

AN ABSTRACT OF THE THESIS OF

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Title System of Zirconium-Tantalum Alloys--Zirconium Rich _____
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The purpose of the thesis project was to make a preliminary investigation of the zirconium rich portion of the zirconium-tantalum system.

The alloys of zirconium and tantalum have several possibilities such as superior corrosion resistance, excellent workability, formation of heat treatable alloys, high temperature properties, and outstanding physical properties.

Tantalum and zirconium are similar in certain respects such as corrosion resistance, workability, susceptibility to oxidation at high temperatures, and high melting points but vary considerably in properties such as density, atomic weight, position in the periodic table, and crystal structure at ambient temperature.

Zirconium experiences a phase change from hexagonal to body centered cubic at 862C. Tantalum is body centered cubic at room temperature and is not believed to be allotropic. According to theories postulated by Hume-Rothery and others, conditions are favorable for solid solution formation at temperatures above the transformation temperature of zirconium but discouraged at low temperatures.

Methods of analysis used in the project included, thermo-analysis, dilatometry, X-Ray analysis, microscopic observation and hardness. Physical properties and heat treating characteristics of the 15% tantalum alloy were also determined in order to obtain an indication of physical properties of the alloys.

The strong affinity for oxygen at elevated temperatures of each of the metals makes it necessary that all heat treating operations be carried on in a vacuum or protective atmosphere.

A limited amount of alloys with 1, 5, 10, 15, 20, 30, and 40% tantalum were available for laboratory tests.

Metallographic and X-Ray specimens were heat treated at a variety of temperatures in order to identify the phases at various temperature levels.

The differential thermal analysis attempted in the project was not particularly successful. Dilation analysis was a very satisfactory method of analysis for the alloy system but insufficient alloy stock plus a shortage of time prevented

extensive investigation with the dilatometer.

From the results obtained by the various methods of analysis several tentative conclusions may be drawn concerning the alloy system of zirconium-tantalum.

A phase change exists at 790°C for all alloys between 5 and 15% tantalum and may extend to greater limits. A second phase change is thought to exist at 910 C. An eutectoid exists between 10 and 15% tantalum which is thought to freeze at the transformation temperature of 790°C.

The solubility of tantalum in beta zirconium at 1100 exceeds 30% but a heterogeneous field exists at 950°C and 30% tantalum. The solubility of tantalum in alpha zirconium under equilibrium conditions is less than 1% at room temperature but increases with temperature as evidenced by the age hardening of the 15% tantalum alloy.

Hardness of the alloys are improved by quenching from the beta solid solution field. The tensile strength of 15% tantalum is improved over that of the annealed state by quenching. The tensile strength is further improved by aging the quenched alloy at 500C for 4 hours. The strength of the quenched and aged alloy reaching an ultimate of 189,000 psi as compared to 91,000 psi for the annealed state.



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WILL BROWN Paper

SYSTEM OF ZIRCONIUM-TANTALUM ALLOYS
ZIRCONIUM RICH PORTION

by

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SYSTEM OF ZIRCONIUM-TANTALUM ALLOYS
Zirconium Rich Portion

INTRODUCTION

Relatively little is known concerning the nature of the tantalum-zirconium system and the nature of the alloys resulting from the combination of the two metals. It is the purpose of this work to make the preliminary investigation of the zirconium-rich portion of the system in order that a completed constitution diagram will eventually be established and the nature of the alloys fully understood.

Tantalum and zirconium each possess outstanding properties which make them applicable for specific uses. The melting point of tantalum is extremely high and the corrosion resistance of either metal is almost without equal. The allotropic transformation from hexagonal to body centered cubic in zirconium makes plausible the possibility of increasing the already attractive strength and hardness of each metal by heat treating the alloys of the elements.

It is generally known that tantalum is among the most ductile metals and the workability of pure zirconium, even in hexagonal state, approaches that of tantalum.

Both tantalum and zirconium suffer from the disadvantage of readily combining with oxygen at high temperature.

It was hoped that the alloying of the two metals might form an oxidation resistant solid solution or stabilize the beta (body-centered cubic) form of zirconium at room temperature which would not be attacked by oxygen at elevated temperatures. The stabilization of the beta phase of zirconium would also improve the bending and forming properties of zirconium.

The alloying of zirconium then has the following possibilities;

1. Improve the already excellent corrosion resistance of each metal.
2. Form solid solutions resistant to oxidation.
3. Stabilize the cubic form of zirconium at room temperature to improve bending and forming properties and oxidation resistance.
4. Improve physical properties of strength, hardness, wear resistance and other related properties.
5. Form a phase stable through a wide range of temperatures thus retaining its strength over this temperature scale in order that the alloy will have useful high temperature properties.
6. Form a heat treatable alloy.

It is apparent then that the alloys possess a rather wide range of possibilities.

It is obvious that the most satisfactory approach to an investigation and understanding of the properties of a series of alloys of two elements is the construction of the constitution diagram of the alloy system. This explains the reason for the investigation of the constitution of the alloys being chosen as the initial point of attack for the study of the alloys.

Previous study of the alloy system has been hindered by the scarcity and cost of each of the elements. Zirconium is now produced by Foote Mineral Company and The United States Bureau of Mines and considerable work is being done on the alloys of zirconium at this time.

The Fansteel Metallurgical Corporation is the sole producer of tantalum at the present and if any work has been or is being done on the alloys of tantalum and zirconium by that company or any other company, the work has not been published.

APPARATUS AND PROCEDURE

Although a survey of the literature indicates that there has been no previous work on the system of tantalum and zirconium, an indication of the procedure necessary to make the proposed study of the two elements can be

obtained by examination of the properties of each metal.

Tantalum and zirconium are similar in certain respects such as workability, corrosion resistance, susceptibility to oxidation at high temperatures, and high melting points; however the two metals are quite dissimilar in many of their properties.

Table 1 shows the various properties of each of the elements.

The melting point of zirconium is given as $1750 \pm 700^\circ\text{C}$ in the Metal Handbook^{1*}; however studies by Fast⁸ indicate that the melting temperature is 1860°C .

Not only do tantalum and zirconium readily form oxides at elevated temperature but each has an affinity for carbon and TaC and ZrC are believed to form a complete series of solid solutions.^{7,11}

Theory

Any indication as to the type of diagram expected from the combination of the two metals would obviously be a great help in outlining a procedure for the study of the system.

According to a hypothesis put forward by Hume-Rothery¹⁰ and others, an extended solid solution field

*References appear in the Bibliography at the end of this thesis.

Table I. Properties of Zirconium and Tantalum

<u>Property</u>	<u>Zirconium</u>	<u>Tantalum</u>
Atomic weight ^{#5}	91.22	180.88
Atomic number	40	73
Valence	4	3 and 5
Group in periodic table	4	5
Density	6.4	16.5
Crystal structure ^{#3}	Alpha HCP Beta BCC (Above 862e)	BCC
Lattice Dimensions ^{#3}	Alpha $a=3.223$ $c=5.123$ Beta $a=3.61$	$a=3.296$

can be expected if the atomic diameters of the solute and solvent do not vary by more than 15%. If the atomic diameters vary by more than this amount, the "size factor" is said to be unfavorable and the solid solution is very restricted. The solid solution formation, with a favorable size factor, is subject to certain other restrictions imposed by valencies of the solute and solvent atoms, type of crystal structure, and position in the periodic table.

According to calculations and postulations made by Meyers¹², the size factor is favorable for an extended solid solution phase in the system of tantalum and zirconium; however as has been previously mentioned, the elements occupy positions in different groups of the periodic table and the crystal structure is different at room temperature.

Harrington⁹ indicates that the type of valence bonding existing between tantalum and zirconium makes the metals compatible in that respect.

Thus in the system of tantalum and zirconium two of four important factors necessary for solid solution formation are favorable while the other two are unfavorable.

Weighing the evidence presented by Harrington and Meyers, the only prediction as to the type of system expected would be that solubility to some extent is

favorable, eutectic reaction probable, with a peritectic type reaction possible.

Methods of Analysis

Of the various possible methods of analysis employed in phase-diagram work, Barrett³ has the following to say:

"Thermal analysis (the use of heating and cooling curves) is particularly valuable for determining the beginning and end of freezing. Dilatometric measurements are excellent for determining the presence of transformations in the solid state. Microscopic observations of polished and etched sections are often sufficient to establish all phases and transformations in the solid state and are more useful than electrical, magnetic, or other physical properties in determining the boundaries of the single and polyphase regions in the reaction, which none of the other methods can do, and makes it possible to determine the nature of most of the high temperature reactions by inference from the microstructures they produce. X-Ray diffraction is capable of determining not only the phases present in an alloy but also the crystal structure of the phases. It excels the other methods in its ability to detect metastable and transitional states and to relate these to the phases

preceding and following them in a transformation permitting in some cases a detailed understanding of the mechanism of transformation and of the strains accompanying it. A diffraction pattern of a polyphase alloy can be made to yield, by comparison with alloys of known compositions, the compositions of the individual phases present, since the spectra of the phases are superimposed in the diffraction pattern for the aggregate.

Conversely we may summarize the principal limitations of these methods as follows: Thermal analysis is a poor method of studying sluggish reactions; the heat of reaction is evolved over such an extended period of time that it is difficult to detect or measure. This method is also likely to fail if the heat of reaction is small. Dilatometric curves are sometimes better than cooling curves for studies of transformations in the solid state, but neither thermal nor dilatometric data are suitable for determining the range of composition of single-phase or polyphase regions in the solid state. Mechanical strength is a good method for determining the beginning of melting and is coming into wide use for this purpose, but otherwise the mechanical methods find little service in phase-diagram work. The valuable microscopic method runs into difficulties when the grain size of a sample is too small or when the products of a reaction

are too finely dispersed. This is likely to occur with reactions in the solid state that take place at low temperatures. It depends upon room-temperature observations and consequently gives only indirect evidence of phases that are not retained by quenching to room temperatures. The microscopic method fails in occasional instances for lack of suitable etching reagent that will distinguish between the various phases present.

The X-Ray method also has its limitations. It is capable of disclosing conditions in an alloy at elevated temperatures in two ways: (1) by quenching the alloy to room temperatures and taking a diffraction pattern at that temperature, in which case it is limited to alloys in which the high temperature form is retained by the quenching action, and (2) by applying the method at the elevated temperature, a procedure requiring special cameras, temperature-control equipment, and inert atmospheres for protecting the sample against oxidation. It is not possible to recognize extremely small quantities of second phase in a binary or polyphase alloy, and so X-Rays are usually inferior to the microscope in this respect. This is not a serious limitation in phase-diagram work. The X-Ray method reveals practically nothing about the distribution of phases, which is evident in microscopic examination and is of vital

importance in practical problems."

