CERAMIC REFRACTORY TECHNOLOGY - I

Introduction of Refractory-

Most of the industries which require high temperature operations depends on refractory materials or bricks made by refractory industries. Among these iron & steel industry is the greatest consumer of refractories. It takes away about 70-75% of the total production of refractories.

India has excellent and huge deposits of raw materials of refractories. In the industrialization of a country, refractory plays an important role. Steel forms its back bone. All metallurgical industries, glass, enamel, pottery, whiteware, cement, power generation etc. ultimately depends on the supply of refractories.

Refractories are the materials which protect industrial equipments (generally kiln or furnace) and vessels. From industrial point of view, refractory materials are those which do not soften by fusion at working temperature. The important features of refractories are excellent resistance to heat and thermal shock, chemical attack (corrosive action of slag or molten mass, fluxes or furnace atmosphere), resistance to abrasion by the charge or flue gases, moving solid particles or molten mass, dust laden gases at high temperature.

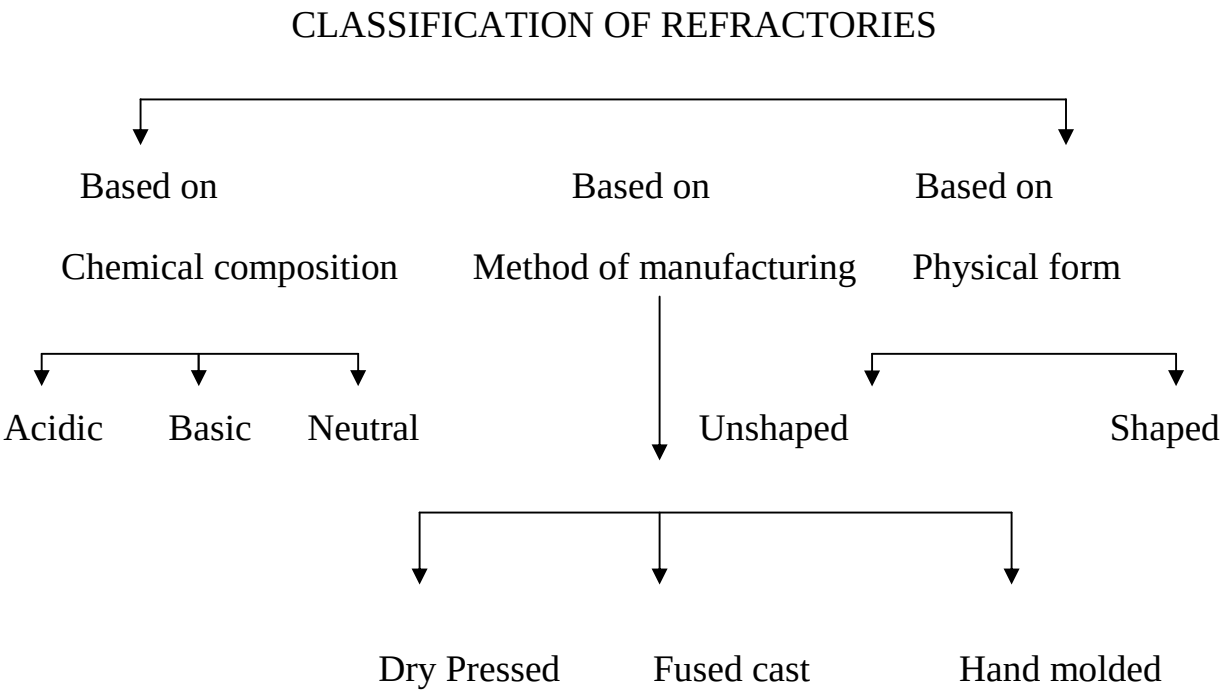
Definition of Refractory-

Refractories are those materials which are hard to fuse. The refractories are those materials which can withstand high temperature without fusing or deformation in shape and size, resist the action of corrosive liquids and dust laden currents of the hot gases.

In technological term “refractory” referred to the materials used to make furnaces, kilns, stoves, driers and critical parts of aircraft, jet engines and aerospace craft operating at high temperature.

Classification of Refractories-

Refractories are classified on the basis of chemical composition, method of manufacture\ing and physical form as shown below-



Classification based on chemical composition-

Refractories can be classified on the basis of their chemical behavior i.e., their reaction to the type of slag. Accordingly, refractory materials are of three types namely Acidic, Basic and Neutral.

1. **Acidic refractory**- Acidic refractories are those refractories which are not attacked by acidic slag (alkaline), but attacked by basic slag (alkalies). These are used in places where slag and atmosphere are acidic.
2. **Basic Refractory**- Basic refractories are those which are not attacked by basic slag's ,dusts and fumes at elevated (high) temperature but attacked by acidic slag.

These have high coefficient of thermal expansion but not much resistant to thermal shock, hence they are not used generally for intermittent (batch) kilns.

These are used in furnance where environment is basic .eg – non-ferrous metallurgical operation, managesite, Dolomite, Magnesite – chrome. Chrome- magnesite refactory etc.

3. **Netural Refractories -** These refractories are neither attacked by acidic slag's nor by basic slag's.

   These are stable to both acids and bases and are used in areas where slag and atmosphere are either acidic or basic.

THESE REFRACTORIES INCLUDES-

1. Various forms of carbon (graphite, charcoal and coke), Chromite.
2. Artificial or synthetic refractories like-zirconiurn carbide, titanium carbide and silicon carbide.
3. Metals used as refractory like iron, copper, molybdenum, nickel, platinum, osmium, tantalum, thorium, tungsten, vanadium and zirconium,

Classification based on method of manufacture

Another way of classifying Refractories is by the manner in which they are shaped. The Refractories can be manufactured by either of the following methods.

1) Dry pressed Refractories - these are those which are pressed with the help of hydraulic or mechanical press in dry state.

2) Hand molded Refractories - these are molded in wooden moulds in plastic condition.

3) Fused cast Refractories- these are those which are first melted and then casted in desired shapes.

Classification based on physical form

Refractories can also be classified according to physical form. These are

Shaped Unshaped Refractories. The former is commonly known as refractory bricks and the later as "monolithics" Refractories.

i) Shaped Refractories

Shaped Refractories are those which have fixed shape. These are also called wall bricks. Bricks shapes can be standard shapes and special shapes. Standard shapes have dimensions that are conformed to by most refractory manufacturers and are generally applicable to kilns and furnaces of same type. Whereas special shapes are specifically made for particular kiln and furnaces. This may not be applicable to another furnace or kilns of same type.

ii) Unshaped Refractories

Unshaped Refractories are without definite form and are only given shape upon application. These are categorized as Plastic Refractories, ramming mixes, castables, gunning mixes, fetting mixes and mortars.

Insulating refractories- A refractory suitable for minimizing heat losses and thus achieving heat conservation in the furnance is called insulating refractories.

