- 20	42.8	42.8	.0267	37.5
14	- 20	37.2	36.5	.0267	40.0
15	- 20	35.7	35.0	.0267	30.5
16	- 20	35.7	35.7	.0267	-
17	- 35	43.0	43.0	.0267	35.5
18	- 35	43.0	43.0	.0267	34.0
19	- 40	46.2	46.2	.0267	32.5
20	- 40	44.4	44.4	.0267	41.5
21	- 45	40.5	40.5	.0267	35.5
22	- 45	43.2	43.2	.0267	37.0
23	- 45	44.0	44.0	.0267	33.0
24	- 45	41.5	41.5	.0267	-
25	- 50	42.2	42.0	.0267	29.0
26	- 50	46.3	46.3	.0267	36.0
27	- 50	47.4	47.4	.0267	29.0
28	- 55	52.5	52.5	.0267	16.0
29	- 55	47.0	47.0	.0267	-
30	- 55	53.0	53.0	.0267	-
31	- 55	48.7	48.7	.0267	-
32	- 60	47.0	47.0	.0267	24.0
33	- 60	47.8	47.8	.0267	32.5
34	- 60	50.4	50.4	.0267	32.0
35	- 70	47.2	47.2	.0267	22.0
36	- 70	54.3	53.6	.0267	12.7
37	- 70	50.2	50.2	.0267	-
38	- 80	51.6	51.6	.0267	20.0
39	- 80	49.0	49.0	.0267	8.0
40	- 90	59.0	59.0	.0267	12.7
41	- 90	54.0	54.0	.0267	13.3
42	-100	57.6	57.6	.0267	9.0
43	-100	58.8	58.8	.0267	-
44	-100	66.2	66.2	.0267	4.5

THE RESULTS OF TENSILE TESTS ON TANTALUM LOT IWC

Test No.	Temp. °C	Ultimate Tensile Stress x 10^3 psi.	Upper Yield Stress x 10^3 psi.	Strain Rate in./in./min.	Percent Elongation
45	-120	68.5	68.5	.0267	4.5
46	-120	65.5	65.5	.0267	-
47	-120	70.8	70.8	.0267	3.0
48	-132	72.2	72.2	.0267	2.5
49	-140	69.6	69.6	.0267	2.0
50	-140	85.0	85.0	.0267	2.0
51	-195	107.0	107.0	.0267	<1
52	-195	108.5	108.5	.0267	<1
53	100	35.0	35.0	2.67	-
54	22	38.0	38.0	2.67	46.0
55	22	36.2	36.2	2.67	39.0
56	0	41.8	41.8	2.67	-
57	0	41.7	41.7	2.67	35.0
58	- 20	49.8	49.8	2.67	10.0
59	- 20	55.8	55.8	2.67	3.0
60	- 40	55.5	55.5	2.67	-
61	- 40	56.0	56.0	2.67	6.0
62	- 60	58.8	58.8	2.67	-
63	- 60	60.5	60.5	2.67	<1
64	- 80	66.7	66.7	2.67	<1
65	- 80	64.4	64.4	2.67	<1
66	-120	78.0	78.0	2.67	<1
67	-120	79.8	79.8	2.67	<1
68	-195	105.0	105.0	2.67	<1
69	-195	107.0	107.0	2.67	<1
70	-195	113.0	113.0	2.67	<1

APPENDIX E

TABLE XI

THE RESULTS OF TENSILE TESTS ON TANTALUM LOT 2WC

Test No.	Temp. °C	Ultimate Tensile Stress x 10^3 psi.	Upper Yield Stress x 10^3 psi.	Strain Rate in./in./min.	Percent Elongation
1	100	40.7	28.8	.0267	25.5
2	50	38.8	31.8	.0267	33.5
3	22	44.8	36.2	.0267	28.0
4	0	47.3	37.6	.0267	33.0
5	- 20	50.2	50.2	.0267	29.0
6	- 40	52.2	51.5	.0267	29.0
7	- 60	63.6	63.6	.0267	21.5
8	- 80	64.7	64.7	.0267	17.5
9	-100	70.0	70.0	.0267	11.0
10	-100	68.3	68.3	.0267	9.0
11	-120	90.4	90.4	.0267	13.5
12	-126	79.4	79.4	.0267	7.0
13	-140	82.2	82.2	.0267	6.0
14	-195	122.5	122.5	.0267	<1
15	-195	129.0	129.0	.0267	<1

