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ARC MELTING OF ZIRCONIUM METAL
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ARC MELTING OF ZIRCONIUM METAL

by
Richard J. Dunworth

The arc melting furnace is used for melting reactive or refractory metals. Zirconium, titanium, vanadium, and hafnium can be melted in graphite or very dense, ceramic crucibles. A considerable loss in corrosion resistance or in ductility of the metal, however, results from contamination by the crucible. Tungsten, tantalum, and molybdenum are melted in arc furnaces because of the high melting points of these metals. Uranium and thorium have been arc melted, but, as suitable crucibles can be used to melt these metals, arc melting is not an important factor in the production of these castings. Titanium, molybdenum, and zirconium castings weighing up to 4000 pounds are produced by an arc-melting process similar to that described in this report. At ANL the castings range in diameter from one inch to six inches and range in weight from 0.1-pound buttons to 130-pound forging ingots.

I. APPARATUS

The arc furnace was originally designed to be either an extractable ingot furnace or a moving electrode furnace. Castings made in the extractable ingot furnace, however, had poor surfaces because the zirconium ingots adhered to the copper mold wall. The mechanical and casting difficulties were similar to those obtained with the continuous casting processes. Consequently, the extractable ingot furnace was discarded, and the moving electrode furnace, shown in Figure 1, was used to make the three-inch and six-inch castings of zirconium. This furnace was constructed from prints supplied by WAPD and was almost identical to the furnace used for their production castings. The metal charge (4) is cut into small pieces and is placed in a large stainless steel hopper. By means of a syntron vibratory feeder the metal pellets are moved to the melting chamber where they drop into the molten pool of zirconium (8) at the bottom of the melting crucible (9). The electrode (6) is raised as the metal level rises in the crucible. Each component of the melting chamber is water cooled to remove the heat radiated from the arc and the molten zirconium.

The electrode was made from 3/4-inch copper tubing with a 0.065-inch wall thickness. The inlet water passes through a 5/16-inch diameter copper tube to within 1/2 inch of the bottom of the electrode. The tungsten tip is screwed into a copper plug which is silver soldered at the end of the electrode. Some electrodes were made by silver soldering the

tungsten tip to the end of the copper tube. If the tungsten was first coated with nickel, a tight joint could be made. The threaded connection, however, is considered a safer joint, and the tip can be changed more easily. The tungsten tip, which is $1/2$ inch in diameter and $1\frac{1}{2}$ inches long, has a $3/8$ -inch, No. 16 ground or machined thread, $1/2$ inch long. The other end of the tip is ground to a point at an angle of 35 degrees to the longitudinal axis with a $1/8$ -inch radius on the point. If the tungsten is heated to 700°C during cutting, the threads can be machined.

The three-inch crucible was made from copper pipe with a wall thickness of $1/4$ inch; the crucible bottom was tapered slightly and silver soldered to the wall. The six-inch crucible was made by rolling $1/4$ -inch sheet copper to a six-inch diameter and by butt welding the seam. Although less machining was required on the copper pipe, satisfactory service was given by both crucibles. In order to keep the water annulus uniform and to prevent hot spots from developing in the wall, the crucible should be concentric in the water jacket. The annulus used in the crucibles was $1/16$ inch, but an annulus of $1/32$ inch would probably provide more efficient cooling. To prevent the crucible from shifting in the water jacket, eight spacers were silver soldered to the wall of the crucible.

The furnace cover, sight glass, and hopper are located on the melting chamber at the top of the furnace. The water-cooled furnace cover is electrically insulated from the furnace by means of a $1/2$ -inch thick rubber gasket between the flanges. The bolts, which are enclosed in rubber tubing, bear against micarta washers at both ends. The sight windows at the top of the furnace were made from polished quartz. Welding lenses of various shades were used to reduce the amount of light from the arc. Since a single dark shade (#14) cracked and shattered in a short time, two lighter shades (#6) were used, giving longer service and equal light reduction. The stainless steel hopper had a capacity of 50 pounds of chopped feed metal in the three-inch furnace and 250 pounds in the six-inch furnace. The push rod, shown at the top of the hopper in Figure 1, was necessary to free metal clogged at the base of the hopper.

Two direct current welding generators furnished power to the furnace through three 4-0 welding cables on each side of the line. At the furnace the cables were fastened to copper bus bars (each $3/4$ inch thick and 4 inches wide). Finally, the electrode was connected to the bus bar through three layers of flexible copper braid, each layer of which was $1/4$ inch thick and $1\text{--}1\frac{1}{2}$ inches wide. Since about 70% of the heat was generated at the anode of a dc arc, the zirconium metal was made the anode in the circuit.

II. FEED METAL AND METAL TREATMENT

The metal melted in these furnaces was mostly crystal bar zirconium. The rods were corrosion-tested before shipment and were graded 1, 2, or 3, depending on the amount of white corrosion product found on the rod. Grade 1 was the most corrosion-resistant material and had no white corrosion product anywhere on the rod. Grade 2 was almost as good, except for a few areas of white corrosion. Grade 3 varied from moderate to heavy layers of white corrosion products over most of the rod.

The concentrations of the chief contaminating elements in zirconium are listed in Table I. Oxygen, aluminum, and silicon are removed most efficiently from sponge zirconium by the iodide deposition process. The amounts of carbon, iron, and titanium are probably less in crystal bar than in the sponge, but the range of values reported in the analyses precludes any positive statement. The oxygen values are considered relative rather than absolute. The principal difference between sponge and crystal bar was in their relative corrosion resistance to water at 300°C. Most of the produced crystal bar corroded much more slowly than sponge metal. Although the concentrations of carbon, nitrogen, aluminum, and titanium in corrosion-resistant crystal bar (Grade 1) were less than in sponge, the concentrations of these elements in Grade 3 crystal bar, which corrodes rapidly, were substantially the same as those in the Grade 1 crystal bar.

