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MAGNETIC AND OTHER PROPERTIES OF IRON-ALUMINUM ALLOYS MELTED IN VACUO

BY
TRYGVE D. YENSEN
AND
WALTER A. GATWARD

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ENGINEERING EXPERIMENT STATION,
URBANA, ILLINOIS
MAGNETIC AND OTHER PROPERTIES
OF IRON-ALUMINUM ALLOYS
MELTED IN VACUO

BY

TRYGVE D. YENSEN
RESEARCH ASSISTANT PROFESSOR OF ELECTRICAL ENGINEERING
AND

WALTER A. GATWARD
RESEARCH FELLOW IN THE ENGINEERING EXPERIMENT STATION
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MAGNETIC AND OTHER PROPERTIES OF IRON-ALUMINUM
ALLOYS MELTED IN VACUO

I. INTRODUCTION

It is the purpose of this bulletin to record the results of experiments to determine the magnetic and allied properties of iron-aluminum alloys melted in vacuo. Previous investigations to determine these properties of pure iron,* iron-boron alloys,† and iron-silicon alloys,‡ melted in vacuo, have been reported in earlier bulletins. Because of the similarity of many properties of aluminum and silicon, the same remarkable results were expected from the iron-aluminum as from the iron-silicon alloys. To some extent these have appeared. The results obtained, although not covering every phase of the subject, include the magnetic, electrical, and mechanical properties. Chemical analysis and photomicrographs are also presented for a number of the alloys.

The chemical analysis was in charge of Mr. J. M. Lindgren of the Chemistry Department; the methods used for the determination of aluminum are described by Mr. Lindgren in the Appendix. The photomicrographs were prepared by Mr. F. E. Rowland, also of the Chemistry Department. Mr. H. R. Fritz, Research Fellow in the Engineering Experiment Station, has rendered valuable service throughout the year as general assistant in connection with the investigation. Besides acknowledging their indebtedness to those persons who have been directly connected with the work, the authors also wish to express their appreciation to many others who have been of service to them. Among these it is particularly desired to mention Professor E. B. Paine, acting head of the Electrical Engineering Department.

Aluminum was first used in the industries in connection with copper to form aluminum bronzes, and it was this application of it that stimulated investigators to develop methods of manufacture capable of producing it at a cost which would warrant its use in large quantities. It is well known today that aluminum occurs in nature

only in the form of oxides and that it is one of the stablest oxides to be found, so stable that the only way of reducing it to its metallic state is by means of electrolysis. This fact early suggested its use for the purpose of reducing other oxides, and it was in the capacity of a reducer, deoxidizer, or degasifier; that it very early became the friend of the iron and steel maker. For this purpose aluminum soon became a serious competitor of silicon, in spite of the high cost of aluminum in its early days. Many were the wonderful qualities attributed to this new metal. A large number of these, however, existed only in the imagination of their advocates and were soon disproved.

Among the well-established properties of aluminum, the following are perhaps the most important: *

a. Aluminum when added to molten iron will reduce iron oxide to metallic iron. Furthermore, it will reduce CO or CO$_2$ gases to free carbon and thus degasify, quiet the bath, and prevent blowholes during solidification and cooling. If the iron is sufficiently liquid, the Al$_2$O$_3$ formed will pass to the surface; if it is not, the Al$_2$O$_3$ will become entangled in the iron, increase the viscosity, and cause the iron to be more or less unsound.

b. When added in sufficient quantities, 2 to 5 per cent, aluminum will cause carbon to be completely precipitated as graphite and thus transform, for example, white cast iron into gray iron. If 10 to 20 per cent aluminum is added, however, the carbon may all be in the combined form Fe$_3$C.

c. As the heat of combustion liberated by the oxidation of aluminum is much greater than that absorbed by the reduction of iron oxides, clearly illustrated by the use of thermite in welding iron, the addition of aluminum to oxidized iron or steel causes a rise in the temperature of the bath. This fact, and not any appreciable lowering of the melting point, is the cause of the higher fluidity noticed when a small percentage of aluminum is added to the bath.†

The metallographic investigation of iron-aluminum alloys was first systematically undertaken by the Alloys Research Committee


of the Institution of Mechanical Engineers,\textsuperscript{*} and later, more extensively, by Gwyer.\textsuperscript{t} The equilibrium diagram according to the latter is shown in Fig. 1. This diagram shows that iron and aluminum, when liquid, dissolve in each other in any proportion. Up to an aluminum content of 50 per cent mixed crystals are at first formed upon solidification, but as the solid solution becomes saturated with

an aluminum content of 34 per cent, any excess above this amount will
be precipitated as solidification proceeds. Alloys containing less than
34 per cent aluminum will suffer no precipitation of any constituent
during cooling and, therefore, at ordinary temperatures will consist
of mixed crystals of iron and aluminum. The lowering of the melting
point of iron due to small percentages of aluminum is very slight.
Even for a 10 per cent aluminum content the melting point is lowered
only about 10 degrees. This shows conclusively that the increased
fluidity of iron, previously referred to, which is obtained by adding
less than 0.1 per cent aluminum, can not be due to the lowering of the
melting point. With an aluminum content of 59 per cent, the com-
 pound FeAl_3 crystallizes out during solidification and leaves behind
liquid aluminum, which freezes at 653 degrees. The theory suggested
by previous writers, that the liberation of heat noticed upon adding
a small amount of aluminum to molten iron might be due to the forma-
tion of compounds of iron and aluminum,* is, therefore, at variance
with the evidence just presented. The only explanation of the
described phenomenon which has stood the test of investigation is that
the heat liberated is due to the formation of Al_2O_3.

From these investigations it appears that within the region
studied in the present research, namely 0 to 10 per cent aluminum,
the alloys all consist of mixed crystals of iron and aluminum, just as
the iron-silicon alloys described in Bulletin 83 all consisted of mixed
crystals of iron and silicon. The photomicrographs of Gwyer, Guillet,
and Hadfield confirm the deductions just stated.

