Fundamental Study on Vacuum Melting of Pure Iron and Stainless Steel. I. On the Crucibles, Ingotism and the Recovery of Alloy Additions.

By

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The relations between vacuum melting practices and the behavior of gases, the magnesia crucibles, ingotism, the recovery of alloy additions etc. were studied. It was found that the B' type magnesia crucibley was most satisfactory in view of its resistance to oxygen contamination. The refining practice using hydrogen gas showed somewhat higher content of hydrogen, while that utilizing immersion of a graphite rod was found to offer difficulties in regard to control of the dissolved carbon content. A sound ingot was obtained by appropriate modifications of the mold assembly. The vacuum melting practices for controlling the compositions of some kinds of stainless steels were confirmed.

1. Introduction

There is a growing interest in the vacuum melting practice and information is required about the effects of this process on the quality of metals manufactured. The present study was performed for the purpose of obtaining fundamental knowledge about the vacuum melting practice particularly as it affects crucibles, stamping refractories, the erosion of crucibles, ingotism etc. Furthermore, the change of the composition of pure iron during vacuum melting, and the recovery of alloy additions in stainless steel melting were also investigated.

2. Vacuum Melting Apparatus and the Melting Practice

The arrangement of the high frequency induction vacuum melting furnace which was used for this investigation is shown in Fig. 1. This apparatus is available for the melting either of 1 kg or of 5 kg iron. The vacuum tank is of a double-walled water-cooled stainless steel construction, and the heating coil and crucible assembly can be tilted in its position by a rotating coaxial system which

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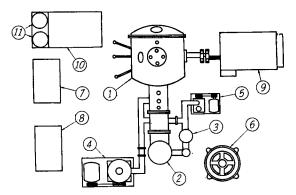


Fig. 1. Arrangement of vacuum melting apparatus.

- 1 Vacuum tank (heating coil, buckets for addition, thermocouples, bridge breaker, gas inlets)
- 2 Oil diffusion pump (10 in ϕ , 1800 *l*/sec)
- 3 Oil booster pump $(4 \text{ in } \phi, 300 l/\text{sec})$
- 4 Rotary pump (1,500 l/min)
- 5 Supplementary pump
- 6 High frequency motor generator (200 V, 15 KW, 10,000 cycle)
- 7 Vacuum system control panel (*a*-tron vacuum gauge, thermocouple vacuum gauge, pyrometer)
- 8 Electric power system control panel
- 9 Condenser bank
- 10 Operation table
- 11 Gas bombe

is used for the electric power supply and for the leading tube of cooling water. After the rough degassing operation, which was done with a mechanical rotary pump, the tank was evacuated by an oil diffusion pump and an oil booster pump backed up by the rotary pump. The pressure was measured mainly by an α -tron vacuum gauge, and sometimes by a thermocouple vacuum gauge. Alloy additions can easily be performed with the aid of the three charging buckets without breaking the vacuum. Two peep windows with protection shield plates, two Wilson sealed tubes with Pt-PtRh immersion

237

couples, a bridge breaker, and two gas inlet valves are attached to the vacuum tank to facilitate the melting operation.

The normal melting operation was carried out as follows:

After the crucible was charged, the tank was evacuated to about 10^{-3} mm Hg, and then the electric power was supplied. The power was controlled by the capacities of the condenser bank and by regulating the exiting field current of the motor generator. The vacuum pumps were kept running during the heating period, but in the case of the melting of materials which contained a considerable amount of gases, violent boiling of the bath was caused by the rapid gas evolution, and the splash of molten metal solidified on the upper part of the crucible. This boiling not only interrupted the melting operation but also detracted from the life of the crucible, and, therefore, it was preferable to suppress the boiling by supplying argon gas to the pressure of 10 to 20 mm Hg until the complete melt down of the charge was attained. After the melt was degassed to a vacuum of about 10^{-3} mm Hg, alloy additions were made by bucket, and the melt was poured into the ingot mold situated at the front of the crucible assembly in the vacuum tank. When volatile metals were added to the bath, the addition and casting were carried out under the pressure of about 20 mm Hg of argon to avoid their vaporization loss. Two examples of melting operations are shown in Figs. 2 and 3.