They have high porosity, low thermal conductivity and high thermal insulation properties. These are produced from asbestos, fire clay, kieselguhr, etc. at low temperatureslagwool. Glasswool and vermiculite are also used as insulating materials. For hightemperature thermal insulation applications foam ceramics and ceramic fiber and wool areused.

Cermet refractories- These refractories are a combination of ceramic materials)eg-oxide,nitride, borides etc) and metallic or metallicalioys materials. It has the combination of goodproperties of both metallic and ceramic materials. Eg-high strength and resistance to hoghtemperature cerets are used in nuclear reactors, missiles and space crafts etc.

Special refractories- These are very expensive refractory materials used for making crucibles and furnances for special/experimental purposes where cost of refractory is no consideration. They are not very common due to their manufacturing limitation.

Special refractories includes pure oxides (eg:- magnesia, silica, alumina, berylia, thoria etc.), borides, nitrides, Silicides, carbides etc.

Other special refractoriers are Sialons, zircon, carborundum, alundum (a mixture of fused alumina and clay), sillimante, lectrocast blocks of mullite, magnesia and mixtures of chromite, bauxite and magnesia.

III) Properties of Refractory materials

1 Refractoriness

It is the ability of a material to withstand high temperature without getting deform or getting fused. It may also be called fusibility of material. This temperature can be found with the help of pyrometer or in terms of the pyrometric cones, called pyrometric cone equivalent (P.C.E.) value. The refractoriness of few pure refractory materials are given in table.

Table – Refractoriness of some pure refractory materials.

Refractory material	Refractoriness
Graphite carbon pure	3600 ^{o}C
Thoria	3000 ^{o}C
Lime(CaO	2570 ^{o}C
Alumina(Al2O3)	2050 ^{o}C

Fireclay(25-30%Alumina)	1650 ^{o}C
Fireclay(35-40%Alumina)	1720 ^{o}C
Silica	1710 ^{o}C
Mullite	1810 ^{o}C
Silicon Carbide	2240 ^{o}C

The refractoriness serves sufficient basis for considering thermal stability of refractory material. This indicates the maximum temperature up to which we can use any refractory material. In labortary, P.C.E. of a refractory is determined by comparing the softening of a test pyramid cone with that of standard pyramid cones of known fusion points under similar conditions. The number of standard pyrometer cone in terms of ASTM cone no. and their corresponding softening point are given in table -

Refractoriness of Standard Pyrometric Cones

ASTM CONE NO. (STANDARD)	**REFRACTORINESS(^{o}C)**
18	1522
19	1530
20	1540
23	1560
26	1646
27	1659
29	1665
31	1683

32	1710
33	1730
34	1785
35	1804
36	1820
37	1835
38	1855
39	1875
40	1900
41	1940
42	1980

2 Refractoriness Under Load (R.U.L.)

It is the property of refractories by virtue of which it resists the combined effects of heat and load lying on this. R.U.L. is of more practical significance than its refractoriness. When the refractories are used in furnaces or kilns in various industries the refractory brick may face the action of various types of mechanical load, tension, compression, scratching, abrasion and also thermal shock. The bottom row of bricks in furnaces or kiln may experience very high pressure due to furnace wall.

The RUL is usually tested under load of 2 kg/cm^2 for dense refractories such as fire bricks and 1 kg/cm^2 for insulating (porous) bricks by heating a test brick or block at a gradually rising temperature in RUL furnace. The temperature at which the test specimen starts to deform or sag and eventually fail usually due to shearing is called RUL.

When a refractory test cone is heated, its lowest melting constituents fuses first. Some fused mass therefore is present in it. long before the fusion point of the refractory cone is reached and that the test cone bends and flattens only

when the amount of fused mass has increased considerably at the expense of the solid and more refractory grains. When a slight pressure is applied during heating of the refractory cone then in that case grains will be forced to slide or slip as soon as some fused mass is formed to separate the solid grains thus resulting in the failure of test cones. So the refractory materials should have either high refractoriness or low fusing constituents (glass) to meet prevailing furnace conditions. The refractoriness in general are made up of two, constituent compounds, fail under load conditions at temperature much lower than the fusion point of the highest melting and predominant (major) constituents. So the RUL of a refractory is much lower than corresponding refractoriness. For example refractoriness of silica brick is 1710 ^{O}C where as RUL is 1650-1660 ^{O}C at 2 kg per centimeter square.

The failure of refractory is also influenced by time of its exposure to heat and load. Even refractory containing small yet viscous fused glass will also fail with their prolonged exposure even at safe load and temperature conditions. Like ways increased load or temperature will cause failure at much lower temperature.

3 Porosity

It is the percentage relationship between the volume of open pore space and the total volume of the refractory. The porosity of refractories determine other properties like slag resistance and spalling resistance. The pores in a refractory material may result as a deliberate addition of various additives. These additives include saw dust, cork and other combustible material. The porosity can also be developed by foaming methods. Increasing the porosity normally reduces the strength.

Porous refractories have high permeability and hence poor resistance to penetration of molten slag, metal and flue gases. Porous refractories act as a good insulator. They have low strength and cannot be used for tall structures.

Porosity is of two types

1. Apparent porosity
2. True Porosity

1. Apparent Porosity

Apparent porosity is the percentage relationship between the volume of open pore space and the total volume of the sample. It can be determined by formula

$$\text{Apparent Porosity} = \frac{W-D}{W-S} \times 100$$

Where D = the constant weight of the Dry Sample
S = the weight of specimen suspended in water
W = the weight of specimen in air including the moisture in its open pores.

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2. True Porosity

True porosity is the percentage relationship between the volume of combined open and closed pore space and the total volume of the sample.
The porosity of insulation brick can be as high as 80%. Fire bricks have porosity of 15-28% . High density products have low density of 2-5 %

4 Density

Is defined as mass per unit volume**.** There are two types of density,

1 Bulk density
2 True density

1 Bulk Density - is the weight per unit volume of refractory sample including volume of open pores space. This factor is very important for overall weight

lying on the foundation of a refractory structure. It is the factor which limits the size of furnace. The furnace structure made by non-porous refractories should be stronger than structures made up of porous refractories to bear the heavy load lying on it.