The mechanical properties of iron-aluminum alloys have been
studied by Hadfield,† Styffe,‡ and Guillet,§ who all agree that
aluminum in quantities up to 5 per cent has very little influence upon
the mechanical properties of iron. When more than 5 per cent is
used, however, the alloys are slightly stronger in tension, but become
brittle. Hadfield, whose alloys contained about 0.2 per cent carbon,
found the limit of forgeability between 5.6 and 9.14 per cent aluminum
content, while Guillet found the limit between 7.18 and 9.25 per cent
for alloys containing 0.8 per cent carbon. The results by these three
investigators are shown graphically in Fig. 6.

The effect of aluminum on the magnetic properties of iron was

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vol. 70, II, p. 16, 1896.
studied to some extent in the early nineties* after the beneficial effect of this element as a purifier of iron and steel had been definitely established. It remained for Hadfield,† however, to make the first iron-aluminum alloys which had better magnetic qualities than those of the purest iron obtainable at the time. His 2.25 and 5.5 per cent aluminum alloys as tested by Barrett showed a higher permeability and a lower hysteresis loss than did the purest Swedish charcoal iron (see Table 1).

**Table 1**

**Magnetic and Electrical Properties of Hadfield's Iron-Aluminum Alloys**

<table>
<thead>
<tr>
<th>Mark</th>
<th>Al</th>
<th>Max. Permeability</th>
<th>Max. Induction for H=8</th>
<th>Retentivity in Terms of B</th>
<th>Coercive Force in Terms of H</th>
<th>Hysteresis loss Ergs per c.c. per cycle</th>
<th>for H=45</th>
<th>for B=9000</th>
<th>Specific Electrical Resistance Microhms</th>
</tr>
</thead>
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<tr>
<td>S.C.I.</td>
<td>.00</td>
<td>4000</td>
<td>1700</td>
<td>10500</td>
<td>1.10</td>
<td>abt. 8500</td>
<td>2334</td>
<td>10.2</td>
<td></td>
</tr>
<tr>
<td>B</td>
<td>.00</td>
<td>1600</td>
<td>6000</td>
<td>9770</td>
<td>1.00</td>
<td>11000</td>
<td>22.0</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1167 D</td>
<td>.75</td>
<td>1500</td>
<td>160000</td>
<td>10500</td>
<td>1.80</td>
<td>11000</td>
<td>22.0</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1167 H</td>
<td>2.25</td>
<td>1700</td>
<td>16900</td>
<td>10500</td>
<td>1.00</td>
<td>8000</td>
<td>39.0</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1167 I</td>
<td>3.30</td>
<td>1200</td>
<td>13000</td>
<td>4150</td>
<td>1.00</td>
<td>6500</td>
<td>70.0</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

From the results shown in the table and those previously obtained with silicon,‡ aluminum and silicon appear to have very similar effects upon the magnetic properties of iron.

Dillner and Engstrom§ have investigated the effect of aluminum and silicon on the magnetic properties of sheet iron as well as of castings. They found that silicon reduced the permeability and increased the hysteresis loss in sheets, but not in castings, and that aluminum had the opposite effect. Furthermore, it was found that a combination of silicon and aluminum gave the best results for sheets.

The main objection to employing aluminum formerly was its high cost. While silicon, in the form of ferrosilicon, could be obtained at a cost of 12 cents per pound of the element, the cost of aluminum was nearly $2 per pound. On this account silicon was employed in making steel for magnetic purposes, and, having given universal satisfaction both with regard to the hysteresis loss and to aging, it has retained its place. In spite of the reduced cost of aluminum, there

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has appeared to be no decided advantage in changing from silicon steel to aluminum steel. Investigators have turned their attention toward improving and perfecting silicon steel while comparatively little investigational work has been recorded regarding iron-aluminum alloys, particularly regarding their magnetic properties. It is hoped, therefore, that the present investigation will prove to be of interest not only on account of the vacuum method employed, but also because it is one of the few systematic investigations on the subject.

II. MATERIAL, APPARATUS, AND METHODS *

The iron used as the basis of the investigation was doubly refined electrolytic iron containing 0.01 per cent carbon or less and about 0.01 per cent silicon. It was cleaned with HCl, distilled water, and alcohol, and then dried by means of ether in vacuo.

The aluminum used as the alloying material analyzed as follows:

\[
\begin{array}{lcccc}
\text{Si} & \text{Fe} & \text{C} & \text{Al (by diff.)} \\
\text{..........} & \text{..........} & \text{..........} & \text{..........} \\
0.20 \text{ per cent} & 0.17 \text{ “ “} & \text{nil} & 99.63 \text{ “ “} \\
\end{array}
\]

The melting was done in magnesia crucibles in an Arsem type vacuum furnace capable of melting 600 grams of iron; the pressure was less than 1 mm. of mercury. Attempts were first made to melt the iron and the aluminum together, but this method had to be given up for two reasons. First, the melting point of aluminum is so low compared with that of iron, that a considerable portion of aluminum evaporated before the iron was in a condition to combine with it. Furthermore, the aluminum vapor interfered with the operation of the furnace in such way that the voltage could not be maintained at a sufficiently high value to melt the iron completely. The result was that the Al\(_2\)O\(_3\) formed, instead of rising to the surface, became entangled in the iron, often completely enclosing pieces of iron and resulting in imperfect ingots. Second, as will be shown, on account of the power of aluminum at high temperatures to reduce CO gas, this method of melting was certain to introduce more or less

carbon into the alloys. It became necessary, therefore, first to melt the iron and then, towards the end of the operation, to drop in the aluminum. This was accomplished without opening the furnace by suspending the aluminum (in the form of a wire or rod) from a very fine wire extended between insulated binding posts which passed through the cover of the furnace. In order to drop the aluminum, the fine wire was fused by connecting the binding posts for a moment to the terminals of the furnace. This method made it necessary to use a cover with a hole in it over the crucible, together with a funnel to direct the rod into the crucible.

