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METHODS FOR DETERMINATION OF SOFTENING — MELTING CHARACTERISTICS OF IRON ORE LUMPS/PELLETS/SINTER

(First Revision)

ICS 73.060.10:77.040.10
FOREWORD

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

Blast furnace burden should have high initial fusion temperature and narrow fusion range to restrict fusion zone and minimize pressure drop and hanging in the stack. Low softening temperature iron ore lead to reduced permeability and attendant blast furnace operational problems. This standard has been brought out with a view to study and indicate the softening behaviour of iron ore pellets to blast furnace operator.

While reviewing the standard, it has been felt by the committee to revise the standard and merge the requirements of IS 11283:1985 in this standard in view of the fact that determination of softening point of Iron Oxide (in powder form) is not relevant and is obsolete now. With the publication of this standard, IS 11283 would be withdrawn. In this revision, guidelines for determination of softening characteristics of iron ore, pellets and sinters under dynamic conditions have also been incorporated.

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

METHODS FOR DETERMINATION OF SOFTENING — MELTING CHARACTERISTICS OF IRON ORE LUMPS/PELLETS/SINTER

(First Revision)

1 SCOPE

1.1 The guidelines prescribe two methods for determination of softening-melting characteristics of Iron Ore, Lumps, Sinter and Pellet. Method 1 of the standard consists of two sections, Section 1 deals with determination of softening characteristics under conditions of increasing temperature at different reducibility and under a constant load. Section 2 deals with determination of softening characteristics under conditions of progressive increase of temperature and reduction under constant load.

1.2 The guidelines are at present applicable to heat hardened pellets but its applicability to other types of pellets such as cold bonded pellets will be considered at the appropriate time.

2 REFERENCES

The following standards contain provisions which through reference in this text, constitute provision of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

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<th>Title</th>
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<tbody>
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<td>Amyl alcohol (revised)</td>
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<tr>
<td>6911:1992</td>
<td>Stainless steel plate, sheet and strip (first revision)</td>
</tr>
<tr>
<td>8167:1989</td>
<td>Method for determination of reducibility index of iron ore oxides, lump ore, sinter and pellets (first revision)</td>
</tr>
</tbody>
</table>

3 GENERAL PROCEDURE

3.1 A sample of pellets of specified size is partly pre-reduced and then packed into a heat resistant steel container which is placed inside an electric furnace under a given pressure. The sample is heated in a neutral atmosphere following a well defined temperature programme, till a shrinkage of 25 percent of the initial height of the sample occurs. The test is repeated on samples pre-reduced at different degrees of reduction. The start of softening temperature of the sample is defined as that corresponding to shrinkage of 2, 3 and 5 percent of the initial height of the sample.

4 APPARATUS

4.1 Cylindrical Container for Softening Test

The crucible shall be of heat resistant steel of 51 mm internal diameter, 5 mm of thickness and 57 mm height. The bottom plate shall fit the bottom of the crucible 6 mm inside the crucible and 5 mm outside the crucible as shown in Fig. 1. A heat resistant cylindrical steel cover 49 mm diameter and 43 mm height shall fit into the top of the container free to move inside it. The crucible with bottom fitted to it shall be kept on the platen of the lower compression piston of 51 mm diameter. The top cylindrical container cover shall be pressed against the upper piston (also of 51 mm diameter) of the compression machine, as shown in Fig. 1. The pressure is applied on the sample by means of the compression machine in such a way that the upper platen remains stationary and the lower platen is pressed from bottom so as to apply a constant pressure on the sample throughout the experiment.

4.2 Compression Machine

The compression mechanism shall be oil driven by hydraulic pump. A gauge shall be attached to the machine for indicating the applied pressure in kg/cm². The platens of the machine compressing the sample shall be of heat-resistant material such as silicon carbide.

4.3 An electric furnace which can provide uniform temperature zone in cylinder space containing the sample, that is 55 mm internal diameter and 40 mm high.

