Iron Making and Steel Making
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Module – 04
Lecture – 20
Testing of Burden Material

In this lecture, we will discuss about the Testing of the Burden Materials.

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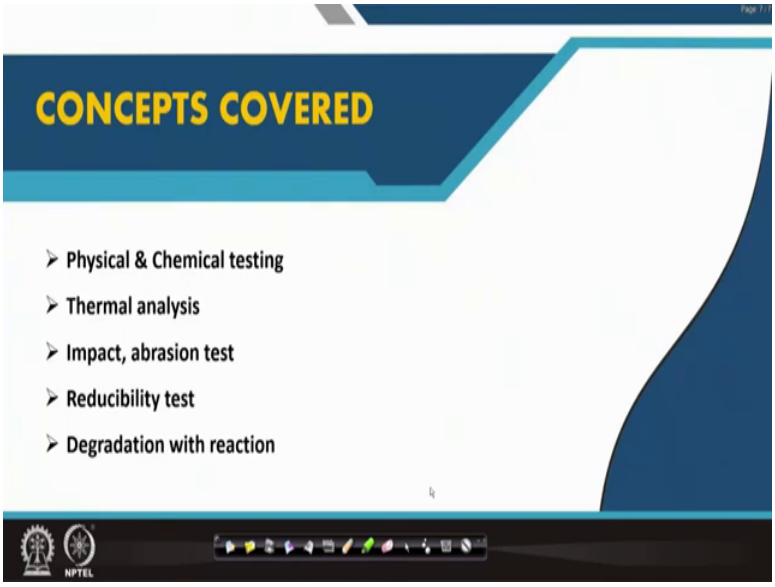

Topics covered will be physical and chemical testing, thermal analysis, impact and abrasion test, reducibility test as well as the test for degradation with reaction.

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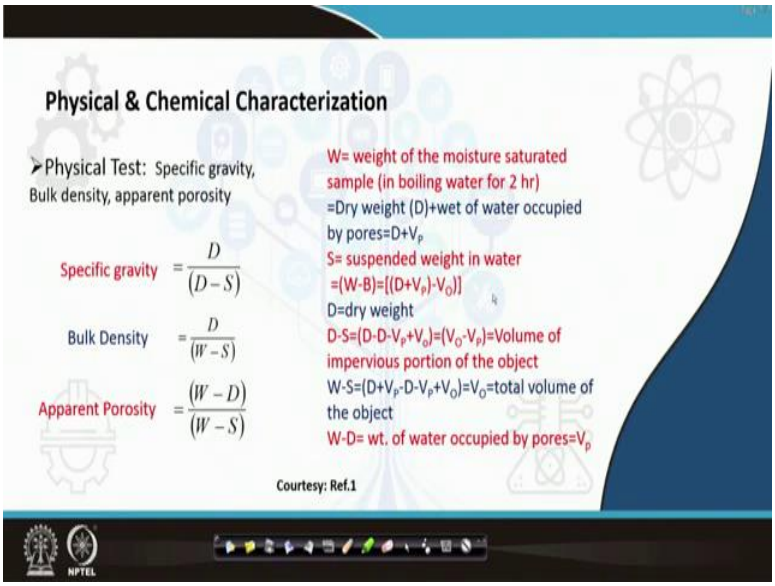

First, let us discuss the physical and chemical characterization of raw material and in this category the most important properties include the specific gravity, bulk density and apparent porosity. And, specific gravity is important for gangue separation in suspended medium in mineral beneficiation. Specific gravity is defined in terms of the density of an object divided by the density of some reference material, and it assess the relative weight between two materials. Water is taken as the reference material and water has a density of 1. So, specific gravity is basically the density of that material. And density of the material is defined as the dry weight of the material divided by the weight of the material excluding the porosity. In contrast, bulk density is defined as the weight of the material divided by the volume of the object including pores into it. So, if the porosity of the aggregate is less it is bulk density is more; if the porosity is more it is bulk density is less. For example, in coke making process during stamp charging, we take the raw mix and then compact it into a cake and their bulk density increases significantly. 30 – 40 percent increase in the bulk density is reported in the cake raw mix, finally leading to better quality coke.