It has already been mentioned as a well established fact that aluminum at high temperatures, when added to molten iron for instance, will reduce CO gas and thus liberate free carbon. That it will do so even at lower temperatures has been shown by Stead,* and in the present investigation this has been further confirmed.

It is evident that whatever gases remain in the vacuum furnace must consist largely of carbon monoxide. During the first trial experiments with the aluminum suspended from the top of the furnace, it was noticed, upon examining the aluminum after the operation, that the lower end of the wire was coated with a grayish substance. This substance was evidently the mixture of carbon and Al$_2$O$_3$ mentioned by Stead. The lower the wire reached, and consequently, the hotter it became, the larger the portion of it which became coated. In order to prevent the wire from becoming hot, it was made as short as possible and, during the later part of the investigation, was protected from direct radiation by means of an iron tube extending down into the furnace. But even with these precautions the tip of the wire or rod was always slightly coated, particularly when more than 5 per cent aluminum was used. Moreover, when the aluminum was dropped in, it obviously became hot and may have had time in a few cases to reduce some CO. In these cases a small amount of carbon was invariably introduced into the alloy, generally not exceeding 0.02 per cent, but in some cases, especially during the early part of the investigation, amounting to 0.10 per cent or even more. One alloy analyzed as high as 0.65 per cent carbon.

It is quite evident from this discussion that in the type of furnace used it would be out of the question to mix the iron and the aluminum, melt them together, and expect to obtain an alloy with

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a low carbon content. At first this method was used without bad effects for very low percentages of aluminum when the iron oxide present evidently was sufficient to oxidize both the aluminum and the carbon, but the method was abandoned for aluminum contents above 0.5 per cent, for the reasons already stated.

The alloys known or suspected to contain more than 0.015 per cent carbon for low aluminum alloys and more than 0.025 per cent carbon for high aluminum alloys have been tabulated separately in order to show the effect of carbon upon the various properties. It appears that the methods employed were not ideal, and that small amounts of carbon may have been introduced with the aluminum. It is possible, therefore, that better results, magnetically, might have been secured had a furnace been employed, having, for instance, a tungsten heating element.

The ingots were allowed to cool while in vacuo, and then were forged into rods and machined into test pieces.

The magnetic testing was done by means of the Burrows compensated double bar and yoke method. It has been previously shown * that the errors made by using this method for very high permeability iron may be much greater than theoretical considerations would indicate, but no corrections have been made in the present case, it being desired that the results given in this bulletin be comparable with those given in the previous bulletin on iron-silicon alloys. Furthermore, the question of corrections is a complicated one and has by no means been definitely settled, for the amount of compensating current needed to equalize the flux along the rod depends largely upon the uniformity of the rods used. This question is further discussed in the Appendix. The Appendix contains also additional data obtained from ring specimens, showing that for permeabilities not exceeding 20,000, the results obtained with the Burrows method may be relied upon as being very close to the correct values.†

The tests for electrical resistance were made by means of a Kelvin double bridge, using standardized iron rods for comparison.‡ For the mechanical testing an Olsen 10,000 pound testing machine was employed, and the usual characteristics were obtained. The rods

‡The standardization was done by the Leeds and Northrup Co.
were tested magnetically and electrically after the following heat treatments:

a. As forged
b. Annealed at 900 degrees C. Cooled at the rate of 30 degrees per hour
c. Annealed at 1,100 degrees C. Cooled at the rate of 30 degrees per hour

Mechanical tests were made on unannealed specimens and on specimens heated to 1,000 degrees C. and then cooled at the rate of 30 degrees C. per hour. The heat treatment was applied in vacuo in order to prevent oxidation, a vacuum of 0.2 to 0.1 mm. being maintained during the treatment. The specimens were packed in powdered magnesia in an electroquartz tube.

III. CHEMICAL ANALYSIS

The marked effect of aluminum and silicon upon the electrical resistance of iron is well known. One per cent of either element added to iron will more than double its electrical resistance, and the increase is nearly proportional to the amount added. Furthermore, the resistance is not appreciably affected by mechanical or thermal treatments. These facts furnish an excellent opportunity for establishing the aluminum content of iron which contains only small amounts of impurities. This opportunity has been used to advantage in the present case. A few alloys were selected the aluminum content of which, as determined approximately by the aluminum added to the iron, covered the range studied. After the magnetic and electrical tests on these alloys were completed, the test rods were placed in a lathe, the surface layer was removed, and fine shavings were collected from along the rods in sufficient quantities for duplicate chemical analysis.

The results of this analysis * are given in Table 2, Column 3; from these figures and those for the electrical resistance, given in Column 5, the curve in Fig. 2 showing the relation between the resistance and the aluminum content was established. The aluminum content of all the other alloys for which electrical resistance measurements could be made, was then determined by means of the curve in Fig. 2. The values thus obtained have been checked by further analysis and found to be within the limits of accuracy desired.

*The method used is described by Mr. J. M. Lindgren in the Appendix.
It was stated in the previous chapter that, due to the conditions of melting and to the reduction of CO by aluminum, a large number of the alloys contained carbon. Therefore, a careful weeding of the alloys was made.

<table>
<thead>
<tr>
<th>No.</th>
<th>Aluminum Content</th>
<th>Carbon Content</th>
<th>Specific Resist. Curve</th>
<th>Remarks</th>
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<tr>
<td></td>
<td>Per Cent As Charged</td>
<td>Per Cent by Chem. Analysis</td>
<td>Annealed at 1100° C. Microhms</td>
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<tr>
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The alloys obtained by mixing the aluminum with the iron before melting includes Nos. 3 Al 05-17. Of these, only Nos. Al 05-08 were retained as being uncontaminated; the ingots for Alloys Nos. 3 Al 13-17 were decidedly unsound. Those in the next series, 3 Al 18-29, inclusive, were prepared by dropping the aluminum into the molten iron. Most of the alloys of this series contain large amounts of carbon, for the aluminum was not protected from undue heating.
Furthermore, when a large piece of aluminum was dropped into the crucible, such agitation resulted that some of the molten iron undoubtedly came in contact with the small graphite funnel, which was used to guide the aluminum into the crucible. Finally, during the melting of the Alloys 3 Al 26, 28, and 29, the MgO crucible cracked and the iron came in contact with the graphite container. In the last series, 3 Al 30-51, inclusive, an alundum or magnesia funnel was used, and the aluminum was protected from undue heating by means of an iron tube. In this series, 3 Al 34-39, inclusive, were classed as contaminated because the aluminum content of the iron was, for
### Table 3
RESULTS OF MECHANICAL TESTS. As FORGED

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**CONTAMINATED ALLOYS**

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Broke outside punch marks.