4.3.1 A temperature recorder shall be connected to the furnace.

4.3.2 The heat shall be controlled according to a pre-established non-linear programme as indicated in 5.3.2 read with Fig. 2.

4.3.3 A recorder to indicate the change in the height of the sample during softening shall be attached suitably to the lower compression platen.
5 TEST CONDITIONS

5.1 Sample Size
Pellets of size between 9 mm and 16 mm shall be used.

5.2 Load Applied to the Sample
A load of 2 kg/cm² held constant throughout the test, shall be applied to the test sample by means of a suitable compression mechanism.

5.3 Heating Rate
A fairly low heating rate within the range of softening temperatures, shall be maintained in order to have a uniform temperature throughout the mass of the sample.

5.3.1 The test sample is placed inside the furnace preheated to 800°C under constant pressure. The temperature of the sample is assumed to be uniform no sooner its volume is stabilized.

5.3.2 The temperature of the furnace is then raised. The rate of heating (see Fig. 2) is decreased from initial 5°C/min at the start of the test to 4, 3, 2, 1 and finally 0.5°C/min linearly till a shrinkage of 25 percent of the initial height of the test sample occurs. The heating pattern may be as follows:

<table>
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<tr>
<th>Rate of Heating (°C/min)</th>
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<tr>
<td>5</td>
<td>20</td>
</tr>
<tr>
<td>4</td>
<td>20</td>
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<td>3</td>
<td>20</td>
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<td>2</td>
<td>20</td>
</tr>
<tr>
<td>1</td>
<td>20</td>
</tr>
<tr>
<td>0.5</td>
<td>Linearly for rest of the time</td>
</tr>
</tbody>
</table>

FIG. 2 RATE OF HEATING FOR THE SOFTENING TESTS
5.4 Pre-reduction of the Sample

The samples are pre-reduced to various degrees of reduction. The samples shall be preserved in vacuum desiccators to avoid reoxidation. Each softening test is carried out on a sample at each degree of reduction in a neutral atmosphere. This makes it possible to determine the softening conditions corresponding to the less favourable degree of reduction.

6 TESTING PROCEDURE

6.1 Introduce the pre-reduced sample into the crucible so as to fill not more than 30 mm height, the crucible being placed on the lower platen of the compression machine with bottom lid in position. Introduce the whole into the furnace from bottom, the furnace being heated to 800°C with N₂ gas being passed into the furnace. Fit the upper cylindrical crucible lid into the crucible and adjust the compression machine so as to have 2 kg/cm² constant pressure on the sample. Wait till the sample attains the temperature of 800°C and note the height of the sample. Start heating the furnace according to the programmed heating pattern in N₂ gas atmosphere.

6.2 Record the heights of the sample and the corresponding temperatures, the pressure being maintained constant during the entire test period.

6.3 Continue heating till a shrinkage of 25 percent of the original height of the sample takes place.

6.4 Then cool the sample in N₂ gas atmosphere. The sample may be used for further tests, if any.

7 EXPRESSION OF THE RESULTS

7.1 The temperatures corresponding to the shrinkage of 3 percent and 25 percent respectively of the initial height of the sample shall be taken respectively as the start and finish of softening of the sample pre-reduced to a particular degree of reduction.

7.2 The test shall be repeated on samples pre-reduced to various degrees of reduction.

7.3 A graph shall be drawn indicating the temperatures of the start and finish of softening at different degrees of reduction starting from 40 percent up to 90 percent with an increment of 10 percent. The behaviour of softening is characterized by two curves indicating the dependence of the softening start and finish temperatures on the degree of reduction.

METHOD I/SECTION 2

8 GENERAL PROCEDURE

8.1 A sample of Ore/Sinter/Pellet as the case may be (about 225 gm) is packed into a graphite container between two layers, one of coke bed at the bottom and other of Al₂O₃ balls at the top (Fig. 3). The packed container is fitted into a stainless steel tube arrangements for passing a gas mixture (30 percent CO, 70 percent N₂) from below, the line pressure of which is recorded with a water manometer. The packed column is introduced into a furnace after fitting to its top to another stainless steel tube and a graphite ram.