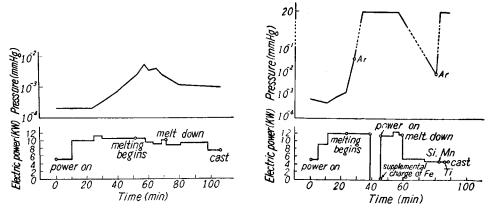


Fig. 2. A heat log of pure iron melting.

Fig. 3. A heat log of stainless steel melting (A.I.S.I. 321).

3. Crucible for Melting

(1) Shape of Crucible

The dimensions of various electromagnesia crucibles used in the present investigation are given in Table 1. All of the crucibles for 5 kg melting have a similar size and there was no trouble as far as their dimensions were concerned, but the crucibles for 1 kg melting were found to be more or less inconvenient in view of the results of test meltings. It was decided, therefore, to use such a crucible as B'-1, the dimensions of which are also shown in Table 1. The B' type crucibles were manufactured from electromagnesia powder with controlled grains which were independent and different from those of K. H. Köthemann et al¹.

Zirconia, magnesia, and mixtures of 50 pct zirconia and 50 pct magnesia were tested as refractories for stamping. It was concluded that stabilized zirconia powder was most excellent for the purpose of laboratory work on account of its

| Crucible | Outer diameter mm | Inner diameter mm | Wall thickness mm | Height mm | Inner volume cm ³ | Note |
|----------------------|-------------------------|-------------------------|-------------------------|--------------|------------------------------------|----------|
| A-1 (Made in U.S.A.) | 52 | - 35 | 8.5 | 100 | 88 | |
| B-1 (Made in Japan) | 66 | 56 | 5.5 | 80 | 174 | For 1kg |
| B'-1 (") | 62 | 48 | 7.0 | 100 | 166 | melting |
| C-1 (") | 55 | 44 | 5.5 | 82 | 116 | |
| A-5 (Made in U.S.A.) | 103 | 83 | 10 | 187 | 950 | |
| B-5 (Made in Japan) | 112 | 86 | 13 | 180 | 1040 | For 5 kg |
| B'-5 (| | Same a | s B-5 | | | melting |
| C-5 (") | 108 | 85 | 11.5 | 186 | 1000 | |

Table 1. Dimensions of the crucibles used.

good thermal insulation and the convenience of handling, because the powder cannot be sintered by heating.

(2) Reaction between Crucible and Molten Metal

It has been observed²⁾³⁾ that the content of oxygen, which had once been decreased by the melting under vacuum, showed a trend to increase again, through the reaction between the crucible and the molten metal. But the time within which the contamination of oxygen in the bath is not recognized is different from crucible to crucible and it depends generally upon the quality of the crucible used. According to W. A. Fischer⁴⁾, the content of oxygen did not show any increase within 2 hrs. From the thermodynamic calculation, however, it is obvious that the oxygen contamination occurs by the dissociation, or reduction, of magnesia. If the bath does not show any contamination of oxygen for a prolonged heating time, the crucible may be sufficient for practical use. The quality of the crucible was judged from this point of view.

The pure iron No. 1 manufactured by a basic electric furnace, the chemical composition of which is given in Table 2, was melted using the various electromagnesia crucibles shown in Table 1 under the vacuum of 10^{-3} to 10^{-4} mmHg

| % | c | Si | Mn | Р | s | Cu | Ni | Cr | Sol.Al | Insol.Al | Mg | N | 0 | н |
|-------|-------|-------|-------|-------|-------|-------|-------|-------|--------|----------|-----|--------|--------|--------|
| No. 1 | 0.025 | 0.09 | 0.013 | 0.010 | 0.007 | 0.022 | 0.021 | 0.004 | 0.001 | 0.0005 | tr. | 0.0030 | 0.0185 | 0.0002 |
| No. 2 | 0.016 | 0.085 | 0.010 | 0.009 | 0.007 | 0.024 | 0.022 | 0.003 | 0.002 | 0.0007 | tr. | 0.0025 | 0.0366 | 0.0001 |

Table 2. Chemical composition of pure iron used as raw material.

at 1600°C, and the changes of composition of the bath were plotted against the holding time up to 2 hrs. The contents of hydrogen and oxygen were determined by the vacuum fusion method, and of nitrogen by the distillation method.