The procedure for cleaning the metal varied, depending on the quality of the casting required. If good corrosion resistance was desired a rather complete cleaning cycle was followed. The black corrosion product on the Grade 1 crystal bars was removed by vapor-blasting. The rods were rolled to 0.300-inch diameter, straightened, and degreased in a trichloroethylene vapor degreaser. Then the rods were pickled in a 1% HF and 50% HNO₃ solution, were given a bright dip in concentrated HNO₃, and were rinsed in water. The hydrogen absorbed by the zirconium during the pickling was removed by vacuum-annealing. By holding the rods at 1100°C for three hours in a vacuum of 5×10^{-6} mm, the hydrogen content was lowered enough to maintain a stable arc when the metal melted. Finally, the rods were chopped into 1/4-inch lengths and put into the hopper for melting. If good corrosion resistance was not required, Grade 2 or Grade 3 zirconium was used, and the rods were vapor-blasted, rolled, straightened, degreased, and chopped. In some cases the vapor-blasting step was omitted.

Corrosion data for eight castings are presented in Table II. Castings #22, #23, and #24, made from melting stock which had the complete cleaning treatment, had good corrosion resistance. After corrosion testing, the surfaces had black tarnishes with no grey film or white corrosion products. The weight gains of the billets were about the same as for crystal bar. Casting #3, on the other hand, had a white film on the bottom sample and a much greater weight gain. This casting was made without

vapor-blasting or acid pickling the feed metal. Casting #12 showed a slightly better corrosion resistance than were exhibited by castings #22, #23, or #24. In this case, the feed metal was vacuum treated but was not vapor-blasted or pickled. Although the evidence was not conclusive, the complete cleaning treatment was considered more desirable for the production of corrosion-resistant castings.

The hardness of the billets did not correlate at all with the amount of cleaning given the feed metal. The hardness values of Grade 1 billets averaged Rockwell B-38; the hardness values of the Grade 3 billets averaged Rockwell B-56. A number of castings comparable to the best Grade 1 castings, however, have been made from Grade 3 zirconium. In spite of the average values, the billet hardness is not considered to be dependent on the grade of crystal bar melted. No specific tests were run to determine if large amounts of white corrosion product contributed significantly to the hardness of the casting. Nevertheless, the white corrosion product was not removed from the feed metal used to make castings #51, #52, and #54, and the hardness (Rockwell B-52) of these billets compared favorably with other Grade 3 billets.

III. FURNACE MELTING PRACTICE FOR CHOPPED CRYSTAL BAR ZIRCONIUM

After the metal had received one of the treatments described in Section II, it was loaded into the stainless steel hopper. The furnace was evacuated with a mechanical pump for several hours, then filled with a mixture of 20% argon and 80% helium to 1 or 2 psi gauge pressure. If greater purity of the inert atmosphere was desired, the gases were purified by passage over uranium and zirconium chips at 850°C at a rate of 1/2 liter per minute. In addition, the furnace was evacuated and filled two or three times so that the remaining traces of air were removed. In most cases, the furnace was filled with the inert gas without purifying the gas or flushing the furnace. Although a number of castings were made at reduced pressures, the contamination from small leaks was less if the pressure was slightly greater than atmospheric pressure.

The tungsten tip used at the end of the electrode was another source of contamination. Though it is probable that this contamination cannot be eliminated completely, it can be reduced. The tungsten concentration in the casting was estimated from the weight loss of the tip. This value varied from 0.3 gram to 2.5 grams, and in a 10-kilogram billet it amounts to 30 to 250 ppm tungsten if the tungsten were homogeneously distributed in the zirconium. Tungsten was reported as less than 400 ppm on the spectrographic analysis and no wet analyses were made. In one instance small particles of tungsten were found in the casting. The large size of the inclusion is shown in Figure 2 at a magnification of only 15X. It was visible on the machined surface, and it appeared as a white corrosion spot after

testing in water at 300°C. In Figure 3, at 75X, the partial solution of the inclusion in the zirconium is shown more clearly. Such insoluble particles were washed to the side of the billet and were frozen near the surface, as shown by an examination of a thorium ingot which contained a large amount of inclusions. Similarly, tungsten particles should be found at the surface of the zirconium billet. Though all the ingots were scalped by machining 1/16 to 1/8 inch off the side, no hard spots were reported. Because tungsten inclusions were found only once, the control of this defect was not a serious problem.

That the tungsten is deposited in the zirconium, however, is almost certain. It is more probable that the tungsten is dissolved in the zirconium than that it is present as discrete particles. Tungsten removal from the tip results from zirconium that splashes from the melt onto the tip. If large or gassy feed metal is used, the tungsten concentration increases markedly. For example, a 4.6-pound billet was made from zirconium sponge that was not completely outgassed, and 5.4 grams of tungsten were lost from the tip - twenty-five times the normal loss. The zirconium, splashed onto the tip, alloyed with the tungsten, and melted off into the pool of molten zirconium. Further evidence to support the splashing mechanism is found in the definite variation of tungsten contamination with the arc length, which is the distance between the tip of the electrode and the molten pool of zirconium. In experiments using various arc lengths, 0.9 gram of tungsten were lost at two inches; 0.2 gram at three inches; and 0.1 gram at four inches. The arc length was maintained, therefore, between three and four inches for most castings. When using the three-inch diameter crucible, five-inch arc lengths resulted in poor arc stability and in too great a power loss to the copper crucible.