Thermal analysis, microscopic observation, X-ray analysis and dilatometer measurement were used in this work. Hardness tests were performed on metallographic specimens and tensile tests were made on one alloy.

One definite complication presented itself in each of the test methods which was not mentioned by Barret³. The strong affinity for oxygen of tantalum and zirconium at elevated temperature made it necessary to carry out heat treatments in a vacuum or protective atmosphere. A vacuum of less than one micron is necessary to prevent serious oxidation of a specimen. Helium atmosphere may also be used satisfactorily to protect the alloys.

Specimens

The alloys used were supplied through the courtesy of the United States Bureau of Mines, Albany, Oregon and were of the composition and shape indicated in Table 2.

Although the alloy preparation is outside the scope of this project, the item is of importance due to subsequent effects resulting from the methods of preparation.

The alloys were melted in a vacuum, three phase, carbon resistance furnace using a carbon crucible. Segregation is minimized in the molten state due to the agitation of the metal created by the rotating magnetic

Table 2. Composition of Available Tantalum-Zirconium Alloys.

<u>Alloy Number</u>	<u>Nominal Composition</u> percent Ta	<u>Chem. Analysis</u>		<u>Shape</u>
		Ta	C	
164	1	1.2	0.14	3/8 Rod
165	5	5.3	0.13	3/8 Rod
166	10	9.7	0.15	3/8 Rod
167	15	14.1	0.26	3/8 Rod
214	15			1/8 Sheet
176	20	20.8	0.25	3/8 Rod
237	30	30.3	0.44	1/2 Rod
301	40	42.5	0.34	As Cast Ingot

field; however this affect does not completely eliminate the possibility of segregation of solidification.

The carbon crucible used in melting plus the fact that both zirconium and tantalum are strong carbide formers accounts for the carbon as shown in the chemical analysis given in Table 2.

After melting, the carbon crucible was turned from the alloy and the metal was then placed in a sheath of mild steel which was welded air tight. The samples, in the steel sheath, were worked at 850°C. The sheaths were then removed and the alloy was ready for use.

From the metallurgy of pure zirconium and the type of diagram expected, it was supposed that a phase change would exist in the region of the critical temperature of alpha zirconium. Therefore it was decided to make a preliminary investigation of the system by heat treating the alloys in the temperature range from 750 to 1100C.

Metallographic

The metallographic specimens used were cut from the swaged rod in lengths of about one eighth of an inch. Sections of the original cast ingot were available so specimens of the cast and rolled material were first prepared for microscopic examination.

The following procedure was followed for preparing

these and subsequent specimens for metallographic analysis.

Rockwell hardness was taken on each sample. They were then mounted in bakelite to facilitate subsequent polishing and ground a 120X belt grinder. After the rough grinding operation, the samples were ground on number 2, 1 and 0 emery paper. After the papers, the specimens were polished on a canvas lap, a felt lap and finally on a velvet lap using No. 280 and 600 mesh carbonundum powder on the first two wheels and levigated alumina as the abrasive on the final polishing wheel. Approximately one minute was necessary on each wheel to obtain a satisfactory polish; however, it was necessary to repolish on the last wheel longer with some of the higher tantalum alloys.

A number of standard and unorthodox etchants were tried; however the only solution that would etch the alloy was one containing hydrofluoric acid. The etchant finally adopted was essentially Vilella's etch.*

The furnace used for heat treating the specimens at temperatures up to 950C is shown in Figure 1.

The furnace has Nichrome elements and operates from 110 volt A.C. circuit. The vacuum system consists of Duo Seal vacuum pump and a Distillation Products oil diffusion

*Vilella's Etch. Hf 2 parts, H₂O 25ml, HNO₃ 1 part, Glycerin 3 parts.

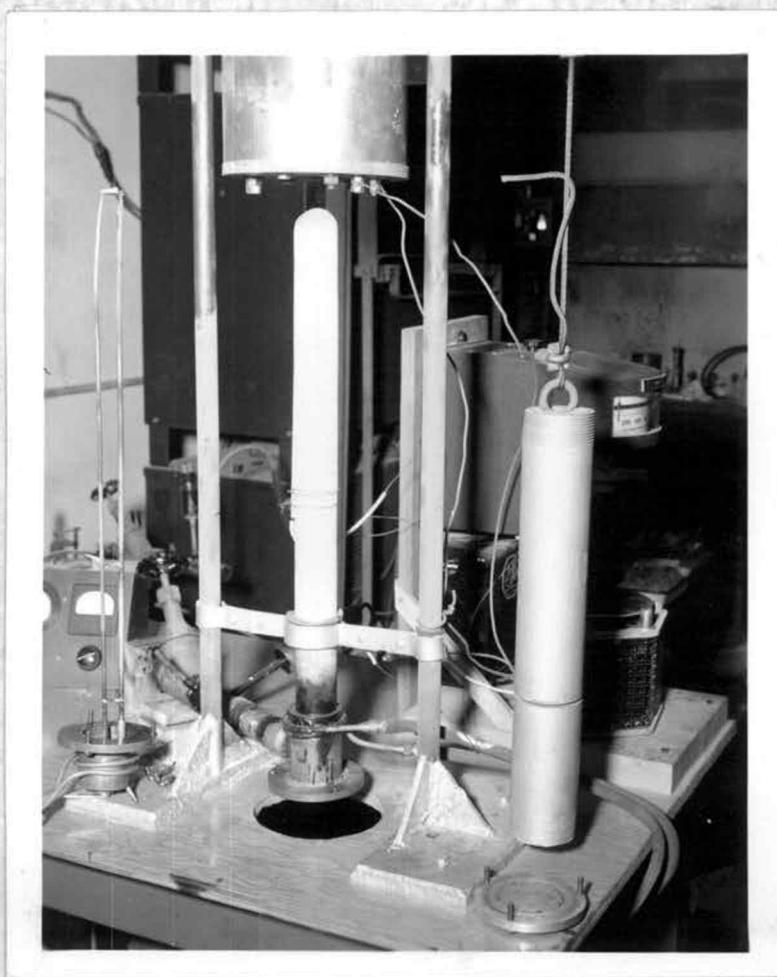


Fig. 1 Vacuum quenching apparatus
for temperatures up to 950C.

pump. Quenching is accomplished by turning the small rod supporting the small pan shown in Figure 1. This allows the pan to pivot about its hinged support thus letting the specimens fall into a shallow well of oil at the base of the quartz tube. The center "trip" rod and the casing around the well of oil are joined by a flexible diaphragm which is soldered air tight, one end to the rod and the other to the well casing.

For heat treating from 950-1100°C the furnace shown in Figure 2 was used. The furnace elements are silicon carbide energized by 220 volt A.C. circuit. A protective atmosphere was established by evacuation of the quartz tube with a vacuum pump and back filling with helium several times. Quenching was accomplished by means of an oil filled receptical connected to the quartz tube by flexible brass tubing. Gravity held the oil receptical vertical while the tube lay horizontal as shown in Figure 2.

The specimens were quenched by taking the quartz tube from the furnace and raising it to a vertical position thus allowing the specimens to fall into the oil. Caution was taken, when evacuating the system, to hold the flexible tube in the vertical position as the outside pressure tended to compress the tube as the system approached a complete vacuum which would cause

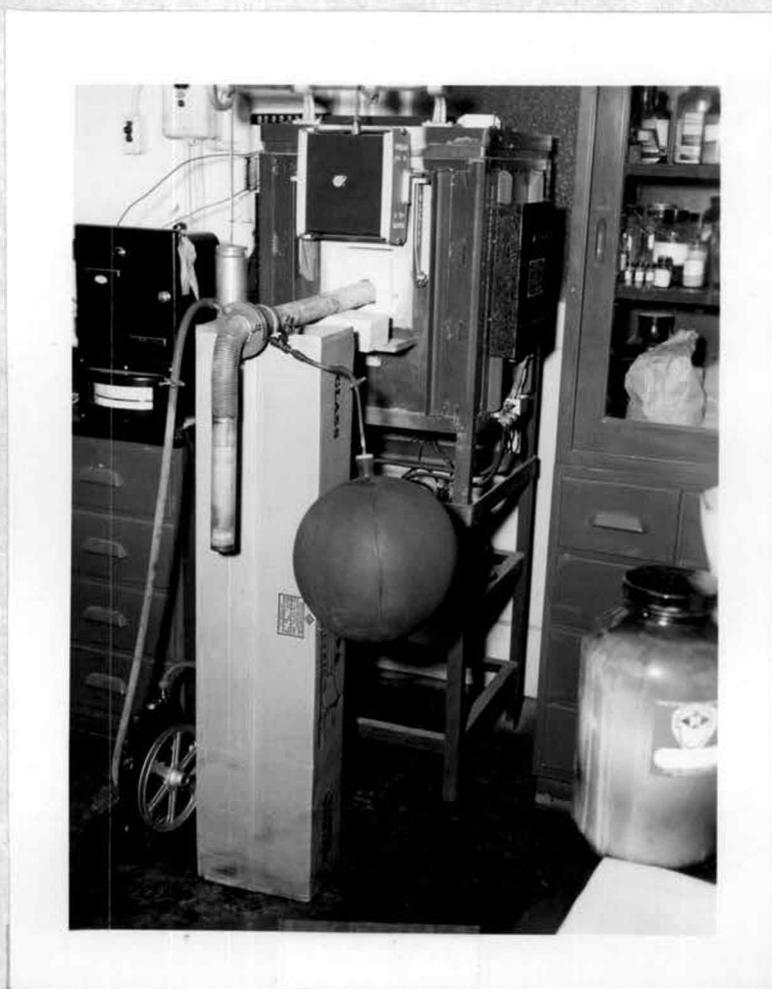


Fig. 2. Apparatus for oil quenching in a protective atmosphere from temperatures up to 1200°C.

the oil receptacle to assume a horizontal position and permit the oil to enter the quartz tube.

Both furnaces were controlled at temperature with a Brown Controller. The high temperature furnace using a platinum-platinum rhodium thermocouple and the lower temperature furnace using a chromel-alumel thermocouple.

In general all alloys were included in a heat treating operation but where previous investigation indicated that a certain alloy could be excluded from a particular heat treatment, it was left out in order to conserve the alloy stock.

Table 3 shows the heat treatment used in preparing the metallographic specimens and the samples to which the heat treatment was applied. All samples were polished and etched by the same procedure used for the as cast and as rolled specimens, with minor variations.

It was noted that samples of 166 and 167, furnace cooled, stained badly with the etch. Variations of the etchant were tried without success. In the case of these alloys it was necessary to remove the stain caused by the etching by giving the surface a light polish after etching. This method is not desirable but had to be used to get any indication of the microstructure.