Broke outside punch marks.

Broke outside punch marks.
**TABLE 4**

RESULTS OF MECHANICAL TESTS. ANNEALED AT 1000 DEGREES C.

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CONTAMINATED ALLOYS

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Broke outside punch mark.
Fig. 3. Mechanical Properties of Iron-Aluminum Alloys Melted in Vacuo. As Forged
FIG. 4. MECHANICAL PROPERTIES OF IRON-ALUMINUM ALLOYS MELTED IN VACUO. ANNEALED AT 1000 DEGREES C.
some unaccountable reason, too low on comparison with the aluminum added. If the carbon introduced is in proportion to the aluminum added, then the carbon content of these alloys was quite out of proportion to the aluminum content.

As a check on this weeding, a number of these alloys were analyzed for carbon; the results of this analysis are shown in Column 4 of Table 2. For low aluminum alloys a carbon content of 0.015 per cent has been allowed; for high aluminum alloys, 0.025 per cent.
IV. Results

1. Mechanical Properties.—The results of the mechanical tests are shown in Tables 3 and 4 and in Figs. 3, 4, 7, and 8. For the
Fig. 7. Mechanical Properties of Iron-Aluminum Alloys Melted in Vacuo, More or Less Contaminated, Compared with Uncontaminated Alloys. As Forged
FIG. 8.  MECHANICAL PROPERTIES OF IRON-ALUMINUM ALLOYS MELTED IN VACUO, MORE OR LESS CONTAMINATED. COMPARED WITH UNCONTAMINATED ALLOYS. ANNEALED AT 1000 DEGREES C.
sake of comparison, the mechanical properties of the corresponding iron-silicon alloys are shown in Fig. 5.* In Fig. 6 the results of the present investigation are compared with those obtained by Hadfield† and Guillet.§ Fig. 9 is a photograph of some of the mechanical test pieces after testing.

From the data thus presented, it may be stated in general that aluminum in the absence of carbon increases the strength of iron in almost direct proportion to the amount added. Furthermore, aluminum appears to affect the toughness of iron only slightly. To what aluminum content these rules can be applied is somewhat uncertain, as no alloy containing more than about 6 per cent aluminum uncontaminated by carbon was obtained. How carbon affects these properties may be judged from Figs. 7 and 8. It is apparent that for alloys containing in the neighborhood of 0.1 per cent carbon, aluminum, up to at least 8 per cent, has no marked effect either upon the strength or upon the toughness of iron. This is especially evident in the case of the annealed alloys. The effect of small amounts of carbon is particularly great upon nearly pure iron; it increases the strength of iron about 50 per cent. The strength curves for alloys containing small amounts of carbon will, consequently, be nearly horizontal, and will cross the curves for the more nearly carbon-free alloys at 4 to 5 per cent aluminum, where the effect of carbon seems to be very small. In general the effect of small amounts of carbon is to conceal the true effect of aluminum upon the mechanical properties of iron. If the results given here for alloys with a carbon content of about 0.1 per cent are compared with those given by Hadfield and Guillet (see Fig. 6), they will be found to be very similar. They are more like Guillet's results, which represent alloys containing about 0.1 per cent carbon, than Hadfield's, whose alloys contained about 0.2 per cent. For carbon contents of 0.4 to 0.6 per cent the alloys are forgeable up to at least 13 per cent aluminum. This also agrees with the results of Guillet. For such carbon contents, however, the alloys are quite brittle.

If the effect of aluminum (Fig. 4) upon the mechanical properties is compared with the effect of silicon (Fig. 5), it will be seen that, up to 4.5 per cent, silicon increases the strength much more than does aluminum. On the other hand, although 2.5 to 4.5 per cent silicon

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Fig. 9. Some of the mechanical specimens after being tested
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markedly increases the brittleness of iron, aluminum has no such effect. Furthermore, silicon beyond 4.5 per cent rapidly decreases both the strength and the toughness, while aluminum continues to add strength to the iron without materially affecting the toughness.

2. Magnetic and Electrical Properties.—The results of the magnetic and electrical test are shown in Tables 5 to 7 and in Figs.

### Table 5

**RESULTS OF MAGNETIC AND ELECTRICAL TESTS**

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PROPERTIES OF IRON-ALUMINUM ALLOYS MELTED IN VACUO

TABLE 7
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10, 11, 12, 14, and 15. In Fig. 13 the properties of the iron-silicon alloys annealed at 1100 degrees are given for comparison with the corresponding iron-aluminum alloys.

The specific electrical resistance unaffected by any heat treatment, which is about 9.8 microhms for pure iron, is seen to increase by 12 microhms for each per cent aluminum added up to an aluminum content of 3 per cent. For higher values the increase falls off gradually.