The three-inch diameter ingots were made at 1250 amp and 54 volts, and the six-inch ingots were made at 1600 amp and 64 volts. The fastest melting rate consistent with good surface quality was needed for low tungsten contamination. In Figure 4 the tungsten loss is plotted against the melting rate for 23 castings. The circle points designate castings that were melted using a four-inch arc length, 1250 amp, and an atmosphere of 20% argon and 80% helium. The square points represent castings that were melted using a two-inch arc length, 100 amp, and a 100% helium atmosphere at an absolute pressure of 15-20 inches of mercury. The triangular points refer to castings that were melted using a four-inch arc length, 1150 to 1450 amp, and an atmosphere of 100% helium at an absolute pressure of 10 inches of mercury. As may be seen, the tungsten loss is affected more by the melting rate and the arc length than by the current, atmosphere, or pressure. For billet #22, which was made at a $2\frac{1}{2}$ -to 3-inch arc length, the tungsten loss is found between the two curves. The points for billets 34, 77, and 78 belong on the four-inch curve, but these castings were made with a 3/8-inch diameter tungsten tip, which probably reached a higher temperature than the 1/2-inch diameter tip used for the other castings and caused

a high tungsten loss. The high tungsten contamination in billet 14 could be due to a variety of causes, such as a small leak, gassy feed metal, or a piece of zirconium hitting the electrode tip. Insufficient data does not allow more definite conclusions to be drawn. On the other hand, the low tungsten loss in making billet 68 can be explained by a possible error in weighing the tungsten tip. With the exception of these two billets, the points fit the curve remarkably well, considering the different atmospheres, currents, and pressures which were used when making the castings. The effects of these variables on the tungsten loss are masked by the more important factors of arc length and melting rate. The slight dependence of the tungsten loss on the amount of current used is exemplified further by the six-inch diameter castings. These billets were made at currents of 1600 to 2000 amp; yet these tungsten losses compared favorably with the losses for the three-inch castings.

IV. DRIP ARC MELTING OF ZIRCONIUM RODS

A process called "drip arc" melting, that reduced the melting time and the tungsten contamination, was developed. Instead of feeding small pieces of solid zirconium, molten drops were added to the pool. By using this method, less splashing and shorter melting times resulted, which contributed materially to lower tungsten losses in the castings. For example, in casting 70 (Table III) the tungsten loss that resulted from melting chips was six times the loss that resulted from drip melting rods. The rods were set in a circle and were welded at the top. A six-inch extension of one rod served as a holder for the assembly. The welded rods are shown in Figures 5 and 6 before insertion in the crucible. The rods were $5/8$ to $3/4$ of an inch in diameter and 29 inches long. The bundle used in the three-inch crucible (Figure 5) consisted of a single layer of rods. It was necessary to butt weld an extra ten-inch length to most of the rods in order to obtain the required length of 29 inches in the assembly. In Figure 6 the assembly was made with a double layer of rods for use in the six-inch crucible. Some of the rods in the inside layer may be seen near the center and bottom of the picture. Two layers were used so that as much zirconium as possible could be packed in the 29-inch length. On some of the rods white corrosion product may be noticed near the bottom of the picture. At the beginning of the melt, the bundle was held about one to two inches above the molten pool of zirconium. The heat radiated from the arc and from the molten zirconium melted the ends of the rods. The casting was built up by these drops, and the assembly was lowered as the rods melted away. There was no splashing observed as the drops of molten zirconium fell only one or two inches into the melt. Furthermore, most of the heat that was formerly lost by radiation to walls of the crucible was absorbed by the zirconium rods and the melting proceeded at a rapid rate.

Several difficulties were encountered in the use of this method. When the three-inch crucible was used, the rods were only $3/8$ of an inch from the electrode. Consequently, the rods had to be straight and strain

free to keep them from shorting against the electrode when they were heated by the arc. A few times the rods did short against the electrode, the copper tube was punctured, and the casting was flooded with the cooling water in the electrode tube. When melting the double layer of rods, the inner layer would melt off before the outer layer. The outer rods were an inch longer than the inner rods and the melting rate had to be decreased so that the outer rods could be melted. As the sight glasses were not properly located for this type of melting, it was difficult to observe the location of the arc. Consequently, the arc burned through the crucible a number of times when it wandered unobserved to the wall of the crucible. No explosions resulted from the reaction of water with molten zirconium because of the small amount of molten zirconium. The 29-inch long assembly produced a casting about 12 inches in length. If a single layer of rods was melted in the six-inch crucible, less difficulty would be experienced, but the assembly would have to be 45 inches in length to make a 12-inch long casting.

The greatest advantage of the drip arc melting process was the shorter melting time. When the feed metal was cut into chunks, the proper melting rate was 11 pounds per hour for the three-inch billets and 25 pounds per hour for the six-inch billets. When the drip arc melting method was used, the three-inch castings were made at 20 pounds per hour, and the six-inch billets were made at 45 pounds per hour. In Figures 7 and 8, two views of casting 63 are shown. This six-inch diameter billet was made at a rate of 70 pounds per hour. At the bottom of Figure 8 the unmelted rods on one side of the billet were due to the arc not being centered and to a fast melting rate. The surface of the six-inch billet was smoother than the surface of the three-inch billet (54) in Figure 9, which was melted at a rate of 35 pounds per hour. The pictures of the top (Figure 10) and bottom (Figure 11) of this casting are typical for these surfaces on arc-melted billets. The arc was started at a low power level on a 1/2-inch to 1-inch layer of chopped zirconium. Since this layer could not be melted completely because of the danger of melting through the copper bottom of the crucible, full power was not applied until the casting was about one inch thick. The bottom of the billet appeared as shown in Figure 11 with unmelted chunks of zirconium which protected the crucible bottom from the molten zirconium. The surfaces of the billets are shiny because of nonmetallic impurities dissolved in the zirconium.

V. SPONGE MELTING

Several attempts were made to melt compacted sponge zirconium from the Bureau of Mines. These tests met with a notable lack of success. Whenever a small chunk of the metal was added to the molten pool of zirconium, the arc was blown out. The amounts of magnesium, hydrogen, and halide salt contained in the metal apparently were sufficient to change the atmosphere surrounding the arc: the stability of the arc was disturbed and the arc was extinguished. Since the currents used in nonconsumable

electrode melting were low, the arc was not stable enough to resist much disturbance. When melting crystal bar, a similar effect was obtained if the hydrogen pickup from acid pickling was not removed by vacuum annealing. Heating the sponge metal at 1400°C for six hours at a pressure of 5×10^{-6} mm, however, did not remove enough of the volatile constituents to permit melting. If small pieces of sponge were melted by repeated arcing on each piece, the arc could then be maintained on remelting such pieces, but this method resulted in considerable tungsten contamination.