Another important property is the porosity; it is defined as the volume of pores in an object divided by total volume of the object that gives the volume fraction of pores into that object and is called the porosity.

During measurement, it is difficult to assess the closed pores and only open pores could be measured and therefore, porosity is addressed as apparent porosity. Now, let us discuss a method, which can be used to estimate apparent porosity, specific gravity, bulk density of a material, based on dry weight (D), suspended weight (S) and moisture saturated weight (W). For measuring W, the object is immersed in boiling water and keep it for 2 hours such that the object gets saturated with the water; in other words all open pores are filled with water. The object is then taken off the water and surface moisture is rubbed off and its weight is taken and noted as W.

For measuring S, the object is hung into the water by string attached to a load cell. The reading in the load cell will give the suspended weight, S.

Now, W can be expanded as:

$$W = D + V_P \tag{20.1}$$

Where, V_P represent the weight of the water occupied by the pores, or, the volume of the open pores wetted by water. This pore volume, obviously, does not account for closed pores that is not accessed and wetted by water. The suspended weight, S can be expanded as:

$$S = W - B = (D + V_P) - V_O \tag{20.2}$$

V_O is the total volume of the object, or, the weight of the displaced water.

So, D-S =V_O-V_P = volume of the impervious portion of solid object ignoring the closed pores.

W-S = V_O = total volume of the object including the pores in it.

So, apparent porosity (ε) may be defined as:

$$\varepsilon = \frac{V_P}{V_O} = \frac{W - D}{W - S}$$

(20.3)

The bulk density (ρ) can be defined as:

$$\rho = \frac{D}{V_O} = \frac{D}{W - S}$$

(20.4)

Apparent specific gravity (γ) can be defined as:

$$\gamma = \frac{D}{D - S}$$

Specific gravity is a very useful knowledge for gravity separation of lighter gangue, bulk density is useful in case of the stamp charging and apparent porosity is useful because it gives a reflection how reactive a material is. For sinter or pellet, a large micro-porosity makes those more reactive by facilitating mass transfer through micro-pores.

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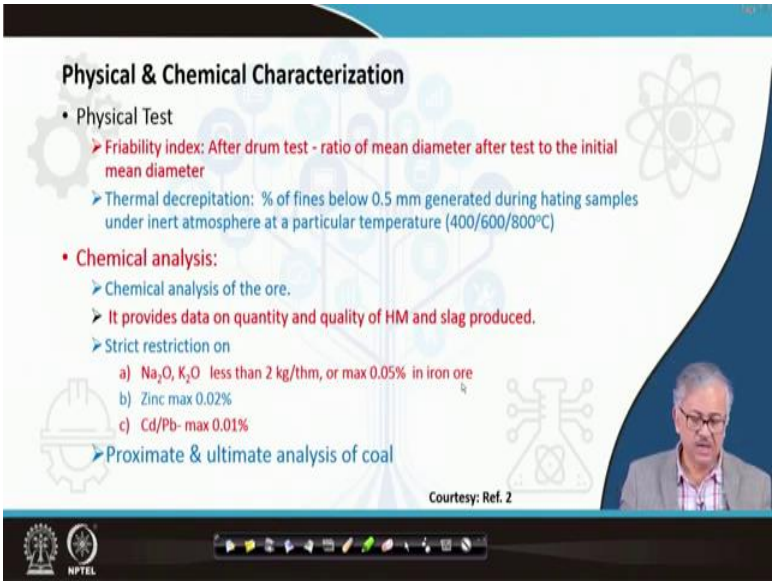


Now, Friability index and thermal decrepitation comes under physical tests.