Apparently the zirconium rich alloys become increasingly difficult, if not impossible to polish mechanically.

Table 3. Heat Treatment Given Various Zirconium-Tantalum Alloys.

<u>Heat Treatment</u>	<u>Alloy Heat Treated</u>
1100C, 1 hour furnace cool	165, 166, 167, 176, 236
1000C, 1 hour furnace cool	164, 165, 166, 167
1100C, 1 hour oil quench	165, 166, 167, 176, 237
950C, 1 hour oil quench	164, 165, 166, 167, 176, 214, 237
900C, 1 hour oil quench	164, 165, 166, 167
900C, 1 hour furnace cool to 850C and oil quench	164, 165, 166, 167
900C, 1 hour furnace cool to 800C and oil quench	164, 165, 166, 167
900C, 1 hour furnace cool to 750C and oil quench	164, 165, 166, 167
850C, 4 hours oil quench	164, 165, 166
Melt 1630+250	176
Melt 1630+250	237
950C, 1 hour oil quench, reheat to 500C $3\frac{1}{2}$ hours, slow cool	214

with increasing tantalum content. Consequently in an attempt to observe the microstructure of the twenty and thirty percent alloys, electrolytic etching was tried using a variety of etchants; however the electrolytic polishing and etching met with less success than did the mechanical polishing and chemical etching.

The microstructure of all specimens shown in Table 3 were examined visually and photomicrographs were taken of each type of structure.

Differential Thermal Analysis

Samples of alloys 164, 165, and 166 were turned to thermal analysis specimens $3/16$ inch in diameter and $1/2$ inch long with a $1/16$ inch hole drilled longitudinally $3/8$ of an inch into the specimen.

The furnace used had Nichrome elements and the heating and cooling rates were controlled by a 30 amp 110 volt A.C. variac.

The specimen and neutral body were protected from oxidation by placing them in a quartz tube, the end of which was far enough out of the furnace to be cool at all times. The open end of the tube was sealed with a rubber stopper. Thermocouple leads and a copper tube were placed through the stopper, the copper tube being used to evacuate the quartz tube and to allow back

filling with helium. Temperature was measured with a Brown potentiometer and the temperature differential was measured with a Leeds and Northrop reflecting galvanometer.

Figure 3 shows the arrangement of the apparatus used to obtain the differential curves.

Heating and cooling curves were obtained for the three alloys previously mentioned and at least two runs were made on each alloy in an attempt to verify the curves obtained.

Dilatometric Analysis

Dilation vs temperature curves were obtained for alloys 165 and 167. Lack of the necessary stock eliminated the possibility of obtaining curves for other alloys.

Dilatometer apparatus consisted of an $\frac{1}{2}$ inch quartz tube 13 inches long and sealed on one end. The tube was supported vertically with the closed end down. A nickel body 3 inches long which fitted snugly into the tube, had a longitudinal hole 0.200 inches in diameter drilled completely through. This body held the specimen vertical and stabilized it from horizontal motion. The specimen was in the form of a rod 0.190 inches in diameter and 2 inches long. The bottom of the specimen was held away



Fig. 3. Differential Thermal Analysis Apparatus

from the quartz tube by an 1/8 inch x 1/2 inch quartz rod. An 1/8 inch x 12 inch quartz rod was also placed on top of the specimen and inside the nickel body. Thus the dialation of the specimen was transmitted through the 1/8 inch quartz tube to an Ames guage which measured the dialation to 1/10,000 of an inch.

A small 110 volt A.C. furnace with Nichrome elements was used for heating. The furnace was regulated by a variac and the temperature measured with a chromel-alumel thermocouple, the bead of which was in the nickel body adjacent to the specimen.

Dilation analysis was carried on without protective atmosphere. Consequently to check what effect oxidation had on the critical points, two runs were made on each alloy. A section of the specimen was also mounted, polished, and examined microscopically after the test in order to observe the depth of oxygen penetration.

X-Ray Analysis

One sample each of alloys 165, 166, 167, 176, and 237 to be used for X-Ray analysis were quenched from temperatures of 950 C and 1100 C. The specimens to be quenched were cut in lengths of 1/16 inch from the swaged rod. After quenching the specimens were cut in one half. One half of the sample was prepared for

microscopic examination in order to ascertain whether or not they were contaminated with oxygen. The other half of the section was filed into X-Ray wedges 3/8 inch long and 1/8 inch wide. The wedges were then ground on 0 polishing paper and thence etched in order to relieve stresses caused by filing. One wedge of alloy 301 was also prepared from the as cast ingot.

The X-Ray machine used to obtain a Debye-Sherrer pattern from the wedges was a General Electric XRD-1 machine using a tube with a copper target and a nickel filter.

The $\sin^2 \theta$ and d values were determined for all visible lines. Identification of the pattern was attempted by comparison of the plotted $\sin^2 \theta$ values of the lines with the Hull-Davy charts. Lattice dimensions of the structures were calculated from the d values of the identified atomic planes.

Physical Properties of Alloy 214

The small 1/8 inch sheet of alloy 214 was welded into an air tight mild steel sheath and rolled at 850 C to 0.030 inch sheet. The sheath was then removed and standard sheet tensile specimens were prepared. Two of the specimens were quenched from 950 C and two furnace cooled from the same temperature. The tensile specimens

were tested with a Baldwin tensile machine with a stress strain recorder. From the tests were determined the tensile strength, yield strength and modulus of elasticity of the alloy in the quenched and furnace cooled condition.

Two small samples of alloy 214 were quenched from 950 C after soaking at that temperature for one hour. Hardness readings were taken on each of the samples. The specimens were then heated to 500 C for $3\frac{1}{2}$ hours, cooled in air and hardness readings again taken. A tensile specimen of the alloy quenched from 950 C and aged at 500 C for 4 hours was also prepared and tested.

Melting Point of Alloys 176 and 237

The approximate melting points of alloys 176 and 237 were determined by melting small samples of the alloys in a vacuum high frequency furnace. Carbon crucibles were used to hold the specimen and the temperature was measured with a Leeds and Northop optical pyrometer.

An attempt to approach black body conditions was made by measuring the temperature at the bottom of a small hole drilled into the specimen. The approximate melting temperature was determined by noting when the

specimen darkened on heating.

DISCUSSION OF RESULTS

Metallographic Analysis

Quenching pure zirconium from above the alpha to beta transformation temperature results in a Widmanstatten type of microstructure similar to that produced by quenching gamma iron. In the extensive work on the alpha to beta transformation of zirconium, Burgers⁴ indicates that this type of structure will always result if a beta zirconium solid solution is quenched.

In the light of work done by Burgers it was assumed, during the course of this project, that the phase was beta zirconium solid solution at temperature if the microstructure of a quenched specimen was Widmanstatten in design.

As mentioned in the procedure, metallographic specimens of the alloys were prepared for microscopic observation. Figures 4 to 12 show the typical microstructures of the various specimens in the as cast and as rolled condition.