The du Pont sponge was melted readily after a vacuum treatment for three hours at 1000°C and a pressure of 2×10^{-5} mm. No bursts of hydrogen occurred during heating, nor was a large deposit found on the furnace tube. As 5.4 grams of tungsten were lost from the electrode tip in making a 4.6-pound casting, the metal should have been outgassed for a longer time and at a higher temperature. The melting data for this casting (21) are given in Table III. The hardness of Rockwell B-80 might be due more to the 0.26% tungsten content than to impurities in the sponge. Casting 31, for example, had a hardness of Rockwell B-83 at the top, B-37 at the bottom and B-20 on the sides. A zirconium pellet that hit the electrode tip near the end of melting undoubtedly caused the greater hardness at the top of this casting.

Microscopic examination of the casting made from du Pont sponge showed a number of sonims, which were not found in castings made from crystal bar. These inclusions (Figure 12) were quite different from the tungsten inclusions (Figures 2-3) in appearance. As there were no areas of diffusion visible around the sonims, it might be presumed that these inclusions were zirconium halides. It is not likely that the sonims are oxides or nitrides, because of their ready solubility in zirconium. Most of the metal, however, appeared as shown in Figure 13. The structure is similar to arc-melted crystal bar.

It may be concluded that arc melting sponge is more difficult than melting crystal bar. If the sponge is porous and well outgassed, it should be possible to make sound castings. The drip-arc melting process might be used with some advantage to melt sponge. It has been shown, though, that compacted sponge is not purified sufficiently by vacuum treatment, and the arc cannot be maintained.

VI. MELTING OF ZIRCONIUM ALLOYS

Homogeneous alloy castings are more difficult to make by arc melting than by crucible melting methods. Since only a 1/4-inch layer is molten as the billet is made, the solute and solvent metals must be added in exact alloy proportions to prevent the formation of alloy-rich layers in the billet. Moreover, because of the long time required to make an arc-melted casting (the casting time may be considered as a slow

freezing rate), an alloy system with a large spread between the liquidus and solidus might produce a severely segregated casting. In addition, it is practically impossible to alloy a solute metal that melts 500-600°C above the melting point of the solvent metal. Little superheat can be produced in the solvent, and the arc is not attracted to a solid piece of solute metal. Then, too, at the melting point of zirconium, the volatility of some metals makes alloying impossible. In spite of these difficulties, if the casting is remelted two or three times, certain alloys can be produced to a rather narrow range of composition. The zirconium-tin alloy is an example of such a system.

A special hopper was built so that the feed metal could be added to the melt in the correct proportions of the alloy. A circular plate was divided into twenty segments. About 100 grams of solute and solvent metal, chopped into 1/4-inch lengths, were weighed to the alloy composition and were put into a segment. As the melting progressed, each segment was dumped into the feeding hopper and was melted completely before another segment was dumped. In this way, the casting was made in 100-gram increments of correct alloy composition. The hopper was built of six segmented plates, so that about 25 pounds of metal could be melted.

The drip-arc melting process was used to make a few alloy castings. The first attempts were made using rods of the solute metal fixed in the rod assembly. As the low-melting solute rods melted too rapidly, this method proved impractical. When using high-melting solute rods, unmelted rods were found at the sides of the billet. A few castings were made by using vibratory feeder to add the solute as the rod bundle melted. While the distribution of solute was more uniform than that obtained by using rods, the rate of rod melting could not be proportioned to the rate of solute addition. As the rods are melted, a mechanical method should be used to insure that the proper amount of solute is added. Then it should be possible to make good alloys by the drip-arc melting process.

VII. MELTING FACTORS - ATMOSPHERE, CURRENT, ARC LENGTH

In the practice of arc melting, the atmosphere, current, and arc length may be varied considerably, and an excellent casting may still be obtained. The billets listed in Table III represent a considerable variation in these factors, although the billets showed little difference in quality. With the exception of billet 73 and of the top of billet 31, all the castings were apparently of good quality, although some billets had surfaces with deeper and more numerous cold shuts, and other billets had varying hardnesses and tungsten losses. The reasons for such differences were not immediately obvious. A study was undertaken, therefore, to determine more exactly the effect of the melting factors on the billet produced.

Certain properties of helium and argon were known from earlier melts: helium has a greater electrical resistivity and thermal conductivity than argon;¹ a decrease in pressure (to 27 inches of mercury vacuum) lowers the resistance of either gas. At the same current, the arc melts a shallow, large diameter pool in helium and a deep small diameter pool in argon; a long arc length requires more current than a short arc length to produce the same melting; a long arc length is necessary for low tungsten contamination.

In the first place, the relative power absorbed by the billet is related to the amount of corona around the electrode. When the composition of the atmosphere is changed to argon-rich mixtures, this corona is quite noticeable around the electrode. The corona produces no heat in the billet and is, in effect, a short circuit between the electrode and the crucible. The power lost by the corona increases as the argon fraction of the atmosphere and as the arc length increase. For example, in an atmosphere of 100% argon, at an arc length of 3-3/16 inches, and at a current of 1600 amp, the metal is frozen, most of the power being dissipated by the excessive corona. Similarly, the metal is frozen in 80% argon at a 3-3/4-inch arc length and 1250 amp. This corona is a visible indication of an excessive loss in power. Although the discharge is visible first at the points marked with a 'C' in Figure 14, some power undoubtedly is lost before the corona starts. Hence, melting under conditions near these points is not desirable. If an arc length of four inches is used, 30% argon should be the maximum amount used in the atmosphere. At a three-inch arc length, however, the composition of the atmosphere could be increased to 40% argon. The loss in power due to the corona will be very slight for these atmospheres and arc lengths. If a five or six-inch diameter billet is being melted, either the arc length or the percentage of argon may be increased, because the electrode is at a greater distance from the crucible wall.