The sample is heated in a flow of reducing gas mixture (6 l/min) following a pre-defined temperature programme. The shrinkage, the inlet and outlet gas composition, the inlet gas line pressure and the temperature are recorded.

9 APPARATUS

9.1 Cylindrical Container

The container opens at both the ends and will be made of graphite (for example from broken arc furnace electrodes, etc.). The dimensions shall be:

\[ ID = 50 \text{ mm}, OD = 70 \text{ mm}, \text{ and } L = 100 \text{ mm}. \]

A perforated bottom plate having five holes (one at the centre), also made of graphite, fits to the graphite container to the lower stainless steel pipe as shown in Fig. 3.

9.2 Lower Stainless Steel Pipe

The SS pipe should fit tightly into the graphite bottom plate and should be long enough to extend outside the furnace tube in the ambient. It rests upon a rigid ground support and bears all the load. It has a side tube for inlet of reducing gas. The gas line is also connected to a manometer and its pressure is recorded throughout the test.

9.3 Upper Graphite Ram

After the Graphite Cylinder is fitted with alternate layers of coke/iron bearing material/Al₂O₃ balls to a height of 90 mm, a graphite pressure ram perforated axially like the bottom plate is placed over the upper Al₂O₃ ball layer. The central hole is for introducing thermocouple as shown in Fig. 3.

9.4 Upper Stainless Steel Pipe

This pipe should fit tightly into the graphite bottom plate and should be long enough to extend outside the furnace tube in the ambient. It rests upon a rigid ground support and bears all the load. It has a side tube for inlet of reducing gas. The gas line is also connected to a manometer and its pressure is recorded throughout the test.
10 TEST CONDITION

10.1 Sample Size
Pellets/Sinter/Ore of size 10-15 mm shall be used.

10.2 Load to be Applied
A load of 2 kg/cm² held constant shall be applied to the test sample by means of a static weight.

10.3 Heating Rate
Heating rate shall be same as in 5.3.

11 TESTING PROCEDURE

11.1 After fitting the bottom plate to the graphite cylinder a coke bed shall be kept on the bottom plate to a height of 10 mm. Over this coke bed will be filled, the test sample of stated size to height of 85 mm including the coke bed. The sample now should be covered by a 10 mm thick bed of Al₂O₃ balls. Having prepared thus, the graphite crucible is fitted to the lower SS cylinder and is introduced into the furnace from below. The lower SS cylinder is secured rigidly over a ground support. The height of the graphite crucible is adjusted so that it is placed in the uniform temperature zone of the furnace.

The graphite ram is then introduced and fitted into crucible as shown in Fig. 3.

A thermocouple is introduced into the central hole and rests over the Al₂O₃ balls layer.

To the graphite ram is fitted the upper SS tube as shown in Fig. 3.
11.2 Weight is kept or load is applied by lever or any other system at the rate of 2 kg/cm² on the SS cover fitted on the top of the upper SS tube.

11.3 Heating is commenced, after fitting the apparatus with the rate as given in 5.3 and the temperature is recorded continuously.

11.4 After the temperature of 350°C is reached CO/N₂ gas mixture (30/70) is passed through the lower SS tube, at the rate of 6 l/min. The pressure of the inlet gas is continuously recorded with a water gauge till the end of the test.

11.5 The outlet gas and the inlet gas are continuously analyzed either by collecting it in gas pipettes or more accurately by connecting an infrared gas analyser in series. The extent of oxidation of gas as measured from the difference of inlet and outlet gas compositions provides the extent of reduction, and rate of reduction at the time of collection of gas samples.

A pointer attached to the top SS cap moving against a scale indicates the setting due to softening during the course of reduction under load. This reading is noted at regular intervals till the end of the experiment.

The end of the experiment is indicated by the constancy of the outlet gas composition for a period of half an hour at the final temperature.