Fig. 4. 1% Ta as cast
250X



Fig. 5. 5% Ta as cast
250X

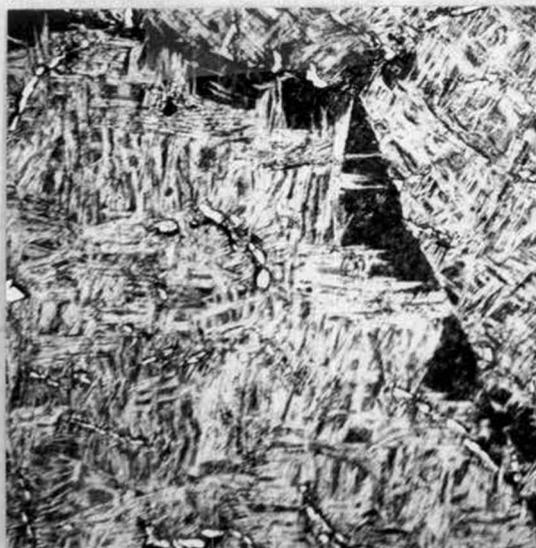


Fig. 6. 10% as cast
250X

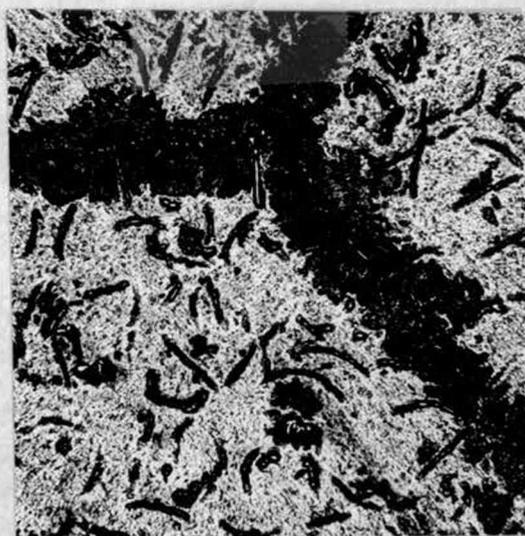


Fig. 7. 15% Ta as cast
250X

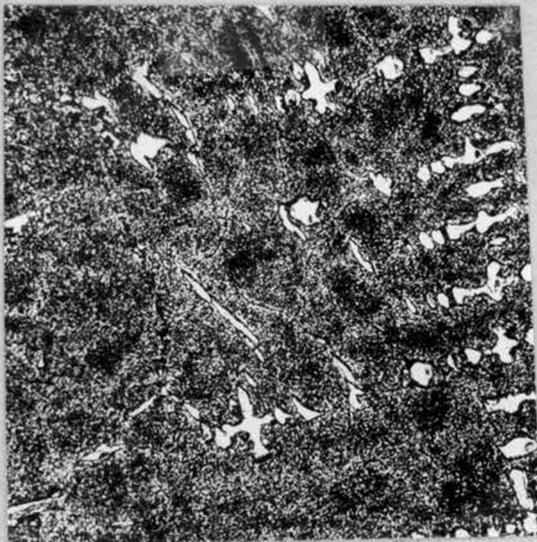


Fig. 8 30% Ta as cast
250X

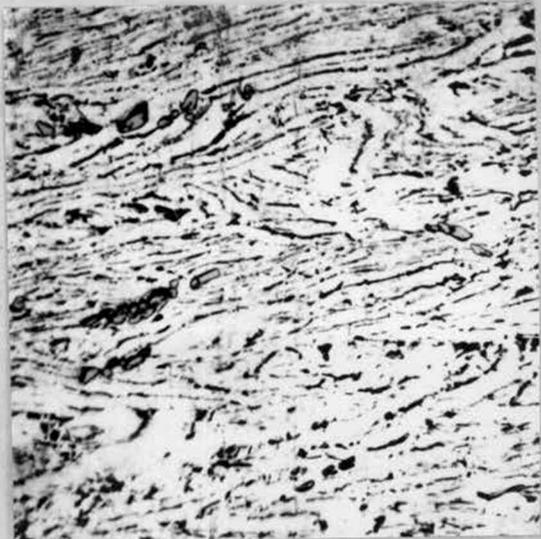


Fig. 9 1% Ta as rolled
250X



Fig. 10 10% Ta as rolled
250X

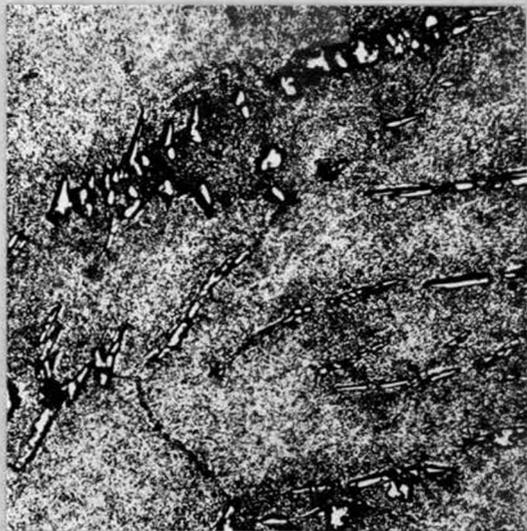


Fig. 11 30% Ta as rolled
250X

Alloys 164, 165 and 166 appear to be quite similar in both the as cast and as rolled condition except that with increasing tantalum there is an increasing amount of a dark second phase which is evident in the grain boundary of the as cast specimen of alloy 166 represented by Figure 6.

The as cast structure of alloy 167 shown in Figure 7, appears to be quite different from the lower composition alloys in that a Widmanstatten structure is not evident in this alloy as it was in the alloys 164, 165, and 166. This would indicate that this alloy was outside the beta field or that the cooling rate from the beta region was too slow to permit the formation of the Widmanstatten pattern. The dark phase oriented at random is zirconium and tantalum carbides which is evident in all specimens. The quantity of carbides present is exaggerated by extreme difference in the etching characteristics of the matrix and the carbides.

The as rolled structure of alloy 166 shown in Figure 10 appears much darker than the as cast structure which indicates that the second phase and the carbides had been dispersed into the light matrix.

The microstructure of alloy 176 in the as cast condition is similar to that of 237 shown in Figure 8. The matrix of either alloy is a dark eutectoid-appearing

phase. A small amount of a white second phase is evident in either specimen with a larger amount of the light phase in alloy 237.

The as rolled structure of 237 shown in Figure 11 compares with that of alloy 176 with the exception of the appearance of more lighter phase.

Alloy 176 appears similar to the photomicrograph of alloy 237 shown in Figure 16. The two phases present appear quite different than do the lower tantalum composition alloys. As mentioned before these alloys are extremely difficult to prepare for microscopic examination and the darkness of the matrix may be due to staining rather than true etching. The spheriodized particles in the matrix which are more abundant in the alloy 237, indicate that a phase is present in these alloys which is absent in the lower tantalum content alloys.

The cast structure of alloy 301 shown in Figure 12 shows a dark matrix similar to alloys 176 and 237 although the light phase in the form of long stringers apparently oriented at random, is much different.

Although the cast and as rolled structures shown are not indications of equilibrium, since the conditions imposed on the metals are not closely controlled and are far from any equilibrium condition, the

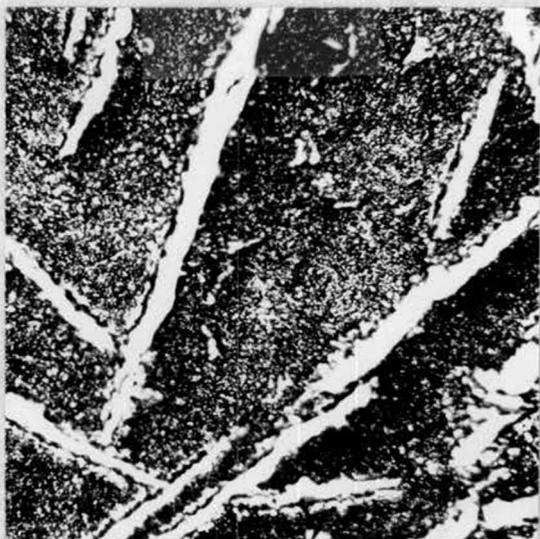


Fig. 12. 40% as cast
250X



Fig. 13. 1% Ta 1100
1 hr. F.C.
250X



Fig. 14. 5% Ta 1100
1 hr. F.C.
250X

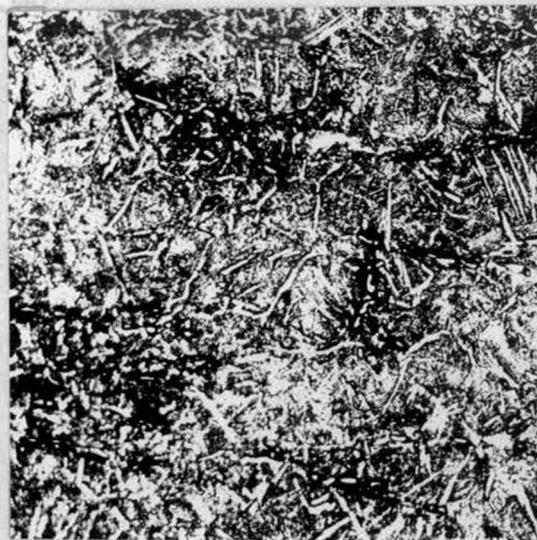


Fig. 15. 10% Ta 1100
1 hr. F.C.
250X

structures are important. The as cast structure serves as an indication of the homogeneity and grain size of the original material. As rolled structures are necessary for comparison with alloys of subsequent heat treatment in order to check recrystallization and phase changes.

In order to obtain an indication of the microstructure for alloys in the near-equilibrium conditions, the specimens were furnace cooled from 1100 C. The structures resulting from this treatment are shown in Figures 13 to 16. Alloy 164 is nearly a single phase alloy with somewhat elongated grains typical of slow cooled alpha zirconium. A small amount of a dark phase is evident between the alpha grains.

Alloy 165 with the same heat treatment is shown in Figure 14. More of the dark phase is present in the alloy and the light phase contains a Widmanstätten pattern. Evidently the critical cooling rate, necessary for the formation of the needle-like pattern on cooling, is less than for alloy 164.

Alloy 167 appears similar to the photomicrograph of 166 shown in Figure 15. Both alloys exhibit a large amount of the dark phase with alloy 167 apparently nearly all eutectoid.

In order to obtain an indication as to the nature of



Fig. 16. 30% Ta 1100
1 hr. F.C.
250X



Fig. 17. 1% Ta 950
1 hr. quench
250X

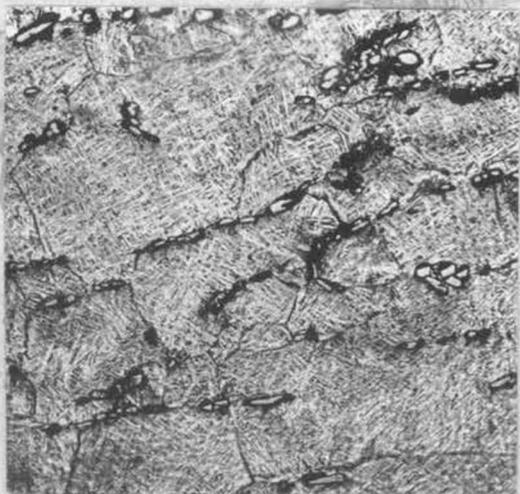


Fig. 18. 5% Ta 950
1 hr. quench
250X

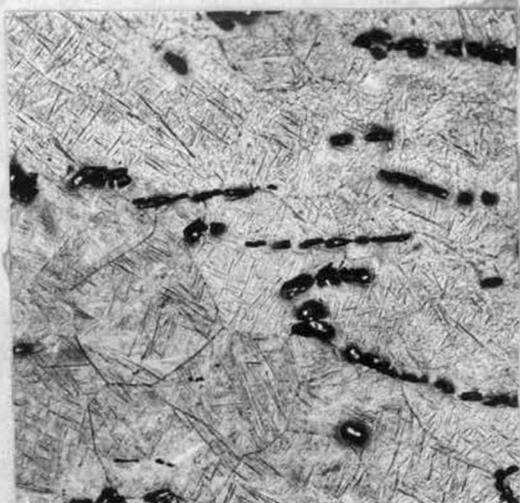


Fig. 19. 10% Ta 950
1 hr. quench
250X

the alloys at temperature, a complete set of the available alloys were quenched from various temperature levels. The group of alloys quenched from 950C indicated that up to 10% tantalum, the structures were beta zirconium at temperature. It is noted in Figures 17 to 22, that with increasing tantalum content the Widmanstatten pattern becomes finer and less distinct.

Alloy 167 quenched from 950C shown in Figure 20 appears to be two phase with the carbides oriented to some extent along the grain boundaries. The alloy was difficult to polish and etch and it is possible that the microstructure is a single phase solid solution with a very fine Widmanstatten pattern. Hardness values for the alloys, shown in Table 4, indicate that alloy 167 is the hardest of the alloys quenched from 950 C.

Figures 21 and 22 show the microstructure of alloys 176 and 237 respectively. Grain boundaries are visible in alloy 176 but not definitely outlined in alloy 237. Evidently recrystallization has taken place since the cold worked structure is no longer visible. The increase in hardness of the alloys over that of the as rolled specimens indicates that some solid solution formation had taken place prior to the quench.