The friability test is done in a rotating drum. Some amount of material is taken in a drum and rotated at a particular rpm for a certain amount of time. After that material is taken

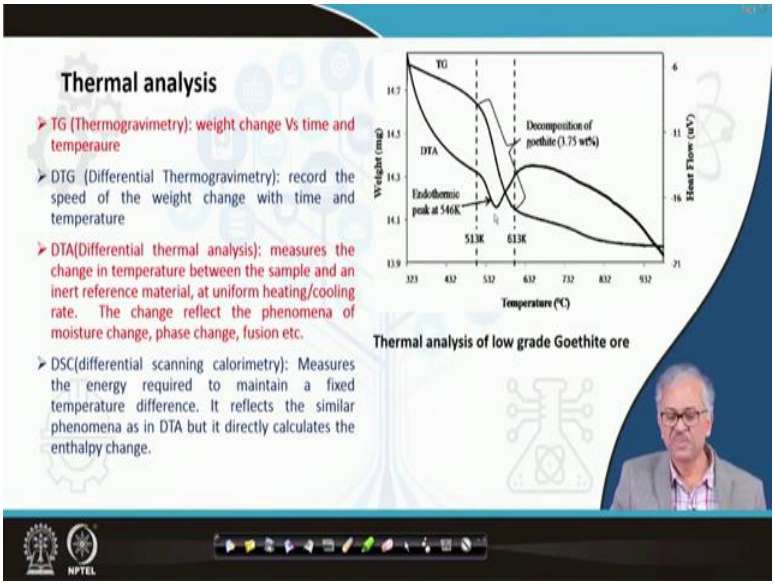
out, sieved and the mean diameter is estimated. Before the drum test also the mean diameter of the charged material is noted. The ratio of the mean diameters before and after test, is defined as the friability index. Obviously, if the ratio is close to 1, then material is very strong and lesser the ratio, more is the propensity of to produce dust. So, charge material with friability index close to 1, will yield better bed permeability in the dynamic bed of blast furnace. Thermal decrepitation means generating cracks in material with sound when heated. It is another important property of the burden material because several burden material decrepit at comparatively lower temperature in the range 400 to 800^{o}C in the upper part of the blast furnace. As a result they generate the fines under the action of heat.

Decrepitation index is measured by heating material in a retort under inert atmosphere. Retort is heated to 400 –800^{o}C and kept under inert atmosphere like N_2 or Ar and kept for some time. After the test, material is taken out and sieved. Fraction of material generated below 0.5 millimetre is defined as an index for decrepitation.

Chemical analysis is important because there are some strict restriction on alkaline oxide like sodium oxide, potassium oxide; those should be less than 2 kg per ton of hot metal or in the ore it should be 0.05 percentage maximum. Maximum allowable percentage of zinc in ore is 0.02%. Cadmium, lead also should be less than 0.01 because the cadmium, lead are very poisonous. Zinc vapor is not good because they may condense on the cooler walls at upper part of the furnace and form accretion.

Proximate and Ultimate analysis of the coal are important. While proximate analysis gives the percentage of ash, volatile, moisture and fixed carbon content of the coal; ultimate analysis provides the elemental analysis of the coal. Both these analysis are very important because for judging the coal for various process requirements. For coke making some

other tests like coal reflectance index (indicating caking property), dilatometry test for swelling of coal are also required. (Refer Slide Time: 14:37)



Next come to the thermal analysis and it is a very important analysis to understand different physical, chemical process a material undergoes during their heating.

Thermo-gravimetry (TG), Differential thermal analysis (DTA), and Differential Scanning Calorimetry (DSC), are some of the thermal analysis techniques regularly used. All these test are based on very small material (of the order of few mg), representing the whole sample. During TG, material is heated and its weight change is recorded against temperature. The weight loss during the process might indicate physical moisture loss, weight loss due to decomposition, or phase transformation.

Another form of thermo-gravimetry is called differential or derivative thermos-gravimetry. Sometimes by TG curve (weight loss versus temperature plot) may appear very gradual and may not separate different processes that is occurring. Recording speed of weight change at a particular rtemperature might separate those processes in terms of different peaks because different processes are likely to occur at different rates. For example, moisture removal may be little slow and phase transformation may be quite fast, which might reflect in terms of separate peaks.

In case of DTA (differential thermal analysis) two samples are taken; one is the test sample under consideration and another is the reference sample. Both the samples are provided heat at a particular rate per unit area (i.e., with the same heat flux) and the

temperature difference between the two samples are recoded. Reference sample should be very inert because they should not participate in any kind of reaction or phase transformation.