As shown in Table 3, the content of carbon was decreased from 0.005 to 0.008 pct after holding for 30 min, and about 0.004 pct after holding for 1 hr, from the initial value of 0.025 pct, but little change was observed by further holding under vacuum. The content of copper was decreased from the initial value of 0.022 pct to 0.007 or 0.008 pct after holding for 30 min and thereafter it was unchanged. The change of the content of aluminum was investigated for crucible A-1 only. Both the contents of acid soluble aluminum and acid insoluble aluminum showed little change from vacuum treatment. The content of hydrogen was decreased from the initial value of 0.0002 pct to 30 min, but little decrease was observed afterwards. This trend is similar to the results reported by W. A. Fischer⁴). Although the content of oxygen was decreased from the initial value of 0.0185 pct to a value less than

240 Hiroshi SAWAMURA, Toshisada MORI, Masao YAKUSHIJI and Hiromasa INOUE

| Heat No. | Crucible | | Treatmen | t | c | ο | N | Н | Sol.Al | Insol.Al | Cu |
|-------------|----------|---------|----------------------|-----------|-------|--------|--------|---------|--------|----------|-------|
| 64 | A-1 | 1600°C, | 10 ⁻³ mmH | lg, 30min | 0.007 | 0.0049 | 0.0015 | 0.00018 | 0.002 | 0.001 | • |
| 69 | " | " | " | " | 0.006 | 0.0039 | 0.0008 | tr. | 0.003 | 0.002 | |
| 65 | " | " | " | 60min | 0.004 | 0.0017 | 0.0010 | 0.00004 | 0.004 | 0.001 | |
| 68 | " | " | " | " | 0.004 | 0.0203 | 0.0019 | 0.00007 | 0.003 | 0.001 | |
| 66 | " | ** | ** | 120min | 0.004 | 0.0099 | 0.0009 | 0.00007 | 0.003 | tr. | |
| 67 | " | " | " | " | 0.006 | 0.0285 | 0.0009 | 0.00006 | 0.004 | 0.001 | |
| 96 | B-1 | 1600°C, | 10 ⁻³ mmH | g, 30min | 0.006 | 0.0103 | 0.0010 | 0.00010 | | | 0.010 |
| 99 | " | " | " | " | 0.008 | 0.0077 | 0.0017 | 0.00004 | | | 0.007 |
| 97 | , ,, | " | " | 60min | 0.004 | 0.0262 | 0.0013 | 0.00005 | | | 0.008 |
| 100 | " | " | " | " | 0.003 | 0.0072 | 0.0011 | 0.00006 | | | 0.008 |
| 98 | " | " | " | 120min | 0.003 | 0.0184 | 0.0015 | 0.00009 | | | 0.009 |
| 102 | " | " | ** | " | 0.004 | 0.0178 | 0.0012 | 0.00006 | Ì | | 0.006 |
| 139 | C-1 | 1600°C, | 10 ⁻³ mmH | g, 30min | 0.005 | 0.0063 | | 0.00009 | | | |
| 142 | " | " | " | " | 0.015 | 0.0073 | | 0.00010 | | | |
| 140 | " | " | " | 60min | 0.006 | 0.0073 | | 0.00007 | | | |
| 141 | " | " | " | 120min | 0.005 | 0.0104 | | 0.00008 | | | |

Table 3. The change of chemical composition of pure iron during vacuum melting.

0.01 pct by holding under vacuum for 30 min, the tendency of its gradual increase was also observed when the holding time was prolonged. This is quite the same trend as the results obtained by J. H. Moore²⁾ and K. Bungardt et al³⁾. The content of nitrogen was decreased from the initial value of 0.003 pct to about 0.001 pct or lower by holding for 30 min, and little or no further decrease was observed after prolonged holding.

It was proved from the above mentioned results that all of the crucibles tested were durable and the minimum content of oxygen in pure iron was attained after holding for about 30 min under vacuum.