Figures 23 to 25 represent the microstructures of alloys 165, 176, and 237 respectively, quenched from

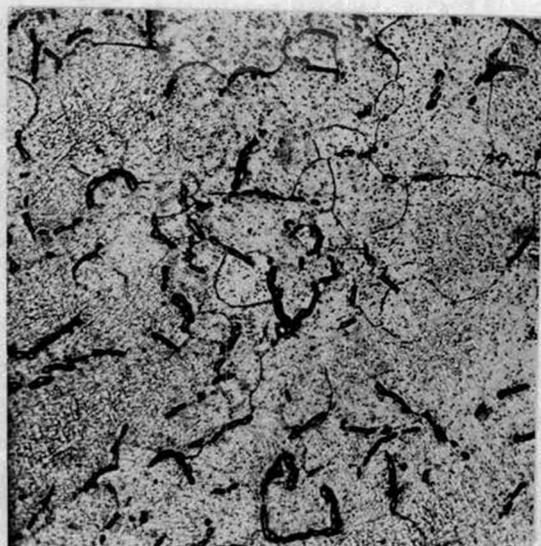


Fig. 20. 15% Ta 950
1 hr. quench
250X

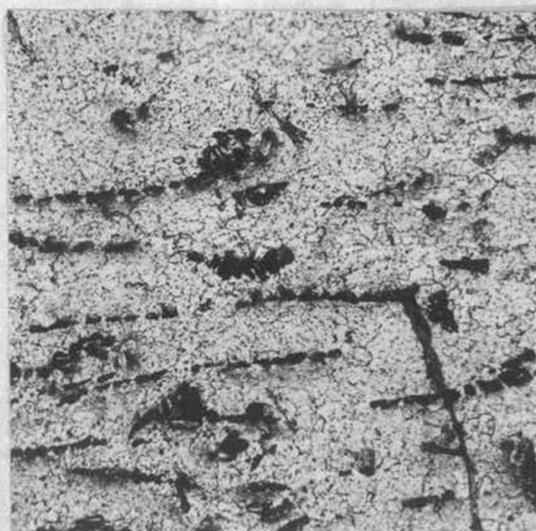


Fig. 21. 20% Ta 950
1 hr. quench
250X

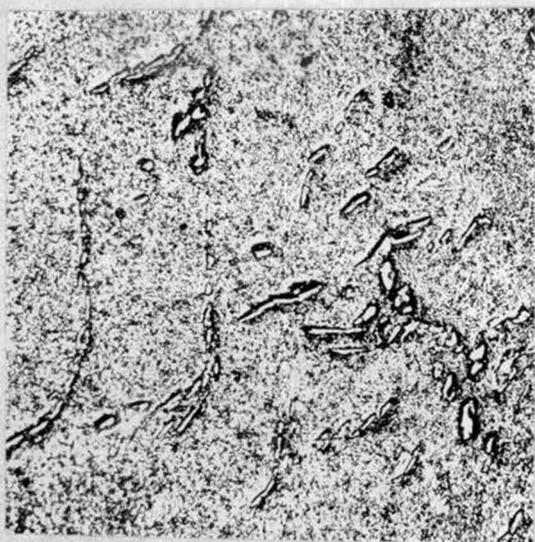


Fig. 22. 30% Ta 950
1 hr. quench
250X



Fig. 23. 5% Ta 1100 C
1 hr. quench
250X

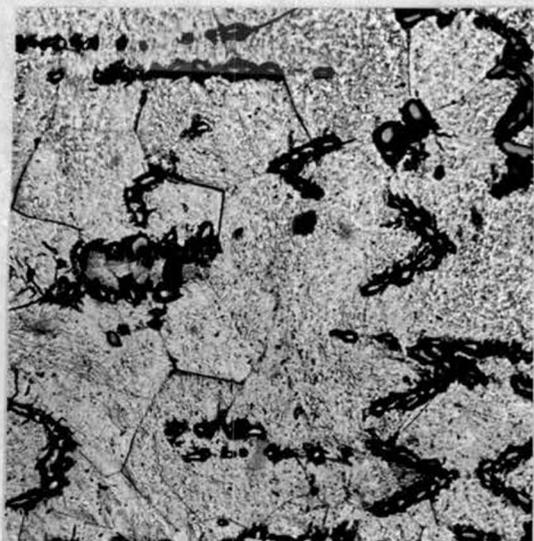


Fig. 24. 20% Ta 1100 C
1 hr. quench
250X

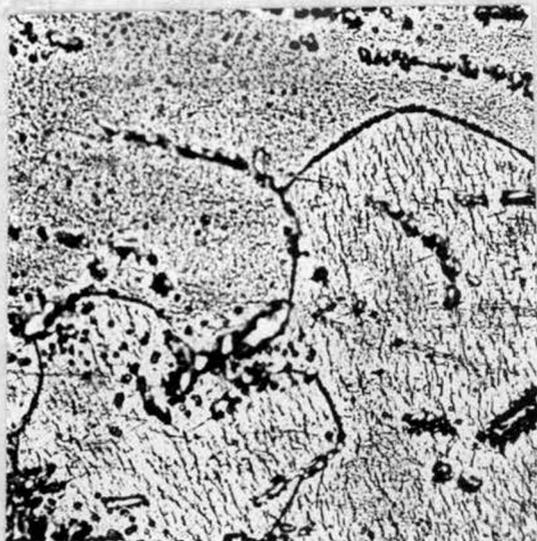


Fig. 25. 30% Ta 1100C
1 hr. quench
500X



Fig. 26. 1% Ta 900
1 hr. quench
200X

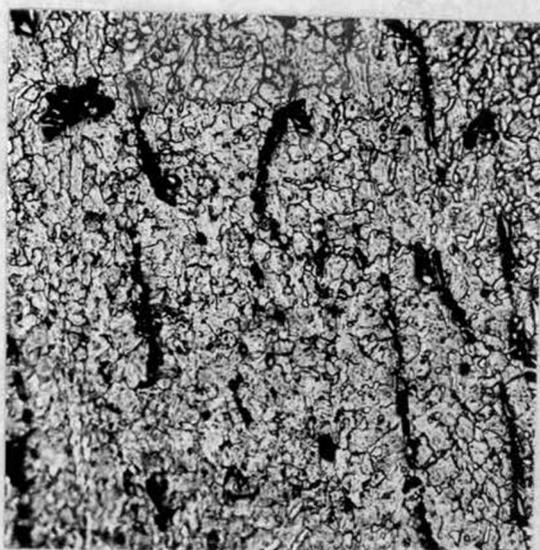


Fig. 27. 5% Ta 900
1 hr. quench
250X

Table 4. Hardness of Metallographic Specimens

<u>Alloy</u>	<u>H.T.</u>	<u>Hardness</u>	
164	As Cast	88.5	RB
165	As Cast	95	RB
166	As Cast	22	RC
176	As Cast	93	RB
237	As Rolled	20.5	RB
165	As Rolled	89.5	RB
166	As Rolled	22	RC
167	As Rolled	20	RC
176	As Rolled	15	RC
237	As Rolled	18	RC
164	1000° 1 hr. F.C.	90.5	RB
165	1000° 1 hr. F.C.	95	RB
166	1000° 1 hr. F.C.	97.5	RB
167	1000° 1 hr. F.C.	100	RB
165 1/16" thick	1100°C 1 hr. quench	39	RC
166 1/16" thick	1100°C 1 hr. quench	34.5	RC
167 1/16" thick	1100°C 1 hr. quench	36.5	RC
176 1/16" thick	1100°C 1 hr. quench	42.5	RC*
237 1/16" thick	1100°C 1 hr. quench	37	RC
164	1 hr. 950° quench	87	RB
165	1 hr. 950° quench	90.5	RB
166	1 hr. 950° quench	33	RC
167	1 hr. 950° quench	38	RC
176	1 hr. 950° quench	30	RC
237	1 hr. 950° quench	31.2	RC
165	1100°C 1 hr. fur. cool	97	RB
166	1100°C 1 hr. fur. cool	25	RC
167	1100°C 1 hr. fur. cool	26	RC
176	1100°C 1 hr. fur. cool	100	RB
237	1100°C 1 hr. fur. cool	20	RC

*Specimen was heat treated separately and may have been oxidized.

Table 4. (Continued)

<u>Alloy</u>	<u>H.T.</u>	<u>Hardness</u>
164	900 1 hr. V.Q.	91 RB
165	900 1 hr. V.Q.	21 RC
166	900 1 hr. V.Q.	33 RC
167	900 1 hr. V.Q.	36 RC
214	900 1 hr. V.Q.	
164	900 1 hr. F.C. 850 V.Q.	91.5 RB
165	900 1 hr. F.C. 850 V.Q.	20 RC
166	900 1 hr. F.C. 850 V.Q.	32.5 RC
167	900 1 hr. F.C. 850 V.Q.	32 RC
164	900 1 hr. F.C. 800 V.Q.	94.5 RB
165	900 1 hr. F.C. 800 V.Q.	23 RC
166	900 1 hr. F.C. 800 V.Q.	25 RC
167	900 1 hr. F.C. 800 V.Q.	29.5 RC
164	900 1 hr. F.C. 750 V.Q.	88.5 RB
165	900 1 hr. F.C. 750 V.Q.	97 RB
166	900 1 hr. F.C. 750 V.Q.	96.5 RB
167	900 1 hr. F.C. 750 V.Q.	92.5 RB
164	850 4 hr. O.Q.	93.5 RB
165	850 4 hr. O.Q.	22 RC
166	850 4 hr. O.Q.	25 RC
176	Melt 1640°C	31 RC
237	Melt 1620°C	39 RC
176	1100°C 4 hr. O.Q.	38.5 RC
237	1100°C 4 hr. O.Q.	54 RC
167	950°C 1 hr. quench	21 RC
167	950°C 1 hr. quench draw at 500 for 3½ hrs.	34 RC
214	950 1 hr. fur. cool	81 RB
214	950, 1 hr. oil quench	22 RC
214	950 1 hr. oil quench draw at 500 for 3½ hrs.	22 RC

1100 C. A distinct increase in grain size, over that of the alloys quenched from 950 C, is noted in the case of each alloy. Alloy 165 appears as a solid solution plus carbides and without the distinct Widmanstatten pattern evident in the alloy quenched from 950 C.

Alloys 166 and 167 quenched from 1100 C differ from those quenched from 950 C in that the Widmanstatten structure and the grain boundaries are not clearly discernable. The structures are somewhat lighter but alloy 167 still appears to be two phase and alloy 166 is single phase. The hardnesses of alloys 166 and 167 quenched from 1100 C and 950 C are very nearly the same.

Alloys 176 and 237 appear more as a single phase solid solution when quenched from 1100 C than when quenched from 950 C. The increase in hardness over that of the alloys quenched from 950C, indicates that more or all of the second phase present in the alloy quenched from 950 C is taken into solution at 1100 C.

Each of the alloys 164, 165, 166, and 167 quenched from 900 C resulted in a microstructure consisting of two phases. The Widmanstatten pattern typical of the alloys quenched from 950 C is not distinct if present as can be seen from Figures 26 to 29.

