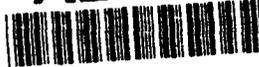


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PRODUCTION AND PHYSICAL METALLURGY OF
PURE METALS -- PART V

- USSR -

by A. I. Yevstyukhin, G. A. Leont'yev,
V. V. Nikishanov, N. V. Borkov,
V. S. Yemel'yanov and Yu. G. Godin

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PRODUCTION AND PHYSICAL METALLURGY OF PURE METALS -- PART V

Arc Melting of Refractory Metals and Alloys under
Laboratory Conditions

This is a translation of an article written by A. I. Yevstyukhin, G. A. Leont'yev and V. V. Nikishanov in Metallurgiya i Metallovedeniye Chistykh Metallov (Production and Physical Metallurgy of Pure Metals), No. 1, Moscow, 1959, pages 106-121.

It is known that melting of rare and refractory metals is carried out either in a protective atmosphere or under vacuum so as to prevent oxidation. However, the melting of these metals in ceramic or graphite crucibles is difficult in practice, and sometimes even impossible. This is especially true of metals subjected to iodide refining. From experiments confirmed by thermodynamic calculations it appears, for example, that zirconium can be melted only in thorium oxide crucibles. Magnesium and aluminum oxides are reduced by zirconium which, in addition, is contaminated by oxygen, cancelling the effect of the iodide refining. Zirconium oxide is easily soluble in zirconium, and the latter becomes contaminated by oxygen. When melted in beryllium oxide crucibles, the metal becomes brittle. Melts in graphite result in carbon contamination up to 0.25%.

Therefore, zirconium melting in induction or resistance furnaces by ordinary methods is very difficult and requires a fundamentally new method of melting.

Such a method is arc melting in a water-cooled copper crucible. The essence of arc melting consists in the ignition of an arc between the specimen placed in an intensively cooled copper crucible, and the electrode. The current is applied to the crucible and the electrode. The specimen maintains a thin unmelted skin in contact with the crucible. Due to this interface there is no reaction between the melted specimen and the crucible. It was found that the unmelted layer assures reliable contact between the specimen

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and the crucible, and no welding occurs.

Both consumable and nonconsumable electrodes can be used in melting. Consumable electrodes are prepared of the metal being melted and consist of a pressed rod or of a bunch of rods secured together at several points. The consumable electrode melts in the course of the operation and drips into the crucible, forming a monolytic ingot. The casting thus obtained is usually of cylindrical shape.

As a rule consumable electrodes are used in industrial arc furnaces adapted for the casting of cylindrical specimens of a certain diameter. The weight of the casting varies between a few kilograms and one ton. There is one industrial furnace that can cast ingots up to 600 mm in diameter, consuming 14,000 amps.

Nonconsumable electrodes are prepared from some refractory metal, most frequently tungsten. They are made reasonably short (30-100 mm), adequately massive (diam 10-20 mm) and are cooled intensively to prevent melting.

In melts using a nonconsumable electrode, only the metal charged into the crucible is melted, and there is no increase in the weight of the ingot due to contribution from the electrode. Nonconsumable electrodes are used in arc furnaces of the laboratory type in cases where small ingots of variable compositions are to be prepared.

Both AC (from welding transformers) and DC are used in melting operations. DC is produced with selenium or germanium rectifiers or by generators with characteristics such as to permit shorting, the current being no more than 20 to 50% of the rating. Welding generators are used predominantly in laboratories and in small industrial furnaces.

Melting by DC arc is preferable, as the arc in this case is more uniform, and about two-thirds of the heat energy is delivered at the anode. Bombarding the anode, the electrons give up not only their kinetic energy but also the work function -- "the latent heat of electron evaporation." The cathode is reached by fewer ions (which bombard and heat it) than the number of electrons reaching the anode. In addition, heat energy is spent for the ejection of electrons from the cathode. Therefore, power consumption being the same, DC can heat the specimen to a higher temperature than AC. In a DC arc the transfer of matter occurs mostly from the anode to the cathode, which precludes the contamination of the specimen by the material of nonconsumable electrode.

Tests have shown that there exists a definite dependence of the voltage differential in the arc, which is usually 20-35 v, upon the width of the gap between the electrode and the molten specimen. The voltage differential increases

with the increase of the gap. This circumstance is utilized in the operation of consumable electrodes in industrial arc furnace melting. Visual observation of the course of melting is done with one eye through the port, i.e., nonstereoscopically. It is therefore difficult to evaluate the gap. Moreover, the gap is frequently concealed from the operator by the nonmelted portion of the consumable electrode. It is therefore preferable to maintain constant the size of the gas gap by observing the voltage of the arc. Its increase above normal indicates that the gap is large, its decrease below normal, that the gap is small.

This dependence is much more complicated in vacuum furnaces.

Thus, utilizing arc melting, one avoids the difficulties connected with the use of ceramic crucibles.

Contaminations in arc melting are very low. It is known that iodide zirconium hardness depends on oxygen content. The addition of 0.01% by wt. increases the Brinell hardness by 10 points. Laboratory tests of arc melting of zirconium in an inert atmosphere using a nonconsumable electrode showed a 5 point increase in Brinell hardness.

Relative elongation of iodide chromium specimens remelted in an arc furnace amounts to 8-10%, with a relative cross section reduction area of 8-16%, proving that such specimens are ductile.

According to data in the literature, the contamination of samples by tungsten does not exceed 0.01%, and that by copper 0.03%, even when melting is done under most unfavorable conditions (new, rough-surfaced crucible, arc ignition by contact with the specimen).

Arc melting requires much less time than induction or vacuum melting in a resistance furnace, i.e., it is more productive. Arc melting eliminates the necessity for high-cost ceramic crucibles which can be used only once, and eliminates metal losses resulting from crucible impregnation.

All these advantages of arc melting prompted our laboratory to work out the design of a universal laboratory arc furnace, (A) as only schematic diagrams were given in the literature and they referred to special furnaces for melting specimens of definite shape, without giving descriptions of furnace elements.

It must be noted that the design described below is the result of a number of experiments and many modernizations of separate assemblies; this work still continues, along with the operation.

(A) The furnace was designed by G. A. Leont'yev, Engineer

Since our object was the preparation of refractory metal alloys into which high vapor-pressure alloying additives were to be introduced, we decided in favor of melting in inert gas at atmospheric pressure, as in this case the changes in charge composition due to evaporation are minimal.

The general view of the universal laboratory arc furnace is shown in Fig. 1. It consists of a melting chamber mounted on a bench by means of studs. The studs make a girder over which a hoist lifts the body of the melting chamber, giving access to the crucible. At the same time these tubular studs convey cooling water to the melting chamber.

The hermetic melting chamber (Fig. 2) consists of a body (1) with electrical leads (2) and bottom (3) with crucible (4).

A broad branch (5) with an observation glass port (6) at its end is used for following the course of melting. Frame (7) with a dark filter can be tilted over the inspection glass of the port so as to protect the operator's eyes. The slot diaphragm (8) protects the dark light filter in front of the observation port from overheating.

The furnace is lighted inside by means of a small left branch while the small right branch (1) is provided for the introduction of a manipulator (11) with which the specimen can be turned over or a bunker handled, which is installed when cylindrical specimens are being melted. Electrical lead (2) with the nonconsumable tungsten electrode (13) is introduced through the upper wall of the furnace. The electrode can be lowered to any point at the surface of the crucible by means of handles (14) without disrupting the hermetic seal. For this purpose the corrugated joint (15) with corresponding rubber gaskets and ball support (16) are provided. The limiter (9) prevents creasing of the corrugated joint during evacuation. The electric lead is counterbalanced by a weight which permits its installation at any distance from the specimen.

The crucible has a set of easily exchangeable copper bottoms which are affixed to the crucible by bolts (22) made water-tight by a rubber gasket (21).

The set consists of a bottom (18) for casting cake-shaped specimens, a bottom for casting rod-shaped specimens and one for casting open rings.

Initially, crucible parts (3), (4), (19), (24), (25) were joined by welding or hard solder, which caused warping of these parts. At present they are joined by threads filled with tin while hot. This permits easy detachment of any part if required.

The chamber, leads, and crucible are water-cooled, and the cooling can be regulated by valves.



Fig. 1. Universal laboratory arc furnace with cooled copper crucible:

Legend continued on
next page

Fig. 1 legend continued from Page 57

1 - melting chamber, 2- bench, 3 - studs for lifting the melting chamber and for cooling the furnace, 4 - bunker, 5 - lighting device, 6 - vacuum pump, 7 - argon bottle, 8 - high frequency exciter, 9 - tank of welding transformer, 10 - argon purifying furnace.

The specimens being melted are placed at the edges of the crucible near the safety ring (20). One after the other they are shifted into the melting cavity by means of the electrode.

For melting the opposite side, the specimen, while still hot, is grasped by the manipulator, turned over, and melted again. The completed specimen is pushed out by the manipulator or electrode into its initial position, and the next specimen is shifted into the melting cavity.

If a rod must be melted, the metal cake on the bottom is placed over the casting channel, and, when melted, it fills the channel contour. For better equalization, the sample is turned over several times by the manipulator.

Cylindrical specimens are melted in a special device. To do it one must 1) dismount the crucible bottom and install the device for melting cylindrical specimens; 2) affix the bunker supports; 3) exchange the manipulator flange for the bunker flange, the bunker being filled in advance with melting material (not more than 5-8 g); and 4) connect the cooling system.

A schematic diagram of the melting chamber with a device for casting cylindrical samples ⁽¹⁾ is shown in Fig. 4.

We use the top-melting principle with simultaneous sinking of the specimen. For this purpose the crucible bottom (3) was made movable. Before the melt, it is sunk 10-15 mm by means of a hand wheel (5). A light knock on bunker (6) charges a layer of metal 6-10 mm high into the crucible. When melted, a portion of the metal fills the dovetail groove in the upper part of the bottom, solidifies there, and forms a reliable bond between ingot and crucible bottom. Further charge additions raise the level of the molten bath, while the lower part of the ingot solidifies due to intensive cooling of crucible bottom and walls.

The melting is conducted in such a way that the molten metal does not rise above the cylindrical (calibrating) part

(1)

The device is designed by V. V. Nikishanov, Engineer

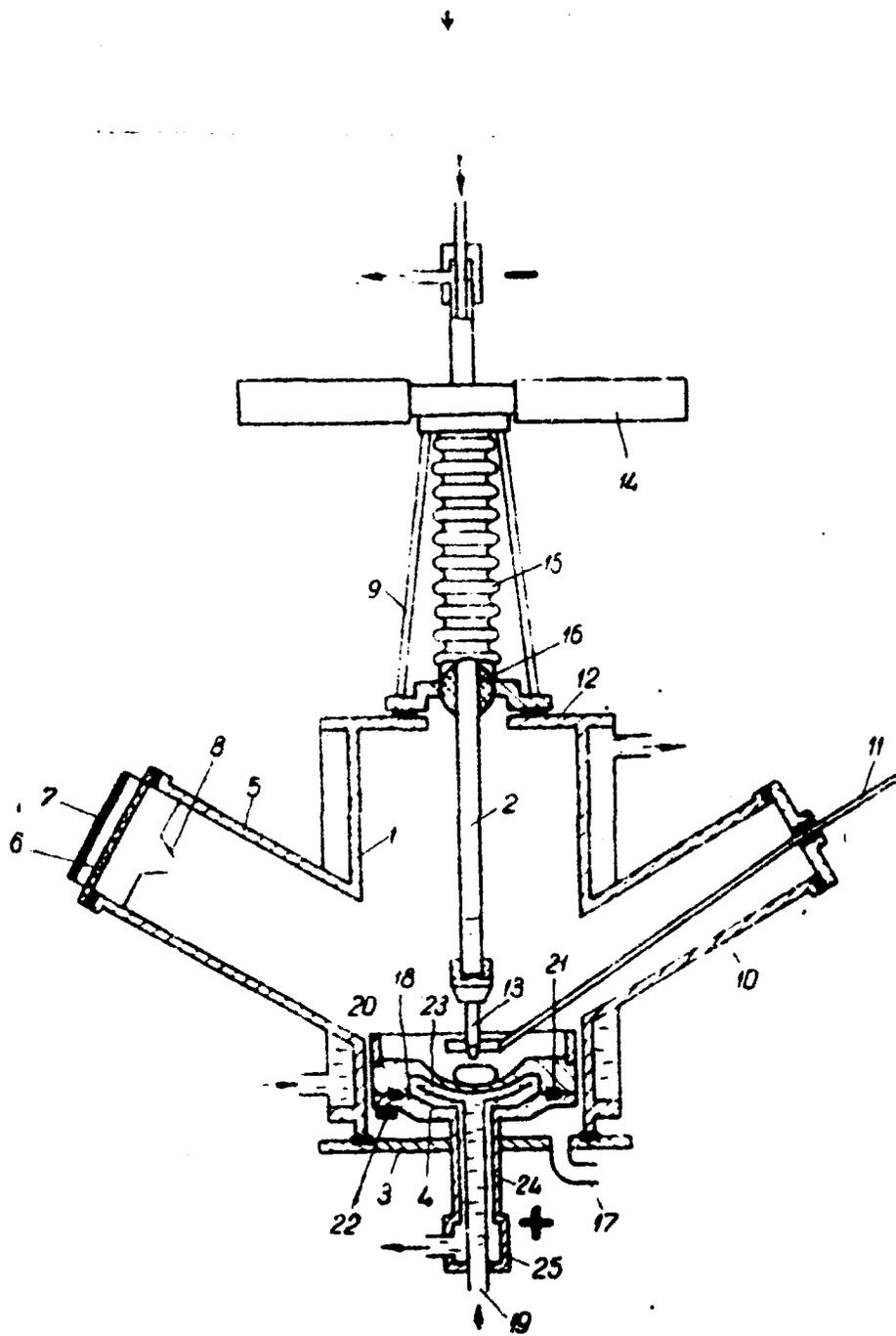


Fig. 2. Schematic diagram of melting chamber in universal laboratory arc furnace:

1 - chamber body, 2 - current lead, 3 - sole of melting chamber, 4 - crucible, 5 - side branch, 6 - glass port, 7 - swivel frame with dark filter, 8 - slot diaphragm,

[Legend continued on next page]

[Fig. 2 legend continued from page 17

9 - limiter, 10 - minor right branch, 11 - manipulator,
12 - upper chamber wall, 13 - tungsten electrode,
14 - electrode moving handles, 15 - corrugated junction,
16 - ball support, 17 - elbow for evacuation of chamber
and filling with inert gas, 18 - bottom for cake-
shaped castings, 19 - cooling water pipe, 20 - ring
preventing specimen from falling to furnace bottom,
21 - watertight gasket, 22 - bolts securing crucible
bottom, 23 - diaphragm for uniform bottom cooling,
24 - electric lead to crucible, 25 - crucible contact.

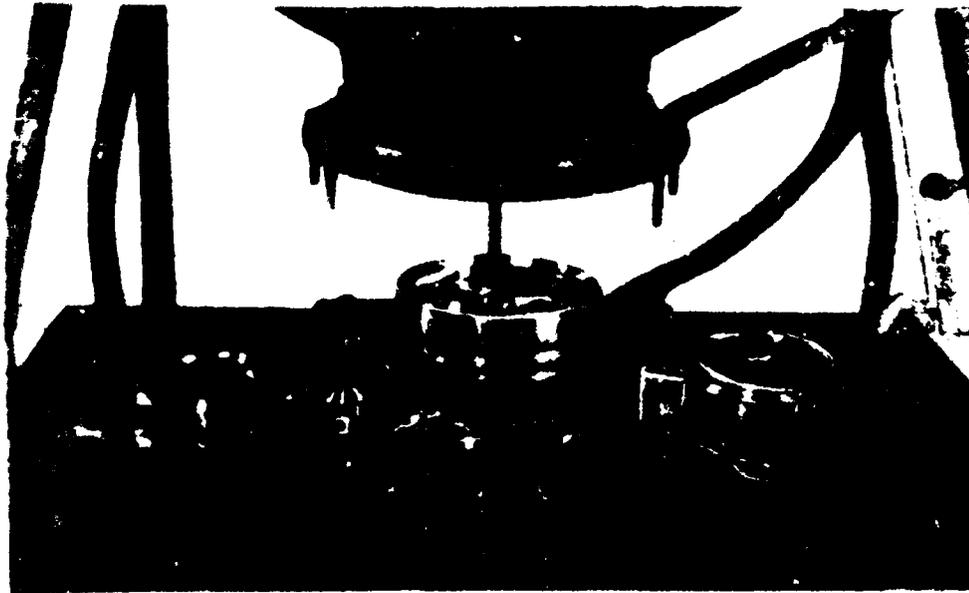


Fig. 3. Arc furnace crucible.

The crucible has a bottom installation for rod casting. Next to the safety ring are previously cast specimens. A cake-shaped specimen in the casting cavity is shown in the first equalizing, remelting stage. In the casting channel is an alloy remelted into a rod. To the left is a bottom for the cake-shaped castings. In front of it are different types of nonconsumable electrodes. At the right is a bottom for open-ring castings.

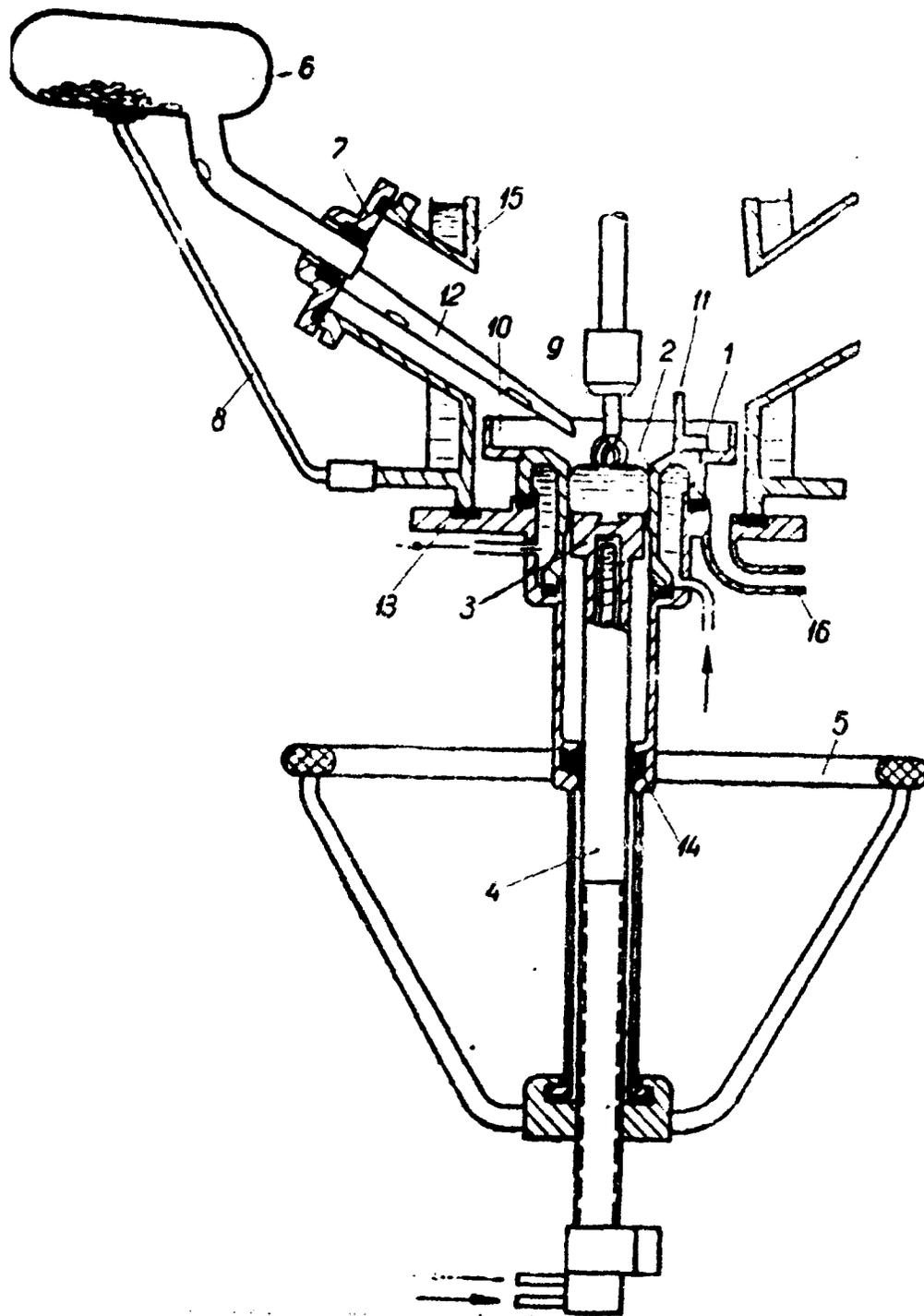


Fig. 4. Schematic diagram of device for melting of cylindrical samples:

Fig. 4 legend continued from page 97

1 - exchangeable crucible body, 2 - calibration part of crucible, 3 - crucible bottom with dovetail groove, 4 - shaft for moving the bottom, 5 - handwheel for actuating the shaft, 6 - bunker, 7 - bunker flange, 8 - adjustable bunker bracket, 9 - tungsten electrode, 10 - ring preventing the charge from dropping to the bottom, 11 - deflector preventing scattering of charge over the crucible, 12 - metal chute from bunker to crucible, 13 - bottom of melting chamber, 14 - shaft vacuum gasket, 15 - body of melting chamber, 16 - elbow for evacuation of melting chamber and filling with inert gas.

(2) of crucible body (1). To achieve this end the ingot is periodically sunk along with the addition of charge. A uniform melt of the upper surface of the ingot is achieved by the operator who moves the tungsten tip (9) along a circular path at a distance of 5-10 mm from the crucible walls. The molten specimen is cooled in the crucible, pushed out of the device, and removed by sliding it along the groove.

When alloys must be prepared, weighed portions of charge of prescribed composition undergo prior melting into rod ingots. The rods are cut into 5-8 g pieces, a required quantity of which is charged into the bunker and melted into an ingot of cylindrical shape. Such a method produces a uniform alloy.

Crucibles are interchangeable so that specimens of different diameters can be prepared.

For zirconium-based alloys, the time required for the melting of one specimen, depending on its weight and shape as well as the number of specimens simultaneously charged into the furnace, is shown in the table.

Exterior views of different shapes of ingots are shown in Fig. 5.

As already mentioned, DC is preferred for the operation of laboratory furnaces. In such a case the nonconsumable electrode is heated to a lesser extent, thus reducing the chance of ingot contamination by tungsten.

The glowing gas column between the electrode and specimen is clearly seen through a protective filter. Where it touches the melt, a cavity is formed due to the energetic bombardment by electrons escaping from the electrode at this point. The latter becomes overheated as compared to the rest of the surface, and convection currents are formed which are clearly visible and which promote better mixing and more

Table

Shape of specimen	Cakes						
	15-25	40-50	80-100	110-160	180-200	230-250	
Weight or size							
Melting time of one specimen, minutes	2-8	8-12	15-20	25-30	30-45	35-40	
Number of simultaneously charged specimens	11-30	7-5	3-2	2-1	1	1	
Current in amps	80-100	140-160	250-300	280-320	320	370	

(A) Upon acquiring certain casting experience it becomes possible to prepare rod specimens of 10-12 mm diam., 50 to 80 mm long, using the casting channel of the furnace bottom.

/continued on next page/

Table continued from page 117

Rods (15 mm diam., length to 85 mm l (A))	Cylinders diam., 30,40,50 mm length to 150 mm)		
20--30	30 50	35 60	40 80
3	1	1	1
320--100	320		

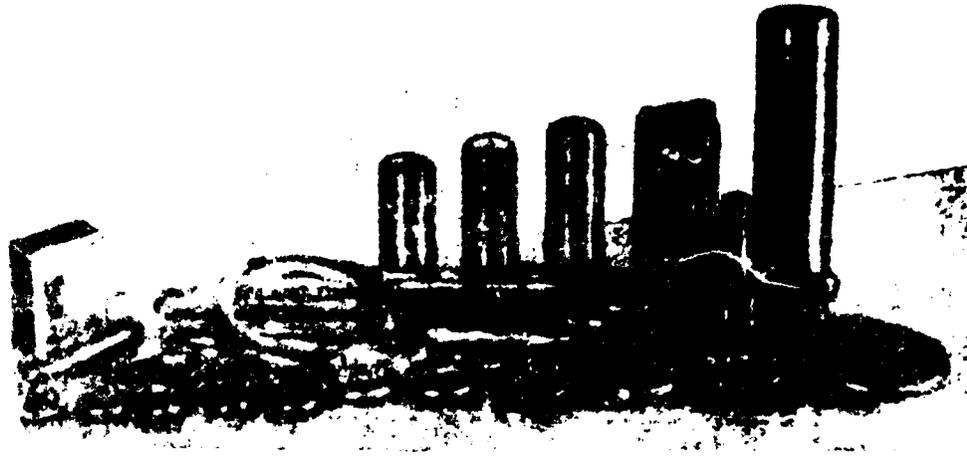


Fig. 5. Ingots cast in universal arc furnace

Long rods are prepared by straightening open rings. Shortened Gagarin samples are machined from rolled rods. An unmachined cylinder shows the protrusion which connected it to the bottom during casting.

homogeneous distribution of the alloying components.