Although the electrical resistance is unaffected by heat treatments, the magnetic properties, as is well known, are extremely sensitive to them. From previous investigations on this subject, it seems probable that the factor which determines the magnetic properties of a certain iron or iron alloy is the internal strain existing in the metal, and that it is the removal of this strain by annealing and slow cooling that is the real cause of any increase in permeability and decrease in hysteresis loss. The transformation of all the β- and γ-iron into α-iron is undoubtedly essential; but in dealing with pure iron, or with iron-aluminum or iron-silicon alloys, this transformation is supposed to take place quite readily, and does not necessitate as slow cooling as 30 degrees per hour. Almost any rate of cooling, short of quenching, seems to effect this transformation, though it has been shown repeatedly that as far as the magnetic properties are concerned, a cooling at the rate of 100 degrees per hour is much inferior to a rate of 30 degrees. A slower rate than 30 degrees per hour does not appear to be of any further advantage. Again, although the transformation from γ- to β-iron (if the β modification actually does exist) takes place at 900 degrees for pure iron, it has been shown that the most noticeable increase in permeability for low and medium densities takes place upon annealing at between 700 and 800 degrees C. A further, but much smaller, increase takes place upon annealing at 900 degrees. Finally, it was shown in Bulletin No. 83, and it is again shown here, that slow cooling from 1100 degrees produces a higher maximum permeability and a lower hysteresis loss than cooling from slightly above 900 degrees. These facts all favor the theory that, for the kind of iron dealt with in this series of investigations, it is the removal of internal strain from the iron which is of importance in

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†"Magnetic and Other Properties of Electrolytic Iron Melted in Vacuo." Univ. of Ill. Eng. Exp. Sta., Bul. 72, p. 30, Fig. 3, 1914.
Fig. 10. Magnetic and Electrical Properties of Iron-Aluminum Alloys Melted in Vacuo. As Forged
Fig. 11. Magnetic and Electrical Properties of Iron-Aluminum Alloys Melted in Vacuo. Annealed at 900 Degrees C.
Fig. 12. Magnetic and Electrical Properties of Iron-Aluminum Alloys Melted in Vacuo. Annealed at 1100 Degrees C.
FIG. 13. MAGNETIC AND ELECTRICAL PROPERTIES OF IRON-SILICON ALLOYS MELTED IN VACUO. ANNEALED AT 1100 DEGREES C. (FROM BUL. 83)
FIG. 14. FLUX DENSITY FOR VARIOUS MAGNETIZING FORCES
FIG. 15. MAGNETIC PROPERTIES OF IRON-ALUMINUM ALLOYS MELTED IN VACUO, MORE OR LESS CONTAMINATED, COMPARED WITH UNCONTAMINATED ALLOYS. ANNEALED AT 1100 DEGREES C.
the production of high permeability, low hysteresis iron. The transformation of all the iron into α-iron is then a foregone conclusion.

Turning now to Fig. 11, it is seen that the maximum permeability for the unannealed alloys varies from 3500 for pure iron to 600 for a 6 per cent alloy. After the alloys have been annealed at 900 degrees these values (See Fig. 12) increase to 24,000 and 13,000, respectively. The annealing at 1100 degrees (Fig. 13), although not materially affecting the pure iron, raised the maximum permeability of the 6 per cent alloy to 17,000 and that of the 0.4 per cent alloy to about 40,000. The maximum permeability is thus increased ten to thirty times, and the permeability for \( B = 15,000 \), two to ten times by annealing at 900 degrees. No further improvement takes place by annealing at higher temperatures: in fact, alloys low in aluminum show an actual decrease in annealing at 1100 degrees (see Fig. 14). In regard to the latter point, it has generally been found that if pure iron previously annealed at 900 degrees is annealed at a higher temperature, it decreases in permeability for values of \( H \) between 20 and 100 gilberts per cm. For \( H = 200 \) (Fig. 14) the permeability is the same for the 900- and 1100-degree annealing; the improvement due to annealing amounts to from 1 to 2 per cent. The saturation value is known to be practically unaffected by annealing.*

Annealing lowers the coercive force and the hysteresis loss in much the same ratio as it raises the maximum permeability, the minimum values occurring with an aluminum content of about 0.25 per cent.

When the iron-aluminum and the iron-silicon alloys (Figs. 12 and 13) are compared, the curves, as might be expected, are similar for the low alloys; both have a maximum in the maximum permeability curve and a minimum in the coercive force and hysteresis curves for about 0.2 per cent. The two sets of curves are, however, distinctly different for high alloys. The silicon curves have a second maximum or minimum for from 3.5 to 4 per cent silicon, but the aluminum curves are without such points, the maximum permeability gradually decreasing and the hysteresis loss and coercive force gradually increasing from the 0.5 per cent aluminum point.

Although the iron-aluminum series thus furnishes a high permeability, low hysteresis alloy (similar to the low silicon alloy), it furnishes none which combines these properties with a high electrical resistance in as marked a degree as does the 3.5 per cent iron-silicon

alloy. Nevertheless, the 3.5 per cent iron-aluminum vacuum alloy has a maximum permeability of six times, and a hysteresis loss of only one-half, that of the commercial 3.5 per cent silicon steel. It has been shown, furthermore, that the high aluminum alloys have the advantage over the corresponding silicon alloys of being much tougher mechanically; thus they are more easily worked and lend themselves to some purposes where the silicon alloys are unsuitable on account of their comparative brittleness. The effect of carbon on the magnetic properties is illustrated in Fig. 15, where the maximum permeability, retentivity, coercive force, and hysteresis loss are shown for a number of alloys containing from 0.02 to 0.16 per cent carbon, and are compared with the uncontaminated alloys. The bars have all received the same mechanical and thermal treatment, finally being annealed at 1100 degrees. The maximum permeability attained by the alloys containing about 0.10 per cent carbon is about 5000 as compared with 20,000 for the uncontaminated alloys. The retentivity for these same alloys is much lower, and the coercive force, except in one case, much higher than for the purer alloys. The hysteresis loss is from 50 to 100 per cent and, for the low alloys containing 0.05 per cent carbon, even 200 per cent higher than for the corresponding uncontaminated alloys.

3. Photomicrographs.—Photomicrographs of a number of the alloys representative of the iron-aluminum series are shown in Figs. 16 and 17. Fig. 16 shows the uncontaminated alloys and Fig. 17 some of the contaminated ones. On the left hand side are shown the alloys as forged, and on the right the same alloys annealed at 1100 degrees C.