The alloys of 164, 165, 166, and 167 held at 900 C for one hour, furnace cooled to 850 and quenched are

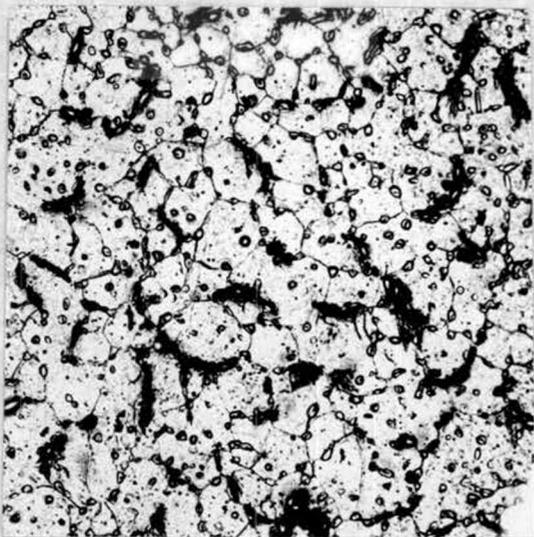


Fig. 28 10% Ta 900 C
1 hr. quench
250X

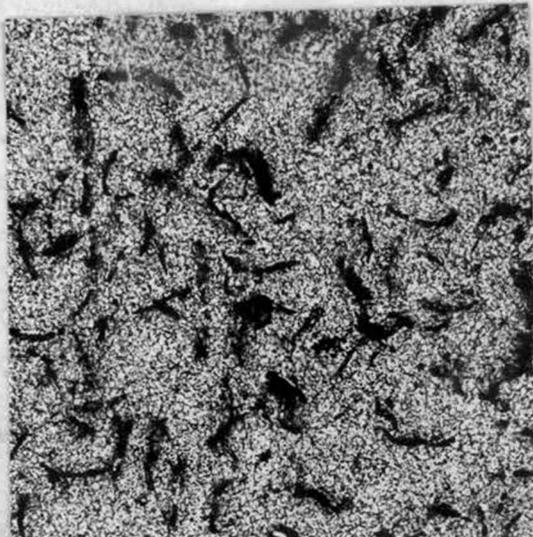


Fig. 29. 15% Ta 900 C
1 hr. quench
250X

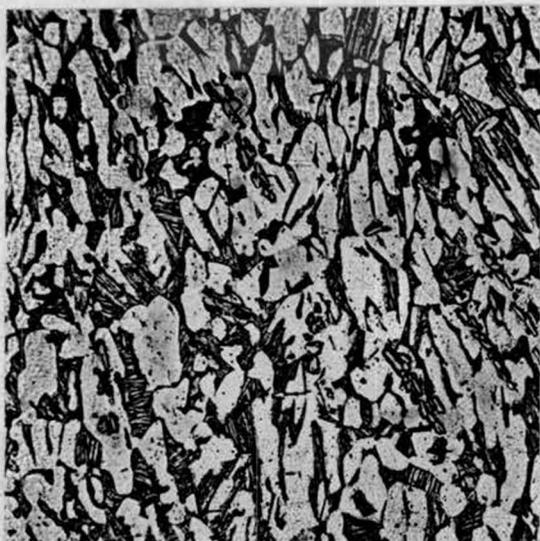


Fig. 30. 1% 900 C 1 hr.
FC to 850 C.
1 hr. quench
250X



Fig. 31. 5% Ta 900 C
1 hr. FC to 850C
1 hr. quench
250X

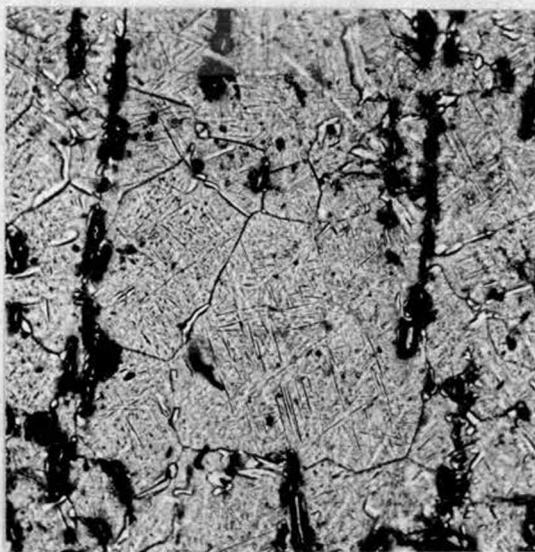


Fig. 32. 10% Ta 900 C 1 hr.
FC to 850 C 1 hr. quench
250X

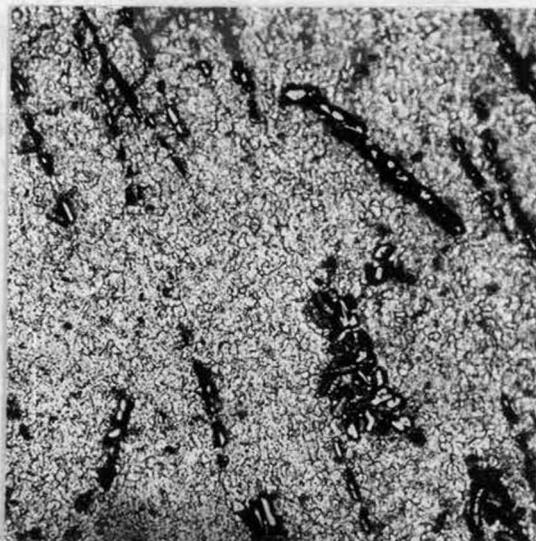


Fig. 33. 15% Ta 900 C 1 hr.
FC to 850 C 1 hr. quench
250X

shown in Figures 30 to 33. The alloys all appear two phase excepting alloy 166 which appears to be solid solution plus carbide. Alloys 165 and 166 seem to exhibit a fine Widmanstatten structure also. Alloy 167 appears similar to the same alloy quenched from 900 excepting the grains are very much finer. The hardness of alloys 166 and 167 is below that for the alloys quenched from 900 C while the hardness of alloys 164 and 165 are nearly the same for each heat treatment.

The alloys heated to 900 C, furnace cooled to 800 C and quenched are essentially the same as those heated to 900 C, furnace cooled to 850 C and quenched. The hardness of alloys 166 and 167 shows a decrease over the alloys quenched from 850 C after being furnace cooled from 900 C. This would indicate that less of the second phase was in solution at the time of the quench.

The specimens heated to 900 C furnace cooled to 750 C and quenched appear quite different from the alloys quenched from 800 C. They appear more nearly like the furnace cooled specimens which is indicative that no further phase change takes place below 750°C.

Comparison of the various alloys in the as cast and furnace cooled conditions indicates that alloys 237 and 176 contain different microconstituents than do the lower tantalum composition alloys.

The increasing amount of dark eutectoid-appearing phase with increasing tantalum content up to 15% tantalum leads to the belief that an eutectoid exists in the region of 15% tantalum.

The difference in appearance of the phases present in alloys 166 and 176 may be explained on the basis of the alloys being hypo and hyper eutectoid respectively. This is logical in that the matrix of alloys 167 and 176 furnace cooled from 1100 C are similar and the marked difference in the two alloys is in the excess light phase; this phase being lamellar in alloy 167 and globular in 176.

From this it would appear that the constitution diagram consists of an eutectoid in the range of 15% tantalum with an intermediate phase beyond 30% tantalum. This possibility is strengthened by the characteristic of the microstructures for the alloys quenched from 1100°C since the alloys appear to be very nearly a single phase solid solution even in the higher tantalum composition. The existence of a distinctly different phase in the as cast structure of alloy 301 indicates that an intermediate phase does lie between 30 and 40% tantalum.

Examination of the alloys heat treated below 1100 C does not appear to verify the existence of the simple

diagram discussed above. The absence of the Widmanstatten pattern in the series of alloys quenched from 900 C and the reappearance of the structure in the alloys heated to 900 C furnace cooled to 850 C and quenched is not explained by the simple diagram. The Widmanstatten pattern is also absent in the alloys quenched from 1100 C and the alloys of 167, 176, and 237 quenched from 950 C.

It is altogether possible that the cooling rate and composition influences the formation of the Widmanstatten pattern even though the alloy is quenched from the beta solid solution field. This would account for the absence of the Widmanstatten pattern in some of the quenched alloys.

The marked increase in hardness of alloy 165 quenched from 1100°C over that of the alloy quenched from 950°C is not in agreement with the simple diagram and is without explanation.

Differential Thermal Analysis

The test results of the differential thermoanalysis attempted during the course of the project was not particularly successful, due to a multiplicity of factors.

The apparatus used was not particularly satisfactory. The cooling rate was controlled by a hand-operated variac. Obviously a constant change in resistance of the variac will not result in a constant temperature change rate for

the specimen due to the difference in heat loss from the furnace at low and elevated temperatures.

The size relationship between the specimen and the neutral body may not have been the most desirable to give a maximum differential effect. Cooling rates in a thermoanalysis test are critical but the necessary cooling rate to give the best differential curve may not be the same even within the same alloy system due to the different heats of solution and relative ease with which the transformation takes place. Apparently in the tantalum system the heats of transformation or solution are low and the transformation rather sluggish.

Although thermoanalysis was attempted on alloys 164, 165, and 166, only the data obtained for the 166 alloy was reproducible.

From the thermoanalysis attempted on alloy 166 it was noted that the break in the curve would occur at successively lower temperatures and become less pronounced, if a single specimen was subjected to a series of heating and cooling cycles. De Boer and Fast⁶ indicate that oxides present on the surface of a specimen will quickly diffuse into the specimen if heated in a vacuum. They also indicate that the effect of oxygen in zirconium is to make the transformation from alpha to beta more sluggish and possibly change the transformation

temperature.

Thus it appears that oxygen contamination accounts for the fact that the transformation temperature of alloy 166 was seemingly being lowered with each repeated heating and cooling cycle. When a new specimen was prepared for each run, the results were quite reproducible.

Figure 34 shows the differential curves obtained for alloy 166. The data represented by the curves indicate that a phase change began at 790 C with another change taking place at 855 C. On cooling the break in the curves was at 822 C and 757 C. Each point fell 33 degrees below the breaks in the heating curve. This would be expected since the cooling rate was more rapid than the heating rate with the result that some supercooling took place during the cooling cycle.

Dilation Analysis

The apparatus available for dilation analysis did not provide a protective atmosphere for the specimen. For this reason dilation analysis was avoided until late in the project. As a last resort in searching for a method to establish the critical temperature of the phase change thought to exist in the region of 750-800 C, dilation analysis was attempted on alloy W165 and the results appear in Figure 35.