To ignite the arc one usually touches the electrode to the specimen and then retracts it. This method permits the ignition of the arc at relatively low voltages, of the order of tens of volts, whereas auto-ignition in vacuum requires hundred of volts, and autoignition under atmospheric pressure, on the order of tens of kilovolts. A slow withdrawal of the electrode results in welding of the electrode to the specimen whereas lifting both of them ignites an arc between the specimen and the crucible. If small pieces get welded to the electrode a melted drop forms at its end and, as it contains tungsten, there is a hazard of contamination of the melted specimen.

This difficulty was overcome by igniting the arc without contact by ionizing the gap with the aid of a spark generator. To prevent hf discharge through generator windings (Fig. 6), a reactive resistance was incorporated into its circuit.

Most suitable for arc melting are welding generators of the SAM-250 (rated up to 300 amps) and SAM-400 (rated up to 500 amps) types. They are pro-

vided with excitation rheostats by means of which the current and, consequently, the melting temperature, can be regulated within a wide range.

One must keep in mind that the whole current passes through the reactive resistance, and, therefore, the reactor of RTS-500 transformer, rated at 500 amp, must be used.

During melting, the arc is switched on and off many times, and it is expedient to use a push-button switch placed at the bench or affixed to the handles of the leads. Usually magnetic contactors of corresponding ratings are used. If more than 350 amps are required for melting, a KT 3025/2 m contactor is required.

A melting is done in an inert atmosphere, the chamber must be pumped out to a residual pressure no higher than 10^{-2} mm Hg before it is filled with an inert gas. Melting is usually done in an atmosphere of argon or helium. The latter is purer but more expensive. Argon of the "chemically pure" type contains up to 0.05% oxygen and 0.2-0.27% nitrogen. To eliminate these admixtures the gas passes through calcium chips heated to 700° , which at such temperature are able to absorb oxygen, nitrogen, hydrogen, and moisture. A still greater degree of purity can be achieved by subsequent passage of the gas through zirconium chips heated to $700-800^{\circ}$.

As the inner walls of the chamber and the crucible contain gases absorbed from the air, the first specimen to be melted must be a sufficiently refractory absorber of these gases. Usually iodide zirconium, or, preferably, its titanium alloy, in 1:1 proportion, is used as an absorber.

During the melting of such an absorber all sections of the melting chamber which are within the zone of heat radiation are heated through and the crucible bottom is degassed. The liberated gases are moved by convection currents and react with the molten metal, being absorbed by the latter.

During specimen melting, elimination of the gases absorbed also takes place. Around the arc there appears a characteristic colored halo, which disappears upon absorption of the gases.

The gases generated in the chamber are absorbed at three locations. A portion of the gases is absorbed by the glowing absorber which, prior to the melt and upon elimination of the atmosphere, is placed on the shoulder of the crucible, where it is melted slightly. Another portion of gases, expanding with argon in the chamber due to heat, reaches the chamber with the heated calcium chips and is absorbed there. The flexible container (Fig. 6) expands, and, after cessation of the melt, contracts, thereby forcing the purified argon back into the melting chamber.

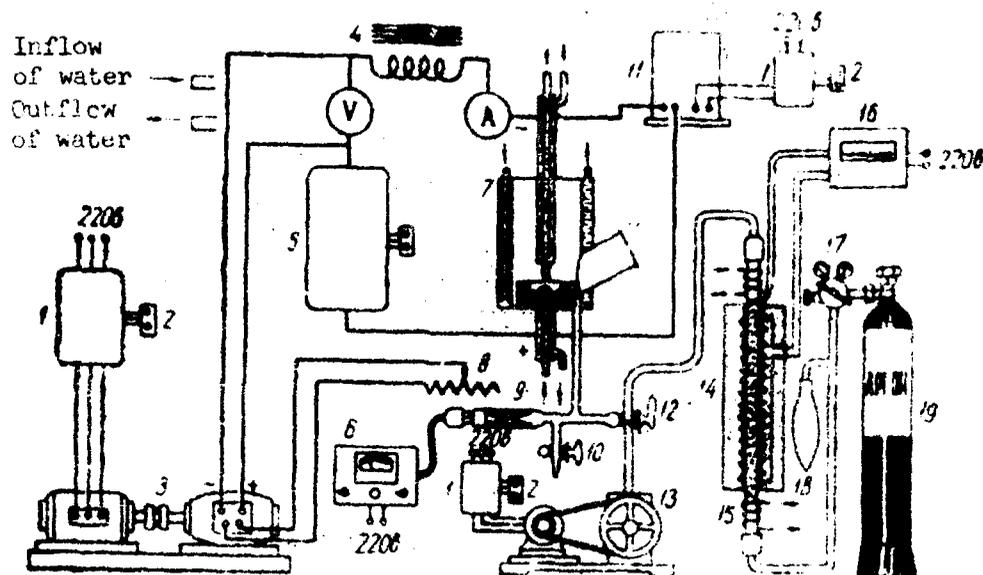


Fig. 6. Diagram of arc furnace connections:

1 - magnetic contactor, 2 - push button, 3 - welding generator, 4 - reactor of welding generator, 5 - contactor, 6 - vacuum-meter, 7 - melting chamber, 8 - excitation rheostat, 9 - gauge tube, 10 - valve to atmosphere, 11 - hf exciter, 12 - three-way valve, 13 - vacuum pump, 14 - furnace for argon purifying, 15 - steel chamber filled with Ca chips, 16 - automatic temperature regulator, 17 - pressure reducer, 18 - flexible container, 19 - argon bottle.

The remainder of gases is absorbed by the specimen itself. Practical experience shows that the contamination of the ingot by nitrogen and oxygen does not exceed a few hundredths of one percent.

In the case of melting under vacuum, the specimen does not absorb liberated gases and the quality of the melt is superior, if the vacuum system has no leaks. However, vacuum melting is possible only for metals with low vapor pressure, and is thus ruled out for our purposes.

In arc melting in an inert atmosphere, the rate of contamination by gases depends to a considerable extent on the granulation of the charge. Such contamination can be considerably decreased by preliminary

degassing of the charge. This is particularly true of metal powders and metals in noncompact state such as are obtained from electrolysis (scales, pieces, etc.). Owing to their great surface area they absorb especially large amounts of gas which can contaminate the melt.

When using powders it is advantageous to remelt them separately after prior degassing. The resulting compact metal is expedient to handle for many reasons. First, powders are frequently coated with oxides which float to the surface of the ingot and must be eliminated mechanically later. Second, spattering of the powder all over the crucible can contaminate specimen awaiting melting. Third, it is difficult to introduce powders into an inhomogeneously granulated charge.

When it is necessary to melt powders and small pieces, an arc is ignited between the electrode and the absorber placed on the shoulder of the crucible until the absorber is melted slightly. The arc glides easily along the metal and can be rapidly transferred to the powder by moving the electrode, so that, the powder is heated and partly degassed. Gases eliminated are taken up by the absorber. By increasing current slowly the spattering of powder is avoided and melting is achieved. In making up the charge it is more expedient to use lumps, rods, foils and wire, as it is easier to prepare from them melt specimens which do not spill.

The more compact the metal, the more quietly it melts, and, conversely, the more porous it is, the more it sputters during melting.

It was established experimentally that homogeneity of the alloy is achieved more rapidly if alloying additives are melted with a portion of the alloyed metal and this product is then melted with the balance of the metal.

The weight of the metal influences the choice of melting procedure. As a rule, one begins with low current so as to avoid melting the whole charge simultaneously. Thus the charge can eliminate the major part of the absorbed gases, and no spattering takes place. Under these conditions only the upper half of the specimen is melted. After turning it over the second half is melted under the same regime. Then the current is increased so that the whole specimen becomes a liquid drop of regular shape.

The complete melting of a zirconium specimen weighing 80-100 g requires 280-300 amp current. In this melting stage alloying additives become uniformly distributed in the melt due to intensive self-mixing of the metal by convection currents. Before the end of melting the current is decreased while uninterrupted motion of the electrode over the specimen continues, so as to cool the latter, uniformly.

In most cases the melts become uniform after 5-7 turn-overs, i.e., after 6-8 remelts.

Arc melting has its peculiar features. Thus, for instance, one cannot melt a compact sample without preheating it. A cold specimen subjected to high current (300 amp for 100 g of a zirconium alloy) melts only to a slight depth directly under the arc, and only after heating does the melted zone widen to cover the whole specimen. If melting is carried out with low current (80 g for a 100 g zirconium ingot), a narrow molten zone is formed directly under the electrode upon heating. This peculiarity is used for "smoothing" the specimen, i. e., for eliminating accumulated metal bulges. The radius of curvature of the sample surface is less than that of the casting cavity when specimens are under 80 g. Such specimens, when turned over, have contact only at the center, and molten metal flowing into the gap between the edges of the specimen and the copper bottom forms an annular bulge on the lower surface of the ingot. When rolling such a specimen into a sheet, torn edges will result. To avoid this, bulges are smoothed by melting with low current.

Specimens weighing over 150 g come out flat, as they have good contact with the bottom. The center is well melted through, and a bulge is formed on the opposite side, which is smoothed by melting with low current.

If the arc furnace is fed from a welding generator of limited rating (current up to 320 amp), samples of over 100-120 g cannot be wholly melted. When heavier (200-250 g) specimens are required, two portions of charge of the same composition must be prepared each weighing, 100-125 g and melted separately into cakes with uniform distribution of additives. Then both cakes can be melted together to make a specimen of required weight.

For better homogeneity the remelted specimen, after being turned over, is pushed out of the center of the casting cavity, instead of being placed over it. It then lies on an inclined surface and, when melted, flows downward, causing additional mixing.

In certain cases homogeneity of the alloy is achieved only after a great number of remelts. This refers especially to zirconium alloys with tungsten, the homogeneity of which is achieved only after 8-15 remelts. In such cases it is especially helpful to prepare an alloy with prior melting of alloying additives (see above). It is possible to prepare in an arc furnace alloys of metals having considerably different melting points -- for example, zirconium (1860°) with tin (232°) -- provided that the metals have low vapor pressures.

In the initial moment of melting, while the sample is

being heated and absorbed gases are being eliminated, a characteristic halo forms around the arc, which disappears after the absorption of gases eliminated from the specimen. Subsequent repeated melting of the specimen does not form a halo and, apparently, does not influence the hardness. When previously degassed specimens are melted, no halo is formed around the arc.

Different metals behave differently in melting. Such metals as zirconium and titanium, prepared by iodide refining, do not leave any traces on the crucible after melting. Tungsten, molybdenum, tantalum, and niobium, although they have low pressure vapors, leave a dark, easily removable film on the crucible. Chromium, silicon, and beryllium leave a film both on the crucible and on the walls of the chamber. This makes cleaning of the furnace after each melt necessary. The chamber walls are wiped with a clean linen rag soaked in some volatile organic solvent which was previously purified by distillation. The crucible is cleaned in the same way, while the iridescent tarnish caused by the contact of the arc flame with copper is removed with fine emery paper.

When the furnace is charged with several different specimens, the sequence of their melting is selected so that samples containing alloying additives of low vapor pressure are melted first, and those of high vapor pressure come last. In such a case the remainder of the samples will not be contaminated by volatile additives. When the melting program comprises first variations of one alloying additive and then, variations of another additive, etc., it is more expedient to melt during one charge ingots with the same additive.