It is clearly seen from these figures that iron and aluminum form a solid solution as has also been shown by previous investigators. In the forged specimens the structure varies greatly, the size of the crystals generally decreasing as the aluminum content increases. The structure of 3 Al 21 is the only exception to this rule, the crystals in this case being unusually large. After the alloys have been annealed at 1100 degrees, this nonuniformity disappears and the alloys exhibit crystals of approximately the same size. For the pure iron, or for the very low alloys, 3 Al 08, 05, and 32, the structure consists, as is usual, of large crystals subdivided into many small ones.*

FIG. 16. PHOTOMICROGRAPHS OF IRON-ALUMINUM ALLOYS, AS FORGED AND ANNEALED AT 1100 DEGREES C. X 60
Figure 17. Photomicrographs of some of the contaminated Iron-Aluminum Alloys, as Forged and Annealed at 1100 Degrees C. × 60
For aluminum contents of 0.40 per cent and above, this subdivided structure does not appear, and the remainder of the alloys are made up of more or less regular crystals.

In the structure of the uncontaminated alloys there is no sign of impurities present; the spots that appear are evidently due to imperfect polishing. In the contaminated specimens the presence of graphite is evident only in the forged specimen of 3 Al 25, containing 0.16 per cent carbon. After annealing at 1100 degrees this evidence of graphite entirely disappeared. What becomes of the carbon in this case has not been ascertained. According to previous investigators carbon in the presence of 4.5 per cent aluminum should be completely precipitated as graphite; but, from the appearance of 3 Al 25 annealed, one is led to believe that the carbon has combined with the iron and aluminum, unless it is precipitated in such finely divided state that it is invisible under the magnification used. It is hoped that this point may be cleared by further investigation.

V. SUMMARY AND CONCLUSIONS

The results recorded in the previous pages may be summarized as follows:

a. The iron-aluminum alloys used in this investigation, prepared by the vacuum method, are less contaminated by impurities than alloys used by previous investigators. The alloys classed as uncontaminated contain only 0.01 to 0.02 per cent carbon. Other alloys containing more carbon are classed as contaminated and are used to show the effects of carbon.

b. Aluminum is more powerful as a deoxidizer than is silicon, for it does not commence to combine with iron until all oxides present are reduced. Aluminum forms a solid solution with iron throughout the range studied.

c. The tensile strength of the vacuum alloys increases in direct proportion to the aluminum content up to at least 6 per cent, the ultimate strength of the latter being 85,000 pounds per square inch (60 kg. per sq. mm.) in the unannealed state and 70,000 pounds (50 kg.) in the annealed. The corresponding figures for pure iron are 48,500 pounds (34 kg.) and 35,000 pounds (25 kg.). The toughness is only slightly affected by the aluminum content.

d. With regard to the magnetic properties aluminum, like silicon, has a very beneficial effect when added in small quantities. The
best alloy obtained, containing about 0.40 per cent aluminum annealed at 1100 degrees, has a maximum permeability above 35,000. The hysteresis loss for $B_{\text{max}} = 10,000$ and 15,000 is 450 and 1000 ergs per cc. per cycle, respectively. For higher aluminum contents the magnetic quality decreases gradually, so that the alloy containing 3.5 per cent aluminum has a maximum permeability of 20,000 and a hysteresis loss for the given densities of 1000 and 2200 ergs, respectively. This loss is only one-half that of the 3.5 per cent commercial silicon steel.

e. The specific electrical resistance increases about twelve microhms for each per cent aluminum added. When the aluminum content exceeds 3 per cent, however, the rate of increase falls off gradually.

By the application of the vacuum process to the iron-aluminum series, an alloy, containing about 0.4 per cent aluminum, has been produced which has remarkable magnetic properties. In this respect aluminum acts like silicon. Aluminum, however, unlike silicon, yields no alloy with similar characteristics for higher percentages. On the other hand, the high aluminum alloys have the advantage over the corresponding silicon alloys of being much less brittle; and this characteristic combined with a fairly high permeability, low hysteresis loss, and an electrical resistance equal to that of the silicon alloys, may make aluminum alloys suitable for certain purposes where the silicon alloys can not be used.
APPENDIX

4. Magnetic Testing with Burrow’s Permeameter.—During the past two years the attainment of permeabilities above 30,000 in vacuum-fused iron-silicon alloys has made the question of accuracy in results of prime importance. There are three effects which may lead to errors: (a) effect of strain, (b) end effect of the permeameter coils, (c) consequent pole effect due to nonuniformity.

a. The effect of strain has been considered in a previous bulletin.* When the rod is properly clamped, this effect is eliminated.

b. The effect upon $H$ of the ends of the various permeameter coils has also been discussed in a previous bulletin.† The Bureau of Standards’ correction of the Burrows method for the theoretical end effect, when the compensating current is not more than twice the current in the two main solenoids, is less than 0.1 per cent. In Bulletin No. 83 of the Engineering Experiment Station of the University of Illinois, it is shown that in some cases the current in the compensating circuit may be thirty times that in the main solenoid. In such cases the correction factor is 2.3 per cent; that is, the measured $H$ must be increased by 2.3 per cent.

c. The effect of nonuniformity of the specimen is one for which no correction can be offered. It is of the same nature as the effect due to the ends of the coils, but it depends only upon the homogeneity of the rod, and, therefore, the error introduced cannot be calculated. Any nonuniformity means that lines of force will leave the iron path and complete all or part of the magnetic circuit in air. Wherever a line of force leaves or enters the iron path, a pole is developed. A line that leaves and re-enters the iron causes two poles of opposite sign. The effect of each pole at the center of the test bar varies inversely as the square of the distance from the center. Hence each pair of poles due to a leakage line has a resultant effect at the center, which depends upon the distance from the center of the points of leaving and of entering the iron. This may be illustrated by the leakage that occurs at the joint of the bar and the yoke. If the lines

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of force leave the rod close to the joint and enter the yoke just across the air gap, the resultant effect on the center of the bar will be small for two reasons: (a) the distance from the center of the resultant pole is great, and (b) the two poles tend to neutralize each other. On the other hand, if the leakage is close to the center of the bar, and the distance is great between the points of leaving the iron and re-entering it, the magnetizing or demagnetizing effect may be great. Leakage of this kind leads to a high percentage of error and is caused by the nonuniformity of the iron. A section of the iron having low reluctance is called a soft spot; a section having high reluctance is called a hard spot.