Secondly, the relative power absorbed by the billet is shown by the diameter and the appearance of the molten pool of zirconium. If the arc length and the composition of the atmosphere are fixed, an increase in the current will increase the diameter of the molten pool. The pool becomes wider with increasing current until the crucible wall is reached. At this

¹ The thermal conductivity of argon is $3.9(10^{-5})$ and of helium is $3.4(10^{-4})$ cal/sec/cm²/cm²/°C. Hdbk. of Chem. and Physics, 37th Ed. p. 2253.

The ionization potential (1st state) for argon is 15.68 volts and for helium is 24.46 volts. Ibid. p. 2341.

The resistance of argon is 0.006 ohm/inch and of helium is 0.034 ohm/inch as calculated from the data in Table VI of this report. The crucible is three inches in diameter and the arc length is two inches (no corona discharge) for this calculation.

point, the surface of the pool is smooth and quiet. With the exception of an occasional ripple, the metal seems hardly molten. Further increases in current, however, impart a definite fluidity to the metal. The pool becomes more and more agitated as the current is raised, until the liquid metal is blown out of the center of the pool. The metal is pushed to the wall of the crucible where it freezes into a splash ring. If the arc length is shortened, the fluidity and diameter of the pool will increase. In an atmosphere of helium, for example, the pool has the same fluidity at 900 amp and a one-inch arc length as at 1300 amp and a three-inch arc length. As the percentage of argon in the atmosphere is increased, greater fluidity is obtained in the pool, but the diameter of the pool is decreased. In argon at a three-inch arc length, the pool is violently agitated at 1600 amp but is only 2-1/2 inches in diameter. Since in a three-inch diameter crucible the arc length should be four inches and the atmosphere should contain not more than 30% argon, more fluidity can be obtained only by raising the current. When casting in a three-inch crucible at a four-inch arc length, good fluidity in the melt will be obtained by using the following values: a current of 1100 amp and 30% argon; 1200 amp and 20% argon; 1300 amp and 10% argon; 1400 amp and 100% helium.

Next, since argon produces fluidity and helium produces a wide pool diameter, neither the fluidity nor the diameter can be used to compare the relative merits of different mixtures of argon and helium. The amount of power used in melting the zirconium, therefore, was measured by two thermocouples imbedded in the billet. Both thermocouples were located one inch below the top surface of the billet - one at the center and the other 3/16 of an inch from the side. After several runs the readings from the center thermocouple were erratic, and its use was discontinued. The side thermocouple, however, gave reproducible readings. The different atmosphere compositions were evaluated by means of these temperatures, which are plotted in Figures 14 and 15. As the argon concentration in the inert atmosphere surrounding the arc increases from 0% to 60%, the temperature developed in the billet also increases. Above 1100 amp the rate of temperature rise decreases considerably. This flattening in the curves is due to greater heat losses and to greater electrical losses. The fact that the temperatures in the billet in an 80% or 100% argon atmosphere were lower than in the 60% argon atmosphere could be accounted for by electrical losses-even though not visible as corona. The low temperatures obtained in a 100% helium atmosphere might be partly due to the high thermal conductivity of this gas. Moreover, in an atmosphere of helium the arc seemed to flow over the surface of the molten pool, and in this manner some power might be lost by a short circuit to the crucible. On the other hand, in argon the arc seemed to penetrate the surface and to form a crater at the point of the arc. The total power used in melting is greater in helium because of the high electrical resistance of this gas.

Finally, if the arc is operated at a partial pressure, less heat is developed in the billet. The diameter of the molten pool, the temperature of the billet, and the electrical resistance of the atmosphere decrease regularly as the pressure is lowered. The arc may be maintained to an absolute pressure of two inches of mercury. Below this pressure the arc degenerates into a complete corona. There seems to be no particular advantage to the use of partial pressures, and the danger of contamination is much greater.

CONCLUSION

The arc-melting process is the best method for melting certain refractory or reactive metals. The effects of the following process variables on the quality of the metal produced have been evaluated: the size, the treatment, and the kind of feed metal; the rate of feed; the arc length; the composition of the atmosphere surrounding the arc; melting in a partial pressure of gas; methods of decreasing tungsten contamination and corona losses; and the effects of the current used in melting.

The following melting practice has been used for zirconium castings. The feed was chopped into 1/4-inch cubes from clean, outgassed crystal bar. To reduce tungsten contamination, the arc length was made 3-1/2 to 4 inches, and the melting rate was set at 11 pounds per hour. The atmosphere was 20% argon-80% helium, so that electrical losses due to corona would be low. The three-inch diameter billets were made at 1250 amp and 54 volts; the six-inch diameter billets were made at 1600 amp and 64 volts.

For alloying work, it would be beneficial to have increased fluidity in the melt. Such fluidity may be obtained by increasing the current or the argon to 30% or 40% of the atmosphere. More tungsten contamination may be expected because the splashing increases when the pool of metal is very fluid. The arc-melting process does not remove most elements from zirconium crystal bar. Magnesium, halide salts, and possibly hydrogen, however, can be removed from sponge metal. After arc melting, there is a pickup of tungsten (20 to 200 ppm) and of nitrogen (30 ppm) in the zirconium. The arc-melting process is not suited for purifying zirconium, but it is a good method for casting billets with a minimum of contamination.