Bulk density can by determined by the formula

$$\text{B.D.} = \frac{D}{W-S}$$

2 True density - is the ratio of weight of material to its true volume containing open and closed pore space.

Bulk density is generally considered in conjunction with apparent porosity. It is measure of the weight of a given volume of refractory. For many refractories, the bulk density provides a general indication of the product quality. While evaluating a refractory brand or comparing several products of equivalent type(except insulating type), it is considered that the refractory with higher bulk density(generally with lower porosity) will be better in quality. The structure of a refractory having higher bulk density will be denser, resulting in better resistance to chemical attack, decreased metal penetration, better abrasion resistance and other related benefits. Bulk density of some refractories are given in table

TABLE Bulk density of Some Refractories

Name of refractory	Bulk Density in gm/cc
Silica	1.73
Chrome magnesite	1.93
Fire clay	2.00
Magnesite	1.93
Dolomite	2.58
Fused alumina	2.90
Semi-Silica	3.00

4. Permeability

Permeability of a material is the volume of the fluid which will pass through one centimeter cube of a material under a pressure of one cm of water in one second. Refractories which come directly under the influence of gases and liquids should be impermeable. It will eliminate the leakage of gases and penetration of liquids through the walls of a furnace or refractory structure. There is no direct dependence of permeability on porosity. Uniform permeability is an indication of absence of internal cracks.

6 Spalling resistance

It is also called thermal shock resistance or thermal fatigue resistance. It is defined as the fracture of the refractory brick or block. It is the ability of refractories to retain their original form without cracking splitting or flaking when subjected to sudden changes in temperature.

A refractory may spall due to different reasons:-

1. Failure may occur due to temperature gradients due to uneven heating or cooling of refractory. This sets up stresses causing failure (thermal spalling). This type of failure is common in silica, magnesite and chrome refractories .
2. When refractories are made from non-uniform raw materials having variations in CTE.
3. Structural changes occurring in refractory during service can result in fracture. This can be controlled by proper mixing of raw materials, burning refractories at higher temperatures and preventing slag penetration as far as possible.
4. On heating refractory its grains expands. This result in compression of structure of refractory. This may result results in failure due to shear. Due to

this reason refractory made from materials having low coefficients of thermal expansion and coarse textures, have increased resistance to sudden changes in temperature.

5. Diffusivity also influences spalling resistance. Greater the diffusivity greater will be the spalling resistance and vice-versa. Diffusivity leads to equalization of temperature and hence less temperature gradient. Fireclay, zirconia, high alumina have better spalling resistance than that of silica, magnesite and chromite refractories.

7 Slag resistance

It is very important property of refractories and depends on the nature of slag and refractory which comes in contact with each other. There are two processes of refractory destruction. One is corrosion i.e., chemical reaction and other is erosion i.e., process of breaking and washing away of refractory materials by molten slag or molten metal. The destruction of refractory depends on many factors, which are as follows

1. The temperature and temperature gradient.
2. The chemical and mineralogical composition of slags and lining.
3. Porosity of the lining and sizes and shapes of its pores.
4. Lining is wetted by the slags or not.
5. Viscosity of the slags.
6. Composition of the gaseous atmosphere in the furnace.
7. The rate of reaction between slag and lining.

At high temperature slag always attacks refractory. Slag action also occur when slag material is capable of forming new compounds with refractory materials. The film of slag in contact with the refractory dissolves some of the refractory. Its composition changes (may be to very small extent).this results in lowering or rise of melting temperature of slag film. If its melting temperature is raised the films becomes highly viscous, sticky, inactive and incapable of dissolving the refractory any further. It protects the refractory

wall as long as slag films does not get detached from the refractory surface. This viscous film gets diffused into slag very slowly and diluted by fresh slag. It becomes less viscous and fluid enough to further attack the refractory. On the other hand when the melting point of the slag films gets lowered by the dissolved refractory, the films becomes more fluid. Due to this its rate of diffusion into slag increases and hence it is replaced by fresh slag rapidly. This results increase in slag attack. Acidic refractory addition to acidic slag and basic refractory addition to basic sag increases the melting point. However Basic refractory addition to acidic slag and acidic refractory addition to basic slag lowers melting point. So basic slag attacks acid refractory and acidic slag attack basic refractory very rapidly. A neutral refractory is very useful because it resists both acidic and basic slags roughly to same degree

8 Strength Cold **crushing**

Cold crushing strength of a refractory material represents its strength or how strong the brick is. In other words it tells how much load it can withstand in cold condition. It is the load in pounds per square inch or kilogram per square centimeter, at which the refractory breaks. Dense and fine grained refractories have good crushing strength. However porous and coarse grained refractories have poor crushing strength.

It indicates the maximum combined load (charge and refractory lining) a refractory can withstand. So this property puts restriction on capacity and size of equipment.

9 Abrasion resistance

The abrasion resistance of a refractory is very important especially when refractory lining comes in contact with moving charge which rubs against it and subjects it to wear. E.g. blast furnace. Abrasion resistance largely depends on hardness and the bonding between particles of the refractories. So

under these conditions the refractories must be dense, fine grained and wear resistant refractory lining to ensure the resistance to abrasion. The abrasion resistance diminishes if the surface of refractory lining is softened.

10 Erosion resistance

It is the measure of resistance offered by a material to the chipping off the particles from it. Erosion takes place when molten metal or gas carry dust and slag particles. When these slag and dust particles strikes against refractory lining it results in chipping off particles from it. Splashing of slag erodes the refractory walls whereas slag particles carried along by flame or gases erode the arches and bends of the structure in particular.

11 Thermal expansion

It is the internal property of the refractory products to expand on heating and contract on cooling. As you can deduce, this property determines the structural stability of equipment. Can you think of how? This is so because refractories are closely packed in equipment with little space in between. If given refractory expand or contract more than expected, it can lead to failure of structure.

12 Modulus of rupture (MOR)

It is the flexural breaking strength of a refractory. It is measured at room temperature and expressed in kilogram per square centimeter.

13 Thermal conductivity

It is the ability of a material to conduct heat and is measured by coefficient of thermal conductivity. Thermal conductivity depends upon the chemical and mineralogical composition of refractories and porosity. As the porosity increases the thermal conductivity decreases. The refractories having air entrapped in their pores behave like insulator.

Refractories having low thermal conductivities are used in melting furnaces to ensure least heat losses. However refractories used in heat recuperators should Have high thermal conductivities to ensure maximum heat transfer. Most refractories are poor heat conductors, but graphite, magnesite and silicon carbide are good heat conductors. Insulating materials have low thermal conductivity.

14 Electrical conductivity

Refractories should have high electrical conductivities when these are used in electrical furnaces as heating elements e.g. silicon carbide. Graphite and metals are good electrical conductors among refractories. Other refractories are electrical insulators. The refractory bused in electrical furnace lining should have very low electrical conductivity. Graphite is used as electrodes either in prebaked form or as lining in high temperature electrical furnace. Electrical conductivity also depends on the porosity and porous bodies are less conductive.

15 Thermal expansion

It is the internal property of refractory products to expand on heating and contract on cooling. As you can deduce, this property determines the structural stability of equipment. Can you think of how? This is so because refractories are closely packed in equipment with little space in between. If given refractory expand or contract more than expected, it can lead to failure of structure.

16 Price

Cost of refractories is one of the important parameter in selection of refractories. It should be low as possible. A costlier refractory is having longer life may prove to be cheaper in the long run than a refractory with lower initial cost.