Observations of changes in the weight of ingots after melting showed that specimens of zirconium alloys in the solubility range lose on the average 0.01 g if the alloying additives have low vapor pressure, and 0.02-0.03 g if alloying additives have high vapor pressure.

To avoid absorption of gases by the crucible and chamber walls, the latter is usually kept under vacuum. The purging system is then switched off from the furnace and remains filled with argon.

Considerable interest in arc melting is shown by industry and research laboratories. This explains the fact that the universal laboratory arc furnace described us us was demonstrated at the All-Union Industrial Exhibition in 1956 and 1957.

Construction Of and Operational Experience With The High
Temperature MIFI-9-2 Furnace With A Graphite Heating
Element For Vacuum Melting and Casting of Pure Metals
and Alloys

This is a translation of an article written by A. I. Yevstyukhin, G. A. Leont'yev and N. V. Borkov in Metallurgiya i Metallovedeniye Chistykh Metallov (Production and Physical Metallurgy of Pure Metals), No. 1, Moscow, 1959, pages 122-127.

Introduction

The authors designed and built a high temperature vacuum furnace with a graphite heater (Fig. 1) for the purpose of melting pure metals and prepare melts weighing 50 to 1,000 grams.

The furnace has a device for vacuum distillation in the shape of a long condenser. The molten metal can cool in the furnace or can be poured out into a mold situated below. If necessary, the melt can be carried out in an inert gas atmosphere. The furnace permits melting in graphite crucibles (in cases where low contamination by carbon is admissible) as well as in graphite or purely ceramic crucibles coated inside by oxide. In the latter case, the ceramic crucible is inserted in a graphite crucible suspended at the heater.

The furnace was built in the experimental workshop of Moscow Physical Engineering Institute.

The MIFI-9-2 furnace has been in operation in the laboratory of the institute for more than five years. During this time, more than 250 melts have been made. All parts of the furnace are in working condition and have not needed repairs. Furnaces of this design are operating in laboratories of certain plants and scientific research institutes.

Furnace Design

The vacuum chamber (diameter 500 mm, height 660 mm) of the furnace (Fig. 2) consists of a steel body (1) steel lid (2) condenser for vacuum distillation (3) and copper bottom (4). Steel parts have water jackets for cooling. Two channels in the copper bottom are provided for this purpose; one along the perimeter and another along the electric leads.

The body and the bottom are secured by bolts with the help of a permanent vacuum gasket 22. They are disconnected only when the furnace is dismantled. The lid is affixed to the body through a vacuum gasket by four bolts with hinged nuts. The lid is easily lifted off by a special hand-winch with boom.

At the bottom of the furnace on a layer of rubber are situated two cast copper electrical contacts (5) in form of segments spaced apart at a distance of 10 mm. They have inner channels for cooling with water and occupy the entire bottom of the furnace. Thick-walled copper tubes carrying water to and away from the electric contacts are used simultaneously for the transmission of current.

The graphite heater (7) is a tube which is thinner at its upper part; the height of the heater is 375 mm, the inner diameter 62 mm (Figs. 2 and 3). At its lower part the heater is cut across its diameter, from the bottom up to 30 mm from the upper end. The heater is in solid contact with conical half-rings (8) with cooled electric contacts. During heating, it can expand vertically without breaking contact.

The heater is protected by four screens (9-12) which decrease heat losses; these are affixed to electric contacts (5) through porcelain insulators. For the same purpose, two small hanging graphite screens (not shown in Fig. 2) are suspended on the heater. All screens are provided with lids (13-15). In the lid of the condenser there is an observation port (18) for following the melt and there are openings in the screen lids. Above the observation port is installed a rotation prism (19) with the help of which the operator, using an optical pyrometer (20), watches the melt and makes temperature measurements.

The whole furnace is mounted on a platform 1.5 meters above the floor. This facilitates easy access to the bottom of the furnace and to the vacuum seals (21) and current leads (6).

As was mentioned, the bottom of the furnace is made of copper. The use of non-magnetic material for the bottom is obligatory. Otherwise Foucault currents arising at the bottom would overheat it. The furnace is pumped out to a residual pressure of 10^{-3} mm Hg by means of a para oil diffusion pump with a capacity of 500 l/min connected with a pump for preliminary evacuation (type VN-1).

The pump and the step-down transformer feeding the heater are situated above the platform on which the furnace is located. The 20 kw step-down transformer has seven voltage steps, which permits, with the aid of heat inertia, a rather smooth increase of temperature from 900 to 2000°C.

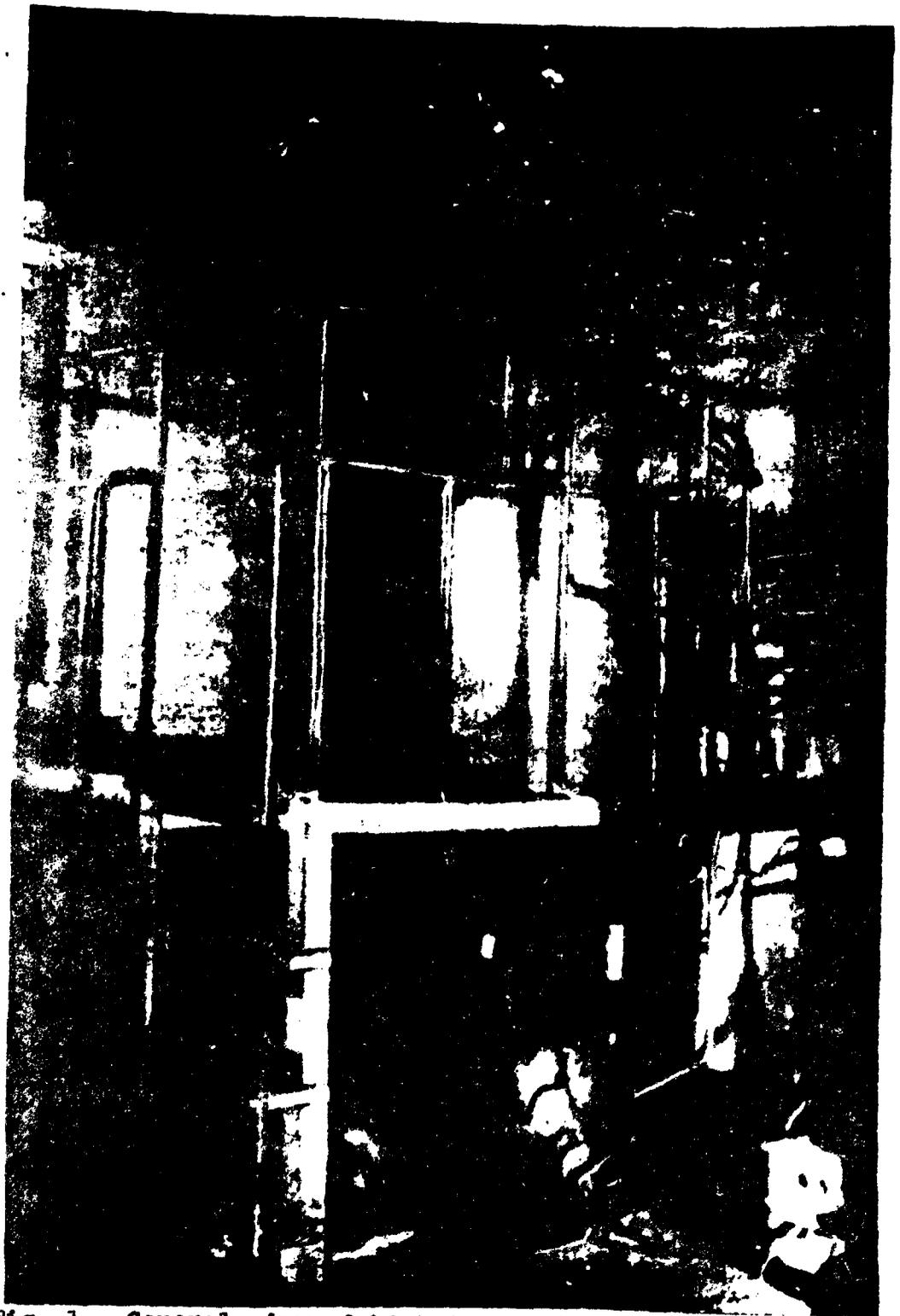


Fig. 1. General view of high-temperature vacuum furnace with graphite heater.

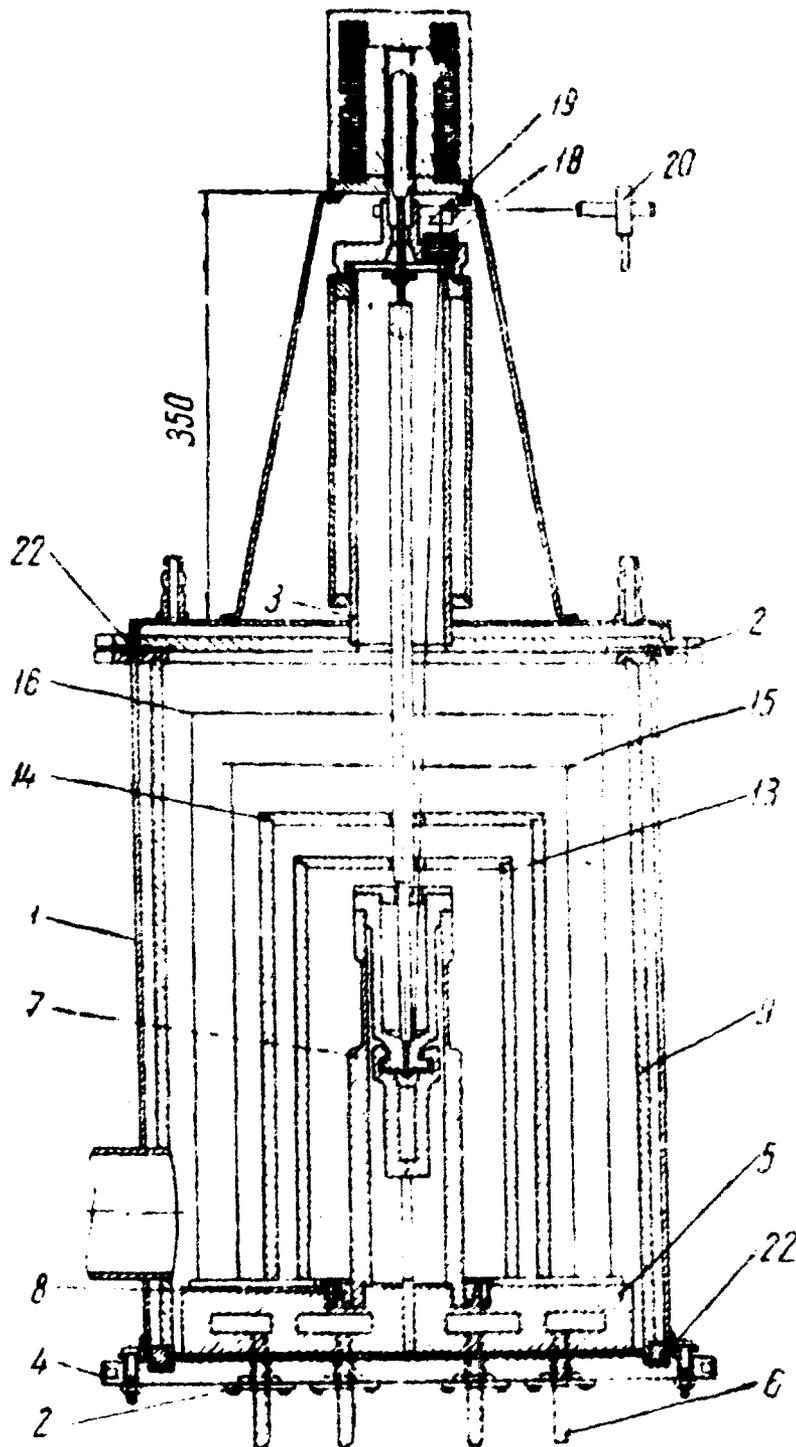


Fig. 2. Schematic diagram of high-temperature vacuum furnace with a graphite heater.