APPENDIX F

TABLE XII

THE RESULTS OF TENSILE TESTS ON TANTALUM LOT IWC

BATCH 1

Test No.	Temp. °C	Ultimate Tensile Stress x 10^3 psi.	Upper Yield Stress x 10^3 psi.	Strain Rate in/in/min.	Percent Elongation
1	100	85.8	80.4	.0267	16.2
2	22	99.4	98.2	.0267	12.5
3	0	112.0	109.0	.0267	10.5
4	- 20	103.2	102.5	.0267	11.5
5	- 40	121.0	121.0	.0267	8.0
6	- 50	126.5	126.5	.0267	-
7	- 70	121.0	121.0	.0267	8.5
8	- 90	136.5	136.5	.0267	8.5
9	-110	145.0	145.0	.0267	2.0
10	-130	151.5	151.5	.0267	4.0
11	-195	208.0	208.0	.0267	2.0
12	-195	199.0	199.0	.0267	3.5

TABLE XIII

THE RESULTS OF TENSILE TESTS ON TANTALUM LOT 2WC

BATCH 2

Test No.	Temp. °C	Ultimate Tensile Stress X 10^3 psi.	Upper Yield Stress X 10^3 psi.	Strain Rate in/in/min.	Percent Elongation
1	100	79.8	76.5	.0267	23.0
2	50	92.3	90.1	.0267	20.0
3	22	102.0	102.0	.0267	24.0
4	- 60	131.5	131.5	.0267	20.0
5	- 60	124.0	124.0	.0267	21.0
6	- 80	138.0	138.0	.0267	-
7	-107	140.0	140.0	.0267	14.5
8	-120	149.4	149.4	.0267	18.5
9	-140	167.5	167.5	.0267	13.5
10	-195	204.0	204.0	.0267	8.5
11	-195	200.0	200.0	.0267	8.5

TABLE XIV

THE RESULTS OF TENSILE TESTS ON TANTALUM LOT 2WC

BATCH 3

Test No.	Temp. °C	Ultimate Tensile Stress x 10^3 psi.	Upper Yield Stress x 10^3 psi.	Strain Rate in/in/min.	Percent Elongation
1	22	45.5	44.6	.0267	24.0
2	0	53.5	53.5	.0267	18.0
3	- 20	60.0	60.0	.0267	18.0
4	- 40	61.5	61.5	.0267	26.0
5	- 60	61.0	61.0	.0267	13.0
6	- 80	71.7	71.7	.0267	12.0
7	-100	74.5	74.5	.0267	8.5
8	-103	78.2	78.2	.0267	9.0
9	-120	92.5	92.5	.0267	6.0
10	-132	-	-	.0267	8.5
11	-136	89.3	89.3	.0267	6.0
12	-195	122.0	122.0	.0267	2.0

TABLE XV

THE RESULTS OF TENSILE TESTS ON TANTALUM LOT 2WC

BATCH 4

Test No.	Temp. °C	Ultimate Tensile Stress x 10^3 psi.	Upper Yield Stress x 10^3 psi.	Strain Rate in/in/min.	Percent Elongation
1	100	46.5	39.6	.0267	26.0
2	51	50.6	48.4	.0267	26.0
3	22	49.4	43.2	.0267	30.5
4	0	50.0	47.7	.0267	30.0
5	- 20	53.2	53.2	.0267	25.0
6	- 40	60.5	60.5	.0267	21.0
7	- 60	67.3	67.3	.0267	14.5
8	- 80	73.4	73.4	.0267	17.5
9	-100	87.2	87.2	.0267	8.0
10	-100	87.8	87.8	.0267	15.0
11	-120	89.3	89.3	.0267	9.0
12	-133	96.0	96.0	.0267	8.0
13	-137	94.4	94.4	.0267	6.5
14	-195	131.5	131.5	.0267	2.0

