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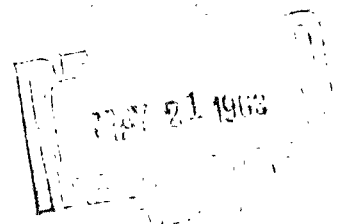
PREPARATION AND PROPERTIES OF CONSUMABLE-ELECTRODE VACUUM-ARC-MELTED ELECTROLYTIC IRON

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ABSTRACT

Oxygen was removed from a relatively pure grade of electrolytic iron by a solid-state, hydrogen reduction treatment prior to vacuum arc melting. Consumable electrode arc melting into a water-cooled copper crucible excluded contamination. The initial oxygen content of 400 ppm was reduced to below 50 ppm in the ingot. The values of various physical (density, electrical resistivity, thermoelectric force, and magnetic) and mechanical (tensile strength, Charpy V notch, and hardness) properties were measured to assess the quality of this iron, which compares favorably with research grades described in the literature.



PROBLEM STATUS

This is a final report on one phase of the problem; work on other phases is continuing.

AUTHORIZATION

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PREPARATION AND PROPERTIES OF CONSUMABLE-ELECTRODE VACUUM-ARC-MELTED ELECTROLYTIC IRON

INTRODUCTION

A wide variety of research requirements for pure iron can be met by using a commercial, high-quality, electrolytic iron and purifying it to desired oxygen and carbon levels by a hydrogen reduction treatment. The treated iron can be consolidated into ingot form, without contamination, by consumable-electrode, vacuum-arc-melting procedures. The processing and fabrication of this high-purity iron into suitable form for full-scale test specimens is described herein.

Evaluation of the quality of the iron, as determined by analyses and characterized by several types of physical and mechanical property measurements, conforms to the methods described in the literature. A general survey article entitled "The Preparation and Properties of High-Purity Iron" by Hopkins (1) outlines the previous work of numerous investigators in this field through 1956. The Hopkins survey provides a compilation and comparison of properties of iron with various levels of known impurities. Despite the similarities in reported analyses, considerable differences have been reported in respect to physical properties sensitive to specific impurities.

HYDROGEN REDUCTION PROCESSING

Electrolytic iron, in flake, nodule, or powder form, containing various levels of impurities is produced commercially on a large scale. Typical nominal impurity analyses of two grades of electrolytic flake form of iron, or "plast-iron," are shown in Table 1.

Of the major impurities listed, only oxygen and carbon in stoichiometric amounts can effectively be removed (as CO gas) by vacuum melting the electrolytic iron. The concentration of certain other volatiles will also be lowered depending upon their vapor pressure and the time, temperature, and vacuum pressure of melting.

Other investigators (2) have employed a solid-state, hydrogen reduction process to reduce the oxygen and carbon content of iron. However their methods were quite elaborate, and extensive periods of time were involved.

To evaluate the effectiveness of a simple hydrogen reduction treatment, a one-pound sample of Grade A101 electrolytic iron, referred to in Table 1, was used in the preliminary tests. These tests were conducted in a hydrogen annealing furnace of a pass-through muffle type with an attached cooling zone. The first reduction treatment was at 850°C for 45 minutes with a hydrogen flow rate of 2.5 cfm. Two additional samples from different lots of electrolytic iron were then treated for 4 hours at 850°C. After examination of the results, shown in Table 2, another two samples were hydrogen treated at 1150°C (temperature limit of the furnace) for one hour. Analytical results were obtained from representative samples taken from 150-gm buttons, helium arc melted in a water-cooled copper crucible in a pure, liquid-nitrogen trapped, helium atmosphere. It can be seen that an equilibrium composition for a given temperature is reached in a relatively short time. Also, the higher temperature (1150°C) is quite effective in reducing the oxygen to the 50-ppm level.