Now in absence of any physical or chemical phenomena, the temperature difference between the two samples would be more or less constant based on density and specific heats of the samples (ignoring temperature dependence of these properties between these samples). Now, if some process involving enthalpy change (moisture removal, chemical reaction, phase transformation) occurs in the sample, this will be indicated by some bulge in the curve representing sudden change of temperature difference between the sample and the reference. In DTA, the temperature difference between these two samples is plotted against the temperature; while both the samples are heated at the same rate per unit area. An endothermic decomposition can be indicated by a decrease in temperature in the sample; but reference will not show any change in temperature. So, temperature difference between sample and the reference will be indicated by a peak.

Similar to DTA, differential scanning calorimetry (DSC) also can be used to indicate such physical and chemical changes in sample. In DSC, the temperature difference between the sample and reference is kept constant by supplying additional heat needed by the sample for moisture removal/chemical reaction/phase transformation etc. So heat flow to the sample and reference differ but temperature difference is maintained constant. In addition to identify the enthalpy related processes, it also provides the amount of enthalpy required to carry out those changes. This is why it has an edge over DTA.

Let us consider the application thermal analysis of goethite decomposition (Figure 20.1).

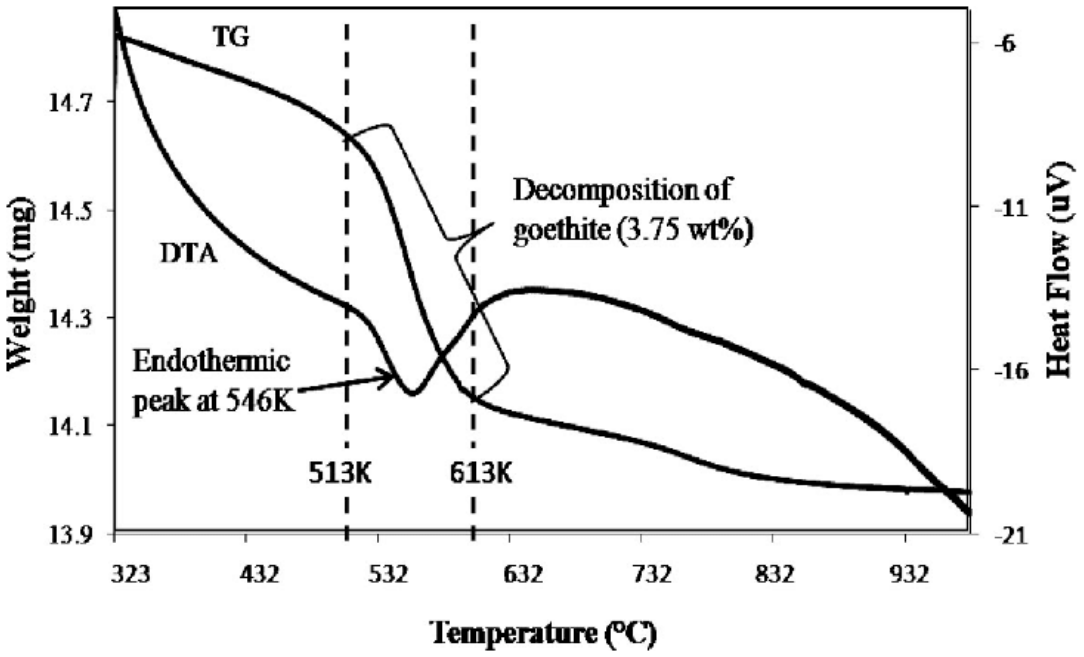
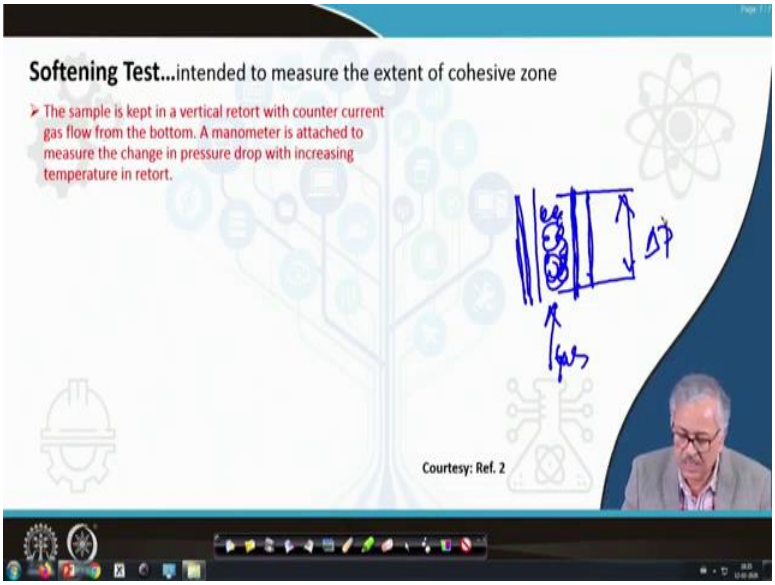