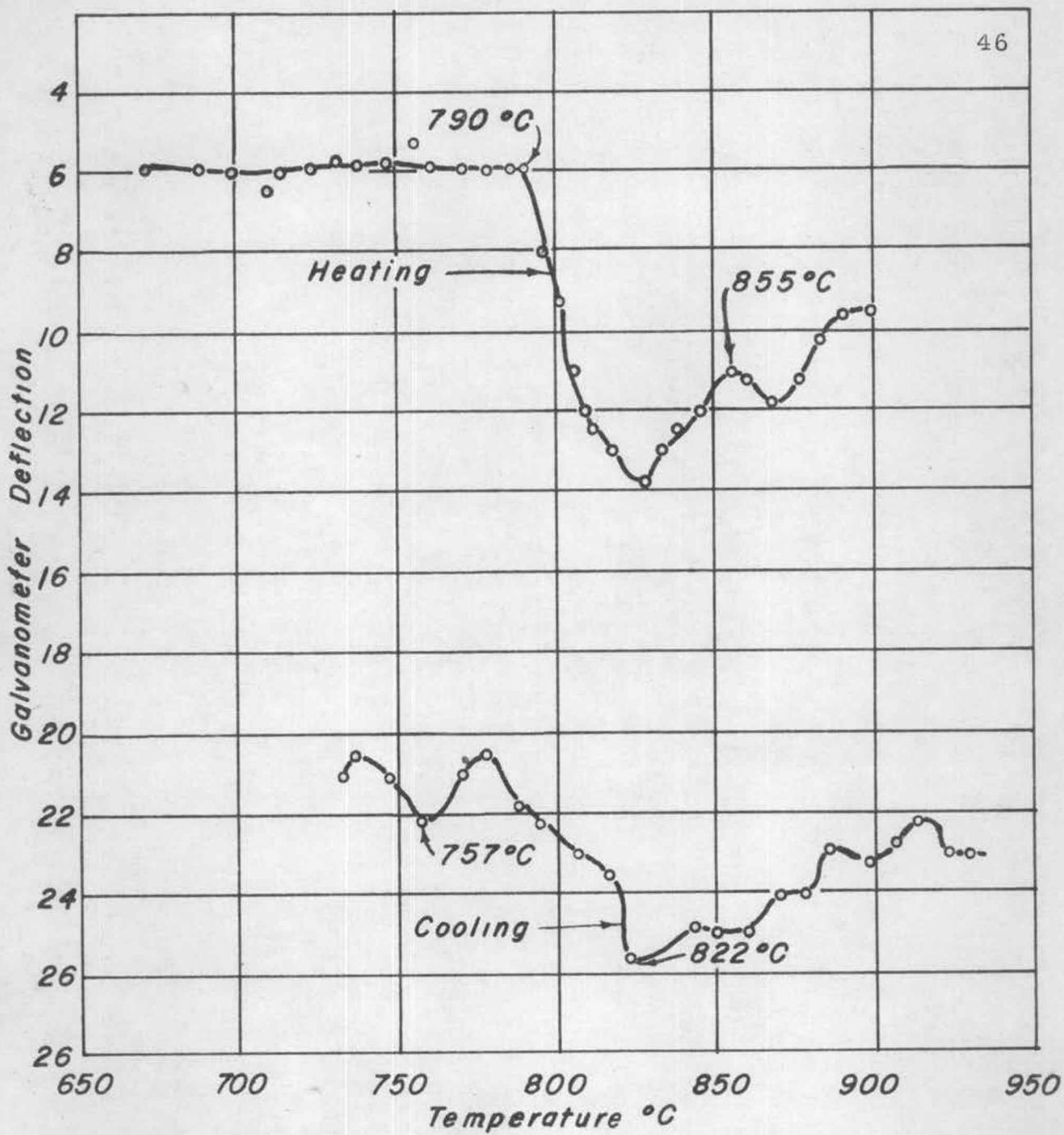


Fig. 34 Differential Thermal Analysis Curve of 10% Ta.-90%Zr.

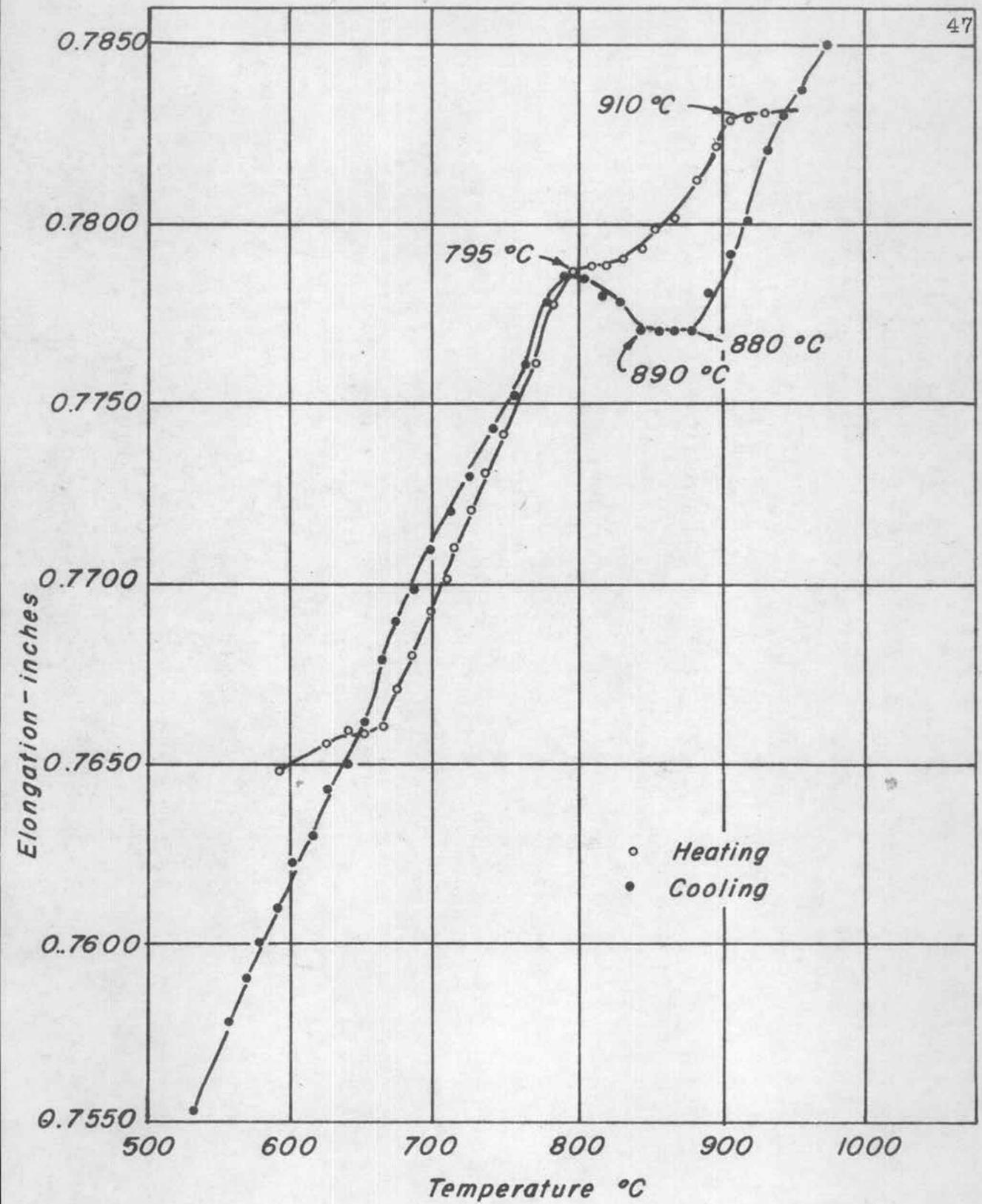


Fig. 35 Dilatometer Curve of 5% Ta - 95% Zr.

The curve of dilation vs. temperature on heating and cooling showed a definite break at two temperature levels but these were viewed with some skepticism due to the possibility of severe oxide contamination.

Dilation vs. temperature data were taken for alloy 167 and represented by the curve shown in Figure 36. Definite breaks in the curve are apparent. From the curve it seems that a phase change began at about 780 C with a second change taking place at about 915 C which was not complete at 980 C.

In order to investigate the possibility of oxygen contamination affecting the transformation points, the same specimen was used to verify the first curve with the result that both sets of curves indicate transformation points at temperatures which corresponded within the limits of experimental error. Figure 37 shows the edge of a section of the dilation specimen used for the analysis of alloy 165. Oxygen penetration is apparently not seriously deep.

Thus in view of the reproducibility of the curves for alloy 167 and also the appearance of the specimen, it is logical to assume that the dilatometry data are reliable.

In view of the assumed reliability of the dilation vs. temperature curves for each alloy, it is obvious

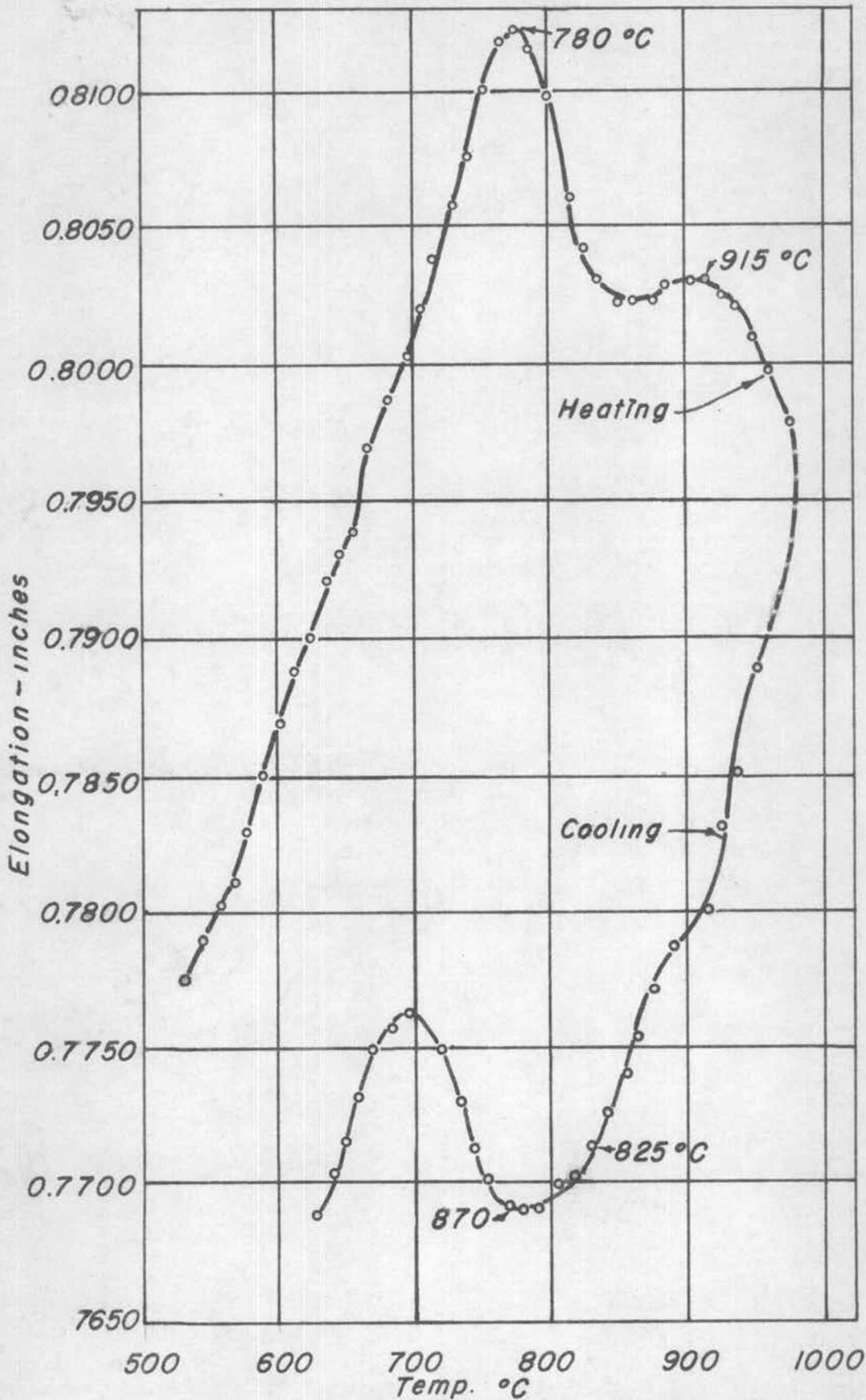


Fig. 36 Dilatometer Analysis of 15% Ta - 85% Zr.