Table 1
Typical Impurity Composition of Electrolytic Iron Flake
and of Vacuum-Melted Ingot

Element	Concentration (ppm)		
	Grade A101*	Grade A104*	VM Ingot D-235†
Aluminum	20	< 30	< 1
Calcium	-	< 10	< 1
Carbon (total)	20	40	< 50
Chromium	20	< 30	1-10
Cobalt	70	20	1-10
Copper	50	< 10	1-10
Hydrogen	100	100	< 1
Lead	~0.1	< 10	-
Manganese	20	< 10	1-10
Magnesium	-	< 5	~ 0.1
Molybdenum	20	< 40	10
Nickel	30	< 10	10
Nitrogen	40	40	11
Oxygen (H ₂ loss)	400	500	-
Oxygen (vacuum fusion)	-	-	50
Phosphorus	30	30	20
Silicon	50	10	1-10
Sulfur (total)	30	40	30
Tin	50	50	-
Titanium	-	< 10	-
Vanadium	-	< 50	10
Zinc	-	100	-
Zirconium	-	< 10	-

*Nominal analysis furnished by supplier of material.

†NRL vacuum melt and analysis.

Table 2
Weight Percent of Some Impurities in Electrolytic Iron
as a Function of Hydrogen Reduction Treatment
(Samples taken from 150-gm helium-arc-melted buttons)

Lot No. and Anneal Treatment	Weight Percent (ppm)			
	Oxygen	Nitrogen	Hydrogen	Carbon
A101 (as-received)	360	15	60	50
A101, 45 min at 850°C	222	16	1	70
A101 } A104 } 4 hrs at 850°C	229	40	1	50
	117	19	10	70
A101 } A104 } 1 hr at 1150°C	98	28	2	50
	56	12	2	50
A104 (duplicate), 1 hr at 1150°C	50	11	5	-

A 100-lb lot of A104 electrolytic iron, with nominal analysis as in Table 1, was selected for processing to ingot. About 16 lb of flake iron per run were spread out in an iron tray which was inserted into the furnace at 1150°C. The hydrogen flow level in the furnace was set at 0.5 cfm for the first 90 minutes. The hydrogen flow was then increased to 2.5 cfm for 30 minutes before withdrawing the tray into the cooling chamber, and then it was maintained at the higher rate for a 1-hr cooling period. The purpose of the low hydrogen flow rate at the beginning was to allow the H₂O product of the reduction to increase the dew point of the hydrogen and assist in carbon removal. The final higher flow rate decreased the moisture content of the furnace hydrogen to near the -70°C dew point of the input gas.

MELTING PROCEDURE

The hydrogen-treated electrolytic iron flake was ductile and was easily compacted into 2-1/2-in.-diam cylinders using a 60-ton press. A dozen compacts were stacked in a vacuum furnace to form an electrode by sintering. With the electrode stinger making contact with the top compact, the sintering current was gradually increased to 5000 amp while maintaining a one micron vacuum. Sintering was completed in about 30 minutes; the temperature of the compacts was above 1000°C, and the final vacuum below 0.1 micron. After cooling the electrode, the vacuum furnace was opened to insert a starting pad of loose iron flake in the bottom of a 4-in.-diam water-cooled copper crucible. When a vacuum of 0.02 micron was re-established in the cold furnace chamber, arc melting was commenced with a current of 1500 amp at 25 volts. The melting current was increased to 1800-2000 amp, and the vacuum was maintained below 0.5 micron during a quiet melt with a minimum of splatter. At the completion of the melt a vacuum of 0.008 micron was measured in the furnace chamber, indicating a tight system having a low leak rate. Four ingots were vacuum arc melted under these conditions. Spectrochemical and vacuum fusion gas analysis of the vacuum-melted ingot, D-235, are included in Table 1. The carbon content listed is near the practical lower limit of the usual combustion method of analysis. It is reasonable to assume a carbon value nearer the listed nominal content of the electrolytic iron.

PROCESSING AND FABRICATION

An etched macrosection of a plate cut through the ingot axis is shown in Fig. 1. A small shrinkage cavity can be seen near the top of the ingot. Several standard-size Charpy V specimens and two tensile specimens were cut from this plate to determine the as-cast mechanical properties.

The large grain structure of the as-cast ingot, while quite ductile, fractures easily. During the initial stages of ingot breakdown by cold forging, it was necessary to work the material very slowly. A 1-1/8 × 1-1/4 × 7-1/2-in. longitudinal section was cold forged into a 3/4-in.-diam bar for tensile test specimens. The part of the bar not used for tensile specimens was further reduced by cold forging to a 1/2-in. square-cross-section bar for Charpy test specimens.