Students after understanding different parameters and properties of refractory materials, you have fair idea how to select a refractory for particular application. But the selection process is not so easy. For example we want to

have material having high abrasion resistance as well as high slag resistance and at the same time high insulation power. For such application you have to choose refractory material having high porosity and high bulk density. Is it possible? Certainly not. For refractory to have high bulk density, refractory must have low porosity. This is so because both are inversely related. Can you think of more such examples?

From above it is clear that properties of refractories play important role in its selection for particular application. So during manufacture of refractories its properties are carefully determined to ascertain its value and to control the variation. Do you know how these properties are assessed? Testing is the tool to assess these properties. Unit No 5 will deal with various methods of testing finished products.

METHODS OF MANUFARACTURING OF REFRACTORY-

1. **Grinding-** Firstly rew materials are ground, so their proper size of material are obtained. Ratio of coarse to fine particles should be even. This ratio is normally maintained at 55:45, equipments used for this purpose are varius types of crushers, pulverisers, hammer mills, ball mills, shaking tables and screens etc.
2. **Pre-treatment-** The main pre-treatment given to refractory raw materials is its firing/calcinations at high temperature for considerable periods of which bring about the complete mineral conversion and hence stabilize the material. The constituents of the finished refractories production between the solid particles of raw materials are rarely completely in equilibrium because it is seldom feasible to actually melt and cast these refractory materials. Natural silica mainly containing quartz which is not stable at high temperature, but undergoes transformation to its allotropic forms tridymite and crystoballite involving high volume changes.

Thus raw silica expands considerably when heated to high temperatures and prestabilization of silica becomes necessary.

3. **Chemical composition –** The chemical composition of the refractory should be such that the surrounding donor chemically attacks the refractory and corrode it. Acidic refractory should not be used in a furnace heating a basic material. Otherwise the brick will react with the furnace stock and corrode, hence it will reduce.
4. **Mixing-** Ground refractory material is mixed with binding material in such a way that plastic materials are equally distributed throught the mass to facilitate easy molding. Mixing is usually carried out in pug mills for even distribution of fine and coarse particles in the whole mass. Precalcalculated amount of water, additives, binding materials and mineralizes are added and the mass is mixed thoroughly to ensure a product of uniform composition and uniform distribution of fine and coarse grains plastic and bond materials.

   For easy moulding and development of useful properties in the fired refractories. To ensure infreased plasticity for easy moulding, the mixture is allowed to remain as remain as such for a day or more.

   Mixing can be dry, semi-plastic or wet type 14-20% water is used wet mixing which are mostly used for hand moulding. Semi plastic mixtures have lower percentage of water to achieve semi-state of plasticity where as the dry mixture include non-plastic basicmixes and clay mixes containing <5% water, since mixing whit so little water is difficult, wateras a fine spray or mist is used to achieve proper mixing due to requirement of high moulding pressures, semi-plastic and dry mixture are machine pressed. Clay mixtures are also obtained in the form of colloidal solutions suitable for moulding by casting.

5. **Moulding –** it is done mechanically by applying high pressure or by hand to infrease density and strength of the refractory.

Moulding -which following mixing refractory materials is done either by hand or machine (by pressing or extrusion). Hand moulding is carried out in wooden boxes and is cheaper than machine on a jobbing basis. However, machine moulding has more practical applications and is cheaper for mass production of standard refractory shapes. Machine moulded refractories have higher strength and density that hand moulded refractories.

Machine moulding can be used for semi-plastic mixture using moderate moulding pressure.

Extrusion is usually used to get the shapes of approximate dimension which are subsequently pressed to exact shapes. Machine moulding of dry mixture requires a pressure of about 1000kgh/cm2 or more.

Density and strength of the refractories increases on de-airing due to decrease in laminations and cracks. Dnsity increase is due to elimination of air of a lower density than the refractory material and or closing of voids.

The method used for moulding refractories of hollow and other special shapes is slip casting in which a colloidal suspension is poured into a plaster of paris moulds which absorbs the water and causes deposition of uniform layer of clay on the walls of mould on deposition of desired thickness of clay, the extra mixture in suspension is poured out of the mould resulting in hollow refractories with irregular interval contours.

Power pressing is used for moulding refractories involving dry compacting or slightly damp refractory power mixture in metallic dies

using sufficint high pressure to produce strong and dense refractory shapes.

6. **Drying-** it is done at slow rate to avoids and high shrinkage. Drying of moulded refractory increases its green strength by removing moisture and making them safe for subsequent handing. Drying is usually carred out under shade on large drying floors. Floors heated by waste heat from kilns are also used where refractories are laid out in open arrays.

For faster drying of refractory of constant and size, tunnel kill are used where raw refractories are stacked in bogies or placed on belts moving through a tunnel against a stream of hot air.

For drying heavy refractories of inticated shapes, controlled humidity driers are specially used Drying rate of fire brick s should be very low to avoid cracks formations as they are liable to maximum shrinkage on drying.

7. **Firing/Burning-** Bricks are burnt in kiln (down draught kiln or tunnel kiln) to remove water of hydration, vitrification and development of stable mineral forms. Shrinkage in volume upto 30% occurs during burning. Burning or firing of refractories which following their drying facilitates developments of stable mineral forms in them and high crusing strength of finished products.

Degree of firing of silica bricks is indicated by its gravity. High specific gravity of 2.5 indicates the presence of considerable amount of unconverted quartz in the refractory where as a specific gravity 2.32 to 3.37 indicates adequate firing and transformation of quartz to crystobalite and tridymite.

Bricks firing are done either in down draught, batch type or tunnel kilns. These killnfiring is time consuming due to involvement of their stage operation namely brick charging firing and cooling.

FIRING TEMPERATURE OF SOME TYPICAL COMMON BRICKS AS FOLLOWS:-

Sr.No	Refractory type	Firing temperature C
1.	Fire clay bricks	1250-1400
2.	High alumina bricks	1400-1550
3.	Silica bricks	1450-1510
4.	Chrome bricks	1450-1650
5.	Magnesite bricks	1450-1650
6.	Direct bonded basic brick	1650-1760
7.	Silicon carbide bricks	1370-1510