The effects of holding time were similarly studied, using the crucible B'-1 which was manufactured with special care (particularly concerning the particle sizes of its magnesia powder). Oxygen contamination was not observed in these experiments within 1 hr at 1600°C as seen in Table 4. Even when the melt was held for 2 hrs, the content of oxygen was kept low enough for the case of heat No. 170. This crucible was superior to the aforementioned three kinds of crucibles. When the melt was kept at 1700°C, however, the content of oxygen increased after holding for only 30 min. When the new crucible was used, the content of silicon showed a trend to increase (see heat No. 166 and 167), but afterwards it was gradually decreased by the vaporization of silicon to about 1/2 of the initial value (see heat No. 169 to 171). The trend to increase the content of silicon

| Heat No. | Crucible | Tr | Treatment | | c | Si | Mn | Р | s | Cu | Ni | Cr | Sol.Al | Insol.Al | Mg | N | 0 | н |
|-------------|----------|------------|---------------------|--------|-------|-------|--------|-------|-------|-------|-------|--------|--------|----------|-----|--------|--------|---------|
| 166 | B′-1 | 1600°C, 10 | ⁻³ mmHg, | 30min | 0.006 | 0.095 | 0.0012 | 0.009 | 0.005 | 0.010 | 0.021 | 0.0015 | 0.0026 | 0.0009 | tr. | 0.0013 | 0.0042 | 0.00007 |
| 167 | >> | " | " | " | 0.005 | 0.099 | 0.0010 | 0.010 | 0.006 | 0.009 | 0.022 | 0.0011 | 0.0026 | 0.0006 | tr. | 0.0012 | 0.0041 | 0.00011 |
| 168 | " | " | " | 60min | 0.003 | 0.071 | 0.0010 | 0.009 | 0.005 | 0.009 | 0.020 | 0.0011 | 0.0024 | 0.0005 | tr. | 0.0016 | 0.0028 | 0.00006 |
| 169 | " | " | " | " | 0.005 | 0.058 | 0.0006 | 0.009 | 0.006 | 0.008 | 0.022 | 0.0017 | 0.0020 | 0.0005 | tr. | 0.0010 | 0.0033 | 0.00008 |
| 170 | " | " | " | l20min | 0.003 | 0.054 | 0.0002 | 0.009 | 0.006 | 0.007 | 0.022 | 0.0005 | 0.0020 | 0.0005 | tr. | 0.0012 | 0.0028 | 0.00005 |
| 171 | " | ** | ** | " | 0.004 | 0.050 | 0.0002 | 0.010 | 0.005 | 0.008 | 0.021 | 0.0009 | 0.0021 | 0.0005 | tr. | 0.0017 | 0.0090 | 0.00009 |
| 172 | " | 1700°C, 10 | ⁻³ mmHg, | 30min | 0.002 | 0.015 | 0.0002 | 0.010 | 0.006 | 0.008 | 0.021 | 0.0013 | 0.0024 | 0.0006 | tr. | 0.0010 | 0.0151 | 0.00003 |
| 173 | " | ** | " | " | 0.003 | 0.015 | 0.0002 | 0.010 | 0.006 | 0.008 | 0.021 | 0.0014 | 0.0029 | 0.0005 | tr. | 0.0015 | 0.0130 | 0.00005 |

Table 4. The change of chemical composition of pure iron during vacuum melting.

Table 5. The change of chemical composition of pure iron treated with H_2 gas and (or) graphite.