Hot forging tests indicated that slow working of the large-grained ingot iron was also required to prevent deep cracking. It is doubtful whether the full-size ingot can be cold forged without using closed blocking dies. A preferred method for the primary breakdown of the ingot into bar or rod would be by extrusion. Atmospheric contamination could be thereby minimized, and a higher yield of usable stock would be realized.

Small pieces of the ingot were also cold forged into a 3/8-in.-diam rod. The rod was stress relieved by heating in a neutral salt bath at 1200°F. The surface of the rod was removed by centerless grinding. Further cold reduction by swaging and wire drawing was employed to produce a 0.020-in.-diam wire for test purposes.



Fig. 1 - Longitudinal macro-section of vacuum-melted iron ingot showing large grain size of the as-cast material

PROPERTIES

Microstructure

The microstructure of the pure iron in the cold worked, stress relieved, and annealed condition are shown in Figs. 2, 3, and 4, respectively. These specimens were prepared by electropolishing. The large grain size of the as-cast ingot referred to in Fig. 1 was reduced by severe cold working, Fig. 2. A relatively short time at 1000°F effectively recrystallizes the cold-worked structure; however, a large range of grain sizes exist, Fig. 3. Heat treatment or annealing at 1800°F increases the grain size, Fig. 4. The absence of any second phase materials, particularly in the grain boundaries, is indicative of a high-purity iron. A subgrain structure can be seen in the background.

Tensile Properties

The large grain size of the as-melted iron ingot precludes the possibility of determining reproducible tensile data from a limited number of specimens. However, a few representative specimens were tested and the results are listed in Table 3.

Charpy V Notch Properties

There is a paucity of published data on the notch properties of pure iron. This dearth is due in part to the difficulties involved in preparing sufficient quantities of pure metals to meet the needs of various test programs. There is a current interest in notch properties, related to fracture studies, and it was deemed important to establish the baseline properties of pure iron.

The Charpy V notch properties of the as-cast ingot material and of the cold-worked iron, vacuum-annealed at 10^{-6} torr for 1 hour at 100°F, then furnace cooled, are shown in Fig. 5. Comparative data from Hopkins' paper (1) are shown as dashed lines (a sharp transition occurs +14°F [-10°C] over a 2°F temperature range for the air-cooled specimen). An explanation was offered for this behavior in the following quotation: "...the fracture of the specimens tested below the transition temperature are of the cleavage type. At temperatures above the transition, iron is very tough, with an impact value of 26-28 m.-kg or about 200 ft.-lb; the test piece does not fracture but merely bends. The transition temperature is lowest (about -10°C) for the air-cooled condition, so that the iron is then tough at room temperature. In the furnace-cooled condition, the transition temperature is well above room temperature (about 60°C), owing partly to a larger grain size and partly to carbide films along grain boundaries on very slow cooling at the carbon level (0.0025%) present in this iron. Grain boundary carbide films tend to promote cleavage rather than intergranular fracture."

The transition from ductile to brittle fracture of the iron produced for the present study occurs at lower temperatures and over a broader range for the two test conditions. The scatter in the notch test data for the as-melted high-purity iron can be attributed to the large grain size referred to in Fig. 1. However, the lower transition temperature is significant and indicative of higher purity. The fracture surfaces of typical Charpy test bars are shown in Fig. 6.



Fig. 2 - Microstructure of cold-worked high-purity iron; electropolished (200X)



Fig. 3 - Microstructure of high-purity iron, stress relieved by annealing for 1 hr at 1000° F after cold working; electropolished (200X)



Fig. 4 - High-purity iron vacuum-annealed for 1 hr at 1800°F, then furnace cooled; electropolished (200X)

Table 3
Tensile Properties of High-Purity Iron for
As-Cast, Cold-Worked, and 1000°F-Annealed Specimens
(0.505-in. Standard Specimens)

Tensile Property*	Type of Specimen		
	As-Cast	Cold-Worked	Annealed
Ultimate Tensile Strength (1000 psi)	29	53	48
Lower Yield Stress (1000 psi)	7	No Discontinuous Yield	-
Elongation (%) (2-in. gauge)	25	22	23
Reduction of Area (%)	10	>90	>90

*Rate of strain: 0.005 in./min below yield point, increased to 0.05-in./min above yield point.