Figure 20.1 Thermal analysis of goethite decomposition

Initial decrease in TG curve as well as DTA curve may be attributed to removal of physical moisture. Between 513 K to 613K, a gradual yet sharper drop in TG curve is observed. DTA curve also shows a sudden decrease followed by increase showing a peak at intermediate temperature between 513K and 613K, which may be attributed to goethite decomposition releasing the chemically bonded water in the temperature range 513 to 613K and reaching a peak decomposition rate at an intermediate temperature corresponding to the peak of DTA curve. (Refer Slide Time: 22:04)



Now, the softening test. It is intended to measure the extent of the cohesive zone. In softening test, some measured amount the sample is taken in an electrically heated retort with a perforated bottom to facilitate gas injection from the bottom through the sample. The test is done non-isothermally, by monitoring the pressure drop across the sample with increase in temperature. The schematics of pressure drop against temperature is shown in Figure 20.2.

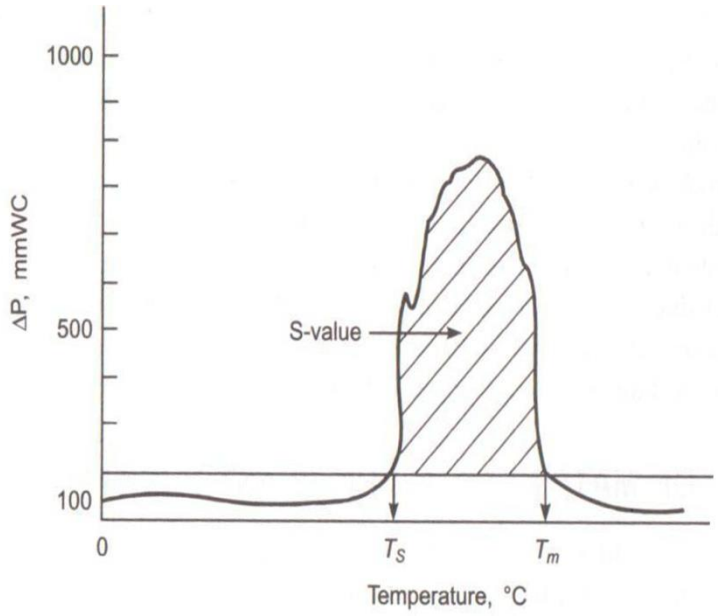
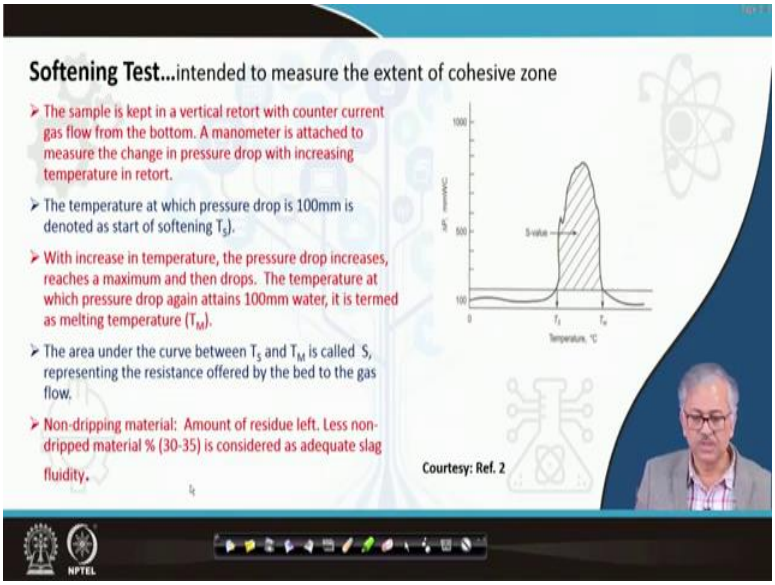