| Heat No. | Crucible | Treatment | c | Si | Mn | Р | s | Cu | Ni | Cr | Sol.Al | Insol.Al | Mg | N | ο | н |
|-------------|----------|---|-------|-------|--------|-------|-------|-------|-------|--------|--------|----------|-----|--------|--------|---------|
| 161 | B'-5 | 30 mmHg H ₂ , 5 min $\times 10$ | 0.003 | 0.081 | 0.0010 | 0.009 | 0.007 | 0.010 | 0.022 | 0.0015 | 0.0021 | 0.0006 | tr. | 0.0012 | 0.0046 | 0.00010 |
| 162 | " | " | 0.011 | 0.083 | 0.0002 | 0.010 | 0.007 | 0.011 | 0.022 | 0.0014 | 0.0024 | 0.0008 | tr. | 0.0016 | 0.0034 | 0.00007 |
| 163 | " | " | 0.005 | 0.090 | 0.0009 | 0.009 | 0.006 | 0.012 | 0.022 | 0.0016 | 0.0016 | 0.0007 | tr. | 0.0013 | 0.0036 | 0.00012 |
| 164 | 33 | 2.5gr. Carbon + 30mmHg H ₂ , 5min \times 5 | 0.007 | 0.110 | 0.0009 | 0.010 | 0.007 | 0.011 | 0.022 | 0.0016 | 0.0022 | 0.0008 | tr. | 0.0013 | 0.0034 | 0.00017 |
| 176 | " | ** | 0.002 | 0.118 | 0.0002 | 0.010 | 0.006 | 0.008 | 0.020 | 0.0016 | 0.0019 | 0.0005 | tr. | 0.0015 | 0.0025 | 0.00004 |
| 177 | " | Graphite rod immersion | 0.398 | 0.179 | 0.0002 | 0.010 | 0.007 | 0.015 | 0.022 | 0.0021 | 0.0022 | 0.0006 | tr. | 0.0015 | 0.0015 | 0.00009 |
| 178 | " | " | 0.127 | 0.157 | 0.0008 | 0.010 | 0.006 | 0.017 | 0.021 | 0.0021 | 0.0019 | 0.0008 | tr. | 0.0013 | 0.0014 | 0.00011 |

242 Hiroshi SAWAMURA, Toshisada MORI, Masao YAKUSHIJI and Hiromasa INOUE

may be due to the reduction of silica which was contained as impurities in the magnesia. Silicon can, therefore, be removed only by heating the bath to high temperature or by holding it for a long time under high vacuum. The content of chromium was decreased by vaporization to about 1/3 of the initial value. The contents of nickel, phosphor and sulphur were entirely unchanged. Traces of magnesium were found in all heats and, therefore, it was proved that magnesium dissolved from the crucibles was completely removable because of its high vapor pressure. Concerning the behavior of copper, aluminum, carbon, hydrogen and nitrogen, the same trend as shown in Table 3 was confirmed.

(3) Refining by Hydrogen Gas and by Graphite Rod

Both groups of results, given in Table 3 and 4, are what were obtained by simple holding under vacuum. It was thought that the experiments in the refining of pure iron melt by reduction with hydrogen gas and by the immersion of a graphite rod were necessary. 5 kg of pure iron No. 2, the composition of which is given in Table 2, was melted in a crucible B'-5 and refining under the pressure of hydrogen and by the immersion of a graphite rod was carried out. The hydrogen refining process was conducted as follows: First, the purified hydrogen gas was blown on to the surface of the molten bath and the bath was held under a pressure of 30 mmHg of hydrogen for 5 min, and then the gas was removed. Hydrogen supply and degassing were repeated (see heat No. 161 to 163). Two of the heats were treated by adding small amounts of graphite (i.e. 0.5 g/kg iron) and also by hydrogen refining (see heat No. 164 and 176).

In the case of heat No. 176, as shown in Table 5, the contents of carbon and oxygen were 0.002 and 0.0025 pct, respectively, and the residual hydrogen showed a low value. However, the content of hydrogen was somewhat higher than for the heats shown in Table 4. It was concluded that in order to eliminate the dissolved hydrogen completely, the degassing time must be prolonged.

The combination of the stoichiometric addition of graphite, corresponding to the oxygen contained in the raw material, together with the hydrogen gas treatment, was not as effective as the simple vacuum treatment. The results of refining by the immersion of a graphite rod are shown for heats No. 177 and 178.

By the immersion of a graphite rod in the bath, the content of oxygen was decreased to an extreme minimum value, but on the other hand the content of carbon was increased to a high value. Furthermore, no advantageous effect was observed concerning the elimination of hydrogen and nitrogen. It is considered that the immersion process is technically difficult for the purpose of manufacturing a pure iron.

