

# Preparation of CrFe Alloys

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## ABSTRACT

High purity, homogeneous samples were prepared for a study of the thermoelectric power and electrical resistivity of the CrFe system. The use of an induction heater for making these samples at first appeared to be ideal because of its ability to generate high temperature and its magnetic stirring properties. However, the chromium component of the molten alloy at this temperature is quite reactive, and a crucible material was not found that would remain inert and intact.

The next device that was tried, an arc furnace, did fill the requirement of being able to contain the melt without reacting with it. The samples were successfully melted on a water-cooled copper hearth with an arc initiated from a water-cooled tungsten tip, under one-half atmosphere of argon. The samples made were of 5-9's purity and were homogeneous to within  $\pm 0.2\%$  of their entire volume of some 2 cc.

Further difficulty was encountered in reducing the rough, as-cast ingot to a usable geometry. In this case a uniform cylinder was desired of approximately 1/8-inch diameter and 2-1/2-inch length. This difficulty was overcome by spark-erosion machining of the rough ingot to near cylindrical form, followed by centerless grinding to a perfect cylinder.

## PROBLEM STATUS

This report completes one phase of the problem; work on other aspects of the problem is continuing.

## AUTHORIZATION

NRL Problem E02-02  
Project RR 010-01-44-5601

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## PREPARATION OF CrFe ALLOYS

### INTRODUCTION

A project was undertaken at NRL to study the thermoelectric power and electrical resistivity of binary alloys of the first long transition metal series. Initially, CrFe alloys in the 10% to 50% range were investigated, using samples that were made in our laboratory. Part of the difficulty in the fabrication of the CrFe alloys arose from the desired sample size and shape. The most advantageous sample for our measuring apparatus (1) is a wire of more or less arbitrary, but small, diameter and at least 2-1/2 in. in length. Accordingly, an ingot size 1/8 inch in diameter by 3 inches in length was selected. This turned out to be one of the smaller impediments in our task of sample fabrication.

At first, we were ill advised as to the ease and method of fabrication. We were told to place the charges in an alumina ( $\text{Al}_2\text{O}_3$ ) crucible, melt in a vacuum with an induction heater, shake out the ingot, and reuse the crucible 7 to 10 times.

### INITIAL PROCEDURE

Thermoelectric power measurements are sensitive to both impurities and homogeneities. For maximum homogeneity, induction melting seemed to be preferable because of its magnetic stirring properties and because it is able to induce temperatures in excess of  $1830^\circ\text{C}$ , the melting temperature of chromium. The initial melts were made in a dynamic vacuum with a 5-kw induction heater. The sample components were put in an alumina crucible which in turn was placed in two other alumina crucibles inside a quartz tube. Since we were working near the temperature limit of the crucibles, the extra crucibles were deemed necessary for protection to personnel should the containing crucible fail (which frequently happened), causing the molten metal to break the quartz and spatter the area with quartz and/or white hot metal. However, the extra thicknesses of crucible considerably increased the induction coil diameter and the coil-to-melt distance, thereby decreasing the coil-melt coupling efficiency. The coupling efficiency was further decreased by the fact that our charge weighed 25 g and consisted of particles on the order of 1/16 in. in diameter.

If the vapor pressures of the primary constituents are sufficiently low, a dynamic vacuum can be used to maintain or improve the starting material purity by permitting continuous outgassing of the crucible and charge during the melting process. However, because the vapor pressure of chromium is about 1 torr at  $1830^\circ\text{C}$ , we succeeded only in vapor depositing chromium over the interior of the vacuum system up to the cold trap. It was then decided to outgas at  $10^{-5}$  torr at temperatures up to  $800^\circ\text{C}$ , then backfill to 500 torr with helium and subsequently proceed to melt. To avoid overheating the sample and crucible, it is necessary to visually observe the melt. Therefore, one must avoid backfill pressures which permit the initiation of glow discharge.

A number of crucibles of various composition and from various manufacturers were tried, the best of which was recrystallized impervious aluminum oxide as made by either Morganite (triangle RR) (2) or Coors. (Some zirconium oxide crucibles were ordered but were 18 months in arriving and thus have never been evaluated.) The most severe problems encountered were reactions of chromium with the crucibles, forming  $\text{Cr}_2\text{O}_3$  and

ruby crystals. As the chromium particles melt, they wet the crucible wall, causing a sizable portion of the charge to adhere to the wall. If the crucibles were substantially larger than the 1/4-in. diameter used, such adherence would represent the loss of a considerably smaller portion of the charge. Since the penetration depth of these crystal-lites is probably not too great, one could remove them by adequate machining of the ingot surface.