Sr.No	Principal Cosistuents%	SIO2	AI203	Fe203	Cr203	Mg0	Ca0
1.	Fire bricks	50-70%	25-38%	2-5%	-	0.5-1%	05-1%
2.	Aliminious Fire bricks	50-55%	38-45%	2-4%	-	0.5-1%	0.5-1%
3.	Semi silica	75-90%	8-15%	1-2%	-	0.5-1%	0.5-1%
4.	Silica bricks	94-95%	0.5-2%	0.5-1.5%	-	0.5-1%	2-2.25%
5.	Sillimanite bricks	25-30%	55-65%	0.5-1.5%	-	0.5-1%	0.5-1%
6	Magnesite bricks	1.5-5%	1-4%	2-8%	-	84-92%	2-5%
7.	Chromo bricks	3-10%	10-25%	12-25%	35-45%	15-35%	2%
8.	Chromomagnesite bricks	3-10%	5-15	8-20%	20-35%	40-60%	1-4%
9.	Doiomite bricks	12-15%	2-3%	2-4%	-	38-42%	38-42%
10.	Periclase	3.0%	1.0%	3.0%	0.5%	98.0%	2.5%
11.	Foresterit bricks	33.5%	1.0%	0.1%	0.5%	54.5%	1.0%

COMPOSITION OF VARIOUS REFRACTORY BRICKS:-

Selection of Refractories- The selection of the refractories for any particular application is made with a view to achieving the best performation of the

equipment furnace, kiln or boier and depends on certain properties of the refractory material for a given application will be determined by the type of furnancer or heating unit and the prevailing conditions. eg- the gaseous atmosphere the presence of slag's, the type of metal charge etc. Therefore, temperature is not only the criterion for selection of refractories.

Any furnace designer or industry should have a clear idea about the service conditions which the refractory is required to face. The furnance manufactuers or users have to consider the following point's before selecting a refractory.

1. Area of application.
2. Working temperatures.
3. Extent of abrasion and impact.
4. Structrual load of the furnance.
5. Stress due to temperature gradient in the structures and temperature fluctuations.
6. Chemical comparability to the furnance environment.
7. Heat transfer and fuel conservation.
8. Cost considerations.

OCCURRENCE OF RAW MATERIALS FOR MANUFACTURING OF REFRACTORIES-

1. The raw material for fire bricks is fire clay. It occurs in the coal firlds of Jharkhand , West- Bengal, Madhya Prasedh, Orisa, Andhra Pradesh.
2. Sillimanie occurs in kjasi hill in Assam , near Pipra village inMadhya prasesh, near the village Pohra in the bhandara district of Maharashtra, as beach sand in Traven core in Kerla State.
3. Kyanite occurs in Chhota Nagpur in Jharkhand.
4. Silica exist in the form of quartzite deposits in Bihar sharif and Monghyr distracts of Bihar Lotaoahar and Brajmade in Orisa.

5. Magnesite occurs in chalk Hills in Salem district and Dodkanya in Mysore state, Idar in Gujrat, Doongarpur in Rajasthan, Almora district of U.P.
6. Chromite of taminadu, sighbum of Jharkhand, ratangiri in Maharashtra and ladakh in J&K.
7. Zirconia is available in the form of Ziorconium minerls, which occurred as beachsend in kerla.
8. Carbone and Graphite are found in Orisa, Bihar and Karnataka

Manufacturing units of Refractories in India

The various manufacturing units in India are as follows

1. Tata Refractories Limited, Belapur,Orissa.
2. Bharat Refractories Limited, Marar, Ramgarh, Jharkhand.
3. Valley Refractories Limited, Chirkunda, Dhanbad, Jharkhand
4. Miathan ceramics Limited, Chirkunda, Dhanbad, Jharkhand.
5. Hind Gulf Refractories, Gandhinagar, Kutch, Gujrat.
6. Prajapati Refractories, Thangadh.
7. Carborrundum Universal Limited, Ranipet, Tamilnadu.
8. Monolithic Refractories, Isanpur, Ahmadabad.
9. Vision Refractories Private Limited, Mumbai.
10. Thermo Technologies Private Limited, Delhi.
11. Alwar Refractories Private Limited, Alwar , Rajasthan
12. OCL india Limited, New Delhi
13. PINC Group, Kilpauk, Chennai, Tamil Nadu.

14. Grind well Norton limited, Devanhalli Road, Bangalore, Karnatka.
15. Reliable Refractories Private Limited, Bhilwara, Rajasthan.

These are names of major Industries. Most of the industries are located near the places of occurrences of their major raw materials.

UNIT-2 TESTING OF REFRACTORIES

1. **Refractoriness-** It is the ability of a material to withstand high temperature without getting deform or getting fused. It may also be called fusibility of material. The temperature can be found with the help of pyrometer or in terms of the pyrmetric cones, called pyrometriccone equivalent (P.C.E.) value. In this test softening characteristics of are compared with standard pyrometric cone in a furnace having or oxidizing atmosphere.

Method:-

1. Take one kg of refractory to be tested.
2. Crush in roll jaw crushers to a size 5mm.
3. Ground in a porcelain or agate and passed through 20 micron I.S. sieve.
4. Pass ground material through magnetic separator to iron particles.
5. Mix the material thoroughly with required amount of water and alkali free dextrin or glue.
6. Make the cones with the help of mould of the shapeof a terrahedron with 8 mm sides on the base and 25mm height.
7. Sintered the cones at 1000 deg Celsius for easy heanding.
8. Arrange the test cones and the standard pyrometric cones of know softening temperature on plaque with the help of some bonding material as show in figure 1. This material should not react with the cones and reduce their fusibility. Standard cones are chosen keeping in view the anticipated fusion temperature of the test cones.
9. Fix each cone on the plaque forming angle of 82 degree with the horizontal.

10. Keep the plaque in a suitanle furnace either gas fires or electrically heated as show in figure 2. The flame should not directly strike the cines and furnace atmosphere should be oxidizing of netural.

Table 2 : Heating Schedule

Room Temp. to ASTM Cone No.	Time interval (Min.)	Cumulative time (Min.)	End Point (^{0}C)
20	45	45	1621
23	16	61	1640
26	7	68	1646
27	7	75	1659
29	8	83	1665
31	10	93	1683
31.5	6	99	1717
32	7	106	1724
32.5	3	109	1743
33	7	116	1763
34	9	125	1785
35	9	134	1804
36	7	147	1820
37	7	148	1835

(xii) Observe the softening of cones through a peep hole. It is indicated by top bending over of the cone and tip of the cone touching the plaque surface as shown in figure

Figure 3 Progressive bending of cones in fusion point furnace (P.C.E.) furnace

(xiii) Record the softening point of the standard Pyrometric cone which most nearly corresponds in time of softening with the test cone as the P.C.E. of the test cone .If the test cone softens in between two standard cones than the P.C.E. of the test cone is taken as the mean of the two cones.

Sometime instead of bonding the cones with the plaque self supporting cones can also be used as shown in figure 4

2 Refractoriness under load (RUL)

RUL is a measure of a resistance of a refractory product to deformation when it is subjected to combined effect of load, rising temperature and time. It is the softening temperature of a refractory under loaded conditions corresponding to breaking of refractory brick.