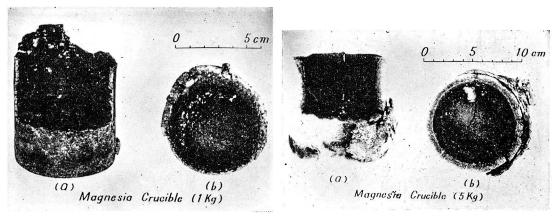


Photo. 1. The appearance of the crucible B-1 after use.

Photo. 2. The appearance of the crucible A-5 after use.

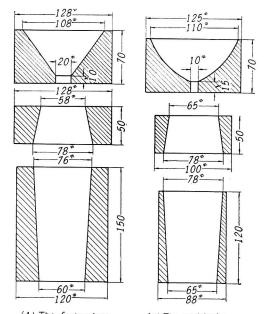
(4) The Life of the Crucible

Photo. 1 shows the appearance of a crucible B-1 which had been used for the melting of eight heats of pure iron and ten heats of iron-titanium-carbon alloys containing titanium up to 4 pct and carbon up to 0.5 pct. When the metal contained a considerable amount of titanium or carbon, erosion on the inner surface of the crucible was observed. Photo. 2 shows the appearance of a crucible A-5 which had been used for the melting of twenty-seven heats of various

stainless steels. Though the crucible was damaged considerably in its appearance, its inner surface was not so eroded. Therefore, these electromagnesia crucibles can be considered sufficiently durable for practical use, but further improvements are desirable in respect to the contamination of oxygen and silicon and the higher resistance to the dissolved active elements.

4. The Effects of Ingot Mold and Casting Temperature on Ingotism

The ingot mold for 5 kg casting made of cast iron with a hot top and funnel gate made of high alumina refractories as shown in Fig. 4(A)



(A) The former type(B) The modified typeFig. 4. The design of ingot mold assembly.

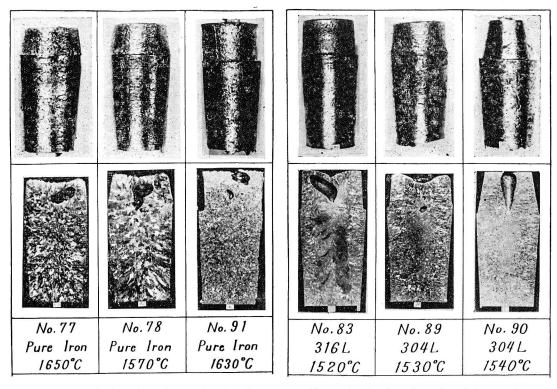


Photo. 3. The ingotism of pure iron by the modified mold assembly.

Photo. 4. The ingotism of stainless steels by the modified mold assembly.

was tested, but a deep shrinkage cavity was often found in the upper part of the ingots, resulting in poor yield. It was considered that the hot top was made ineffective by rapid solidification because of the large heat capacities of the mold assembly.

The thinner walled mold of mild steel and the smaller hot top as shown in Fig. 4(B) were then prepared, and the design of the funnel gate was also improved to prevent erosion by the pouring stream. The ingotism of pure iron and stainless steels was reexamined using a modified mold assembly. Some examples are shown in Photo. 3 and 4. The pure iron melts were poured at three different temperatures, i.e. 1570, 1630 and 1650°C, but the influence of the differences of casting temperature on ingotism was not obvious.

Using two kinds of hot top one of which was made of high alumina refractories and the other of mild steel, stainless steel melts were poured at about constant temperature. The piping was not so deep in all cases, and, therefore, it may be concluded that by the appropriate modifications of the design of the mold assembly, the ingotism was improved.