A traveling coil mechanism was built, and, with practice, the operator could manipulate the heater power and coil location so as to cause most of the melt to work its way to the bottom of the crucible. However, even after obtaining a compact molten ingot, a contraction pipe would be drawn into the top of the ingot on cooling.

In all, some five or six induction heaters of various frequency and output power were used, the most satisfactory being one of 12.5-kw output at 100 kc. Considering all factors involved, the liabilities of induction melting of these alloys seemed to considerably outweigh the assets. A new melting technique was desired, and to that end an arc furnace was employed.

### ARC FURNACE

Preliminary attempts indicated that the arc melting technique would be satisfactory, so a stainless steel furnace was purchased from Vacuum Industries, Inc., for this project and is shown in Fig. 1. Basically, it consisted of a cylindrical Pyrex vacuum chamber, the "stinger" or negative electrode, which is also used for moving the specimen inside the furnace between melts, and the hearth (barely visible), which is both anode and mold for the melt. The "stinger" is brought into the vacuum chamber through a brass and Teflon ball and swivel joint, which permits the operator to preset the penetration depth of the stinger and then move the arc over the entire hearth. The stinger itself is water cooled by means of two concentric copper tubes which form its body. Attached to the end of the outer tube is a tungsten tip, shown in Fig. 2.

The hearth is a 1-in.-thick copper plate and is shown in Fig. 3. The depressions represent an evolution of a number of ideas about suitable molds. Those that have worked the best, for our alloy system, are the two smaller spherical segments, which are 1-1/4 in. across by 1/2 in. deep; the two larger spherical segments which are 1-5/8 in. across by 0.450 in. deep; and the two longest cylindrical depressions, which are near the center of the hearth. The smaller of these is 5 in. long with a 3/16-in. radius, and the larger is 6 in. long with a 1/4-in. radius. The remaining depressions were not found to be particularly useful. The narrowest of these, 3-1/4 in. long with a 1/8-in. radius, was found to be too narrow. The surface tension of the liquid alloy is too great to permit the alloy to flow along the depression and causes it to ball up and flow over the edge of the mold, seeking to form a sphere. The 3/16-in.-diameter depression does not do this and is the mold used most.

Also present on the hearth are two tungsten electrodes which serve a dual purpose. They are used as starting tips to initiate the arc, and are also used as handles to remove the hearth from the furnace. If such starting tips are not used, the hearth would soon be eroded if the arc were struck against it; and the charge to be melted would be scattered if the arc were struck against it: in either case the "stinger" electrode will be contaminated and subsequent melts impure.

### MELTING PROCEDURE

The starting materials used were iodide chromium of 5-9's purity from Chromalloy Corp. and 1/4-in. iron rods of 5-9's purity from Johnson Matthey. The chromium is

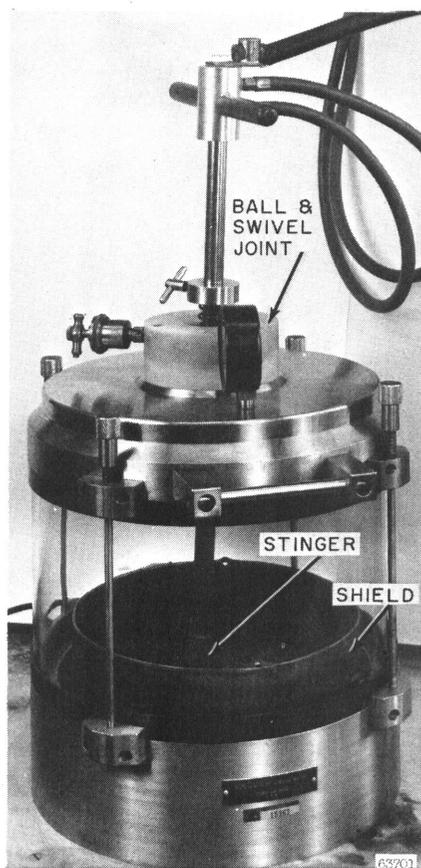


Fig. 1 - The arc furnace

broken up into pieces that range in size from powder to chips on the order of  $1/64$  sq. in. in cross section. The iron rod is cut into wafers from  $1/64$  in. to  $1/16$  in. thick. The constituents are then etched in hydrochloric acid to which hydrogen peroxide has been added at the rate of a few drops per 25 cc. The etched particles are then washed in deionized water, followed by an alcohol wash and subsequently air dried.