NOTE — $\frac{d\theta}{dt}$ can be calculated from the graph. Percentage reduction between 40 percent and 41 percent is calculated from the graph between reduction and time for $\frac{d\theta}{dt}$. From the reduction curve, the time (in minutes) to attain the degrees of reduction of 30 percent and 60 percent are determined. The reducibility index $\frac{d\theta}{dt}$ at 40 percent reduction, expressed in percentage per minute, is calculated as follows:

$\frac{d\theta}{dt} = \frac{33.6}{t_{60} - t_{30}}$

where

$t_{30}$ = time to attain 30 percent degree of reduction, min
$t_{60}$ = time to attain 60 percent degree of reduction, min.

METHOD 2

12 TERMINOLOGY

For the purpose of this standard, the following definitions shall apply.

12.0 Softening Start Temperature ($T_s$)
It is the temperature at which the gas pressure drop begins to show a sharp increase. The temperature at which the gas pressure drop reaches 1 KPa (100 mmWG) has been adopted to represent it.

12.1 Melt-Down Finish Temperature ($T_{mf}$)
It is the temperature at which the sample bed stops contracting. It is the temperature where pressure drop comes back to 1 KPa again.

Precautions

1. The length of the uniform temperature zone should preferably be about 2/3rd the length of the graphite crucible.
2. The crucible should be coated with lime on the inner wall at least above the test sample.
3. Fittings should be tight and ensured to check any leakage of gas.
4. The crucible should be positioned accurately in the uniform temperature zone inside the furnace.

Presentation of Results — Softening Characteristics of Iron Ore Lumps/Sinter/Pellets

<table>
<thead>
<tr>
<th>Time</th>
<th>Inlet Gas Comp</th>
<th>Outlet Gas Comp</th>
<th>Pointer Travel</th>
<th>Temperature</th>
<th>Inlet Gas Pressure</th>
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<tbody>
<tr>
<td></td>
<td>CO% N₂% CO₂%</td>
<td>CO% N₂% CO₂%</td>
<td>( contraction)</td>
<td></td>
<td>mmWG</td>
</tr>
</tbody>
</table>

$\frac{d\theta}{dt} = \frac{16 \times CO_2 \%}{22.4 \times Weight of sample}$ by O₂/(kg samp. min)

Graphs of temperature versus % reduction, carbon rate and inlet gas pressure are plotted to reveal and to compare the behaviour of sample during the test.

Precautions

12.2 Softening — Melting Range ($T_m - T_s$)
It is the temperature range of softening and melting.

12.3 Residual Material (RM)
It is the weight of the metal and slag which have not dripped at the end of the test, expressed as a percentage of the sample weight (corrected for the oxygen associated with the iron).

13 PRINCIPLE OF TEST

13.1 For a bed of given height of any solid material when heated, the height of the bed initially increases because of thermal expansion. This behaviour continues with increasing temperature, till a point is reached when the solid starts softening. This start of softening temperature is generally observed when the gas pressure drop in the bed reaches 1 KPa. Similarly as the material softens, the pressure drop of the gas...
passing through the bed begins to show a sharp increase. With the increase in temperature, softening increases leading to further increase in gas pressure drop till the maximum pressure drop has been attained. Further increase in temperature of heating, leads to a decline in the pressure drop and a point is attained where the pressure drop comes back to the 1 KPa again. This is the temperature where the melt-down has finished. At this temperature the bed height no longer declines and remains more or less unaltered. This test involves compacting the sample in the crucible inside an electrically heated furnace, heated to temperatures of approximately 1 600°C. The loading and displacement measurement device are mounted on the sample. The temperature is raised to 1 600°C as per programmed heating schedule. The sample temperature, degree of sample bed contraction and gases pressure drop across the sample bed are monitored continuously. The test is discontinued when the pressure drop in the bed reaches a level of 1 KPa (100 mmWG) again.