5. Recoveries of Alloy Additions

As a fundamental experiment in manufacturing stainless steel having the required composition by vacuum melting, eight types of stainless steels namely AISI 304, 304L, 316, 316L, 321, 321L, 347, and 347L were vacuum melted and the recoveries of alloy additions were examined. The chemical compositions of materials used as alloy additions are given in Table 6. Silicon, manganese,

| | С | Si | Mn | P | S | Cu | Fe | Мо | N | 0 | н | Others |
|---------------------|-------|-------|--------|-------|-------|-------|---------------|--------|-------|--------|--------|-----------------------|
| Electrolytic Mn | 0.006 | 0.008 | >99.99 | | | | | | | | | Al tr. |
| Electrolytic Ni | 0.002 | 0.005 | 0.005 | | 0.001 | 0.004 | 0.00 7 | | | 0.0014 | | {Co 1.3 {Pb 0.0006 |
| Metallic Si | | 99.7 | | | | | | | | 0.07 | 0.0026 | |
| Metallic Ti | 0.05 | 0.05 | | | | | 0.06 | | 0.009 | | | |
| Metallic Mo | | | | | | | | >99.99 | | | | |
| Low carbon Fe-Cr | 0.056 | 0.50 | | 0.024 | 0.006 | 0.11 | | | 0.018 | 0.022 | | Cr 61.02 |
| Low carbon Fe-Nb | 0.07 | | | | 0.032 | | | | | 0.018 | | Nb 68.64 |

Table 6. The chemical composition of added materials.

titanium and ferro-niobium were added under the pressure of about 20 mmHg of argon, and the casting was also carried out under the same pressure of argon within 3 to 5 min after the final alloy addition. The electrolytic manganese was preliminarily vacuum treated to remove the gases in a graphite crucible at about 800°C for 2 hrs. The change of the content of gases in the electrolytic manganese by this vacuum treatment is shown in Table 7.

Table 7. The change of the content of gases in the electrolytic manganese by vacuum degassing treatment. (800° C, 2hrs, 3×10^{-2} mmHg)

| % | N | 0 | н |
|--|--------|------------------|--------|
| As received After degassing treatment | 0.0038 | 0.0049 0.0422 | 0.0131 |

Hydrogen was almost completely removed and nitrogen was decreased to some extent, while the content of oxygen was considerably increased. In order to prevent oxidation, therefore, it is necessary either to lower the degassing temperature or to treat under higher vacuum.

The compositions aimed at and the results analyzed are shown in Table 8. There was a mistake in heat No. 79 for charge calculation concerning the amount of nickel, and the lower recoveries of manganese in heats No. 79 and No. 80 were

| Heat No. | Type AISI | | с | Si | Mn | Р | s | Cu | Ni | Cr | Mo | Ti | Nb | Mg | ∑N | Sol.N | Insol.N | 0 | Casting temp. °C | Pressure before add mmHg |
|-------------|--------------|-------------------|----------------|--------------|-------------|----------------|----------------|-------|---------------|---------------|--------------|-------------|--------------|-------|--------|--------|---------|--------|------------------------|--------------------------------|
| 79 | 304 | Aimed Analyzed | 0.08 0,086 | 0.70 0.63 | 1.7 1.22 | <0.03 0.016 | <0.03 0.007 | 0.021 | 10.7 12.98 | 18.3 17.41 | | | | 0.001 | 0.0043 | 0.0034 | 0.0009 | 0.0057 | 1570 | 2×10 ⁻³ |
| 80 | 304L | Aimed Analyzed | <0.03 0.008 | 0.70 0.61 | 1.7 1.49 | <0.03 0.015 | <0.03 0.009 | 0.014 | 8.3 8.49 | 20.0 19.63 | | | | 0.001 | 0.0055 | 0.0037 | 0.0018 | 0.0073 | 1550 | 1×10 ⁻³ |
| 82 | 316 | Aimed Analyzed | 0.08 0.080 | 0.70 0.82 | 1.7 2.52 | <0.03 0.016 | <0.3 0.006 | 0.021 | 13.7 12.66 | 16.3 15.62 | 2.75 2.55 | | | 0.001 | 0.0058 | 0.0043 | 0.0015 | 0.0029 | 1530 | 2×10 ⁻³ |
| 84 | 316L | Aimed Analyzed | <0.03 0.006 | 0.70 0.64 | 1.7 2.08 | <0.03 0.015 | <0.03 0.007 | 0.015 | 11.3 11.16 | 18.0 17.75 | 2.75 2.79 | | | 0.001 | 0.0028 | 0.0028 | tr. | 0.0094 | _ | 1×10 ⁻³ |
| 86 | 321 | Aimed Analyzed | 0.08 0.074 | 0.70 0.77 | 1.7 2.21 | <0.03 0.012 | <0.03 0.009 | 0.018 | 12.7 12.22 | 17.3 18.68 | | 0.6 0.58 | | 0.001 | 0.0028 | tr. | 0.0028 | 0.0125 | 1550 | 1×10 ⁻³ |
| 85 | 321L | Aimed Anolyzed | <0.03 0.004 | 0.70 0.68 | 1.7 1.97 | <0.03 0.015 | <0.03 0.006 | 0.016 | 9.3 9.16 | 20.0 19.71 | | 0.6 0.53 | | 0.001 | 0.0033 | tr. | 0.0033 | 0.0074 | | 2×10 ⁻² |
| 88 | 347 | Aimed Analyzed | 0.08 0.074 | 0.70 0.72 | 1.7 2.04 | <0.03 0.010 | <0.03 0.006 | 0.019 | 12.7 12.40 | 17.3 17.48 | | | 0.95 0.95 | | 0.0047 | 0.0027 | 0.0020 | 0.0057 | 1540 | 1×10 ⁻³ |
| 87 | 347L | Aimed Analyzed | <0.03 0.011 | 0.70 0.68 | 1.7 2.00 | <0.03 0.010 | <0.03 0.007 | 0.019 | 9.3 9.14 | 20.0 20.13 | | | 0.95 0.99 | | 0.0039 | 0.0011 | 0.0028 | 0.0069 | 1540 | 2×10 ⁻² |