Our samples consisted of two 12.5-g units of the proper ratio of Cr to Fe. Two 12.5-g unit charges were initially made to allow maximum mixing of the Cr and Fe, resulting in a homogeneous ingot. After suitable mixing, the two units are combined to form the final 25-g ingot.

The initial charges are placed in the smaller of the circular depressions, and a zirconium getter button is placed in one of the larger circular depressions (Fig. 4). The cap is placed on the furnace, and the chamber is rough pumped and backfilled several times. The final backfill is stopped at about  $2/3$  of an atmosphere. Either argon or helium can be used for the inert atmosphere, but argon seems to be preferable. Helium permits chromium to boil off and deposit itself on the interior of the chamber, considerably clouding the Pyrex and making visual observation difficult.

The power source is a variable current welding power supply with a 40 to 400 amp range. A description of this device will be given in the "modifications" section. Starting at about 80 amps, the operator first melts the getter button and then starts to work on one of the unit charges. As the top pieces of the charge melt and form a liquid skin over the solid chips beneath (Fig. 4), the current is increased and the stinger is moved rapidly over the area of the charge. Thus, the molten iron and chromium are mixed by the pressure of the arc. Since the hearth is considerably cooler than the melt, and because one does not want to contaminate the melt, there is usually a layer of charge on the bottom of the depression which is not melted. After the operator is satisfied with the state of the melt, the current is reduced and the arc is transferred to the second unit charge. The process is repeated; then the arc is extinguished.

After the color leaves the tip of the "stinger" and the ingot, one of the charges is transferred to the cylindrical depression 5 in. long with a  $3/16$ -in. radius, while the other is inverted in its own mold. Again the same melting procedure is carried out (Fig. 5), whereupon the ingots and molds are interchanged. Thus, the liquidus-solidus interface does not get chased up and down, as it would if the ingot were merely inverted in the same mold. Rather, the interface history is destroyed during each melt. The alternating between small circular and small cylindrical mold continues until each unit has been melted about 10 times, whereupon one of the charges is run out in the cylindrical depression until it is just a thin skin about 5 in. long. This requires very fast traversing of the melt with the arc at about 200 amps. This skin is then placed in the cylindrical mold 6 in. long with a  $1/4$ -in. radius, and the second charge undergoes the same treatment (Fig. 6), whereupon it is placed beside the first ingot. With the arc at about 100 amps, the two unit charges are then slowly fused together. After they are joined, the arc is increased and moved over the length of the melt, causing it to decrease in length and increase in diameter because of the surface tension.