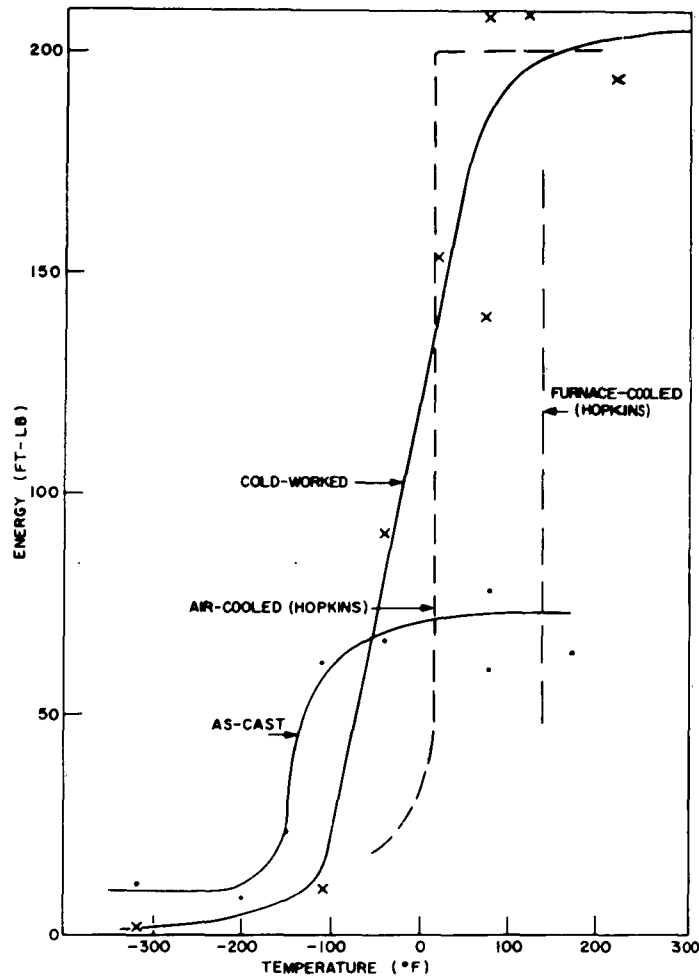


Fig.5 - Charpy V notch properties of as-cast and of cold-worked iron, vacuum-annealed at 10^{-6} torr, 1000°F for 1 hr. Dashed lines show data from Hopkins' paper (1).

Fracture in the as-melted iron specimens occurred along the grain boundaries and revealed a large-scale fibrous structure. Only one Charpy test specimen at -320°F broke completely through. The refined grain structure of the cold-worked and vacuum-annealed iron effectively increased its plasticity. Considerable deformation occurred at temperatures above -40°F , with the fracture progressing only about half way through the Charpy specimens..

Hardness

Brinell and Vicker's hardness values between 49 and 52 were measured on the iron ingot material.

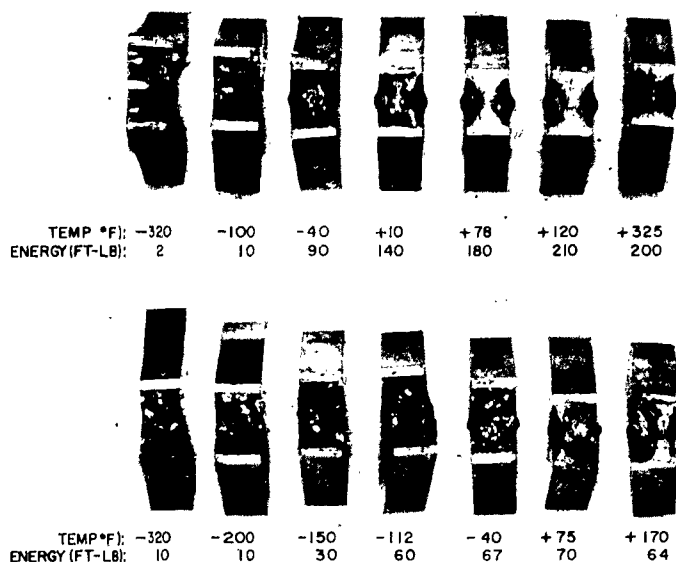


Fig. 6 - Fracture surfaces of (a) cold-worked, vacuum-annealed and (b) as-melted Charpy impact test bars