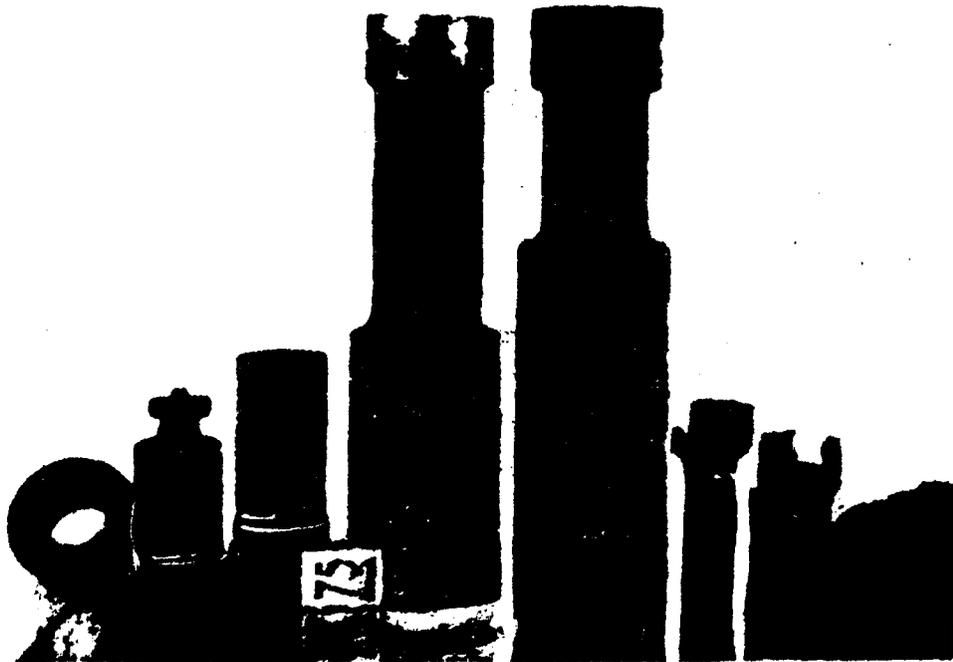


Fig. 3. Crucibles, heaters and molds for high-temperature vacuum furnace:

a, b, parts of sectional crucible with casting channel; a - upper part of ordinary graphite crucible; b - lower part of dense graphite crucible; c - melting crucible, no casting provided; d - exterior view of heater with elongated heating part; e - half of the heater with sectional crucible in it; ordinary mold is affixed to crucible; f - ordinary mold with the metal cast in it (rod diam. 12 mm, length 70 mm); g - specimen cast in mold with shaped insert; h - mold with insert for simultaneous casting of three rods; next to it - castings.

For smooth control of temperatures to $1,000^{\circ}\text{C}$, a voltage regulator is coupled with the primary circuit of the transformer.

There is a hydraulic contact which switches the heat off when furnace cooling ceases.

When melting takes place in ordinary crucibles (Fig. 3, b), metal is placed in the bunker at the upper, wider portion of the crucible. When melted it runs into the lower part of the crucible which is the

mold. The metal is cooled together with the crucible and the heater. When the melt provides for casting, a crucible with a casting hole is used (Fig. 3, d). The mold to be used is suspended from the crucible. The casting hole is plugged with a carbon rod which can be lifted by a magnetic device (Fig. 2). The metal is cast in a cold mold and solidifies immediately.

Operation of the Furnace

Practice has shown that melts can be considerably speeded up if the crucible and the mold are degassed prior to use by heating them in the furnace to 1900°C. In this case the probability of melt contamination by gases from the crucible is greatly reduced. The operation takes 5-7 hours, including cooling of the furnace. It is not recommended that a hot furnace be opened, as all its interior parts easily can absorb a large quantity of gas, the elimination of which will require considerable time.

Heating of a sample to its melting point (1800°C) in a degassed crucible takes 2-3 hours, after which the furnace cools for 4-5 hours.

When melting zirconium alloys containing Al, W, Mo, Cr and other metal additives, 9 to 20% of weight of the charge was expended for impregnation; in alloys with Ti, Ta, and Nb additives, impregnation used up 30-50% of the charge.

Contamination of alloys by carbon did not exceed 0.25%. The mold or the insert in it must be broken to extract the cast specimen. The upper 0.5 mm layer of the specimen, which contains carbides, is usually eliminated.

The selection of the type of graphite for crucibles is of great importance. The less the porosity of the crucible, the less it will absorb metal. The best types of graphite having no through pores are the ARV and PK-0.

For molds, heaters and inserts, graphite of which electrodes for steel melting furnaces are made is used.

For purposes of saving dense graphite, crucibles were made in two sections (Fig. 3, a and b) and were joined by threads. The upper portion, which is not in contact with the melting metal, was made of the ordinary, lower-density graphite ARV.

In certain cases the crucible (Fig. 3) was made of ordinary graphite and had an insert of dense ARV graphite or PK-0. In this case, the melt was cooled in the crucible together with the furnace. Due to the limited power of transformer (20 ks), the heating section of the heater had to be made thinner so as to reach a temperature of 2,000°C.

(Fig. 3, d). In this case the heater carried a current of 1000-1100 amp at 17.5 v with a current density of 3.7 amp/mm².

One of the heaters worked for about 200 hrs. During this time no fewer than 50 heats were made. The breakdown of a heater consists in the cracking of its heating part. Heaters can be exchanged easily and rapidly.

Mechanical Properties of Binary and Ternary Alloys
of Zirconium With Tantalum and Niobium At Room and
Elevated Temperatures

∕This is a translation of an article written by V. S. Yemel'yanov, Yu. G. Godin and A. I. Yevstyukhin in Metallurgiya i Metallovedeniye Chistykh Metalloy (Production and Physical Metallurgy of Pure Metals), No. 1, 1959, Moscow, pages 128-143.∕

Introduction

The mechanical properties of binary and ternary zirconium alloys with tantalum and niobium have been described in a series of works.

Anderson and others ∕1∕ determined the mechanical properties of forged specimens of zirconium alloys with 1.2, 5.3, 9.7 and 14.1% tantalum and with 0.6, 5.1 and 12.9% niobium at room temperature and 650°. The alloys were prepared from zirconium sponge by induction melting in graphite crucibles.

The mechanical properties of binary alloys of zirconium with tantalum (0.5-27.5%) and niobium (2.5-27.5%) were investigated by Litton ∕2∕. Tantalum alloys were investigated after annealing at 700 and 725°, heat treatment, rolling, and pressing, while niobium alloys were studied after annealing at 700°. Specimens were prepared by induction melting in graphite crucibles.

Schwoppe and Chubb ∕3∕ determined the mechanical properties of binary alloys of zirconium with tantalum and niobium at 500° prepared by melting in graphite crucibles. They also found that tantalum alloys at temperature above 600° possess greater hardness than niobium alloys. Saller, Stasy and Poreinbka ∕4∕ arrived at the same conclusions.

In the review article ∕5∕ it is indicated that niobium influences hardness of zirconium alloys to a greater extent than does tantalum.

Yu. V. Bykov, A. N. Rozanov and D. M. Skorov have plotted the curve showing the dependence of strength on composition of Zr-Nb alloy systems annealed at 700°, with a maximum at about 7.5% Nb. Curves were also plotted for the dependence of hardness on composition of alloys cast and hardened from 750°, showing a maximum at the same level of niobium. In the curves of hardness vs composition for specimens cooled slowly from a temperature of 650°, no maximum

was observed.

The published data show that the influence of tantalum and niobium on the mechanical properties of zirconium has not been fully clarified. Data concerning the influence of heat treatment and temperature on mechanical properties of binary alloys are also scant. There are no data at all relating to mechanical properties of ternary alloys of zirconium with tantalum and niobium.

The authors of this article have studied the hardness and strength of cast and heat-treated binary and ternary alloys of zirconium with tantalum and niobium at and above room temperature.

Test Preparation and Procedure

The hardness of zirconium alloys with tantalum (0-100%) and with niobium (0-20%), as well as that of ternary alloys containing up to 18% tantalum and niobium -- the compositions of which lie in polythermal cross section of the system Zr-Ta-Nb with ratios $\frac{\text{Nb}}{\text{Ta}} \% = 0.2, 0.5, 1.0, 2.0, 5.0$ (the serial numbers of these cross sections being 1, 2, ..., 5) -- were studied in cast and hardened state (after isothermal annealing) with the use of samples which were investigated for state diagrams [7], [8]. Specimens of pure zirconium and its alloy were prepared separately according to methods described in [8] for hardness studies at elevated temperatures. They were subjected to homogenizing annealing at a temperature of 1200° for 40 hours and were forged at 600-800° in air. Alloys with low tantalum and niobium contents could be forged easily; with increasing quantities of alloying elements, however, malleability deteriorated. The scale formed on the surface of the ingots during forging was removed on an emery wheel. Annealing of samples was done in vacuum at temperatures lower than the eutectoid lines for systems Zr-Ta and Zr-Nb in the region of α, β, γ systems Zr-Ta-Nb. Annealing regimes are shown in Table 1.

The hardness of binary and ternary zirconium alloys containing 1% Ta and 1.5% Nb and 2% Ta and 3% Nb was measured on flat samples, while alloy cross sections 1, 3, and 5 were tested on cylinders about 10 mm in diameter and 60-80 mm long.

The installation shown in Fig. 1 was designed for measurements of hardness at higher temperatures. A pobedite cone with an angle ground at 90° was used as indenter. In the course of the work, the indenter was checked periodically against a standard block for hardness. In cases of

incorrect data, the cone was reground.

Alloy hardness was measured in an atmosphere of chemically pure argon at slightly above atmospheric pressure. Samples were honed and washed in acetone before being placed in chambers. Six to ten imprints were made on each specimen for each temperature. A constant temperature for each measurement was maintained with the help of a contact galvanometer. Samples were kept at the temperature of the test for 45 minutes before measurements were taken.

Loading was done on a Rockwell instrument. During measurements the load was 60 kg, applied for 1 min. The diameter of the imprint was ascertained with a cathetometer. The value of hardness was calculated as the relation of imposed load to imprint area according to the formula.

$$H_k = \frac{p}{1.1 \cdot d^2},$$

After the measurement of hardness at elevated temperatures, strength characteristics were studied on cylindrical alloy specimens belonging to polythermal cross sections 1, 3 and 5. In addition, melted specimens of pure zirconium and binary alloys containing 1, 2, 3, 5, 7 and 10% tantalum and niobium were also tested.

Specimens of binary and ternary alloys were forged into rods of 6-7 mm diameter. The scale on the rods was cleaned off with an emery wheel, and the rods were then subjected to heat treatment. Binary alloys in the annealed (below the eutectoid line) and in the hardened (-range) states were investigated at room temperature. Ternary alloys were investigated only in annealed state (lower than the range of the Zr-Ta-Nb system at 350°. Heat treatment regimes are shown in Table 2.

The mechanical properties of small Gagarin specimens were measured on an IM-ChA testing machine. The elongation diagram was recorded and determined as the average value of three measurements. For measurements at 350°, a device was used, after small alterations, which was designed for hardness measurements at elevated temperatures. Measurements were carried out in an atmosphere of chemically pure argon at pressures slightly above atmospheric. The size of the device permitted its installation on the testing machine, and an elongation diagram was recorded. Prior to the measurements, specimens were kept at test temperatures for one hour.

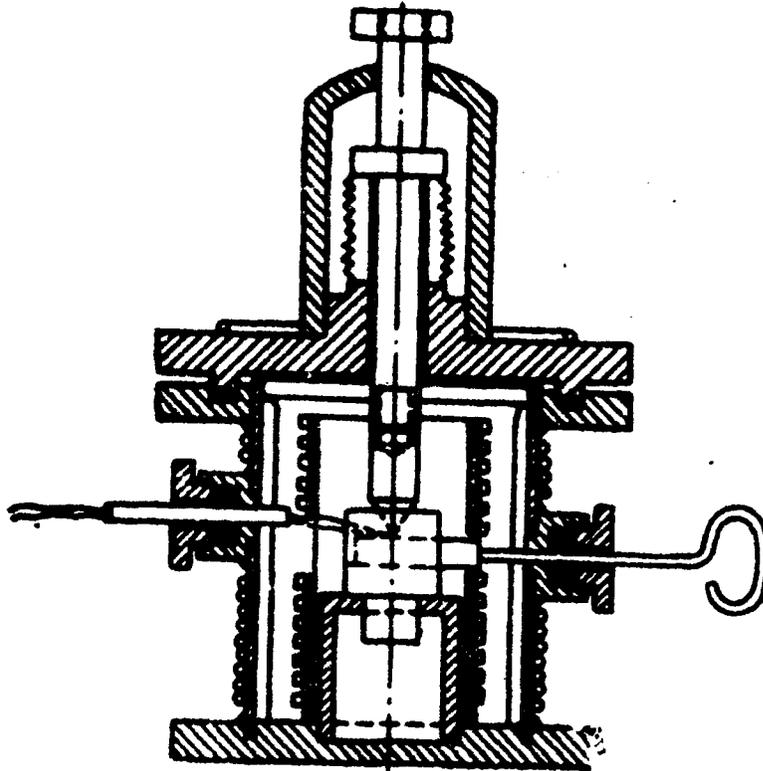


Fig. 1. Installation for measurement of hardness at elevated temperatures.