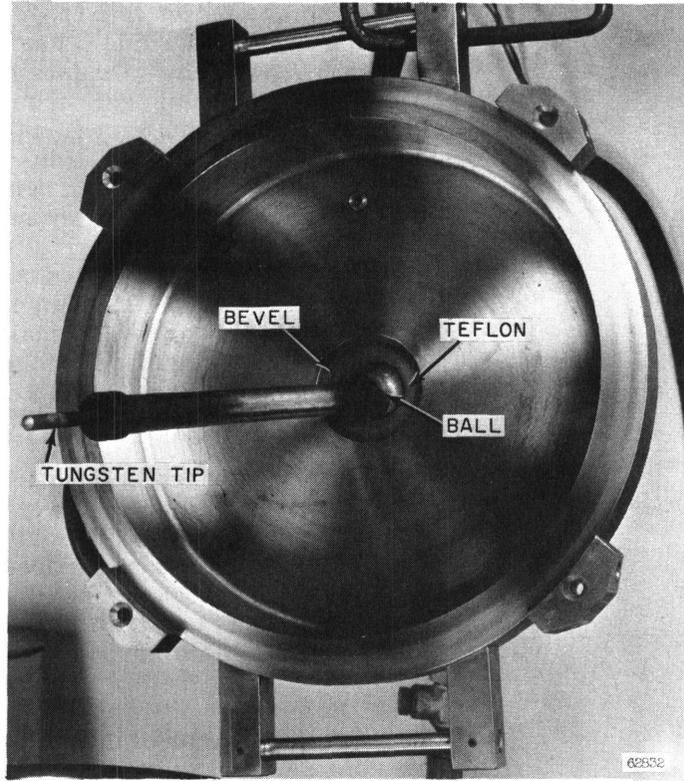


Fig. 2 - The stinger-cap configuration

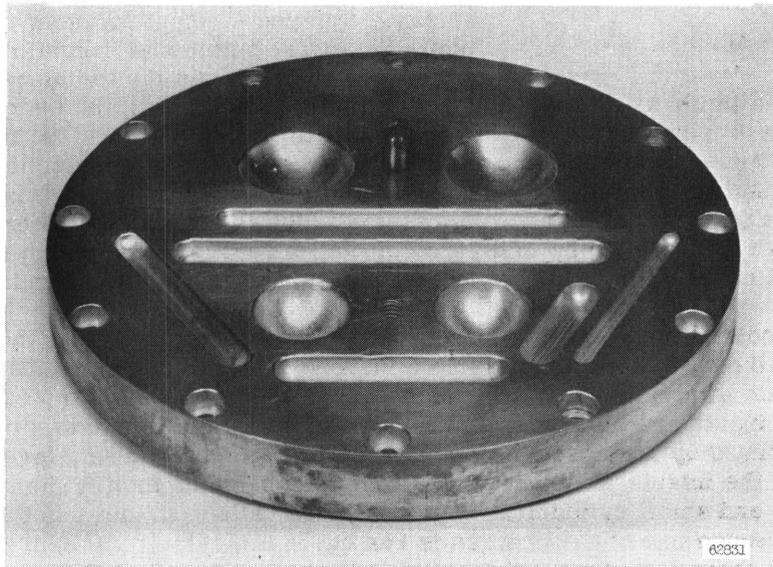


Fig. 3 - The hearth plate configuration

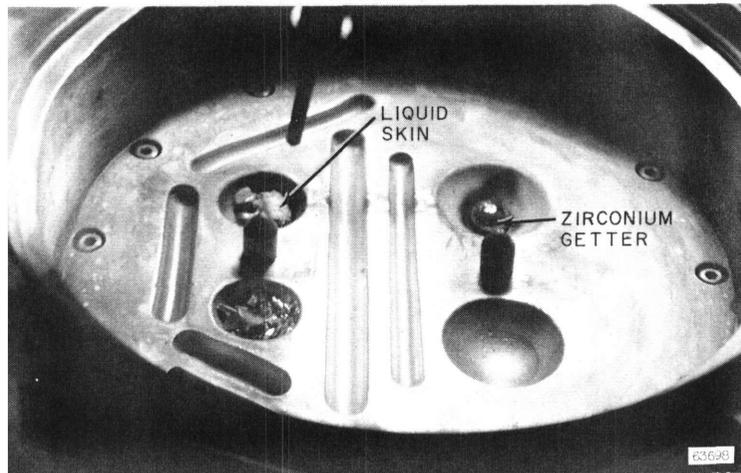


Fig. 4 - Initial charge placements

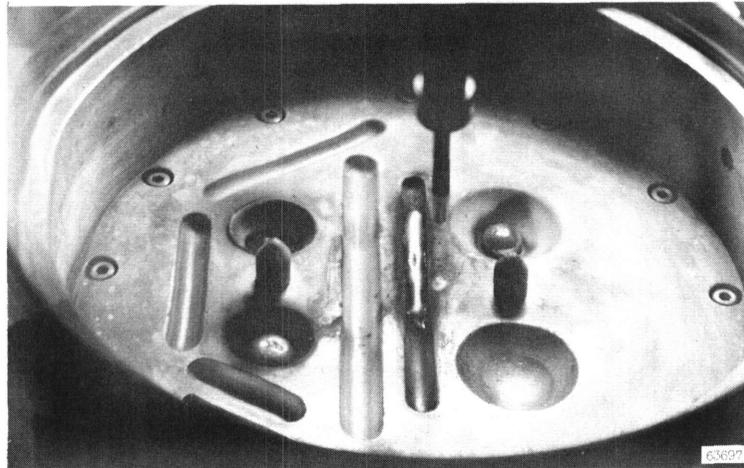


Fig. 5 - First melting stage

This cylindrical ingot is then placed so that it bridges the remaining larger circular depression. With an arc of about 160 amps, the center of the bridge is melted, causing it to collapse, and, as soon as it falls, the arc is transferred to the two ends sticking up. By alternating ends, one can melt the ingot so that the molten metal will flow along the solid portion (Fig. 7), and all will eventually end up in the bottom of the mold. However, if the ends are melted too fast, the ingot may fall to the hearth in drops and splatter, causing great difficulty in their recovery.