Density

In order to eliminate the problem of minute air bubbles and the rusting of the specimens resulting from measurements in water, trichloroethylene was used for the displacement liquid. The measured density of the trichloroethylene was 1.455 gm/cc at 25°C. The density of an iron cylinder machined from a 3/8-in.-diam cold-worked rod was determined by weighing in air and in trichloroethylene at 25°C and found to be 7.895 gm/cc. This density is 0.26 percent greater than the accepted value for iron of 7.874 gm/cc.

A coil of 0.020-in.-diam cold-worked iron wire had a measured density of 7.934 gm/cc, which decreased to 7.924 gm/cc after vacuum annealing at 850°C. For comparative purposes, density measurements were made on a sample of 99.999 percent iron wire (vacuum annealed at 850°C) and was found to be 7.900 gm/cc.

Electrical Resistivity

Samples of 0.020-in.-diam wire, prepared as previously described by total cold reductions of greater than 90 percent, were used for these measurements. Potential leads were attached to the wire at a separation of one meter. The sample was loosely coiled around a quartz tube and suspended in a water bath. Resistance measurements at temperatures of 0°, 20°, and 100°C were made on a Kelvin Bridge. The cross-sectional area was computed by a liquid displacement method, which gave a corrected average wire diameter of 0.02045 in. The measured electrical resistivity of a coil of the cold-worked iron wire was 9.725 microhm-cm.

The temperature coefficient of resistance in the range 0°-100°C was 0.00637 $\Omega/\Omega/^\circ\text{C}$ at 0°C. This coil of iron wire was vacuum annealed at 10^{-6} torr for two hours at 800°C and remeasured. The electrical resistivity after annealing was increased to 9.829 microhm-cm, and the temperature coefficient was lowered to 0.00634 $\Omega/\Omega/^\circ\text{C}$. These results are indicative of redistribution of impurities in the iron wire during the annealing heat treatment.

Another suggested and convenient index of purity and crystal perfection is the ratio of the electrical resistance at 300°K to that at 4.2°K (liquid helium). This ratio eliminates the necessity of measuring the specimen dimensions at the two temperatures and is related directly to sample purity and crystalline perfection. The resistance of a one-meter coil of 0.5-mm-diam iron wire was measured at liquid helium temperature (4.2°K) and at 300°K. The electrical resistivity of this coil of wire at 20°C was 9.765 microhm-cm. Results of these measurements gave an electrical resistance ratio ($R_{300}/R_{4.2}$) of 60 and compares favorably with the values reported for 99.99 percent pure iron. A more recent report on high-purity zone-refined iron prepared at the Franklin Institute (3) gives a resistance ratio value of only 22.8.

For comparative purposes, a sample of reputedly 99.999 percent pure iron (zone-refined) procured from a commercial source was swaged and drawn into 0.5-mm-diam wire. The $R_{300}/R_{4.2}$ resistance ratio of this cold-worked wire was 33.7, and its resistivity at 20°C was 10.34 microhm-cm. Both coils of wire (with $R_{300}/R_{4.2}$ ratios of 60 and 33.7) were annealed at 2200°F (1200°C) for four hours and furnace cooled to eliminate the effects of cold working. The coils of wire were wrapped in tantalum foil and encapsulated for heat treatment in Type 304 stainless steel tubing that had been evacuated and back-filled with high-purity argon. Resistance ratios ($R_{300}/R_{4.2}$) of the two samples of annealed wire were 73.2 and 106, respectively. These final measurements are indicative of the higher purity of the 99.999 percent iron. It was observed however, that the arc-melted electrolytic iron wire had undergone a considerable grain growth during the high-temperature annealing. This fact precluded making resistivity measurements. Some grains were about 4 mm in length. This grain growth was not apparent in the 99.999 percent pure iron wire.

Thermoelectric Force

Determination of the thermoelectric force of this iron against a thermocouple grade of platinum at the temperature of 100°C gave a value of 1.90 mv with the cold junction at 0°C. This value is some 4 percent lower than that reported by Cleaves of the National Bureau of Standards (4). Similar measurements of the 99.999 percent versus platinum gave 1.95 mv for the thermoelectric force.