14 NUMBER OF TEST
Single test shall be carried out on the material in the size range of 8 to 10 mm.

15 SAMPLES
15.1 The total mass of the softening-melting test sample shall be approximately 500 g, crushed to a size according to 5.1.
15.2 The sample shall be placed inside a heat resistant crucible of 48 mm dia and compacted in order to keep the sample height of 150 mm. Vessel dimension ID = 70 mm, OD = 85 mm and length = 150 mm.
15.3 The sample shall be sandwiched between two layers of coke, at the top and at the bottom (Fig. 4).

16 HEATING
16.1 After placing the sample in the crucible, it shall be placed in the furnace. The heating schedule shall be as follows:
- 0-200°C, manual
- 200-900°C, 10°C/min
- 900-1200°C, 3°C/min
- 1200-1600°C, 5°C/min
16.2 Heating to 200°C shall be done under N₂ atmosphere to avoid oxidation of the sample on heating and atmosphere of 30 percent CO and 70 percent N₂ shall be provided for heating from 200°C to 1600°C.
16.3 Gas flow rate shall be kept at 15 l/min at NTP, during the heating schedule.

17 PRESSURE APPLIED
A constant pressure of 98 KN/m² at 900°C onwards shall be applied to the sample by a loading device.

18 APPARATUS
18.1 Crucible
The crucible shall be made of circular graphite. It shall be connected with the lower perforated plate, by means of a screw. The lower perforated plate shall be a graphite plate having holes that makes the test sample meltdown. A graphite stand shall support the crucible and also work as a guide pipe to feed the reduction gas from the bottom. A typical illustration of the crucible is shown in Fig. 5.

18.2 Furnace
It shall be electrically operated furnace with graphite resistance heating elements capable of raising the temperature of the furnace to an operating level of 1600°C. The heating schedule shall be monitored by an external temperature controller.

18.3 Loading Device
It shall be loading device with a capacity to load maximum 60 kg. It shall have an accuracy: less than ±1 percent.

18.4 Displacement Transducer
It shall be a linear type potentiometer that detects the movement length of loading base. The loading base shall be a base for moving the loading device up and down.

18.5 Sample Collecting Device
This device shall be composed of the lid, container case housing, the turn table and container, drive motor and lid lifting device.

The container to receive the specimen, separated in the direction of rotation shall be placed in the water cooled turn table. The container shall be made of SS 316 (Grade X20Cr25Ni20 as per IS 6911). The number of cells shall be 10.

18.6 Sieves
Eight to ten mm sieves conforming to IS 360 shall be used.

18.7 A typical illustration of Softening-Melt down apparatus is shown in Fig. 6.

19 PROCEDURE
19.1 Approximately 500 g of sample shall be put into the crucible and subsequently compacted to have the initial sample bed height of 150 mm. The sample vessel shall be placed at the centre of the furnace with the help of supporting stand. The sample shall be sandwiched between two layers of coke at the top and at the bottom. The sample shall be heated to the maximum
19.2 The progress of reduction of the sample shall be monitored by analyzing the inlet and outlet gases using an infra-red gas analyzer coupled to a recorder. The sample temperature, degree of sample contraction, the gas pressure drop across the sample shall be monitored continuously.

19.3 The temperature at which initially there is a pressure drop of 1 KPa shall be noted. The test shall be continued and all parameters monitored as the pressure drop reaches a maximum and then decreases. The test shall be performed until the pressure drop in the initial bed again reaches 1 KPa (100 mmWG).

19.4 The start of softening temperature shall be taken as the temperature where pressure drop of 1 KPa (100 mmWG) is achieved during the initial period of test.
19.5 The end of melt-down temperature shall be taken as the temperature where the pressure drop again reaches 1 KPa.

20 EXPRESSION OF RESULTS
The following data shall be reported:

a) The Softening Start Temperature \( (T_s) \),
b) The End of Melt-Down Temperature \( (T_m) \), and
c) Residual Material \( (RM) \).

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**Fig. 5 Crucible**
Fig. 6 Experimental Melt-Down Determination Apparatus
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Amendments Issued Since Publication

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