METHOD-

1. Take a specimen of refractory whose RUL is to be determined.
2. Obtain a cylindrical shape having diameter 50 ± 0.5 mm and height 50 ± 0.5mm by cutting, boring and grinding the central portion of refractory being tested. One of the face of specimen should be original faces of the refractory sample taken forming one of the faces of the finished test specimen.
3. Check that the refractory specimen must be free from cracks and other macro defects.
4. Heat the sample in an electric furnace consisting of a refractory tube of 100 to 120 mm internal internal diameter,120 to 150 mm outer diameter

and about 500 mm length. This tube can be of corundum, magnesite or mullite as shown in figure.1

5. Ensure to keep central 100 to 120 mm length of tube is placed in the hottest zone of the furnace.
6. Place the sample in the central portion between carbon or mullite rods with about 5mm thick carbon plates in between sample and rods.
7. Apply the load of 2kgf/cm^2 is applied with the help of rods.
8. Heat the furnace at the rate of 15^0C per minute up to 1000^0C and at the rate of 8^0C above 1000^0C.
9. Plot the change in height is against the time on rectangular co-ordinates. As the temperature is raised at approximately constant rate and change in height is plotted against the time , the chart will give temperature deformation curve.
10. Measure the temperature with the help of an optical pyrometer. It is sighted obliquely through a 20mm (max) ID radial tube or adjusted upon the bottom of refractory tube closed at its bottom and suspended in the furnace at the beginning of the test at about middle of the specimen . This is tightly closed by total reflecting prism interposed for giving allowance for diminution of intensity There should not be more than 30^OC variation in horizontal plane.

11. Change in the height of the specimen on heating is plotted against temperature on 10:1 scale to represent temperature deformation curve for the test. The typical curve for some of the refractories is shown in figure.

12. From the plotted curve, note the temperature (T_a) corresponding to the point at which the curve has dropped 3mm below its highest point(denoted by bend of curve downwards with respect to horizontal tangent).The temperature (T_e) cooresponding to the point at which the height of the tse specimen has decresed by 20mm from its original height is also noted. The

temperature (T_b) at which premature breaking of the test specimen, without its actual softening takes place denotes the breking ponit of the test specimen.

3. Resistance to chemical attack

Chemical attack on refractories takes place due to slags, gases like carbon monoxide and glasses etc. at the high working temperature.

1 Resistance to slag attack:-

The resistance to slag attack can be determined by one of the following methods:-

A Running slag test:- In this test specimen refractories in the form of single brick or column of bricks are heated to a uniform temperature. The temperature is near to the temperature at which refractory is finally to be used. Then the powdered slag dropped regularly on top of refractory. The slag run from here in groove downward provided for the purpose. At the close of test the depth of groove is again determined. The enlargement of groove determines the resistance of the refractory to slag attack. This test gives excellent results. However, this test cannot be used to determine quantitative results.

B Spray test:- (In this test refractory sample is maintained at uniform temperature. The powdered slag is then sprayed on heated refractory surface with the help of revolving burner.)

The slag powder is thus impinged uniformly and under similar temperature and other working conditions over the entire surface of the refractory under test. The decrease in thickness gives the slag resistance.

C Molten bath slag test:- This is another test to determine the slag resistance. In this test the refractory to be tested is kept in bath of molten slag. The slag is stirred to expose the refractory surface to fresh slag. The decrease in dimension of brick gives the measure of slag resistance.

2 Resistance to carbon monoxide attack:-

Carbon monoxide have disintegration effect on refractories. This effect depends on amount of ferric oxide present in refractories. The carbon monoxide gas is generated by reaction of formic and sulphuric acid or by the reaction of charcoal at 1000 . You must purify the nitrogen and carbon monoxide for carbon, oxygen and water vapors. Measure the flow rate carbon mono oxide with the help of manometer and a flow meter in the circuit.

METHOD-

Cut two specimens cut in cylindrical shapes of 50 mm length and not less than 30 mm diameter. One specimen is cut from interior of refractories and other from exterior of other refractory shape. You can cut specimens in prismatic or rectangular shape.

Place the test specimens in wire wound electrical furnace.

Purge the furnace with nitrogen.

Heat the furnace at 450 and pass purified carbon monoxide through the furnace at the rate of 2 litres per second.

Carry out the test for 100 hrs or when refractory gets disintegrated if it occurs earlier. Examine the test pieces regularly for discoloration, carbon deposition and disintegration that may take place during the course of test.

Record the observations for each of the two specimens.

Maintain the test temperature between 450 to 500 .

Note the time at which disintegration or deposition of carbon monoxide takes place. This is taken as measure of resistance of refractory to carbon monoxide.

4. Permeability:-

It is a measure of rate at which a fluid flows through a porous body. It is denoted by the volume of gas or air in cc that passes through one centimeter refractory cube at a unit pressure difference(gm/cm sq.) per minute. Schematic representation of the permeability determination apparatus is shown in figure.

Pass the purified air through a cubic refractory sample of known surface area and thickness. Fix the sample in mercury seal. The pressure difference is measured by manometer. Note the Volume of air passed in a given time. Then Permeability of refractory sample can be calculated by using the following formula:-

$$\text{Permability} = \frac{V.H.}{A.\ t.\ \Delta p}$$

Where V= Volume of air passed through specimen(cc)

H= thickness of sample(cm)

A= cross sectional area of the sample(sq.cm)

t= time for which air is passed through the sample(min)

Δp= pressure difference across the two surfaces of vthe sample(gm/sq.cm)

5. APPARENT POROSITY

Apparent porosity can be determined by two methods

A Boling point method

Cut the test specimens of size 6.5cm×6.5cm×4cm with the help of cutting wheel. The sample should be cut from interior of the brick

Remove the any loose adhering particles.

Dry the sample in oven at 110□ to a constant weight. Note it as 'D'.

Suspend dry sample in distilled water. The sample should not touch sidesor bottom of the vessel.

Boil the specimen in suspended condition for two hrs and the cool it.

Take its weight in suspended condition and note it as ‘S’.

Remove the specimen from water.

Wipe of extra water with blotting paper

Weigh in air and note it as ‘W’

Then calculate apparent porosity (P) by using following formula: -

$$P = \frac{W-D}{W-S} \times 100$$

Where W-D= actual volume of open pores of the specimen (CC)

W-S= external volume of the specimen (CC)

Evacuation method

Cut the test specimens of size 6.5cm *4cm with the help of cutting wheel.

Cut the sample from interior of the brick and remove any loose adhering particles.

Dry the sample in oven at 110^0C to a constant weight. Note this weigh asdry weight ‘D’

Place the dried sample in empty vaccum desicator.

Evacuate the desicator to a pressure less than 25 mm of Hg.

Then admin the immersion liquid (water or farction of liquid paraffin dolling above 200^0C)

Place the sample in such condition for 2-3 hrs under reduced pressure.

Allow the air to enter the desicator.