Figure 20.2 Schematics of pressure drop against temperature

It may be observed that initially there is no appreciable change in pressure drop with temperature up to the point of start of softening of the sample. In this initial period of heating, there is no physical change in the sample that can influence flow resistance through the sample bed. But beyond the temperature when the sample starts softening, the resistance through the sample bed increases and pressure drop reaches a maximum

followed by a decrease, as the liquid flows out of the sample bed. (Refer Slide Time: 25:02)



So, from this curve you can have two measurements. First is the softening range; start indicated by sudden increase in pressure drop and the end of softening indicated by the point where pressure drop arrives to its original value. The area under the curve as indicated by hatched line represents the extent of flow resistance through the bed. It will vary from material to material because it is the material softening characteristics. If the softening range is low the area under the curve will also be low. Besides, the height of the curve will also depend on the viscosity of the softened material. More viscous material will provide high peak pressure drop and higher area. The second parameter is the amount of non-dripping material, which can be assessed from the amount of reside left over after the test. This residue will never melt. So, this is called the non-dripping material, which remain solid after the test. Such non-dripping material will increase the viscosity of slag in the blast furnace. Non-drip material with 30 to 35 percent is considered to maintain an adequate slag fluidity in blast furnace. So, if the non-dripping material estimated less than 30 to 35 from the test, it may be considered okay.

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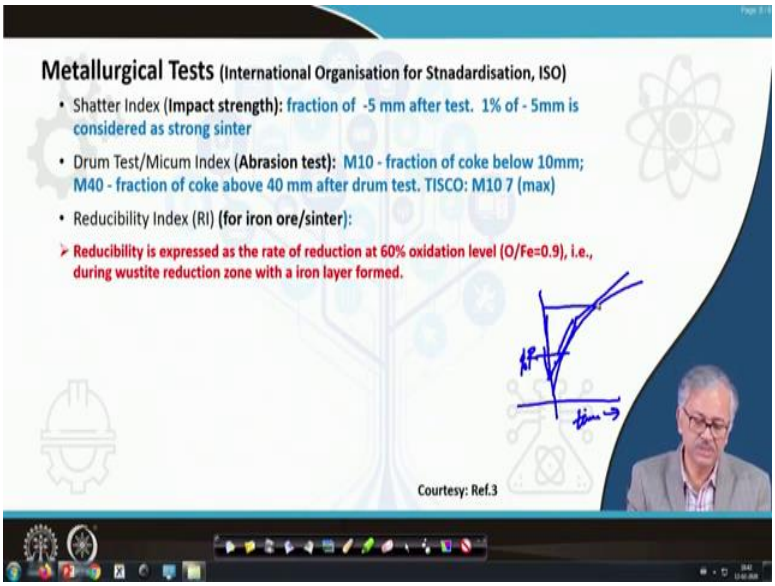

Some metallurgical tests that are the based on international organization of standardization (ISO test) are described now. First test is called the shatter index. It measure an impact strength of the material.

Certain amount of burden (say ore) material is taken and then lifted to a certain height from the ground and then allowed to free fall on a prescribed ground for certain amount of times. After the test, solid residue from the test is sieved and the fraction of minus 5 millimeter particles is defined as the shatter index and 1 percent of minus 5 millimeter is considered as a strong sinter.