The button ingot is now put back into the same smaller cylindrical mold, melted, and returned to the circular mold. This process is repeated another eight to ten times. After the operator is satisfied with the state of the ingot, the final button to cylinder transfer is made, and a smooth ingot is cast. It may be necessary to turn the cylinder over once and, with an arc of about 110 amps, very lightly melt and smooth the rough surface which was just previously in contact with the hearth.

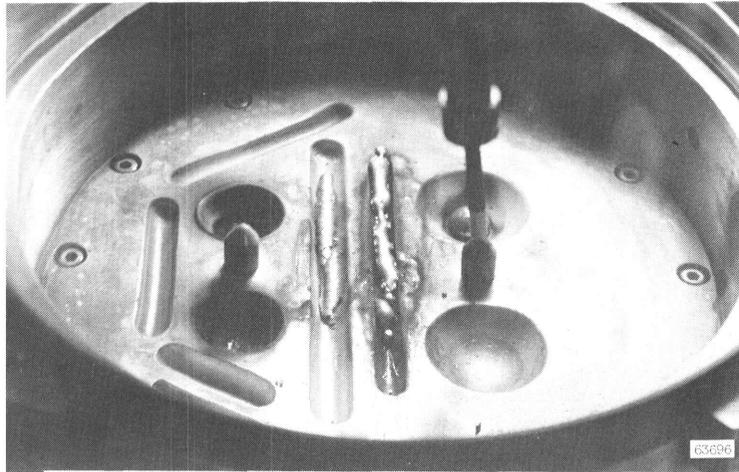


Fig. 6 - Preparation for joining charges

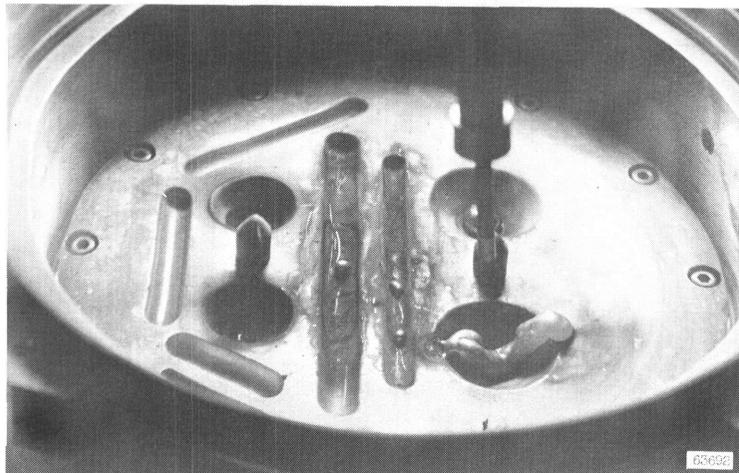


Fig. 7 - Homogenizing the full charge

With the single 25-g charge, it is frequently helpful to break the cylinder into thirds prior to transferring it to the circular mold. This can be done by drawing the melt out to about 4 in. in length, and then concentrating a fairly strong arc at two points along the molten ingot. The ingot will easily melt through at these places and one can then mix the three pieces in the circular mold (Fig. 8).

We now have an ingot that is about 3 in. long with a 3/8-in. diameter. After things cool down, the top of the furnace is removed. Prior to removing the ingot, however, a screwdriver or some other suitable device should be used to scrape any surfaces of the interior of the furnace that are covered with a sootlike deposit. Especially with a helium atmosphere, it was found that an apparent unstable chromium oxide is deposited which, when disturbed at all, will form a stable oxide by slowly burning.