Takes its weight in suspended condition with the help of siling thread. Note it is suspended

Weigth’S’

Remove the specimen with the help of sling slow. Wipe of extra water with blotting paper

Weight in air and it as saturated weight ‘W’

Then calculate apparent porosity (P) by following formula:-

$$P = \frac{W-D}{W-S} \times 100$$

Where W-D =actual volume of open of the specimen (CC)

W-S = external volume of the specimen (CC)

6. Bulk Density

Bulk density can be measured by either of two mwthods

1. Direct measurement method
2. Direct volume measurement method

A Direct measurement method:-

This method is used to determine the bulk density of regular shape refractories. First determine the volume of refractories by measuring its dimensions directly. Then take its weight. The bulk density is then determined from the formula.

$D_B = \frac{W}{V}$(g/cc)

Where W = weight of specimen in grams

V= volume of specimen in cc.

B Direct volume measurement method:-

This method is used to determine the bulk density of the refvractories which are having intricate shapes. In this method value of dry weight (D), suspended weight(S) and saturated weight (W) is determined. The same procedure is used, which was used for apparent porosity determination. Then calculate the bulk density by using the following formula:-

$D_B = \frac{D}{W-S}$(g/cc)

Where D = Dry weight of sample (in gram)

w-s = external volume of specimen(in cc)

7. True Specific Gravity and True density:-

This test is conduvted when materials does not dissolve in or attacked by water.

1. Take two test specimens of refractory to be tested
2. Cut two test piecese to a size from interior of refractory
3. Crush the piecede to a size not exceeding 3 mm.
4. Mix and reduce the crushed sample to 50 gram by coning and quartering
5. In case of pre ground material, draw 500 gram of representative sample and reduce to 50 gram by coning and quartering.
6. Grind cone material in agate so that it pass through 149 micron IS sieve.
7. Remove magnetic fraction with the help of magnetis separator introduced during crushing and grinding.
8. Dry the material to constant weight at 105-110^0 C
9. Take 8-12 gram of sample and place in glass stoppered weighting bottle.
10. Take a pycnometer dry it at 105- 110^0C, coll in dessicator and note down itsweight as ‘WP’
11. Fill the pycnometer with distilled water at room temperature place the stopper.
12. Wipe the extra water outside and note down its weight as ‘W’ empty the pycnometer, dry it and with material sample from weighing bottle. Weight it and not it as ‘W’ with stopper in position.

    fill half of pycnometer wit distilled water and boil it for 10-15 minutes. This is done to avoid loss of sample due to popping.
13. Then fill the remaining part of pycnometer with distilled water and cool room temperature in water bath.

    Place the stopper in position and wipe of extra water,

    Weigh the pycnometer and note its weight as ’ w_2’

    Calculate specific ravity and true density by using following formula

True Specific Gravity = $\frac{weight of\ sample}{weight\ of\ equal\ volume\ of\ water}$

$$\frac{w - wp}{w - wp - (w2 - w1)}$$

True density = true specific gravity X (d_w - da)g/cc

$$\frac{w-wp\ (dw-da)}{w-wp\ -(w2-w1)}\text{g/cc}$$

Where $d_{w\ and}$ d_a are densities of wqater and air repectively at the temperature at which..

Test is conducted.

8. Cold crushing strength:-

CCS is determined by standard hydraulic or mechanical compression testing machine show in figure. Various steps involved are

1) Cut the test specimens from refractory shapes preserving the original surface as far as possible. Sixe shuld be kept equivalent to size of 230 mm standard brick. If smaller and other special refractory shapes the test Specimens the smaller size are used. In this case sample size can be cube with side of 75 mm are used.
2) Keep the specimens between two rams (bearing block having plane surface of size equal to or more than of specimen) of machine. In between ram and specimen 5mm thick asbestos fiber or cardboard are kept. As show in figer.
3) Switch on the machine to move the rams. The machine indicates the pressure in terms of tonnage on pressure gauge. The pressure will be show foe every moment.

4) Note down the pressure when the brick fails. At this point indicator returned back to zero.

The value of machine gives the value of CCS. At the time off failure, cracks will appear on the surface of sample.

The assembly used in test is show in figure-

9. Modulus of rupture test (MOR):-

Failure of refractory brick generally takes place due to bending moments. In practice failure of brick due to crushing rarely takes place.MOR test is used to determine resistance offered by brick to bending moments. This test is performed with the help of standard mechanical or hydraulic compression machine as show blow:-

To perform this test cut the test specimen of size 22cmX6.5cm or 7.5cm. the specimen is cut from desired refractory shap. Place the specimen on the bearing edges of the machine. These edge are positioned 18 cm apart. Apply the load at the middle of the specimen uniformly. The rate shuld be 10 kgf per minute (±10%) in case of mechanical pressing machine. Load shiuld be applied without any jerk. Note the load (W) at which the specimen fails. The use following relation to calculate MOR.

$$\text{M.O.R} = \frac{3WL}{2BR2} \text{ KGF/SQ.CM}$$

Where L = the distance between bearing edges (cm)

B= width of specimen (cm)

T= thickness of specimen (cm)

10. Permanent liner change (PLC)- is important to determine Because it indicates the volume stability i.e, expansion or shrinkage, or the refractories takes place during service (use as lining). It also indicates reversible thermal expansion i.e., the material expands on heating and contract on cooling due to phase transformation during use. PLC and reversible thermal expansion value is used in designing refractory lining for providing joints. PLC is can be determined by two methods.

1. **Permanent liner change on reheating (PLC) test;-** This test is carried out in the laborary furnace. The sample is heated to particular temperature for particular time. This temperature and time is different for different refractory materials. The test results can be reported in two ways.

Liner change:- The PLCR is expressed as percentage of increase/decrease in length as under

$$\text{PLCR \% (LINER)} = \frac{increse-decrease\ in\ length}{original\ lengt} \text{ X } 100$$

Volumetric change:- The PLCR is expressed as percentage of increase/decrease in volume as under

$$\text{PlCR \% (volume)} = \frac{increase-decrease\ in\ volume}{original\ volume} \text{X } 100$$

MOTHOD-

Cut test specimen of size 5X5X12.5cm by cutting wheel. In case of smaller refractory shap, the specimen of largest size is cut.

Retain maximum possible original face.

Determine its volume and place it is kiln having oxidizing atmosphere. Keep the specimen is such a way that flam does not imping directly on specimen. The laggest face of the specimen should rest on a supporting refractory brik drawn from same lot of refractories that are under test.

Place fused alumina or kyanite of – 85 micron I.S sieve between supporting refractory and test specimen. Keep the specimens 4cm apart.

Heat the test specimen according to set time – timeperature schedule of particular refractory.

Hold the refractory for given time at maximum temperature as per stipulated time.

Cool the refractory specimen in the kiln itself in 10 hours.

Measure its dimensions again and calculate its volume.