Magnetic Properties

Only those magnetic properties of pure iron that are greatly affected by impurities and by mechanical and thermal treatments were determined.

Test specimens were prepared by cutting rings (1-1/4 in. O.D. × 1 in. I.D. × 1/8 in. thick) from as-melted and cold-rolled plate. A low-temperature (1000°F, or 538°C) stress relief and a higher temperature (1600°F, or 871°C) vacuum annealing treatment were given to the specimens from the cold-rolled stock. Two coils consisting of 100 turns each of copper wire were wrapped on the iron ring specimens. Typical B-H magnetization curves, or hysteresis loops, were measured and are shown in Figs. 7 and 8. The B-values were adjusted to an assumed induction of 15,500 gauss, which is typical for high-purity iron. The hysteresis curve for the cold-worked iron is quite broad, as expected, and the low-temperature stress-relief treatment decreases the magnetic losses. Annealing at 1600°F decreases the magnetic losses still further and raises the residual induction by a factor of two. The area (hysteresis loss) of the large-grained, as-melted pure iron curve is comparable to the annealed specimen. However, there is a marked lowering of the residual induction in the as-melted specimen.

The coercive force (B_{max} about 15,500) for the annealed and as-melted samples was 0.1 oersted and is similar to the result obtained by Cleaves (4). This value could be lowered by long-time, high-temperature heat treatment. Cold working increases the coercive force

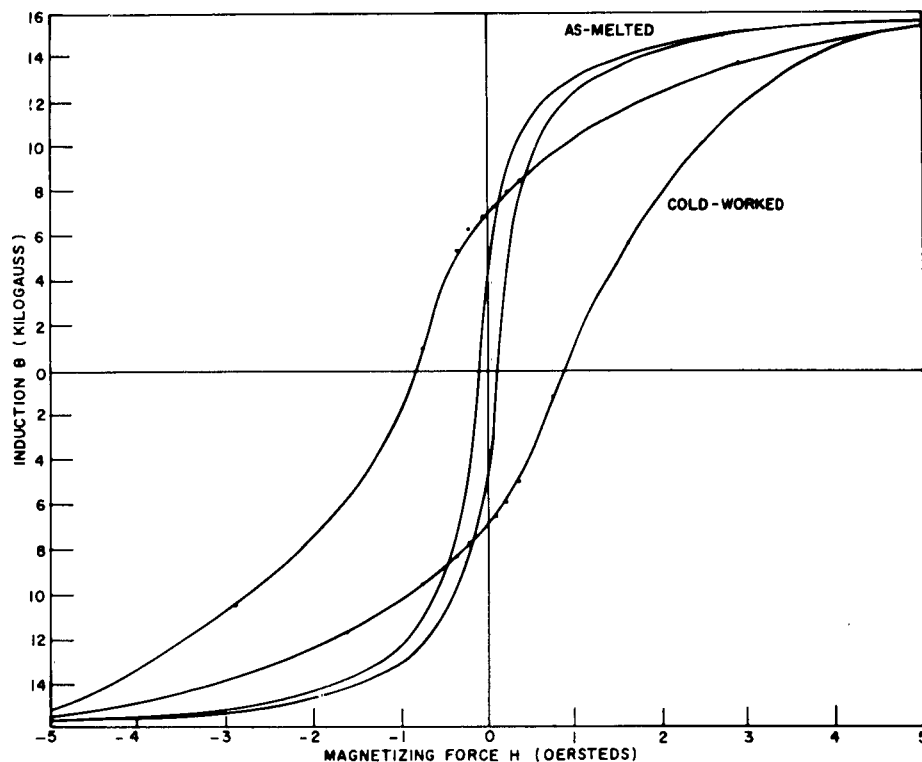


Fig. 7 - B-H magnetization curves for cold-worked and as-melted high-purity iron

to 0.85 oersted, and the stress-relief anneal at 1000°F lowers the coercive force to 0.35 oersted. Values of the coercive force and residual induction are listed in Table 4.