In order to investigate the uniformity of rods, a special permeameter was constructed having movable test coils on the outside of the main solenoids. There was only one layer of winding on the solenoids and the maximum value of \( H \) obtainable without heating the coils was 60 gilberts per cm. By properly connecting the test coils it was possible to investigate the density at points along the bars and to calculate the amount of leakage at any point at any density up to that corresponding to \( H = 60 \).

Fig. 18 shows the results obtained with Rod No. 3 Si 27C. For each value of \( H \) the leakage along the bar was measured and plotted with the density at the center as the reference point. It should be noted that the diameter of this rod varies as much as 1 per cent, and that at densities above 15,000 the distribution of flux follows the variations in diameter. At lower densities, however, the uniformity of the material has the greater effect, because the iron is more sensitive at densities falling on the steep part of the magnetization curve. From Fig. 18 it can be seen that the assumption that compensation neutralizes only the demagnetizing effect of the leakage at the joints is entirely without justification. In this case a strong magnetizing effect is introduced because of a soft spot about four inches from the center of the rod. If the test coils marked c-c, which are wound over each end of the test bar, were placed three inches from the center they would indicate that a negative current was required for compensation. Such a current would increase the leakage at the joints and would plainly lead to error. If the coils c-c were placed five and one-half inches from the center or close to the yokes, they would then indicate that a positive current was required. However, in raising the ends of the leakage curve, the flux at the point \( +4 \) would be raised still higher.
than for the uncompensated case and the effect would be to increase $H$ at the center beyond its indicated value. When the two coils, c-c, are connected in series and opposed to the test coil at the center, a balance indicates that the average flux at the two ends is equal to that at the center, even though the flux at one end may be much higher than that at the center and the flux at the other end correspondingly lower. Since when the c-c coils were placed half way between the center and the ends, a negative compensating current was sometimes required, it was decided preferable to place them nearer the ends.

Fig. 19 shows the effect of compensation on the value of $H$ at the center, even though the flux at one end may be much higher than that at the center and the flux at the other end correspondingly lower.
FIG. 10. CURVES SHOWING EFFECT OF COMPENSATION ON FLUX DISTRIBUTION
center for Rod 3 Si 40C. The diameter of this rod is very uniform and the nonuniform flux distribution is due to soft spots near the ends. Two conditions are shown for two values of $H$. In (a) $H$ is 0.5, and in (b) $H$ is 2.0. The test coils were connected so that the flux at either end of the test bar could be balanced against the center. The solid curve in both figures shows the distribution when the rod is uncompensated. Since the leakage is less for the negative than for the positive end, a smaller compensating current was required at the negative end in order to raise the flux to an equality with the center. The two ends were balanced separately, and the distribution for the two cases is shown by the two dashed curves. Compensation for the positive and negative ends raised the density at the center from 7,870 to 14,280 and 13,680, respectively, for $H = 0.5$; and to 14,280 and 14,580, respectively, for $H = 2.0$. An increase was desired after the leakage at the joints had been neutralized, but there was no way of knowing how much magnetizing effect the "soft spot" had on the center. The density at the center is probably exaggerated, especially for $H = 0.5$.

The double bar and yoke method is based upon the assumption that the rods are uniform. If the rods are uniform, when the c-c coils are placed about half way between the center and the ends, a balance of the average of the flux between these points and the center will result. There may still be some leakage near the joints, but for reasons stated previously its effect is small. As shown in Fig. 18, the distribution can not be assumed to be uniform, and so results by this method are likely to be inconsistent. Nonuniformity is most troublesome in rods of high permeability; with rods of ordinary quality this effect is not so important. For commercial work where only commercial iron is tested, this method is probably sufficiently accurate; but for laboratory testing of high permeability iron, the limiting accuracy should be carefully considered.

5. Results Obtained with Rings.—In Table 8 will be found results obtained with rings made of pure open hearth iron, remelted in vacuo. For the sake of comparison, the results obtained with some of the same iron in the form of rods tested by the Burrows method have been included. All the specimens have been annealed at 1100 degrees C. From these results it is seen that for permeabilities not exceeding 20,000, when the possible variation in
mechanical and heat treatment of the various specimens are considered, the two methods check very well.

**TABLE 8**

**COMPARISON BETWEEN THE RING METHOD AND BURROW’S METHOD OF MAGNETIC TESTING**

<table>
<thead>
<tr>
<th>Specimen No.</th>
<th>Kind of Specimen</th>
<th>Max. Density</th>
<th>Max. Permeability</th>
<th>Permeability for B = 10000</th>
<th>Permeability for B = 15000</th>
<th>Permeability for B = 20000</th>
<th>Permeability for B = 25000</th>
<th>Permeability for B = 30000</th>
<th>Hysteresis Loss Ergs per c.c. per Cycle</th>
<th>Retentivity Gausses</th>
<th>Coercive Force Gilberts per cm.</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>4-01 Ring</td>
<td>14300</td>
<td>5850</td>
<td>13700</td>
<td>5700</td>
<td>986</td>
<td>2063</td>
<td>8400</td>
<td>12300</td>
<td>0.33</td>
<td>0.39</td>
<td>0.38</td>
<td></td>
</tr>
<tr>
<td>4-01 Rod</td>
<td>14180</td>
<td>5850</td>
<td>13200</td>
<td>5350</td>
<td>1080</td>
<td>2190</td>
<td>8700</td>
<td>15900</td>
<td>0.37</td>
<td>0.40</td>
<td>0.38</td>
<td></td>
</tr>
<tr>
<td>4-02 Ring</td>
<td>16500</td>
<td>9500</td>
<td>16450</td>
<td>6400</td>
<td>950</td>
<td>2010</td>
<td>8700</td>
<td>13900</td>
<td>0.30</td>
<td>0.35</td>
<td>0.30</td>
<td></td>
</tr>
<tr>
<td>4-03 Ring</td>
<td>17300</td>
<td>9000</td>
<td>10700</td>
<td>8250</td>
<td>852</td>
<td>1755</td>
<td>8400</td>
<td>12900</td>
<td>0.28</td>
<td>0.33</td>
<td>0.30</td>
<td></td>
</tr>
<tr>
<td>4-03 Rod</td>
<td>20900</td>
<td>9000</td>
<td>20200</td>
<td>7500</td>
<td>865</td>
<td>1760</td>
<td>9300</td>
<td>13600</td>
<td>0.30</td>
<td>0.34</td>
<td>0.30</td>
<td></td>
</tr>
</tbody>
</table>

