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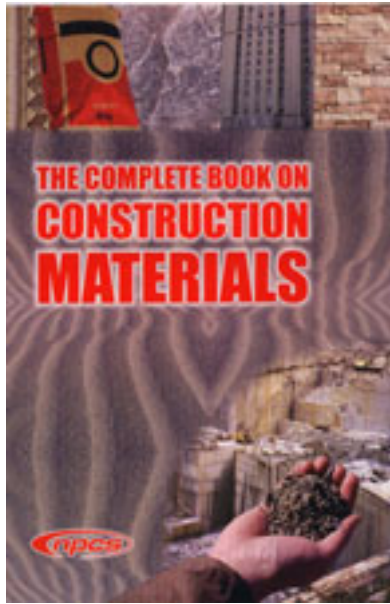
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The Complete Book on Construction Materials



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Construction industry is the largest consumer of material resources, of both the natural ones (like stone, sand, clay, lime) and the processed and synthetic ones. Each material which is used in the construction, in one form or the other is known as construction material (engineering material). No material, existing in the universe is useless; every material has its own field of application. Stone, bricks, timber, steel, lime, cement, metals etc. are some commonly used materials by civil engineers. Selection of building material, to be used in a particular construction, is done on the basis of strength, durability, appearance and permeability. The stone which is used in the construction works, in one form or another is always obtained from the rocks. The rocks may be classified in four ways; geological classification, physical classification, chemical classification and classification based on hardness of the stone. Various kind of rocks come under these classification for example; igneous rocks, plutonic rocks, sedimentary rocks, silicious rocks, stratified rocks etc. brick is the most commonly used building material which is light, easily available, uniform in shape and size and relatively cheaper except in hilly areas. Bricks are easily moulded from plastic clays, also known as brick clays or brick earth. Bricks can be moulded by any of the three methods; soft mud process, stiff mud process and semi dry process. There are various kinds of bricks; specially shaped bricks, burnt clay bricks, heavy duty bricks, sand lime bricks, sewer bricks, refractory bricks, acid resistant bricks etc. lime is an important building material, it has been used since ancient times. Lime is used as a binding material in mortar and concretes, for plastering, for manufacturing glass, for preparing lime sand bricks, soil stabilization etc. Concrete is a construction material obtained by mixing a binder (such as cement, lime, mud etc.), aggregate (sand and gravel or shingle or crushed aggregate), and water in certain proportions. Based on the binding materials, the common concretes can be classified as; mud concrete, lime concrete, cement concrete and polymer concrete. World demand for cement and concrete additives is projected to increase 8.3 percent annually in next few years.

This book basically deals with rock and stone, formation of rocks, classification of rocks, geological classification, metamorphism physical classification of rocks, chemical classification, classification based upon hardness of the stone composition of stone (rock forming minerals), igneous rock forming minerals, sedimentary rock forming minerals, texture of the rocks, types of fractures of rock, uses of stone, natural bed of stone, aluminium and magnesium alloys, mechanical properties of a partially cured resin, DMA characterization, chemical advancement of a partially cured resin, differential scanning calorimeter characterization, chemical mechanical relations, moisture content as a variable, wettability and water repellency of wood, fungal and termite resistance of wood etc.

The book provide wide coverage of building materials such as stone, bricks, lime, mortars, concrete, asbestos, gray iron, cast iron, steel castings, aluminium, wood, architectural paints and so many others with their applications in building construction. The book is resourceful for all professionals related to construction field, technocrats, students and libraries.

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**Sample Chapter:**

# STONE

## Introduction

All the engineering structures are made from some materials. Each material which is used in the construction in one form or the other is known as engineering material. Engineering materials are also sometimes termed as building materials or materials of construction. Every engineer has to come across various materials in carrying out various engineering works and projects and as such he is supposed to be fully conversant with their properties and behaviour.

No material existing in the universe is useless. Every material has its own field of application. An engineer has to be conversant with the properties of most of them. Stone bricks timber steel lime cement metals etc. are some commonly used materials by a Civil Engineer. Even engineers in branches of Mechanical Electrical Electronics etc. are required to know the properties of these materials. Selection of building material to be used in a particular construction is done on the basis of strength durability appearance and permeability. In order to carry out safe constructions some standards for the materials to be used are fixed. These standards are fixed by Indian Standards Institutions (ISI). These standards are continuously reviewed and modified from time to time to suit to the changed conditions. All the commonly used Engineering Materials have been discussed in this book in regard to their properties place of occurrence manufacture and uses. In the first chapter stone has been discussed.

It is likely that our country may face shortage of common building materials like cement lime bricks aggregates plywood plastics etc. It is therefore an urgent need to handle the situation by manufacturing cheap building materials and also by developing new building materials. Shortage of building materials and the high costs are likely to hamper many projects and developmental programmes. It is therefore imperative to lay greater emphasis on the growth of such industries which use local raw material resources for producing less costly building materials.

### Rock and Stone

Rock is the term used to name a solid portion of the earth's crust. It has no definite shape and chemical composition. It is generally very big in size. The rocks have one or more than one minerals. Rocks having only one mineral are known as mono mineralic rock and those having several minerals as Poly mineralic rocks. Quartz sand pure gypsum magnesite are examples of mono mineralic rocks and granite basalt etc. those of poly mineralic rocks. The rocks are named after the predominant mineral present in it.

A rock having calcium carbonate mineral as predominant mineral is termed as calcareous rock. Similarly rock predominant in clay is called argillaceous rock. Quartz felspar hornblend mica augite dolomite are some of the common rock forming minerals.

