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IS 12052 (2007): Method for determination of re-oxidation rate of sponge iron/direct reduced iron (DRI) in contact with humid air and in direct contact with water [MTD 30: Sponge Iron and Smelting Reduction]
Indian Standard

METHOD OF DETERMINATION OF RE-OXIDATION RATE OF SPONGE IRON/DIRECT REDUCED IRON (DRI) IN CONTACT WITH HUMID AIR AND IN DIRECT CONTACT WITH WATER

(First Revision)

ICS 77.080.10
FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Sponge Iron and Smelting Reduction Sectional Committee had been approved by the Metallurgical Engineering Division Council.

Sponge iron because of its high iron content, reactivity and porosity can re-oxidize quite easily and this tendency depends on the process conditions during manufacture, cooler discharge temperature in particular, nature of the material and conditions prevailing during handling and storage of the final product.

Because of the very nature of the product, there is some degree of risk involved in the handling of sponge iron. The risks are of two types:

a) Risk of loss of the metallic value by slow conversion of sponge iron to a higher oxidized state, and

b) Risk of massive overheating to such an extent that a bulk mass of sponge iron stored/transported may spontaneously ignite (spontaneous combustion).

The first phenomena represents only a modest economic loss while the second involves a large loss plus the definite possibility of damage to equipment, such as bins, railroad cars, trucks and ships. Both can be totally prevented, if proper precautions are taken during storage and transport.

In order to assess the re-oxidation characteristics of sponge iron in contact with humid air under different temperature conditions and/or in direct contact with water, a simplifier laboratory test can be used, and these test methods have been prescribed in the following two sections of the standard:

a) Section I (A) — Re-oxidation test in contact with humid air

b) Section I (B) — Determination of crossing point temperature

Section II — Re-oxidation test in direct contact with water

This standard was first published in 1987. While reviewing the standard in the light of the experience gained during these years, the Committee decided that the standard may be revised. In this revision, the title of the standard has been changed and additional test for determination of cross point temperature has been included in Section I.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2:1960 'Rules for rounding off numerical values (revised)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.
Indian Standard

METHOD OF DETERMINATION OF RE-OXIDATION RATE OF SPONGE IRON/DIRECT REDUCED IRON (DRI) IN CONTACT WITH HUMID AIR AND IN DIRECT CONTACT WITH WATER

(First Revision)

SECTION I (A) RE-OXIDATION TEST IN CONTACT WITH HUMID AIR

1 SCOPE
This section of the standard prescribes the test method of determining the re-oxidation rate of sponge iron/DRI in contact with humid air under given temperature conditions. The test should be conducted at ambient temperatures of 25, 30, 40 and 50°C and an elevated temperature of 100°C to simulate the total range of storage conditions.

2 PRINCIPLE OF METHOD
The test sample is inserted into a reaction vessel which in turn is placed in an air oven capable of developing a temperature of up to 300°C. The reaction vessel is a one litre bottle which is connected to an oxygen generator through a glass tube fitted at the top. The volume of oxygen consumed is an indication of the rate of oxidation.

3 SAMPLING
3.1 Sampling of Sponge Iron Direct Reduced Iron (DRI)
For measuring the re-oxidation rate, a sponge iron sample of 1 kg of 6-50 mm size shall be taken (it is to be noted that pellets typically 9-16 mm and lump ore upto 50 mm in size are charged together in shaft furnace).

4 APPARATUS
The apparatus shall consist of the following:
4.1 Air Oven — It is a temperature controlled air oven where the temperature can be raised up to 300°C. The precision for controlling the temperature should be ± 5°C.
4.2 Oxygen Generator — It is an electrochemical oxygen generator capable of producing oxygen to a maximum of 60 mg/h. An electrical system controls the operation of the generator. When a definite pressure exists in the vessel, the generator remains OFF but when, due to consumption of oxygen, the pressure falls below the desired limits, the auto switch puts the system ON. The operating time of the generator is recorded in the hour meter. As the amount of current passed is known from the ammeter and the time of operation of oxygen generator will be given by the hour meter, so the amount of oxygen consumed by sponge iron for re-oxidation may be determined.

4.3 A contact type of pressure gauge shall be used.
4.4 Reaction Vessel — The reaction vessel is a one-litre bottle which is connected to the oxygen generator through a glass tube fitted at the top.
4.5 Reagents — Chemicals used in the oxygen generator are given below:
   a) Distilled water,
   b) Mercury for pressure indicator, and
   c) Saturated solution of CuSO₄.5H₂O acidulated to pH 2.5.

4.6 A schematic diagram of the set-up is given in Fig. 1.

5 TEST PROCEDURE
5.1 The thermostat is set at the desired temperature and sufficient time is allowed for it to attain the set temperature.
5.2 One kilogram of the sponge iron/DRI sample is introduced into the reaction vessel and the cover is immediately closed.
5.3 The oxygen generator is switched on and the operation is allowed to continue for 24 h.
5.4 After the completion of the test, the unit is switched OFF.

6 TEST CONDITIONS
6.1 For the purpose of this test, the following test conditions shall apply:
   a) Amount of sponge iron (DRI) : 1.00 kg
   b) Duration : 24 h
c) Measuring intervals: 1 h for first 5 h and then 4 h for the rest of period

d) Temperature: 25, 30, 40, 50 and 100°C (all data to be reported)

7 TEST RESULTS
After the test ends, the graph of volume of oxygen consumed (cm³) at NTP/kg of sponge iron versus time (hours) is drawn. The oxygen consumption at the end of 5 h serves as a reference value for comparison of re-oxidation between various sponge iron grades in contact with humid air.

8 NUMBER OF TESTS
The test shall be repeated three times and the results shall be reported as the average of three readings obtained provides each does not vary by more than 10 percent from the mean value.