The completed ingot is then cut on a spark erosion cutter, which, in our case, produced a nearly uniform cylinder. The rough ingot is glued into an arbor with woods metal and is

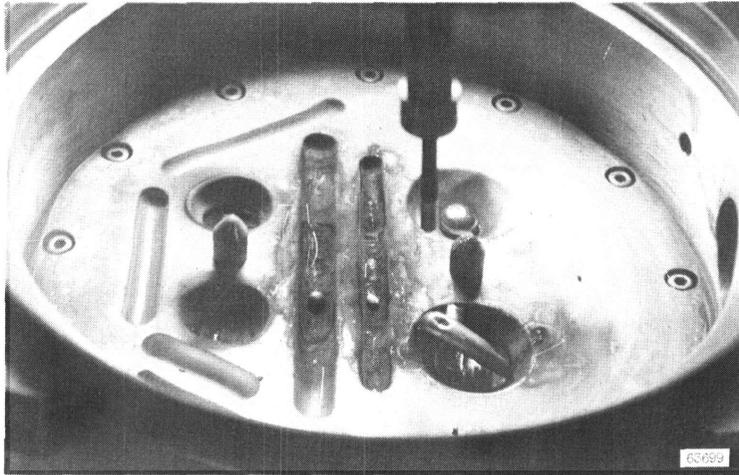


Fig. 8 - Breaking up the full charge.

then spun through a hole in a brass plate, eroding the ingot into a cylinder. The hole in the brass is usually 1/64 in. or so larger than the desired finished cylinder. The specimen will emerge from this process with a somewhat pitted surface and a slight taper. It is then placed in a centerless grinder and polished to the desired diameter. The cast ingot is compared with a polished ingot in Fig. 9.

The cylinder is then cleaned in a liquid honing machine to clean away the sludge deposited on the surface from the grinding lubricant. It is then electrolytically etched in a 50-50 solution of nitric and acetic acids. Once again it is washed, in deionized water and in alcohol, and air dried. It is now ready to be annealed.

The sample is wrapped in a 0.002-in. molybdenum foil for the anneal. The molybdenum foil is usually very brittle and will crack if wrapped around such a small piece. However, if one first anneals the molybdenum in a vacuum (red heat is adequate) the foil is quite ductile. The foil-wrapped specimen is then inserted in an alumina crucible inside of a quartz tube. The tube is attached to a vacuum system which is evacuated and backfilled twice, with a final atmosphere of 500 torr of helium. The sample is annealed at 1250°C for 2-1/2 hours and then water quenched. The heat for the anneal can be supplied by either an induction coil or by a Kanthal heater. The temperature is monitored by a thermocouple set between the molybdenum foil and the crucible.

The water quench is accomplished in the following manner. The quartz tube is connected to the vacuum system vertically by means of a quick coupling and a bucket of water is placed underneath it. After the tube is evacuated, the coupling nut is unscrewed just a bit, and the vacuum is adequate to hold the tube in place. After the anneal, a 2 to 5 psi overpressure of helium is imposed on the vacuum system, whereupon the tube is shot out of the furnace into the water. After the sample cools, it is taken out of the foil, etched, and cleaned once again, and is now ready for measurement. The annealing apparatus used is the same apparatus that was used in our induction melting attempts.

Samples prepared in this manner have been analyzed both spectrographically and quantitatively. The impurities noted in the spectrographic analysis are within the tolerances specified in the analysis received with our starting material, including the presence of oxygen. Quantitative analyses have shown the samples to be homogeneous throughout their length and cross section to the accuracy of the analysis, plus or minus 0.2%. The final sample ratio of chromium to iron is typically 0.3% less chromium than was started

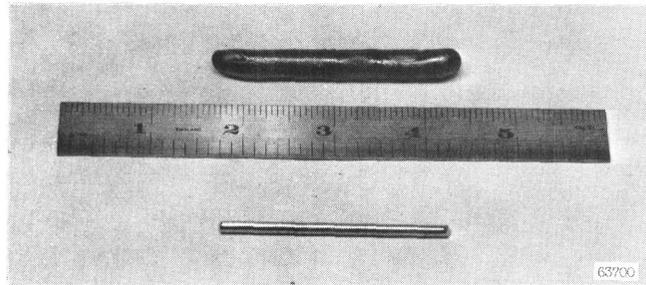


Fig. 9 - Comparison of the cast and polished ingots

with. This is due, at least in part, to the evaporation of chromium forming the black deposit referred to previously.

#### MODIFICATIONS

Several modifications have been made on the arc furnace since its purchase. The prime means of cooling the furnace is a flow of cold water under the 1-in.-thick copper hearth plate. Originally, there was only an inlet and an outlet hole diametrically offset in the stainless steel base of the furnace and a single circular baffle at the center of the cooling chamber. The bottom of the hearth plate was smooth, permitting laminar flow of the cooling water, which allowed the hearth to run rather hot. To change this, a series of baffles were attached to the bottom of the hearth plate, causing turbulent flow (Fig. 10).