Fig. 37. 5% Ta Dilation
Specimen
250X

that a transformation begins in the region of 780 C-795 C with a second transformation taking place at about 910 C and not complete at 950 C. It is most likely that the critical points are identical for both alloys and the discrepancy in the curves is caused by factors such as rate of cool and effect of dissolved gases.

X-Ray Analysis

As mentioned in the procedure, Debye-Sherrer patterns were obtained for alloys 165, 166, 167, 176, and 237 quenched from 950 C and 1100 C. Two patterns were also obtained for alloy 301 in the as cast condition. The samples of 301 were different in that one sample was taken from the top of the ingot and one from the bottom of the same ingot.

The possible constituents in the alloys of sufficient proportion to give an X-Ray pattern are (1) Alpha zirconium (2) beta zirconium (3) tantalum (4) intermediate phases (5) oxides of tantalum or zirconium (6) carbides of zirconium (7) carbides of tantalum.

Table 5 shows the lattice dimensions and crystal structure of the various possible constituents. The X-Ray pattern for the quenched alloys indicated that two phases were present, the principal phase being alpha

Table 5. Crystal Data of Possible Constituents in Zirconium-Tantalum Alloys

<u>Phase</u>	<u>Crystal Structure</u>	<u>Lattice Dimensions</u>
Alpha zirconium	H.C.P. ¹³	a = 3.223 c = 5.123
Beta zirconium	B.C.C.	a = 3.16
Tantalum	B.C.C.	a = 3.296
Intermediate phases	unknown	
ZrC	Simple Cubic ¹²	4.673
TaC	Simple Cubic ⁵	4.44 - 4.56
TaC ₂	H.C.P. ²	a = 3.09 c = 4.93
Zr oxide	Complex	
Ta oxide	Complex	

zirconium. The second phase present in the quenched alloys was identified as a face centered cubic lattice with lattice dimensions of about 4.61. The intensity of the lines for the second phase increased slightly with increasing tantalum which would lead one to believe that the second phase present was an intermediate phase, probably a compound of tantalum and zirconium.

The alpha zirconium lattice dimensions remain quite constant regardless of heat treatment or tantalum content which would indicate that the solubility of zirconium in tantalum was less than the composition of alloy 165 and the balance of the tantalum was present in the intermediate phase.

The X-Ray pattern obtained for alloy 301 indicates that the structure of the major phase was a body-centered cubic structure with a lattice dimension of 3.29. This rather upsets the possibility of an intermediate phase existing with the body centered cubic type of lattice identified in the quenched specimen.

The second phase present in the quenched alloy might well be carbides or oxides or either metal, but referring to Table 5, it is evident that neither the carbides nor the oxides are face centered cubic although the lattice dimension of zirconium carbide is similar to that of the face centered cubic structure.

Burgers⁴ indicates that the transition from beta to alpha zirconium tends to favor the formation of an intermediate face centered cubic phase on cooling. It is possible that the presence of tantalum tends to stabilize this phase at room temperature. This would account for the existence of the face-centered-cubic-phase in the quenched specimens.

The supposition that the second phase present in the quenched specimen is formed on cooling does not account for the tantalum in the alloy. Reference to Table 5 shows that the lattice dimensions of beta zirconium and tantalum are quite close which means that tantalum present in zirconium may not have a great effect on the lattice dimensions of alpha zirconium.

Assuming this thinking is correct the solubility of zirconium in tantalum at temperature extends well toward an intermediate phase which lies between 30% and 40% tantalum.

Physical Properties of Alloy 214

Table 6 shows the physical properties determined for alloy 214. From the table it is evident that the hardness of the quenched alloy is substantially that which was expected in view of the hardness data shown in Table 4.

Table 6. Physical Properties of Alloy 214

	<u>Quenched from 950</u>	<u>Furnace cooled from 950</u>	<u>Quench from 950 draw at 5000 f for 3½ hours.</u>
Hardness	21 RC	81 RB	34 RC
Ultimate Strength psi	127,500	91,000	189,000
Yield Strength psi	91,400	68,000	
Proportional Limit psi	67,400	52,000	
Modulus of Elasticity psi	10.9x10 ⁶	14.5x10 ⁶	
Percent Elongation	5.5	15	

The alloy quenched from 950 C and drawn at 500 C shows quite an increase in hardness over that of the quenched alloy. The tensile strength of the alloy was increased to 189,000 by aging as compared to 127,500 for the quenched alloy. Time did not allow further investigation of this phenomenon.

Melting Points of Alloys 176 and 237

The melting point of alloys 176 and 237 were determined, within the range of accuracy of the optical pyrometer, to be 1630 C, which is over 200 C less than the melting point of pure zirconium.

Correlation of Results

Microscopic analysis indicates that a phase change exists in the alloys between the temperatures of 750 and 800 C. This is verified in the case of alloys 165, 166, and 167 by thermoanalysis and dilatometry.

The extended solubility of tantalum in zirconium at elevated temperatures is apparent from the photomicrographs of the quenched specimens. If the solid solution field thought to exist at elevated temperature is a phase of beta zirconium containing tantalum, then the existence of the Widmanstätten structure in the lower quenched alloys and not in alloys above 15%

tantalum, can only be explained on the basis that the tantalum present retards the formation of the Widmanstatten pattern. This may be related to the existence of a body-centered cubic lattice present in the quenched alloy which is thought to form on cooling.

Metallographic analysis indicates that a eutectoid exists between 10 and 15% tantalum. This possibility was not confirmed or disputed by other methods excepting that thermal analysis responded quite well for alloy 166 where it failed for the other alloys, which would be an indication that considerable eutectoid was present in alloy 166.

The possibility of the existence of a second phase change just above 900°C as indicated in alloys 165 and 167 by dilatometry is also reflected in the microscopic observation in that the microstructures of alloys 164, 165, 166, and 167 quenched from 950 C appear quite different than those quenched from 900 °C.

Metallographic investigation indicates that an intermediate phase exists between 30 and 40% tantalum. This is verified by the X-Ray analysis. The intermediate phase is apparently of a cubic crystal structure, probably body centered with lattice dimensions of 3.29 which is similar to the crystal structures of tantalum. It might be assumed that the phase identified

in alloy 301 was tantalum solid solution, but this would not account for the 60% zirconium in the alloy.

CONCLUSIONS

The thorough investigation of the system is beyond the scope of this thesis and considerable work is yet to be done before a complete identification of the phases present in constitution diagram will have been accomplished; however several tentative conclusions may be drawn from this work.

Among the most definite conclusions concerning the diagram is the existence of a phase change in the region of 790 C in all alloys up to at least 15% tantalum.

The transformation taking place at 790 C is thought to be eutectoid temperature and the eutectoid lies between 10 and 15% tantalum. A second transformation takes place on heating at 910 C.

The solubility of tantalum in zirconium is above 30% at 1100 C and at 950 C and 30% tantalum heterogeneous field exists. At 950 C the solubility extends to at least 10% tantalum and at room temperature the solubility of tantalum in zirconium is less than 1%.

The solubility of tantalum in alpha zirconium increases with temperature. This is evidenced by the age hardening characteristics of the 15% tantalum alloy.

An intermediate phase with a cubic lattice exists in the region of 40% tantalum. Concerning the properties of the alloys it can be said that all alloys up to 30% tantalum are heat treatable in that the hardness of specimens quenched from elevated temperatures show a marked increase in hardness over the furnace cooled specimens.

The tensile strength of the alloy containing 15% tantalum is increased substantially by quenching from 950 C. The ultimate strength compares to a hardened medium carbon steel. The alloy of 15% tantalum is also age hardenable.

BIBLIOGRAPHY

1. American Society for Metals. Metals Handbook. 1948 ed. pp. 1142-1144.
2. American Society for Testing Materials. X-Ray Diffraction Cards.
3. Barrett, Charles P. Structure of Metals. McGraw-Hill. p.554.
4. Burgers, W.G. On the Process of Transition of The Cubic-Body-Centered Modification into The Hexagonal-Close Packed Modification of Zirconium.
5. Chemical Rubber Company. Handbook of Physics and Chemistry. 30th ed. p.304.
6. De Boer, J.H. The Influence of Oxygen and Nitrogen on the Alpha-Beta Transition of Zirconium. Received des Travaux Chimiques des Pay-Bas. Vol 59 1940 pp.459-467.
7. Ellinger, Finley H. The Tantalum-Carbon System. Trans. American Society for Metals. Vol 1 1940 p.101.
8. Fast, J.D. The Transition Point Diagram of Alpha Zirconium des Pay-Bas. Vol 58 1939 pp. 973-983.
9. Harrington, R.H. Modern Metallurgy of Metallurgy of Alloys. John Wiley and Sons. Chapters 5,6, and 7.
10. Hume-Rothery Atomic Theory for Students of Metallurgy. Institute of Metals 1946.
11. Journal of Physics and Chemistry. X-Ray Investigation of Pseudobinary System of TaC-TrC, CbC-TrC, TaC-ZrC, CbC-ZrC. 20 pp. 769-72 1946.
12. Meyers, Rupert H. The Constitution and Properties Alloys Containing Tantalum and Columbium.

13. Wyckoff, W.G. Crystal Structures. Chapters 1 and 2. Interscience Publishing Company