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Table I

ANALYSIS OF SPONGE AND CRYSTAL BAR ZIRCONIUM
PARTS PER MILLION OF EACH ELEMENT⁽¹⁾

Metal	O	C	N	Al	Cr	Cu	Fe	Mg	Mn	Ni	Si	Ti
High Purity Crystal Bar	200	190	20	20	L2	10	7	5	50	L5	L50	L50
Crystal Bar	200	70/350	60/150	30/100	2/10	5/20	200/700	1/20	2/7	40/80	L50	L50
Du Pont Sponge	800/1100	125/260	100/400	400/500	L2/10	30	300/700	1/10	L1/5	3/20	L50/200	70/150
Grade A Sponge ⁽²⁾	1000	150	40/70	30/400	2/10	10	200/1000	25/1400	20/50	L20	20/50	L50/300
Grade B Sponge ⁽²⁾	1000	50/150	90/150	500/1000	20	10	800/1600	50/3600	20/50	L20	60	70/150
Grade C Sponge ⁽²⁾	1000	100/300	20/70	80/200	10	10	100	5000/7200	20/50	L20	250	30/70

(1) Ag, As, B, Be, Bi, Ca, Co, K, Li, Mo, Na, P, Sb, Sn, and V below limits of spectrographic analysis

(2) Made by Bureau of Mines

Table II

CORROSION DATA OBTAINED ON CERTAIN CASTINGS

Casting Number	Type of Corrosion Product	Rate of Corrosion, mg/cm ² /month
2 top	white	1.4
bottom	white	69.6
3 top	black	2.7
bottom	white and black	4.1
4 top	white and black	0.7
bottom	white	0.6
5 top	white and black	0.7
bottom	white	15.3
12 top	black	0.2
21 top	white and black	7.6
22 top	black	0.2
23 top	black	0.2
24 top	black	0.3

Table III
MELTING DATA FOR ZIRCONIUM CASTINGS

Casting Number	Billet Diameter, inches	Grade of Crystal Bar	Metal Treatment (1)	Tungsten loss, grams	% Argon	% Helium	Absolute Pressure, inches of Hg	Casting weight, pounds	Melting Rate pounds/hour	Arc Length, inches	Volts	Amps	Hardness, HB (2)	Casting Number	Billet Diameter, inches	Grade of Crystal Bar	Metal Treatment (1)	Tungsten loss, grams	% Argon	% Helium	Absolute Pressure, inches of Hg	Casting weight, pounds	Melting Rate pounds/hour	Arc Length, inches	Volts	Amps	Hardness, HB (2)
1	3		B		100		32	4.5		1	25	500		40	3	3	B	0.4	20	80	32	23.3	11.6	4	54	1400	29-S
2	3	3	B		100		32	14		2-1/2	32	1100	92-T	41	3	3	B	0.4	20	80	32	23.4	10.8	4	54	1400	42-S
3	3	1	B		100		32	15.9		1-3/4	49	625	102-B	42	3	3	B	1.2	20	80	32	23.8	8	4	54	1200	37-S
4	3	2	C		100		32	17.7			50	750	53-T	43	3	3	B		20	80	32			4	54	1200	32-S
5	3	3	C		10	90	32	23			46	875	42-B	45	6	3	B	0.3	20	80	32	53.6	34.5	5	67	1600	47-T
6(3)	3	3	C	1.6	10	90	32	12	9.6		46	875	72-T	46	3	1	B		20	80	32	21.8	6.6	4	54	1250	43-S
7	5	3	C	0.4	10	90	32	42.7	28.6	3-1/2	63	1450	65-B	47	3	1	B	1.3	20	80	32	23.6	18.9	4	54	1250	48-B
8	5	3	C	0.9	10	90	32	35.2		4	63	1400	73-T	48	6	3	P	1.4	30	70	32	85	145	50	1500	22-S	
9	5	3	C	0.5	10	90	32	42.2		3-1/2	65	1400	66-B	49	6	3	B	1.9	20	80	32	64.3	51	4	68	1650	50-S
10	3	3	C	0.8	10	90	32	25.6			45	1050	72-T	51(8)	3	3	B	2.4	20	80	32	27	7.7	4	54	1250	52-S
11	3	3	C	0.4	10	90	20			3-1/4	41	900	61-B	52(8)	3	3	P	5.6	20	80	32	35.2	4.4	4	53	1250	52-S
12	3	1	C	0.5	10	90	20	16			41	1000	58-T	54(8)	3	3	D	0.6	20	80	32	23.4	34.7	3-1/2	50	1250	20-S
13	3	3	B	2.3	100	15	14.6				41	1000	59-B	63	6		D	1.1	20	80	32	130.6	69.6	3-1/2	65	1600	60-S
14	3	3	B	4	100	15	22	17.6			41	1000	63-S	64	6		D	3	20	80	32	89.3	41.8	4	63	1600	68-S
15	3	3	B	1.9	100	15	25.1	10			41	1000	59-T	66	3	3	B	1.0	20	80	32	25.2	5.9	4	54	1250	73-S
16	3	3	B	2.6	100	20	25.1	7.3		2	45	1000	47-T	69	3	3	B	0.6	20	80	32	28.2	10.9	4	53	1250	62-S
17	3	3	B	1.7	100	20	25.1	13.7		2	41	1000		70	6	2 & 3	D	1.0	20	80	32	137	45.6	4	64	1600	34-S
18	3	3	B	1.6	100	20	27.4	20.6			41	1000		73(9)	3	3	B	3.6	20	80	32	32	6.7	3-1/2	51	1250	28-B
19	3	3	B	2.0	100	20	25.2	20.2			42	1000		74	3	3	B	4.1	20	80	32	29.5	6.5	4	54	1250	82-S
21(4)	3	-		5.4	100	20	4.6				42	1000	79-T	76	6	1, 2 & 3	D	2.9	20	80	32	79.2	45	4	63	1600	
22	3	1	A	0.9	100	20	24	16		3	49	1000	80-S	77	3	3	B	3.2	20	80	32	67	18.7	3-1/2	52	1250	55-S
23	3	1	A	0.1	100	20				3-1/2	38	1400	55-T	78	3	3	B	1.6	20	80	32	28.8	14.4	4	54	1250	46-S
24	3	1	A	0.1	100	5	25	13.6		4	47	1250	46-T														
26	3	3	A	0.2	100	10	27.5	13.7		3	44	1150	42-I														
27	3	3	A	0.1	100	10	26.1	11.6		4	53	1150	40-T														
28(5)	3	1	B	0.6	100	10	17.6	10		4	47	1250	38-S														
30(6)	3	1	A	0.7	100	10				4	49	1450	40-T														
31(7)	3	1	A		100	10	16.7	7.2		4	50	1450	41-S														
34	3	1	B	2.7	100	10	25.8	10.3		4	47	1350	34-T														
35	3	3	B		100	10	25.3	10.5		4	45	1250	25-S														
37	5	3	B	3.9	40	60	32	54	15.4	4-1/2	67	2000	17-B														
38	5	3	P	5	30	70	32	55.4	13.9	4-1/2	68	1700	83-T														
39	5	3	B	3	20	80	32	54.4	18.1	4-1/2	66	1700	20-S														
													26-S														
													64-S														