9 TEST REPORT
The test report shall include the following:

a) A statement of the condition, source and porosity of the sample;
b) A screen analysis giving percentage of 6 to 8, 8 to 10 and 10 to 15 mm fraction of sponge iron sample;
c) The measured oxygen consumption at the end of first 5 h for 3 tests and their mean value;
d) Statement of the weighing device used; and
e) Graph of oxygen consumed (cm³) at NTP/kg of sponge iron versus time (hours).

SECTION 1 (B) DETERMINATION OF CROSSING POINT TEMPERATURE

10 TEST PROCEDURE
For determination of the propensity to oxidation in presence of air, a simpler technique called ‘Crossing Point Temperature’ determination technique may be used. This involves keeping a beaker-full of sponge iron in a muffle furnace or air oven previously maintained at about 150°C. The open beaker would have a thermocouple for monitoring the temperature of the central portion of sponge iron kept in the beaker.
The temperature of the furnace/oven would be raised at a steady rate lying between 2 to 5°C/min. The furnace temperature would naturally always be leading the temperature of sponge iron, but after a certain temperature, the temperature plot of sponge iron would 'Cross' or overtake the temperature of the furnace. This would be recorded as the 'Crossing Point Temperature' (CPT).

The CPT of all sponge irons lie between 200 and 350°C. Midrex and HyL sponge irons lie close to the lower end while coal based sponge irons are near the higher end.

SECTION II RE-OXIDATION TEST IN DIRECT CONTACT WITH WATER

11 SCOPE
This section prescribes the test method for measuring the re-oxidation rate of sponge iron (DRI) in an accelerated test under controlled laboratory conditions and a fixed temperature in the presence of moisture.

12 PRINCIPLE OF TEST
The test sample is inserted into the reaction vessel which, in turn, is placed in a water bath of constant temperature. The reaction vessel in connected to the electrolytic oxygen generator. Both the oxygen generator and the reaction vessel are connected to a pressure sensing unit. With absorption of oxygen by the sponge iron, the pressure (Δp) inside the reaction vessel decreases and is sensed by the pressure measuring unit. This drop in pressure is transmitted to the controller which converts it (Δp) in terms of mg of oxygen consumed.

13 SAMPLING OF SPONGE IRON DIRECT REDUCED IRON (DRI)
The sampling of sponge iron for measuring the re-oxidation rate shall be done by taking the desired quantity of 6-15 mm size sponge iron.

14 APPARATUS
14.1 The apparatus shall consist of the following.
14.1.1 Reaction Vessel — This vessel is made of glass and holds the sponge iron under test. It is connected to the electrolytic oxygen generator and pressure sensing unit.
14.1.2 Water Bath — It shall be a temperature controlled water bath. High speed water circulation is provided through the jacket for temperature uniformity. The high delivery of recirculation pump helps in adopting to the set point temperature up to an accuracy of ±0.1°C within a short time. The water bath is fitted with a temperature protective switch which shall be set at 40°C. The cooling water discharge is connected to the water bath by a plastic hose. The cooling unit is having an independent water circuit. The temperature controller is set at the desired batch temperature.

14.1.3 Oxygen Generator — It is an electrolytic cell from which oxygen is generated and the quantity is limited to 60 mg/h. A pressure controller controls the switching ‘ON/OFF’ of the oxygen generator. The oxygen generating set continuous according to the requirements. The amount of oxygen generated and absorbed are measured and recorded. Absorption of oxygen by the sponge iron in the first hour is reported as oxygen consumed per kg of sponge iron.

14.1.4 Pressure Indicator — It is contract type pressure gauge.

14.1.5 A schematic diagram of the test apparatus is given in Fig. 2. Cooling water circuit is not shown in Fig. 2.

15 TEST PROCEDURE
15.1 Check the water levels in the water bath and in the cooling unit and put ON the water bath switch. This also starts the recirculating pump and the temperature controller. Start the cooling unit and after about 30 min, the water bath shall reach the set point temperature.

15.2 Fill the reaction vessel with 50 g of sponge iron sample, and wet by distilled water (for carrying out a further accelerated test, wetting may be done 5 percent NaCl solution). Place the reaction vessel into the water bath. Connect the reaction vessel to the oxygen generator and the measuring unit to the pressure gauge and the oxygen generator.

15.3 Prior to carrying out the experiment, adopt the temperature of sample to the temperature of the water bath. This takes about 30 min. Then close the vent plug at the pressure sensing unit and start the measuring and control unit.

16 TEST CONDITIONS
16.1 For the purpose of this test, the following test conditions shall apply:
   a) Quantity of sample : 50 g of the sponge iron
   b) Wetting of liquid : 2.5 cm² of distilled water
   c) Measuring time : 1 h
   d) Measuring intervals : 0.1 h
   e) Temperature of measurement : 25°C
17 TEST RESULTS
After the test is completed, the measured oxygen absorption are plotted on a log-log graph. In the abscissa, the measurement times are plotted beginning at 0.1 h while the ordinate is presenting the oxygen demand converted to g oxygen/kg of sponge iron. The oxygen absorption measured at the end of first hour, serves as reference value for comparison between various sponge iron.

18 NUMBER OF TESTS
The test shall be carried out in triplicate and the result shall be reported as the average of the three readings, provided each does not vary by more than 10 percent from the mean value.

19 TEST REPORT
The test report shall include the following:

a) Statement of the condition, particularly porosity and the source of the sample;

b) Screen analysis giving percentage of 6 to 8, 8 to 10 and 10 to 15 mm fraction of sponge iron sample;

c) Measured oxygen consumption (at the end of the first hour) for 3 tests and their mean value;

d) Statement of the weighing device used; and

e) Log-log plot of g oxygen/kg of sponge iron versus time.
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