Table 8. The chemical composition of stainless steels manufactured by vacuum melting.

due to the fact that the necessary addition became impossible because of the overturn of a bucket. The lower contents of nickel and chromium and higher content of silicon and manganese in heat No. 82 are due to metal loss through splashing before the additions of silicon and manganese.

From the results in Table 8, the recoveries of each elements may be considered.

It was found from the preliminary experiment that the content of carbon in the ingot was decreased to 0.02 pct when 0.08 pct of carbon was aimed at. In this case the contents of oxygen and carbon in the raw iron, and of carbon in the ferro-chromium, were taken into consideration. The amount of carbon corresponding to 0.06 pct was, therefore, over charged. Satisfactory results were then obtained.

Manganese was added in excess of 25 pct in these experiments. But the addition in excess of 5 pct may be adequate if the addition is made under the pressure of 20 mmHg of argon and followed by casting in the same atmosphere within 5 min.

Silicon was added in excess of 10 pct while nickel, chromium, molybdenum, titanium and ferro-niobium were added just as calculated. In spite of a little scatter in the recoveries, the amounts of these alloy additions are generally appeared to be satisfactory.

The content of copper was decreased slightly when compared with that of the raw iron. This may be due to the presence of 0.11 pct of copper in the low carbon ferro-chromium. The content of magnesium was found to be negligibly small of the order of 0.001 pct in all cases.

6. Summary

A fundamental study of the vacuum melting practice was made, noting effects on crucibles, ingotism, recoveries of alloy additions, etc. The results obtained are summarized as follows:

1) Pure iron was melted and held under vacuum in crucibles made of four kinds of electromagnesia, and the change of composition during the vacuum treatment up to 2 hrs was examined. It was found that all of the crucibles tested were durable. The B' type crucibles was superior to the others; oxygen contamination was not observed within 2 hrs at 1600° C, but the content of oxygen was increased considerably when the melt was kept at 1700° C for 30 min.

2) The results of refining pure iron by hydrogen gas showed the higher content of hydrogen.

3) The content of oxygen was sufficiently decreased by the immersion of a graphite rod into the molten bath, but the content of carbon was increased

247

248 Hiroshi SAWAMURA, Toshisada MORI, Masao YAKUSHIJI and Hiromasa INOUE

rapidly. It is considered, therefore, that the control of the content of dissolved carbon is difficult.

4) By the appropriate modification of the design of the mold assembly, the ingotism can be improved.

5) Stainless steels were satisfactorily vacuum melted by the method in which the addition of silicon and manganese was made under the pressure of 20 mmHg of argon to prevent their vaporization loss.

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