(1) Metal treatment: (A) - corrosion tested, vapor blasted, cold rolled to 0.300 inches, pickled, outgassed 3 hours at 1100°C; (B) - corrosion tested, cold rolled, cleaned with trichloroethylene; (C) - corrosion tested, cold rolled, cleaned in trichloroethylene; outgassed 3 hours at 1100°C; (D) - drip arc melt of corrosion tested rods welded into assembly.

(2) T - top; B - bottom; S - side.

(3) Small pieces (0.2 inches diameter x 0.125 inches long) attracted to electrode.

(4) Melt of du Pont sponge metal.

(5) Large pieces of crystal bar (0.75 inches diameter x 0.125 inches long).

(6) Air leak in furnace.

(7) Zirconium piece hit tungsten tip at end of casting.

(8) White corrosion product not removed from feed metal.

(9) Water leak in furnace.

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- (6) Air leak in furnace.
- (7) Zirconium piece hit tungsten tip at end of casting.
- (8) White corrosion product not removed from feed metal.
- (9) Water leak in furnace.

Table IV

MELTING DATA AT 3 INCH ARC LENGTH, 3 INCH DIAMETER
CRUCIBLE AND A PRESSURE OF 32 INCHES, ABSOLUTE

<u>100% Helium Atmosphere</u>											
Volts	63	62	61	60	58	57-1/2	55-1/2	59	60-1/2	57-1/2	
Amps	1430	1290	1150	990	860	850	720	680	660	530	
Temperature ⁽¹⁾	933	915/942	906/897	888	888	888	897	802	788	843/788	
Pool Diameter ⁽²⁾	3+	3+	3+	3+	3+	3+	3+	3+	3+	3+	
Melt Fluidity ⁽³⁾	VF	VF	F	Q	Q	Q	Q	Q	Q	Q	
<u>80% Helium, 20% Argon</u>											
Volts	47	46-1/2	46	45	44	42-1/2	42	41			
Amps	1490	1290	1240	1110	930	710	490	250			
Temperature ⁽¹⁾	1029	937/1012	1003	1048/978	986/955	975	858				
Pool Diameter ⁽²⁾	3+	3+	3+	3+	3+	3+	2-3/4	1-1/4			
Melt Fluidity ⁽³⁾	VF	VF	VF	F	F	Q	Q	Q			
<u>60% Helium, 40% Argon</u>											
Volts	42-1/2	42-1/2	42	41-1/2	40-1/2	39-1/2	34-1/2				
Amps	1650	1430	1200	960	750	520	290				
Temperature ⁽¹⁾	1089	1063	1029	979	964	879	768				
Pool Diameter ⁽²⁾	3+	3+	3+	3+	3+	2-1/2	1				
Melt Fluidity ⁽³⁾			VF	VF	Q	Q	Q				
<u>40% Helium, 60% Argon</u>											
Volts	40	36	40-1/2	36-1/2	39	36-1/2	39	35	38-1/2	36	38
Amps	1850	1650	1490	1370	1280	1100	1025	860	780	575	525
Temperature ⁽¹⁾	1031	1072	1046	1106/1055	1102/1080	1116/1136	1135	1114/1135	1127	870/924	906
Pool Diameter ⁽²⁾		3+		3+	3+	3+	2-7/8	2-3/4	2-1/2	1-1/4	1
Melt Fluidity ⁽³⁾		S				VF		VF			1/2
<u>20% Helium, 80% Argon</u>											
Volts	29	30	31	31-1/2	30-1/2	30-1/2	29-1/2	29-1/2	29-1/2		
Amps	1950	1670	1290	1000	980	850	700	680	350		
Temperature ⁽¹⁾	1073	1072	1068	1050	1063/1020	996	933	908	744		
Pool Diameter ⁽²⁾	3+	3+	3+		2-3/4	1-1/2	1	1-1/4	Frozen		
Melt Fluidity ⁽³⁾			VF				VF	VF			
<u>100% Argon</u>											
Volts	25	25	25-1/2	25	24						
Amps	2000	1600	1180	800	410						
Temperature ⁽¹⁾	1084	1080	1044	934	818						
Pool Diameter ⁽²⁾	2-3/4	2-3/4	2	1-1/4	1-1/4						
Melt Fluidity ⁽³⁾			VF	VF	VF						

(1) Temperature in °C at the side of the billet.

(2) Measured in a 3 inch diameter crucible.

(3) Degree of fluidity in the melt: Q - quiet; F - fluid; VF - very fluid; S - splashes metal onto crucible wall.