Drum test of coke provides the micum index. In drum test certain amount of coke is taken in a drum, which is subsequently rotated at a particular rpm for a certain amount of time. After the test, sample is taken, and sieved. Minus 10 millimeter fraction is defined as M10 and fraction of plus 40 millimeter is defined as M40. Maximum allowable M10 is around 7, as par TISCO data.

Ore reducibility Test: The reduction rate become critical after iron layer is formed from wustite reduction, because iron layer brings in another kinetics step (diffusion through product iron layer) in series in addition to existing outside film resistance and chemical kinetics at the reaction interface. Reducibility represents the speed of oxygen removal from ore, which varies with time, or oxidation state of the iron. Reducibility is usually measured at 60% oxidation level (O/Fe=0.9), or 40% reduction level at 900^{o}C.

Since in blast furnace the wustite reduction takes place in the isothermal zone at 900°C and it also attains equilibrium in the upperpart of isothermal zone, reducibility is usually measured at 900°C. In one of the commercial method, % wt loss during reduction in 30%CO (70%N_2) at 900 °C for 3 hrs is reported as Reducibility Index. Obviously during 3 hrs of reduction, reduction mostly represents in the wustite reduction regime.

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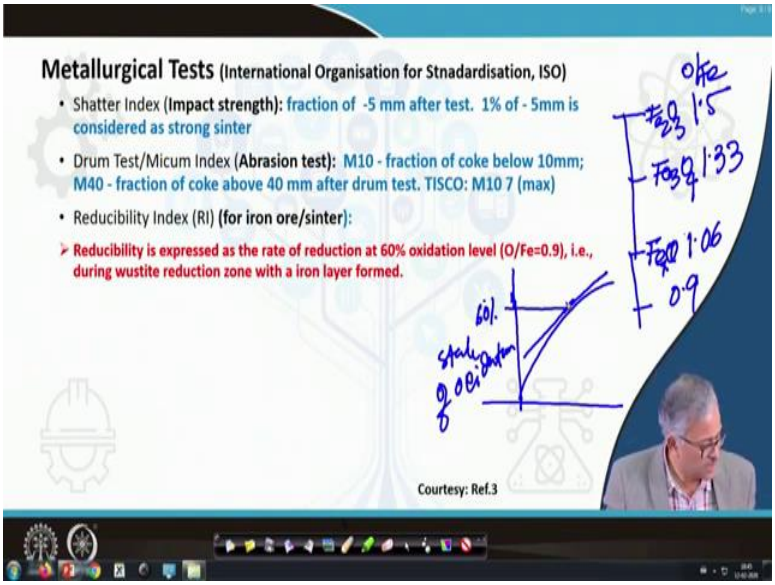

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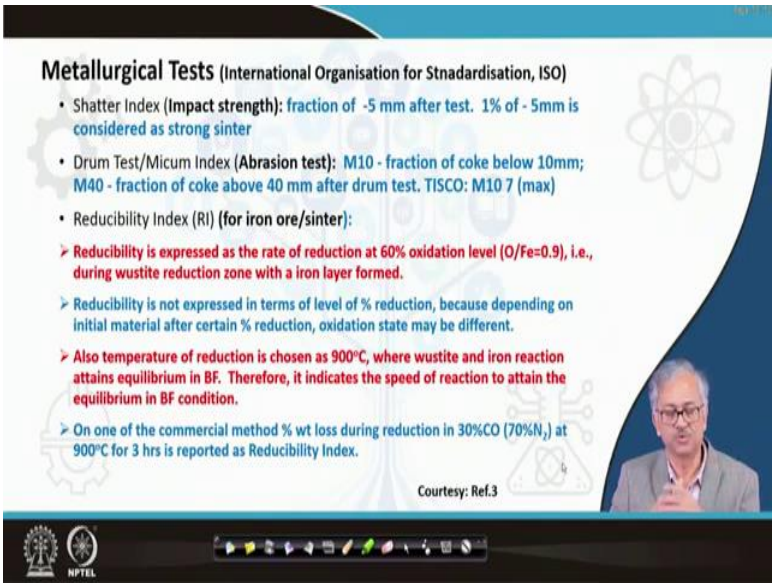