Another cooling problem arose in that occasionally the furnace walls got so hot that the neoprene gasket between the Pyrex cylinder and the furnace base almost became vulcanized to the base. Since this heating is due primarily to radiation, a 5-in. high, 20-gauge stainless steel radiation shield that fits snugly around the perimeter of the hearth was made. There is a gap of about 1/2 in. between the hearth and the wall of the

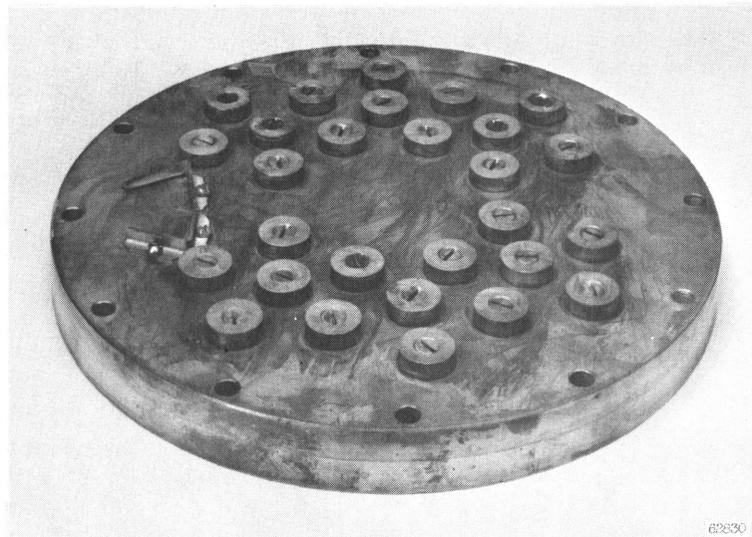


Fig. 10 - Bottom of the hearth plate

furnace, and occasionally particles of the charge, or the entire charge itself, will be knocked off the face of the hearth. This radiation shield thus serves a dual purpose in that it also confines all particles to the hearth face. To further cool the walls of the furnace, a 12-in. fan is usually played on the furnace during the melting operation.

Another modification was made to the feed-through hole in the Teflon block of the ball and swivel joint for the stinger. As was mentioned above, the stinger, with the current off, also serves as a manipulator by which the sample is moved from depression to depression. As purchased, the Teflon block restricted the polar angle through which the stinger could rotate and one could not retrieve ingots pushed to the edge of the hearth. By increasing the diameter of the holes in the Teflon, the entire hearth face could be reached by the stinger tip. It was also necessary to put a greater bevel on the circumference of the hole in the stainless steel furnace cap, to prevent arcing from the stinger to the cap.

It was found very desirable to alter the current during the melt, since if one started the arc at a current high enough to melt, severe damage to the tungsten tips took place when starting the arc. Also, too high an initial current will cause the arc to move the charge in the mold before melting occurs, which results in the loss of small pieces of charge. Finally, if too high a current arc is used when transferring the arc from one depression to another, severe hearth scarring can occur. Therefore, the operator should reduce the arc current before the transfer.

For these reasons, a variable current power source was used. The unit was found in a surplus warehouse and at some point in its history was modified by the installation of a motor-driven secondary coil in the transformer section. In order to take advantage of this feature, a group of four foot pedals was wired into the circuit of the machine. The operator can trip the master on-off solenoid by one foot pedal. Two other pedals are used to increase and reduce the current after the arc has been struck.

The remainnig pedal is used to impose a high-frequency ac signal on the dc arc current so as to enable the operator to initiate the arc without physical contact between the "stinger" and the starting tip. However, the high-frequency arc starter was found to present a back emf to the selenium rectifiers in the power supply sufficient to break them down. To eliminate this problem, the selenium rectifiers were replaced by silicone diodes, and an L filter was inserted between the diodes and the starter. The filter consisted of a 0.100- $\mu$ f capacitor in parallel with the diodes, and a 6-in.-diameter coil of 8 turns of number 0 wire in series with the diodes and the starter. A 200-ohm resistor is also placed in parallel with the high frequency starter.

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14. KEY WORDS	LINK A		LINK B		LINK C	
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Alloys Chromium Iron Induction heating Vacuum furnaces Refining (Metallurgy) Melting Electric arcs Annealing						

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