Table V

EFFECT OF VACUUM ON THE TEMPERATURE DEVELOPED IN
THE BILLET AT CONSTANT CURRENT, ATMOSPHERE
OF 100% HELIUM, AND 3 INCH ARC LENGTH

<u>Current 1000 amps</u>								
Pressure ⁽¹⁾	5	10	15	20	25	30	35	40
Volts	33	41	47	51	55	59	63	68
Temperature ⁽²⁾	899	974	998	1042	1036	1036	1046	1050
<u>Current 850 amps</u>								
Pressure ⁽¹⁾	5	10	15	20	25	30	35	40
Volts	33	40	45	49	53	57	60	64
Temperature ⁽²⁾	881	953	993	1015	1012	1020	1035	1049
<u>Current 700 amps</u>								
Pressure ⁽¹⁾	5	10	15	20	25	30	35	40
Volts	32	39	43	47	51	54	58	61
Temperature ⁽²⁾	770	897	933	982	975	988	1005	1016

(1) inches of mercury, absolute

(2) °C, at side of billet, 3/16" from edge, 1" below surface.

Table VI

EFFECT OF ARC LENGTH ON CORONA AND MELT
FLUIDITY AT CONSTANT POWER (40 KW)

		100% Helium at 30 " absolute pressures					
Atmosphere							
Arc Length, inches	1	1-1/2	2	2-1/2	3	3-1/2	4
Volts	45	49	52	54	58	62	65
Amps	890	820	770	735	690	645	615
Temperature, °C(1)	897	994	959	959	959	950	968
Corona	no	no	no	no	no	no	no
Fluidity(2)	++	Q	Q	Q	Q	Q	Q
		80% Helium, 20% Argon at 30 " absolute pressure					
Atmosphere							
Arc Length, inches	1	1-1/2	2	2-1/2	3	3-1/2	4
Volts	33	38	41	42	44	46	50
Amps	1290	1070	980	940	900	860	795
Temperature, °C(1)	950	986	1029	1038	1072	1003	1012
Corona	no	no	no	no	no	no	no
Fluidity(2)	++++	++	+	+	+	Q	Q
		60% Helium, 40% Argon at 30 " absolute pressure					
Atmosphere							
Arc Length, inches	1	1-1/2	2	2-1/2	3	3-1/2	4
Volts	32	35	37	38	40	42	43
Amps	1290	1130	1070	1050	1000	950	930
Temperature, °C(1)	1012	994	1012	1012	994	986	955
Corona	no	no	no	no	no	no	Slight
Fluidity(2)	VF	++++	++++	++++	+++	+++	+++
		40% Helium, 60% Argon at 30 " absolute pressure					
Atmosphere							
Arc Length, inches	1	1-1/2	2	2-1/2	3	3-1/2	4
Volts	29	30	31	34	36	39	41
Amps	1340	1280	1250	1180	1110	1030	975
Temperature, °C(1)	1038	1080	1106	1106	1097	1089	1012
Corona	no	no	no	no	no	Slight	Slight
Fluidity(2)	VF	VF	++++	++++	++++	++++	+++
		20% Helium, 80% Argon at 30 " absolute pressure					
Atmosphere							
Arc Length, inches	1	1-1/2	2	2-1/2	3	3-1/2	4
Volts		24	27	29	31	32	32
Amps	(3)	1670	1480	1380	1290	1250	1250
Temperature, °C(1)		1003	1020	1003	951	897	861
Corona		no	no	no	Slight	Large	All
Fluidity(2)		VF	VF	VF	++++	++++(4)	(5)
		100% Argon at 30 " absolute pressure					
Atmosphere							
Arc Length, inches	1	1-1/2	2	2-1/2	3	3-1/2	4
Volts			22	23	25		
Amps	(3)	(3)	1820	1700	1600	(3)	(3)
Temperature, °C(1)			986	986	1003		
Corona			no	Slight	Large		
Fluidity(2)			VF	VF	VF		

(1) Measured 3/16" from the side of the billet, 1" below the surface.

(2) Fluidity estimated as quiet (Q), fluid (+ 's) or very fluid (VF).

(3) No data because of operating difficulties - unstable arc, corona, etc

(4) Pool was just melted.

(5) Pool was frozen.

FIGURE 1.

ARC FURNACE FOR 3 AND
 6 INCH DIAMETER BILLETS

Fig. 2. Undissolved tungsten inclusion in zirconium casting. Note area of alloying around the inclusion.

Macro 7,901

Polished

15X

Fig. 3. Enlarged view of center part of Fig. 2. Zirconium matrix is at the upper right section of the picture.

Micro 7,902

Etched

75X

FIGURE 4
MELTING RATE VS. TUNGSTEN LOSS

Fig. 5. Welded rod assembly for 3" diameter crucible. Rods butt welded to make 29" length desired.

Macro 13,246

1/5X

Fig. 6. Assembly for 6" diameter crucible. Note heavy white corrosion product remaining on some rods.

Macro 13,247

1/5X

Fig. 7 Casting #63 made by the 'drip-arc' melting process. Shuts at the bottom of the billet result from low current used at the start of the melt.

Macro 13,218

1/4X

Fig. 8 Rear view of casting #63 shown in Fig. 7. Unmelted rods are due to the arc being off center.

Macro 13,219

1/4X

Figure 9. Casting #54, 3" in diameter, made by 'drip-arc' melting process. Top of billet is at the left side of the picture. Note that the surface is rougher than 6" billet in Figure 7.

Macro 12,952

1/2X

Figure 10. Top surface of casting #54. Zirconium splashed onto the crucible to form the beads at the lower edge.

Macro 12,954

1X

Figure 11. Bottom surface of casting #54. The chopped pieces, used for starting the melt, are shown.

Macro 12,953

1X

Figure 12. Casting #21 made from du Pont sponge zirconium.
The dark areas are sonims which are not found in
castings made from crystal bar.

Micro 11,991

Bright field, etched

250X

Figure 13. Du Pont Sponge Zirconium Casting.

Micro 10,729

Bright Field, etched HF + AgNO₃

60X

FIGURE 14
BILLET TEMPERATURE VS. MELTING CURRENT
IN VARIOUS ATMOSPHERES

FIGURE 15
BILLET TEMPERATURE VS. MELTING CURRENT
IN VARIOUS ATMOSPHERES