Table 1

Aerialing results	V101 13/2 13/2 13/2	1 1 1 1 1 1				
		results 13/2 13/2	gross weight	gross weight	gross weight	
Temporal no. 100	770	600	715	650	600	625
Other no.	2	4	2.5	3	4	4

Table 2

Treatment regime	Alloys				
	with Ta 1, 2, 3, 5, 7, 10% pure Zr	with Nb 1, 2, 3, 5, 7, 10%	Gross Section 1	Ternary Cross Section 2	Gross Section 3
Annealing, °C	770	600	715	650	600
Duration of annealing, hrs.	2	4	2.5	3	4
Hardening after 15 min. aging, from °C	1000	1000	—	—	—

The Hardness of Cast and Hardened Alloys

The introduction of tantalum and niobium into zirconium increases its hardness considerably. Cast alloys, both binary and ternary, show clearly, in the curves of hardness vs composition peaks which are also observed in corresponding curves of alloys isothermally hardened in water from temperatures in the B-range (Figs. 2 and 3). An exception is the first polythermal cross section where this peak is absent and the second cross section where the peak is scarcely noticeable. However, the presence of a peak on hardness vs composition curves for alloys in the Zr-Ta system of the third cross section (corresponding approximately to 28% Ta and 13% Ta and Nb) shows that the peak must also exist on the hardness curves of both the first and the second cross sections; these peaks would occur, however, beyond the range of concentrations being studied.

In the hardening of one alloy from different temperatures, the maximum corresponds to a certain composition, i.e., its position is independent of the temperature of hardening. A slight discrepancy in the peak position on curves of hardness vs composition of cast and hardened alloys is caused by the homogenizing annealing of the latter. However, both in the binary systems Zr-Ta and Zr-Nb and in polythermal cross sections of the ternary system Zr-Ta-Nb, the peaks correspond to different contents of tantalum and niobium. On the curves of hardness vs composition of hardened alloys. The peaks of the dual systems are situated near 28% Ta and 7.5% Nb respectively. For the ternary system, it is on a straight line connecting these compositions. Thus, if one draws a straight line on the plane of the concentration triangle connecting the composition of 28% Ta and 7.5% Nb, a range of concentrations will be obtained in which the hardness and, consequently, the hardness of the alloys increases with increase of tantalum and niobium content, when hardening is done from the δ -phase range. A further increase in concentration of alloying elements beyond the limits of this range results in decreased hardness of alloys hardened from the same temperature range.

The existence of a peak on the curves of hardness vs composition for binary and ternary alloys when the hardening is carried out from δ -phase ranges is caused by martensite transformation of the cubic δ -phase into the hexagonal ϵ -phase.

The curves of hardness vs composition of cast alloys of the Zr-Ta system, isothermally hardened from 1200°, show, in the tantalum-rich portion, a clear decline because of the formation of an area of continuous solid solutions of

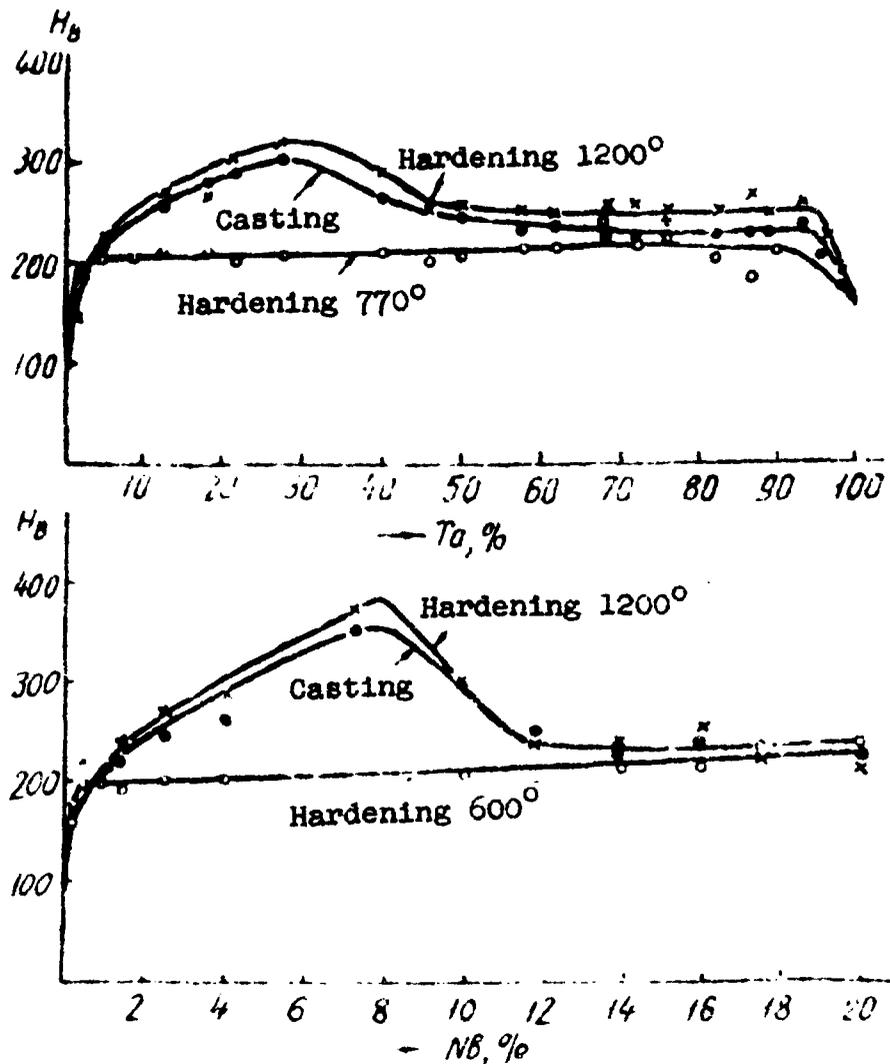


Fig. 2. Curves of composition vs hardness of cast and hardened alloys in the systems Zr-Ta and Zr-Nb.

zirconium in niobium

When binary and ternary alloys were hardened from temperatures lower than those of the β -phase range, no peaks on the curves of hardness vs composition were observed. After a rapid rise of hardness due to the introduction into zirconium of small

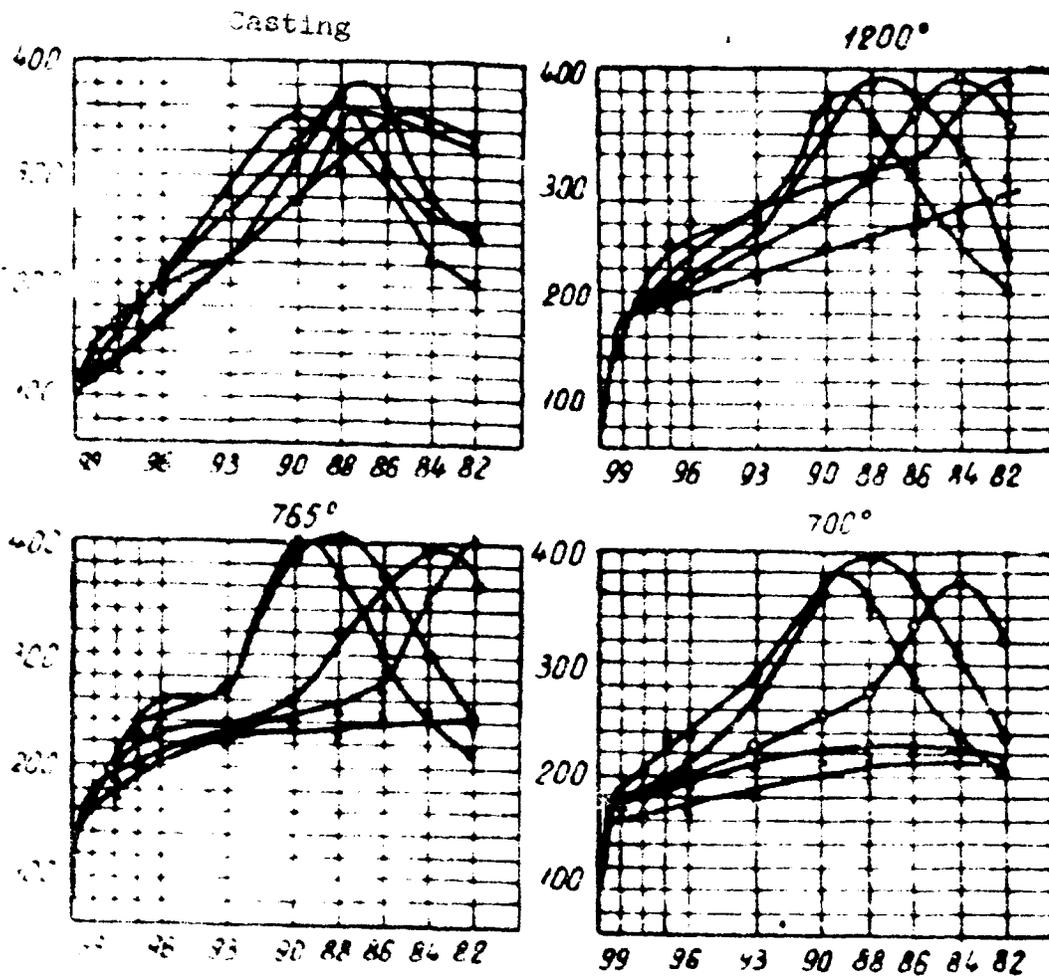
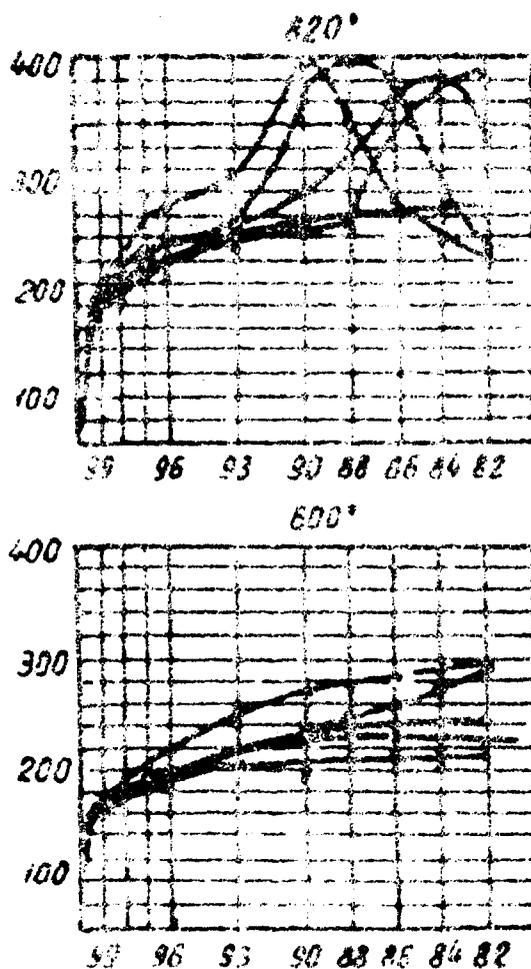


Fig. 3. Curves of composition vs hardness of cast and hardened alloys in the system Zr-Ta-Nb for six polythermal cross sections with a constant Nb/Ta ratio.

[Fig. 3 continued
on next page]

Fig. 3 continued
from page 34



quantities (less than 1%) of tantalum and niobium. The curves show a milder slope on a further increase in alloying elements. Considerable increase in the hardness of zirconium through small additions of alloying elements is caused by the existence of a narrow barrier of solid solutions of tantalum and

niobium in α -zirconium [7], [8].