SUMMARY

1. It has been demonstrated that a relatively simple hydrogen reduction treatment of a commercial electrolytic iron can effectively lower its oxygen content to 50 ppm or less. This iron can easily be formed into an electrode by pressing and sintering, thus overcoming the difficulties of canning and the introduction of impurities from this source. Consumable-electrode, vacuum-arc-melting techniques provide additional refining and eliminate the crucible contamination usually associated with induction melting.

2. The large grain structure of the as-melted high-purity iron ingot can be reduced by careful cold forging. Once the initial stages of breakdown have been accomplished, the high-purity iron behaves in a very ductile manner.

3. Physical properties measurements show that the purity of the processed iron is very high. The anomalously low electrical resistivity of this cold-worked iron wire as compared to the 99.999 percent pure iron may be associated with its higher density; however, this requires further study. A resistance ratio ($R_{300}/R_{4.2}$) of 73 for the annealed wire is a good indication of the purity of this iron although it is lower than some research grades of high-purity zone-refined iron.

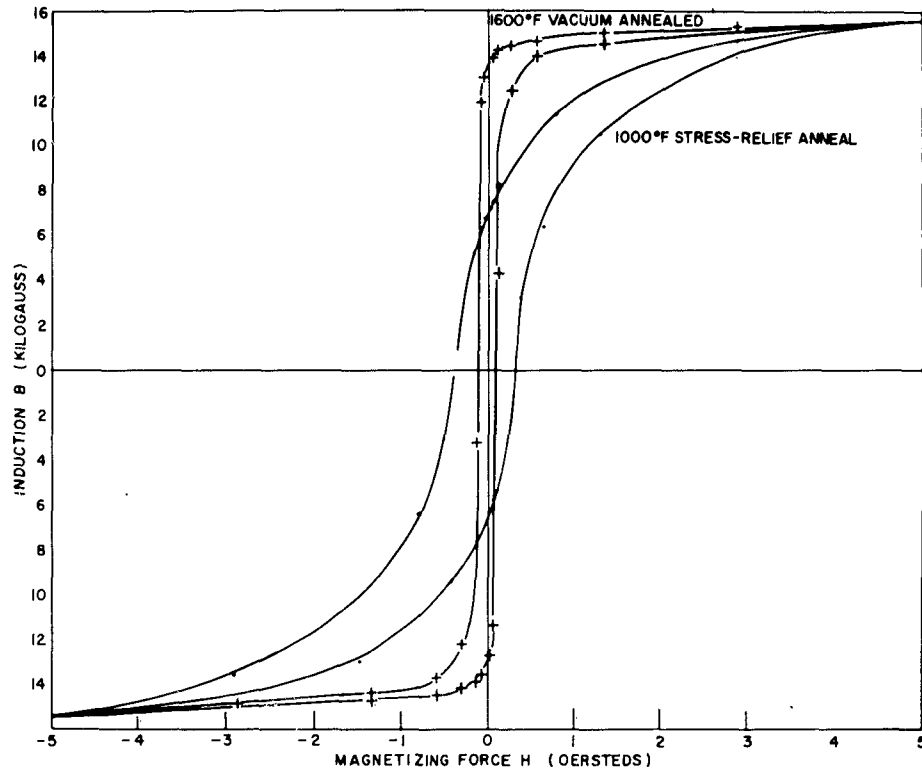


Fig. 8 - B-H magnetization curves for 1000° F and 1600° F annealed high-purity iron

Table 4
Magnetic Properties of High-Purity Iron
Subjected to Various Treatments

Measured Property	Type of Specimen			
	As-Melted	Cold-Worked	Annealed at 1000° F	Annealed at 1600° F
Coercive Force (oersteds), B_{max} about 15.5 gauss	0.1	0.85	0.35	0.1
Residual Induction (kilogauss)	5.5	6.7	6.7	13.4

The recrystallization and large-grain growth of this iron compared to that of the sample of 99.999 percent pure iron cannot be fully explained. However, it might be associated with a discontinuous or selective grain growth phenomenon. This type of grain growth is characterized by a final grain size that is dependent on the number of nuclei and not on the amount of a dispersed phase, nor on specimen size, as is the case with normal grain growth (5).

It must be emphasized that chemical analysis alone cannot be relied upon to characterize high-purity metals. Their physical and mechanical properties must also be determined and related to their processing history.

ACKNOWLEDGMENTS

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