DETERMINATION OF OXYGEN IN IRON POWDERS

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Iron powders constitute the principal component of many sintered materials and consequently their study is of both scientific and practical interest.

The chemical analysis of iron powders for impurities present in solid solution or in the form of chemical compounds is performed by the standard methods used for the analysis of dense metals. An exception is provided by oxygen, which cannot conveniently be determined in iron powders by the methods used for its determination in steels. The oxygen content of steels amounts to hundredth parts of one per cent, while its concentration in iron powders is many times higher. The method of vacuum melting [1], as well as spectral [2] and other methods, are not suitable for this purpose because of their complexity and need for special apparatus. Besides, these methods are used for analyzing steel.

Iron powders in contact with air actively absorb oxygen, and, because of the substantial size of the specific surface of these powders, the amount of resultant oxides is quite appreciable. In addition to oxide films on the surface of iron particles, internal oxide inclusions are observed in the particles of powders obtained by the reduction method. With increasing oxygen content of powders their technical usefulness decreases, and for this reason determination of oxygen in iron powders is of major importance.

At the present time, there are a number of methods for determining oxygen in iron powders [3-5]; essentially, these methods determine the total amount of oxygen, which cannot always accurately characterize the properties of a powder. In order to evaluate the quality of a powder, it is often necessary to know the relative amounts of the phases in which oxygen is combined in the given powder. In the present investigation, the authors determined the phase composition of nonmetallic inclusions in iron powders produced by the Sulinsk factory and the Institute of Materials Problems, Academy of Sciences, UkrSSR.

Figure 1 shows an x-ray diffraction picture of a starting iron powder, obtained in the URS-70 x-ray structural installation with chromium radiation, in an RKD camera used for studying polycrystals. Against the interference lines in this photograph are shown the phases corresponding to these lines according to calculation. As can be seen from this diffraction picture, iron powder contains oxygen combined with iron in the form of various phases, the amount of which, judging from the intensity of the interference lines, is substantial. It was found impossible to determine the amount of this oxygen in iron powder by the methods employed for other materials [6] without an appropriate preparation of specimens.

In order to determine oxygen, nonmetallic inclusions were initially isolated from iron powder. For this purpose, use was made of the sulfate electrolytic method [7]; the anode was provided by a specimen compacted from the powder under examination. Materials compacted from powder have a substantial porosity (up to 20-23%), as a

Fig. 1. X-ray diffraction picture of starting iron powder made by Sulinsk factory.
result of which metallic particles detached from the specimen used to fall during electrolysis among the nonmetallic inclusions. When the separated nonmetallic inclusions were dissolved in acids in the course of analysis, the iron particles also dissolved, thereby disturbing the results of the analysis. Infiltration of specimens with paraffin wax only slightly reduced the amount of metallic iron present with the nonmetallic inclusions. The presence of metallic iron was detected by the mercuric chloride method.

Specimens for the analysis were prepared in the following manner. A cylinder of 10 mm diameter and 25-30 mm length was compacted from the starting powder. In order to protect the specimen against additional oxidation, a rubber tube was pulled onto it in such a way that the air/electrolyte interface passed over this tube and not over the specimen. The same tube firmly pressed a copper wire loop to the specimen, forming a contact, thereby connecting the latter to the positive terminal of the electrolytic installation. Before infiltration, the specimens were heated to a temperature of 70-80°C, the subsequent infiltration being continued until air bubbles ceased to appear. After infiltration, the specimens were removed from the resin and were left for a few hours to polymerize. Before weighing, they were rubbed with a pad slightly wetted with a solvent. Electrolysis was performed under the following conditions:

1. Anode—test specimen.
2. Electrolyte—3% FeSO₄·7H₂O + 1% NaCl + 0.2% KN₃C₄H₄O₆.
3. Cathode—a copper plate.
4. Insulating membrane—a tracing paper bag attached to a glass ring whose projections are placed on the edges of the glass tank; this arrangement enables specimens to be freely suspended.
5. Electrolysis parameters—0.02-0.04 A/cm², duration 1-1.3 h.

Electrolysis may be performed with several specimens at the same time, by increasing the tank size and the battery output through parallel connection. Figure 2 shows a diagram of an electrolytic installation. The difference in specimen weight before and after electrolysis gives the amount of dissolved alloy. When electrolysis is performed as described above, 0.2-0.4 g of alloy is dissolved in the time indicated. The separated nonmetallic inclusions cover the specimen in the form of a crust, and only a very small amount of inclusions may be found in the tracing paper bag. The nonmetallic inclusion powder is quantitatively transferred to a tall cylinder, washed by decanting, dissolved in H₂SO₄ (1 : 4), and subjected to analysis.

For determining the total amount of iron, Fe³⁺ is reduced to Fe²⁺ in an aliquot part of sulfuric acid solution, with hydrogen liberated on addition of metallic zinc. A blank test is carried out with the same amount of zinc and acid. Divalent iron is titrated with 0.01 N KMnO₄ solution, the amount of which is usually 5-10 ml depending on the iron content. Calculation is performed with the formula:

$$Fe_{tot} = \frac{(a - b)T \cdot 100}{B} \%,$$

where a is the number of milliliters of 0.01 N KMnO₄ solution required for sample titration, b is the number of milliliters of 0.01 N KMnO₄ solution required for the blank test, T is the titer of the KMnO₄ solution, converted to iron, and B is the weight of dissolved metal in the aliquot portion of the solution.

Ferrous iron was determined by titrating the aliquot part of sulfuric acid solution of nonmetallic inclusions with 0.01 N KMnO₄ solution, calculation being performed in the same way as in [1]. Ferric iron was determined as a difference.