As mentioned above, peaks on the curves of hardness vs. composition of alloys hardened from the β -phase range shift in the ternary Zr-Ta-Nb system from the boundary Zr-Ta to the boundary Zr-Nb, i.e., toward a smaller amount of zirconium in the alloy. This means that small additions (up to 12%) of niobium influence the hardness of zirconium to a much greater extent than do similar additions of tantalum, if hardening is done from the same temperature. However, when hardening is done from the α and β range, the influence is approximately identical, which can be explained by the similar values of tantalum and niobium solubility in β -zirconium and the identical atomic diameters of these elements.

Hardness at Elevated Temperatures

Iodide zirconium remelted in arc furnaces shows a hardness typical of many refractory elements. However, with increasing temperature, its hardness decreases rapidly, and at 350° it is approximately half as hard as at room temperature. The alloying of zirconium by tantalum and niobium raises its hardness both at room and higher temperatures. Figure 4 shows curves of the dependence of hardness on temperature in some binary and ternary zirconium alloys with tantalum and niobium. For comparison, the same graph shows a curve of hardness vs temperature of iodide zirconium remelted in an arc furnace. Due to the exponential character of the dependence of hardness on temperature, these curves are shown, for convenience, in semilogarithmic coordinates.

The curves of hardness vs temperature of pure zirconium and its alloys consist each of two straight lines and a transition zone in the range 500-600° temperature. The first part of the curve is inclined to the temperature axis to a lesser degree than is the second half. This shows that up to 500°, the hardness of zirconium and its alloys decreases during heating to a lesser degree than at temperatures above 600°. The transition zone of each curve represents a temperature region in which the mechanics of deformation change, and corresponds approximately to the temperature of softening of the alloys. If the softening temperature of pure zirconium is assumed to be 425° [5], it is apparent that it increases upon alloying with tantalum and niobium.

The curve of hardness vs temperature of pure zirconium is considerably lower than analogous curves for alloys, due to the lower hardness values of zirconium at room and elevated temperatures. Its somewhat greater slope in relation to the temperature axis is caused by a more rapid decline of hard-

ness of pure zirconium than of the alloys when heated.

Curves of hardness vs temperature for binary zirconium alloys with tantalum and niobium have nearly the same slope up to 500° relative to the temperature axis. This shows that with rising temperature the hardness of these alloys decreases to about the same extent. However, above 600° the hardness of the niobium alloys declines more rapidly than that of the tantalum alloys, which confirms the data of works [3] and [4]. This phenomenon apparently is due to the fact that at a temperature of 612 ± 15°, alloys of the Zr-Nb system undergo eutectoid dissociation, and, as the temperature rises, the high-temperature β -phase is discerned in their structure. The eutectoid dissociation in the Zr-Ta systems takes place at elevated temperatures (700°) [7], which causes greater hardness of Zr-Ta alloys above 600°. All the above tends to indicate that for temperatures above 600° it is more efficient to alloy zirconium with tantalum than with niobium, and this effect is maintained up to 800°.

The hardness of ternary alloys containing 1% Ta and 1.5% Nb, and 2% Ta and 3% Nb, changes in almost the same way as that of binary alloys with increasing temperature. Somewhat higher values of hardness at elevated temperatures were obtained for the alloy with 2% Ta and 3% Nb, as compared with other alloys. This can be explained by the higher content of alloying elements in the melt. The influence of the content of alloying elements on zirconium hardness at elevated temperatures was studied and curves of hardness vs composition for annealed binary and ternary alloys at different temperatures were plotted. These curves are shown in Figs. 5 and 6. For the temperature range studied (200-700°), they are reminiscent of hardness curves of alloys hardened from temperatures below the β -phase range, i.e., they show a substantial increase in hardness of zirconium when about 1% tantalum and niobium is introduced and a smaller increase in hardness as more of these alloying elements are added. A rapid increase in hardness at low additions of tantalum and niobium to zirconium takes place just as in the case of hardened alloys) because of the formation of a small solid solution area of these elements in α -zirconium. The hardness of binary and ternary annealed alloys at equal temperatures and concentrations of alloying elements over a temperature range of 20 to 600° is also approximately equal. This indicates that tantalum and niobium have the same influence on the hardness of α -zirconium at room and elevated temperatures (to 600°).

Somewhat higher values of hardness in alloys rich in tantalum and niobium of the third polythermal cross section are apparently caused by the oxidation of the surface of the

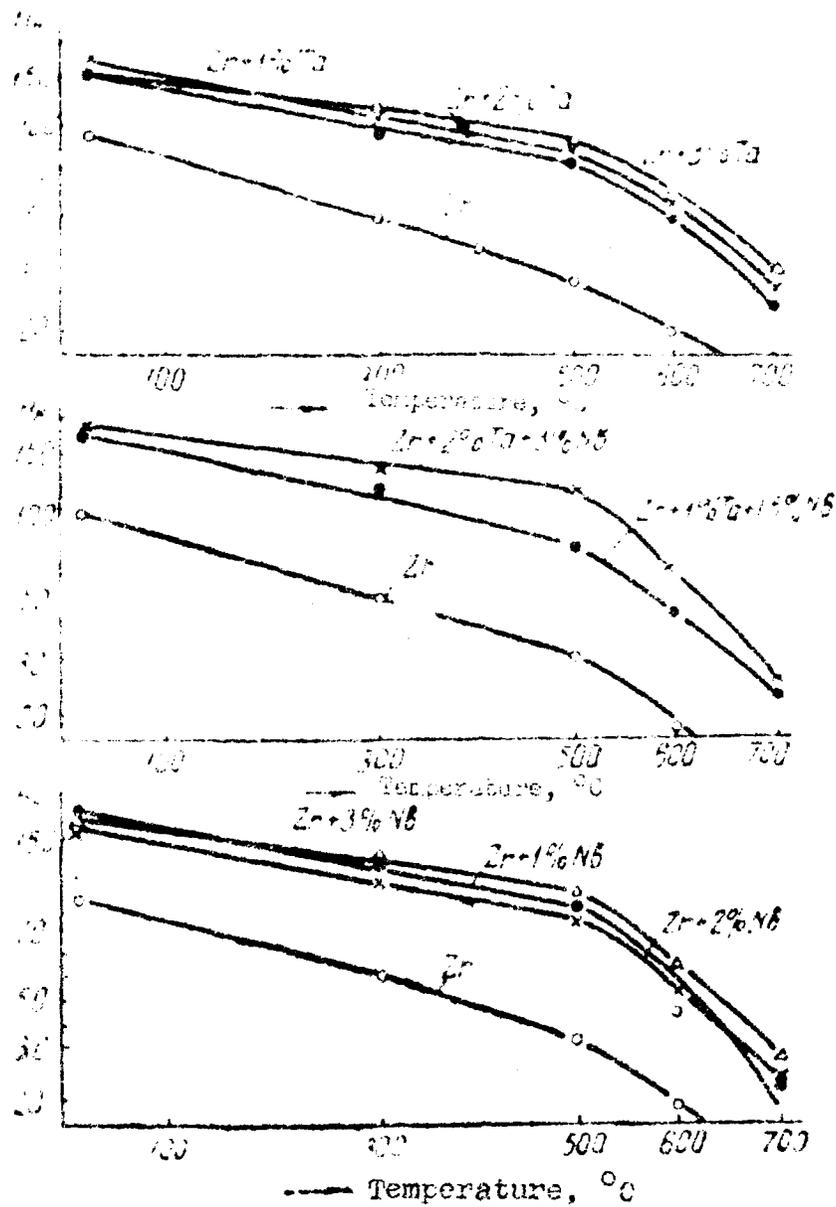


Fig. 4. Hardness of binary and ternary zirconium alloys with tantalum and niobium at elevated temperatures.

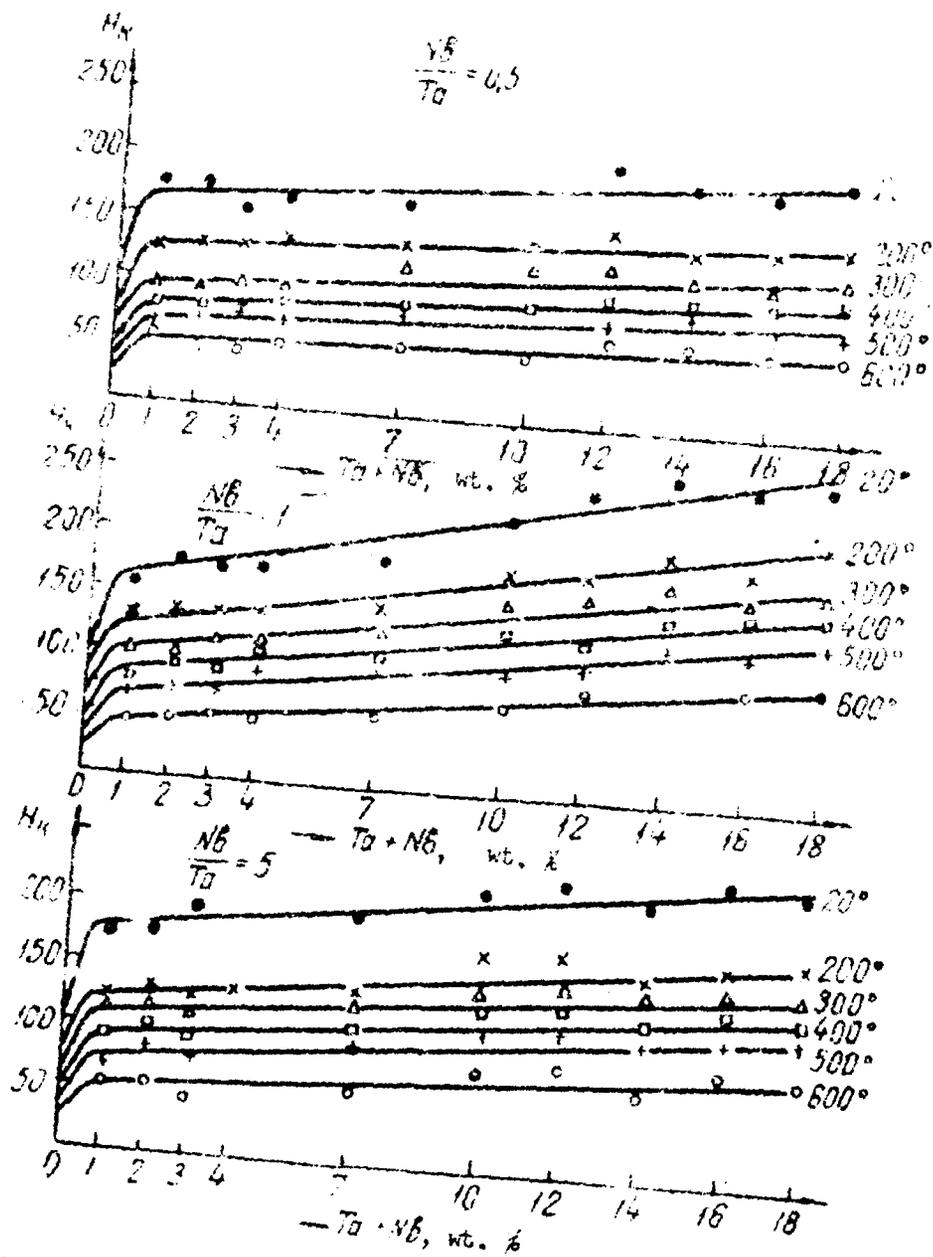


Fig. 5. Curves of composition vs hardness of alloy systems Zr-Ta-Nb in three polythermal cross sections at elevated temperatures.

samples during forging.

From the graph it appears that between 500 and 600°, binary and ternary alloys show a noticeable decrease in hardness. This decrease is caused either by a viscous flow of the alloys or by other undetermined circumstances,

Mechanical Properties of Zirconium and its Alloys Under Elongation

Alloying zirconium with tantalum and niobium improves its strength characteristics and decreases its ductility in the annealed and especially in the hardened state. Figure 7 shows the curves of composition vs tensile strength and relative elongation of zirconium and its binary alloys after hardening and annealing. A comparison of the curve of hardness vs composition of hardened and annealed alloys show that after hardening from temperatures in the β -phase range, the strength of the alloys increases considerably while the ductility decreases correspondingly. Annealing at temperatures below the eutectoid lines in the Zr-Ta and Zr-Nb systems eliminates this strengthening effect.