TABLE XVI

THE RESULTS OF TENSILE TESTS ON TANTALUM LOT 2WC

BATCH 5

Test No.	Temp. °C	Ultimate Tensile Stress x 10^3 psi.	Upper Yield Stress x 10^3 psi.	Strain Rate in./in./min.	Percent Elongation
1	100	75.5	69.0	.0267	21.5
2	58	100.5	75.6	.0267	21.5
3	22	81.4	81.4	.0267	20.5
4	22	82.2	82.2	.0267	22.0
5	0	90.4	90.4	.0267	18.0
6	- 20	94.4	94.4	.0267	15.0
7	- 40	100.3	100.3	.0267	17.0
8	- 60	111.5	111.5	.0267	-
9	- 60	116.0	116.0	.0267	17.0
10	- 80	114.0	114.0	.0267	13.5
11	-100	124.7	124.7	.0267	8.5
12	-120	134.2	134.2	.0267	10.5
13	-136	145.0	145.0	.0267	13.5
14	-195	184.0	184.0	.0267	9.0
15	-195	184.3	184.3	.0267	10.5

TABLE XVII

THE RESULTS OF TENSILE TESTS ON TANTALUM LOT 2WC

BATCH 6

Test No.	Temp. °C	Ultimate Tensile Stress x 10^3 psi.	Upper Yield Stress x 10^3 psi.	Strain Rate in./in./min.	Percent Elongation
1	100	53.9	52.5	.0267	20.0
2	50	47.7	42.2	.0267	24.0
3	22	46.5	44.0	.0267	19.0
4	0	52.2	52.2	.0267	22.0
5	- 20	57.7	53.8	.0267	23.0
6	- 40	66.2	66.2	.0267	19.0
7	- 60	70.7	70.7	.0267	17.0
8	- 80	69.4	69.4	.0267	17.0
9	-100	81.6	81.6	.0267	10.5
10	-124	93.7	93.7	.0267	11.5
11	-140	96.2	96.2	.0267	12.0
12	-195	131.2	131.2	.0267	3.0

TABLE XVIII

THE RESULTS OF TENSILE TESTS ON TANTALUM LOT 2WC

BATCH 7

Test No.	Temp. °C	Ultimate Tensile Stress x 10^3 psi.	Upper Yield Stress x 10^3 psi.	Strain Rate in/in/min.	Percent Elongation
1	100	57.4	52.4	.0267	18.5
2	50	68.6	63.6	.0267	16.0
3	22	68.7	66.0	.0267	20.5
4	- 20	75.5	75.5	.0267	20.5
5	- 40	83.4	81.7	.0267	19.0
6	- 60	88.0	88.0	.0267	19.0
7	- 80	90.4	90.4	.0267	16.0
8	-105	107.2	107.2	.0267	12.0
9	-120	106.0	106.0	.0267	6.0
10	-140	108.5	108.5	.0267	7.0

APPENDIX G

TABLE XIX

THE RESULTS OF TENSILE TESTS ON TANTALUM LOT 2WC

BATCH 8

Test No.	Temp. °C	Ultimate Tensile Stress x 10^3 psi.	Upper Yield Stress x 10^3 psi.	Strain Rate in./in./min.	Percent Elongation
1	104	117.0	84.4	.0267	15.0
2	51	99.7	99.2	.0267	14.0
3	22	98.8	98.8	.0267	16.5
4	0	97.5	97.5	.0267	16.0
5	- 20	104.2	104.2	.0267	14.5
6	- 40	110.5	110.0	.0267	20.0
7	- 60	119.0	119.0	.0267	10.0
8	- 80	112.0	112.0	.0267	4.5
9	-100	126.5	126.5	.0267	4.0
10	-119	141.5	141.5	.0267	3.0
11	-138	153.5	153.5	.0267	2.5
12	-195	200.0	200.0	.0267	5.0

APPENDIX H

TABLE XX

THE RESULTS OF TENSILE TESTS ON TANTALUM LOT 2WC

BATCH 9

Test No.	Temp. °C	Ultimate Tensile Stress x 10^3 psi.	Upper Yield Stress x 10^3 psi.	Strain Rate in./in./min.	Percent Elongation
1	103	43.7	28.3	.0267	23.0
2	22	40.2	40.2	.0267	5.0
3	- 23	46.4	46.4	.0267	1.0
4	- 60	53.2	53.2	.0267	0
5	- 80	60.7	60.7	.0267	0
6	-105	71.3	71.3	.0267	0
7	-131	80.8	80.8	.0267	2.0
8	-195	135.5	135.5	.0267	3.0