Open Hearth Iron Remelted in Vacuum and Annealed at 1100 Degrees C.

<table>
<thead>
<tr>
<th>Specimen No.</th>
<th>Kind of Specimen</th>
<th>Max. Density</th>
<th>Max. Permeability</th>
<th>Permeability for B = 10000</th>
<th>Permeability for B = 15000</th>
<th>Permeability for B = 20000</th>
<th>Permeability for B = 25000</th>
<th>Permeability for B = 30000</th>
<th>Hysteresis Loss Ergs per c.c. per Cycle</th>
<th>Retentivity Gausses</th>
<th>Coercive Force Gilberts per cm.</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>3 Al 22 Ring</td>
<td>23500</td>
<td>7000</td>
<td>16700</td>
<td>900</td>
<td>636</td>
<td>1320</td>
<td>8400</td>
<td>11000</td>
<td>0.19</td>
<td>0.20</td>
<td>0.20</td>
<td>Al-0.45%</td>
</tr>
<tr>
<td>3 Al 18 Ring</td>
<td>19700</td>
<td>6400</td>
<td>12500</td>
<td>350</td>
<td>674</td>
<td>1210</td>
<td>9000</td>
<td>10200</td>
<td>0.20</td>
<td>0.20</td>
<td>0.20</td>
<td>Al-0.33%</td>
</tr>
</tbody>
</table>

Iron-Aluminum Alloys Melted in Vacuum and Annealed at 1100 Degrees C.

6. **Determination of Aluminum in Iron, by J. M. Lindgren.**—Four well known methods for the determination of aluminum in iron were tried. First, precipitation by means of sodium hydroxide; second, fusion in a nickel crucible with sodium hydroxide; third, the electrolytic separation with a mercury cathode; and finally, the method of Rothe,* with ether as a solvent for ferric chloride. The first two methods were objectionable because of the slow, tedious filtrations and the resulting voluminous filtrates. The electrolytic method did not remove all the iron, and so made a determination of that constituent necessary. Here, also, tedious filtrations and voluminous filtrates were encountered. Gooch and Hovenst made use of ether in the determination of aluminum by precipitating AlCl₃·6H₂O with gaseous hydrochloric acid and completing the separation from iron by means of ether.

Rothe demonstrated that by means of ether large amounts of iron could be separated from nickel-aluminum, copper-cobalt, vanadium, and titanium. He employed a special apparatus for the...
separation, which consisted of two separatory funnels joined to a common stem with a 3-way stopcock. By means of pressure applied by the vapors of ether the solution was forced into the second funnel, where the separation was continued by means of fresh ether. Rothe demonstrated that the iron should be present in the ferric state and that the hydrochloric acid present should have a definite strength.

In the present work the separation was carried out in a 200 cc. Jena beaker. It was found necessary, in transferring from one separatory funnel to another during the process of removing all the iron, to use considerable amounts of hydrochloric acid in order to wash the syrupy aluminum chloride from the sides of the funnel; this increased the acid concentration, and made it very difficult to remove all the iron. It occurred to the writer that the separation could be made easier in the original beaker in which solution was effected by decanting the supernatant ether and ferric chloride, and so make possible a cleaner and easier separation by subsequent washings with ether in the same beaker. The decantation was more easy to accomplish than first appeared possible, because the supernatant liquid of ferric chloride and ether was much lighter than the lower solution of aluminum chloride and acid.

Details of the method used are as follows: Dissolve from 1 to 5 grams of iron in a 200 cc. Jena beaker by means of hydrochloric acid, with additions of nitric acid to afford complete oxidation of the iron; evaporate to dryness on a water bath, and take up with just enough hydrochloric acid, sp. gr. 1.19, to completely dissolve the iron. Care should be taken that no iron oxide remain undissolved. Evaporate again on a water bath to a syrup consistency, add 30 cc. of ether, and stir vigorously with a glass rod until the ether has dissolved as much ferric chloride as possible. Allow to stand until the separation is distinct and both layers are clear. At this point it is sometimes necessary to add more hydrochloric acid, two cc. at a time, until the

<table>
<thead>
<tr>
<th>Per Cent Aluminum Taken</th>
<th>Per Cent Aluminum Found</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.732</td>
<td>0.763</td>
</tr>
<tr>
<td>0.732</td>
<td>0.791</td>
</tr>
<tr>
<td>1.464</td>
<td>1.484</td>
</tr>
</tbody>
</table>
solutions become perfectly clear. Carefully decant the supernatant solution of iron and ether and repeat the addition of ether. Usually six additions of ether are sufficient to extract completely all of the iron. Evaporate the ether and precipitate the aluminum in the usual way by means of ammonia. Burn the oxide in a porcelain crucible and finally in a muffle at 1000 degrees C to constant weight. The necessity of a high temperature in burning the aluminum precipitate shows the impossibility of obtaining satisfactory results by any of the methods involving the burning off of a mixed precipitate of aluminum and iron.
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