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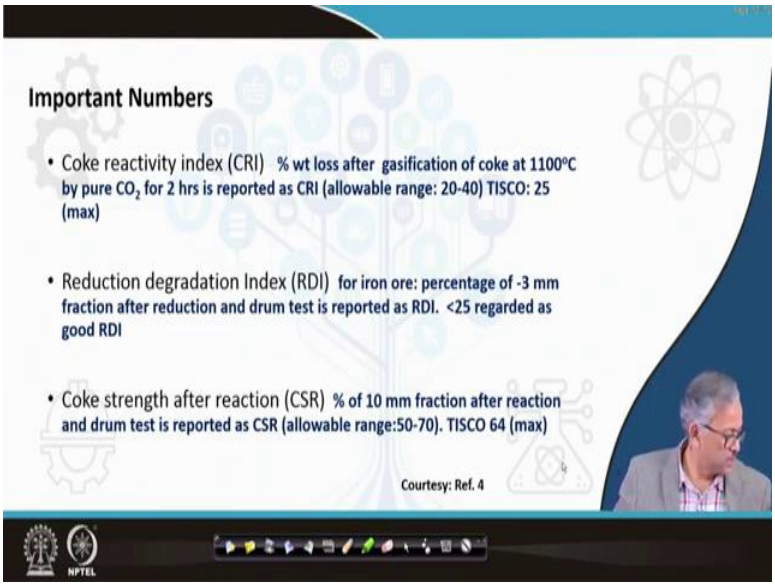

Coke reactivity index (CRI) is defined as the weight loss after gasification of coke at 1100°C by pure carbon dioxide for 2 hours. Reduction degradation index (RDI) is mainly used for the iron ore. In this test, iron ore sample is reduced in reduction atmosphere (30% CO, 70% N_2) for 3 hours followed by a drum test. After drum test the solid residues is sieved and the percentage of minus 3 millimeter is represented as the reduction degradation index (RDI). RDI less than 25 is considered as a good sinter.

Similarly, in estimating coke strength after reaction (CSR), coke is subjected to gasification followed by a drum test and percentage of plus 10 millimeter coke surviving the test is defined as the CSR. Allowable range of CSR is 50 to 70. TISCO CSR is around 64.

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And, these are the references.

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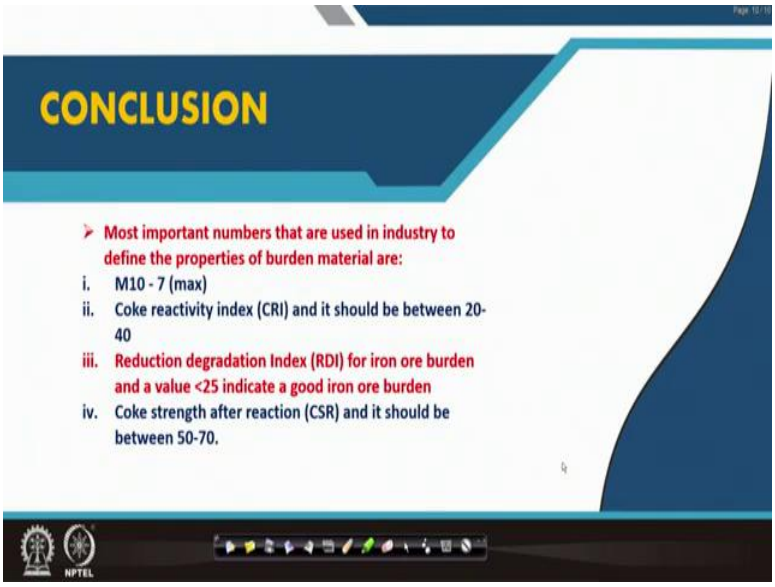

Conclusion: The important numbers to represent coke strength are Micum number, CRI and CSR. M10 should be maximum 7 and allowable CRI range is 20 to 40. Allowable range for CSR is 50-70.

RDI is the most important index to represent the strength iron ore under blast furnace condition and it should be less than 25.