Ix Calculate PLCRby using following formula:=-

$$\text{PLCR \% (LINER)} = \frac{FINAL\,LENGTH - ORIGINAL\,LENTH}{ORIGINAL\,LENGTH} \text{ =x100}$$

$$\text{PLCR \% (LINER)} = \frac{FINAL\,LENGTH - ORIGINAL\,LENTH}{ORIGINAL\,LENGTH} \text{ =x100}$$

2 **Dialtometer method of PLC:-**

This is method is used to measure liner change of refractory of refractory sample with temperature, Dialtometer assembly is show in figure-

In this test a sample is cut in shape of cylinder or bar. Clean the sample from any loose dust particles. Place the cut specimens on supporting tube in such a way that its other end is free to expeand. This free end is joind to

core of an electronic displacement transducer. When sample is heated change in length take place. This make the free end to move and which in true pushes the ceramic rod. This rod pushes the core of the trandsducer. Record the deflection in millivolt. This deflection is proportional to the change in length of specimen. Time to time standardization of dilatometer is done with the help of material of known coefficient of thermal expansion. From this correction factoris determined for expansion which takes place in ceramic tube and carrierrefractory on heating along with specimen, Dialtometeric analysis enables you to determine structure changesand percentage of different phase formeddue to transformation on heating. These changes involve changes in volume e.g .% cristbellite, tridymite and quartz in a silica brick on heating to desired temperature.

Expansion characterstics:-

Expansion characterstics is very important property for the refractories. It is very essential to know about refractories where it will expand of contract during heating ensure the stability of it.

Expanding refractories are not suitable for the most applications. Expension sets up stress causing blowing and bursing of lining. Firebrick, chrome magnesite, forsterite, mangnesite and alumino silicate refractories shrink in service. Silica refractories expand on heating due to critical transformation. Expansion of refracatories depends on composition and temperature. Expansion of refracatory also various from batch to batci and brick to brick. Ordinary or thermal expansion value is very important in furnacedesign. This help for providing expansion joins. Expansion factor also determine thermal shock resistance.

Firebricks have low and uniform expansion up to 1000C. The ≤ 0.5% or less. Silica bricks behave differently. Tridymite shows two irregularties, while cristoballite and quartz show one irregulaty in thermal expansion as show in following figure.

The thermal expansion of managesite refractory is comparatively high 1.3-1.4 % where as that of chorme or chorme managesite brick is much lower except in reduscing atmosphere. Normal fireclay refractories have low thermal expansion of the order of 0.5% up tp 1000^0 C Reversible thermal expansion of some refracatories is show in following figure-

Thermal conductivity test.:-

Thermal conductivity depends on chemical and mineralogical comoposition, glassy phase and application temperature . it does not affect performance but its value determine the thicknese of lining can you guess what should be its'svalue high or low? Well it depends on application. In some application its value should be high e.g.h. in recuperators, regenerators, muffles etc. where heat transfer is required. However where heat conservation is required its value should be low. Thermal conductivity can be determined by calorimetric method developed by CGCRI Calcutta as show in following figure-

This method was developed by C.G.C.R.I. Calcutta. This method is very useful for measurement of conductivity at very low temperature. This method was specifically useful for measurement of thermal conductivity of cold face (low temperature) low insulation bricks. In this method one of brick is heated with the help of hot plate. At the other end is circulated through calorimeter. The inlet and outlet temperature is measured. This gives the rate of heat transfer passing through brick. The water flow is so such that the temperature of cold and is constant.

Spalling resistance:-

Spalling resistance is determined by one of the two standards methods:-

1. Panel test
2. Small prism test

Panel Test

Procedure for determination of spelling by panel test method

Cut 12 to 14 test specimens from original refractories by cutting wheel. The size of specimen should be of 65-70mm thick, 230 mm long and 113mm wide. If size of original brick is 230mm in length then take original shapes for testing.

Label with ceramic paint on the inner face which is not exposed to heating. Each specimen is then dried at 105^0c and weighed.

Then lay test specimens in the penel in the panel frame by kaolin or any other suitable material having PCE value not less than that of refractory specimen.

Selected Material not react with refractory specimens.

Ensure joints of specimens less than 2 mm thick.

Back the test panels are with suitable insulation to allow heat loss of approximately 605 kca/m^2/hr(0_C/m) under operating conditions. Its mean temperature should be about 700^0 C.

Preheat that panel assembly gas or oil fired furnace. Maintain face temperature at 1600c (avarge) for 24 hrs. raise the temperature of preheating i.e. up to 1600^0 C in 5 to 8hrs.

Record the condition of samples after preheating and take photograph for record.

Remove the insulation from panel and transfer the samples in spalling furnace with the help of track or trolley.

Heat the samples in spalling furnace at 1400^0C. maintain the rate of heating in a way that this temperature is attained in 3hrs.

Chang the position of panels at 1000^0 C 1200^0 C and 1300^0 C for uniform heating. Heat the specimens for 10 min. at 1400^0C.

Cool the panel cooled for 10 min. with the help of blast of air-water mist.

This is delivered through vertical manifold having aperature of size 90mmX600mm. Admit the air at the rate of 40m^3 per minute. Admit the water at rate of 11 liters during first 8 minutes of colling. No water is admitted during last 2 minutes. The cooling unit is provided to and fro motion (about 125 times in one minute) over the entire surface of panel by external means.

Subject the samples are subjected to such 12 heating and cooling cycles.switch off the furnace.

Subject the panels to another two cooling cycles without water mist.

I. Then cool the specimen overnight after spacing the panel 25mm face to face.
I. Carefully remove the refractories samples after 24 hrs from the panel.
I. Weigh all the samples and rearrange in original for comparison with photographs. The average loss of weight of panel gives the indication of the extent of spalling resistance.

Small prism test:-

This is another method to determine spalling resistance. The procedure is described below.

Procedure for determination of spalling resistance by small prism test method

Cut three specimens from refractories (of standard shape) under test.

Ground the specimens in shape of prism of size 50mm side and 75mm height.

In case of non-standard shapes (sleeves, nozzles or other pouring type refractories), the test specimens are cut in form of ring of height 50mm.

Dry these specimens to a constant weight.

Then place the specimens in cold or semi-muffle type furnace. Heat the furnace at uniform rate to attain temperature of 450^0C in 3 hrs in case of silica refractories. In case of fireclay , siliceous or refractories heat the specimens to attain 1000^0 C in three hours.

Heat the specimens for ten minutes.

Remove the specimens from furnace with the help of pre-warmed pair of tongs.

Place these specimens on a brick floor for 10 minutes for cooling. The room should be free draughts.

Observe the samples for any crack.

Again reheat the specimens for 10 minutes and cool another 10 minutes.

Again observe the specimens for any cracks.

Repeat the steps eighth and ninth till cracks are appeared. Note the cyclein which crack appear. Number of such heating and cooling cycles is the measure of spalling resistance.