The curve of strength vs composition of hardened zirconium alloys with niobium has a peak corresponding to about 7.5% niobium, which is analagous to the peak of the hardness vs composition curve. This peak, as mentioned before, is caused by martensite-type transformation of the cubic β -phase into the hexagonal α -phase. On the curve of relative elongation vs. composition, this corresponds to a minimum.

In the range of concentration, studied, hardened zirconium alloys with niobium have greater strength than have the alloys with tantalum. As in the case of hardness this is due to a narrower area of martensite-like transformation in the Zr-Nb system as compared with the Zr-Ta system and to the fact that the Zr-Nb system attains maximum tensile strength at a lower content of alloying elements. Among hardened specimens of the Zr-Nb system, the maximum strength and the minimum relative elongation is shown by the alloy with 7.5% Nb, while the corresponding figure for the Zr-Ta system is 10% Ta.

In the annealed state, the strength of zirconium increases rapidly upon the introduction of up to 1% of the alloying elements; further additions of tantalum and niobium results only in a slight further increase in strength. The relative elongation of the alloys varies inversely.

The strengths of annealed binary alloys of zirconium containing equal quantities of tantalum and niobium, respectively, are approximately the same. This shows that the introduction of either tantalum or niobium into zirconium increases

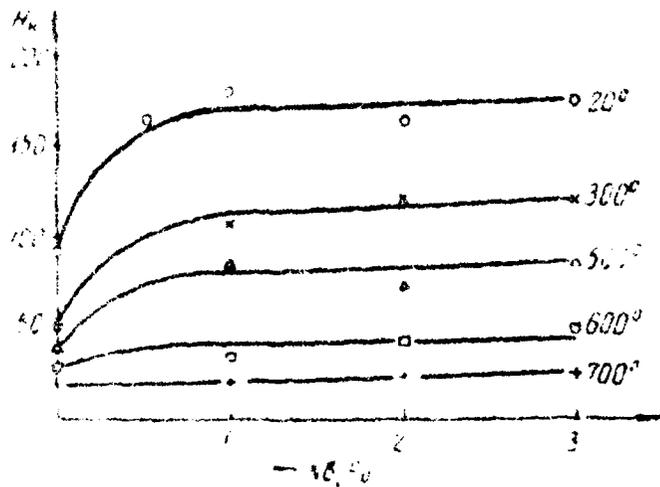
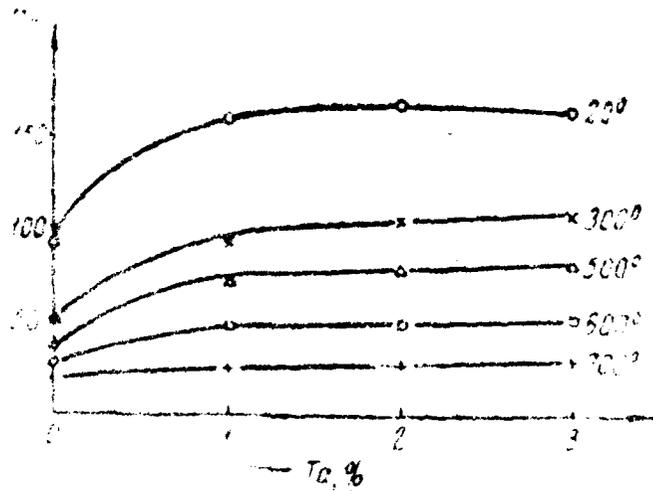


Fig. 6. Curves of hardness vs. composition of alloy systems Zr-Ta and Zr-Nb at elevated temperatures.

its strength to approximately the same extent as could be expected considering that the atomic diameters of these elements are the same, and their solubilities in zirconium are nearly equal [7], [8]. These results confirm neither the conclusions of the review [5] nor the data of works [2] and [6]. The higher

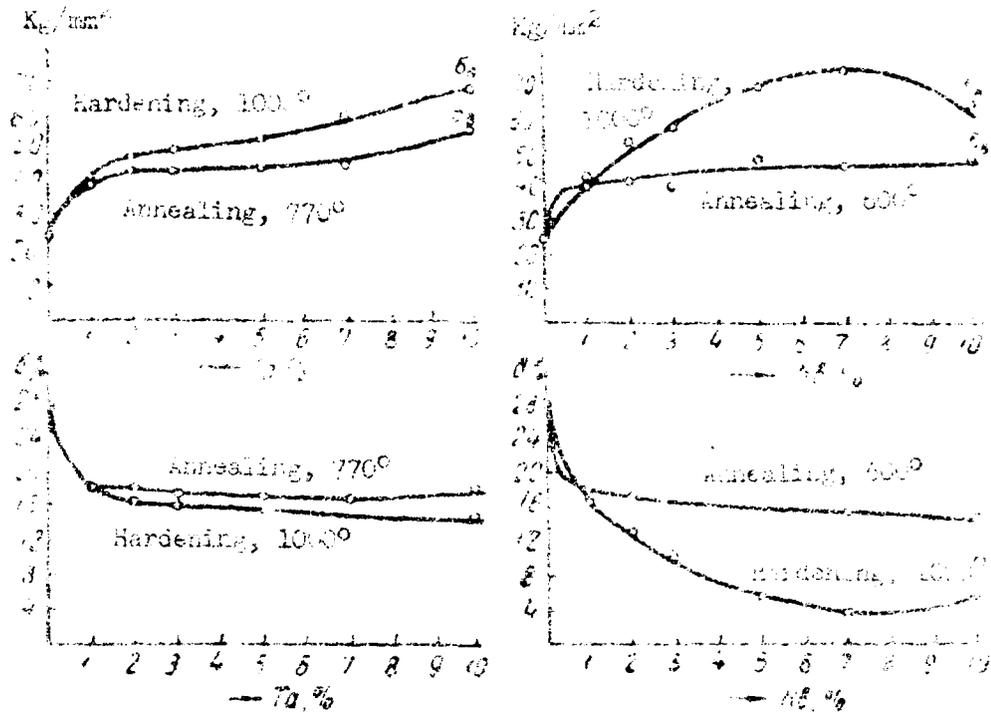


Fig. 7. Dependence of tensile strength and relative elongation of alloy systems Zr-Ta and Zr-Nb on composition in hardened and annealed states.

values of Zr-Nb alloy strength given in these works as compared with our data, and the data of work [2, 7] concerning the Zr-Ta alloy strength, were obtained apparently because, in the works referred to, the annealing was carried out at temperatures of 700-725°, i.e., above the eutectoid line of the system Zr-Nb and below that of the system Zr-Ta. Upon rapid cooling of the Zr-Nb alloys, hardening could have taken place, increasing the strength. This supposition is confirmed by the fact that the curve for the dependence of tensile strength on composition of the Zr-Nb alloy system annealed at 700° [6, 7] has a peak which is characteristic only of alloys hardened at temperatures above the line of eutectoid dissociation.

The results of studies of mechanical properties (elongation tests at 300°) of ternary alloys belonging

to three polythermal cross sections are shown in Fig. 8 as curves of mechanical properties vs composition. At this temperature pure Zr has a relatively low value of tensile strength: $\sigma_B = 11 \text{ kg/mm}^2$. The introduction into zirconium of a 1% global addition of tantalum and niobium increases its tensile strength to 24 kg/mm^2 .

Upon a further addition of alloying elements, the strength continues to increase but at a slower rate. In the range of concentration studied, the greatest strength and the least relative elongation of three polythermal cross sections was shown by alloys containing maximum amounts of (18%) of tantalum and niobium.

Conclusions

As a result of work carried out, the following was established:

1. Binary and ternary zirconium alloys with tantalum and niobium, hardened from temperatures in the β -phase range, have greater hardness and strength than annealed alloys.

2. On curves of strength and hardness vs composition of binary and ternary alloys of polythermal cross sections hardened from temperatures in the β -phase range, a peak is observed which is caused by martensite-like transformation of the cubic β -phase into the hexagonal α -phase.

3. Alloying of zirconium with tantalum and niobium substantially increases the strength and hardness at room and elevated temperatures in both the annealed and hardened states. In the annealed state, a considerable increase in hardness and strength of zirconium is observed when about 1% of tantalum and niobium is added.

4. The introduction of niobium into zirconium (up to 10%) strengthens it after hardening to a greater degree than does similar addition of tantalum.

5. Binary and ternary alloys of zirconium with tantalum and niobium in a temperature range of 500 to 600° show a considerable drop in hardness, apparently caused by a change in the mechanics of deformation.

6. At temperatures higher than 600° , zirconium alloys with tantalum are harder than those with niobium, and this effect is maintained up to 800° .

7. In the annealed state, tantalum and niobium influence the hardness and strength of zirconium to an equal degree.

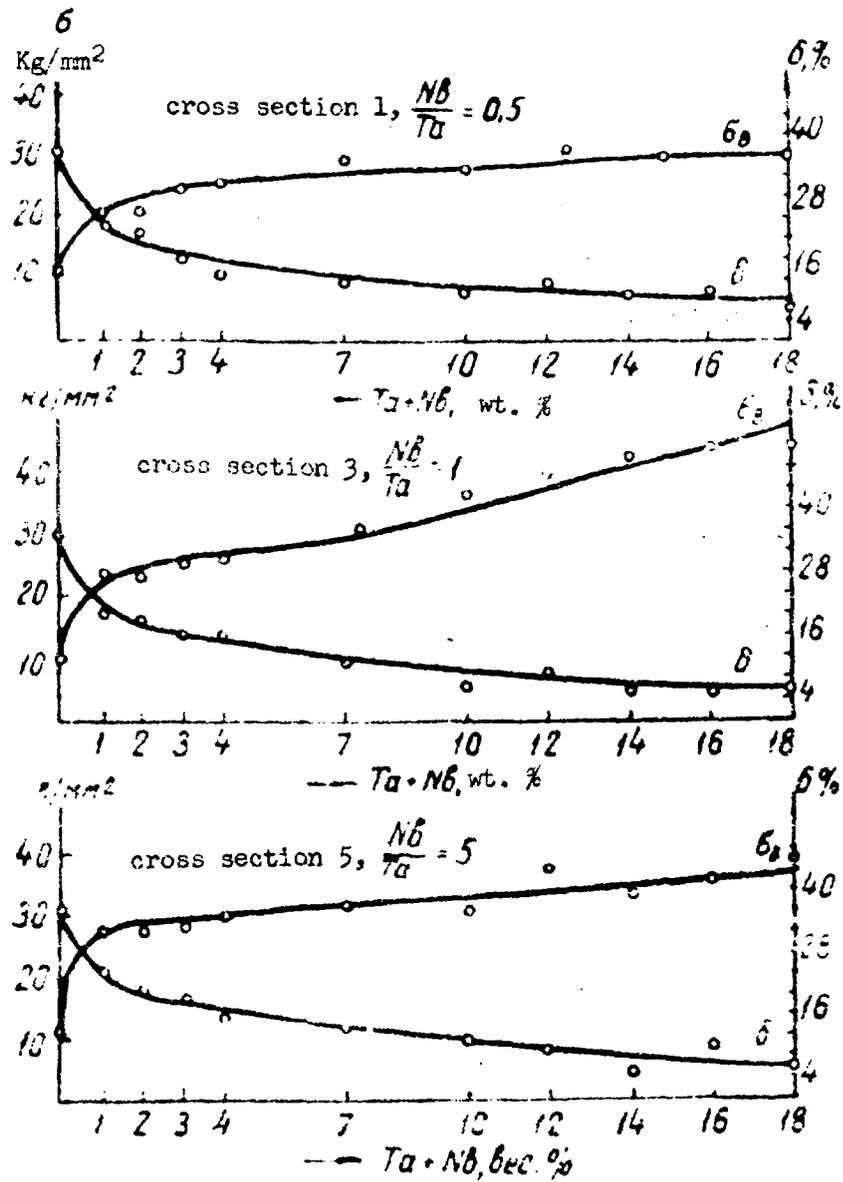


Fig. 8. Dependence on composition at 350° of tensile strength and relative elongation of alloys in three polythermal cross sections of the Zr-Ta-Nb system. wt. %

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