### Stone

The stone is always obtained from rock. The rock quarried from quarries is called stone. Quarried stone may be in form of stone blocks stone aggregate stone slabs stone lintels stone flags etc. Stone has to be properly dressed and shaped before it is used at the place of its use.

### Formation of Rocks

Solar system consists of sun as the centre and all other planets revolve around it. Our earth is one which originally was in form of mass of incandescent gases. The mass of gases after cooling first converted into molten mass and then on further cooling the surface of the molten mass converted into solid crust. The process of cooling of earth is still continuing and thus process of solidification of molten matter is also continuing. Existence of molten matter under earth's crust is reflected by eruption of volcanos from time to

time. The molten matter of which the earth and other planets were originally made up and existence of which is confirmed by the volcanic eruptions is known as Lava or Magma.

## Classification of Rocks

The stone which is used in the construction works in one form or the other is always obtained from the rocks. The rocks may be classified in following four ways.

Geological classification

Physical classification

Chemical classification and

Classification based on hardness of the stone.

Geological classification

According to this classification rocks may be divided into following three categories.

Igneous rocks

Sedimentary rocks and

Metamorphic rocks.

**Igneous rocks.** As already explained in article 1.3 formation of rocks the inside portion of the earth's surface is very hot and it can cause fusion even at ordinary pressures. The molten lava or magma occasionally tries to come out of the earth's surface through cracks or other weak spots. This magma when gets exposed to the outside cooling effect solidifies in the form of a rock known as igneous rock. Hence igneous rocks are formed as a result of solidification of molten lava lying below or above the earth surface due to cooling effect. Depending upon the cooling effect following different types of igneous rocks are formed.

**Volcanic igneous rocks.** This type of igneous rock is formed when molten lava or magma gets exposed to atmosphere at the surface of the earth. In this case cooling of magma is very rapid and hence structures of these rocks are extremely fine grained. This rock may contain some quantity of glass which is non crystalline. Example of Volcanic igneous rock is Basalt.

**Hypabyssal rocks.** This rock is formed when magma is allowed to cool at comparatively slower rate. Such conditions of cooling generally prevail at relatively shallow depth under earth crust. Since rate of cooling is not as fast as in case of volcanic rocks the structure of resulting rocks is fine grained and crystalline but not as fine as in case of volcanic rocks. The best example of hypabyssal rock is Dolerite.

**Plutonic rocks.** These rocks are formed when cooling of magma takes place at a very slow rate. Such conditions of cooling generally exist at a considerable depth from the surface of the earth. The structure of these rocks is coarse grained and crystalline. Stone obtained from Plutonic rock is most commonly used in building industry. The best example of plutonic igneous rock is granite.

All the igneous rocks contain minerals like Augite Felspar Horn blende mica quartz etc. Before solidification all these minerals are in molten state along with some gases forming magma.

**Sedimentary rocks.** The rocks are formed by the deposition of broken up materials like sand clay

Fig. 1. Sedimentary Rock formation

## Stone

Disintegrated rocks dead sea organisms etc. with the aid of water wind frost etc. on the pre existing rocks. Earth's crust when subjected to weathering cause disintegration which results in the formation of clay sand and pebbles. The disintegrated mass is carried by rain water streams wind etc. and settles as and when conditions become favourable to it. The process of deposition of new disintegrated matter continues in regular layers. With age this deposited mass becomes a rock known as Sedimentary rock. Since the sediments get consolidated in horizontal or nearly horizontal layers these rocks show different layers distinctly. All the layers of this rock may have same or different composition colour and structure as all the



layers have deposited under varying conditions. The formation of these rocks is shown in Fig 1. These rocks can be easily split along the bedding plane. Sand stone limestone slate and shale are some common Sedimentary rocks.

Metamorphic rocks. These rocks are formed when igneous as well as sedimentary rocks are subjected to a very large heat and pressure. The process of change due to heat and pressure is known as metamorphism. The rocks change their character due to metamorphism and the resulting mass of rock change into hard and durable foliated structure Marble quartzite and slate are common examples of metamorphic rocks.

## Metamorphism

All the rocks of igneous and sedimentary origin represent a mass of mineral composition. This mass remains in equilibrium under the general atmospheric conditions. When either temperature or pressure or even both are increased the equilibrium of the mass gets disturbed and its minerals realign themselves to re establish the equilibrium. Re alignment of minerals change the texture of the rock. This process is known as metamorphism. It should be remembered that weathering action and sedimentation action are not included in metamorphism.

Heat pressure and chemically active fluids are the three agents which bring about the changes of metamorphism.

Heat may be supplied by the general rise of temperature inside the earth or by hot magma and pressure may be caused due to heavy overlay rocks or due to movement of the earth during earthquakes. Chemical liquids do not take any active part in the process of metamorphism. Following four types of metamorphisms occur.

Plutonic metamorphism.

Thermal metamorphisms.

Cataclastic metamorphism.

Dynamo Thermal metamorphism.

Plutonic metamorphism. The metamorphic change takes place at large depths under the earth. Uniform pressure and high temperature are responsible for this change. This is due to the fact that rocks become plastic mass at certain depths and plastic mass can be in equilibrium only under uniform pressure.

Thermal metamorphism. The changes brought about in this metamorphism are predominantly due to high temperature.

Cataclastic metamorphism. The metamorphism or change is brought about by directed pressure only and temperature uniform pressure do not play any role in it. This change takes place at the surface of the earth. We have used two terms above uniform pressure and directed pressure. Directed pressure can be applied to solids only. Directed pressure when applied to liquids is converted into uniform pressure. Uniform pressure can be applied to liquids and solids both.

Dynamo thermal Metamorphism. Temperature increases with depth inside the earth. The changes brought about in the rock by combination of heat and directed pressure are known as Dynamo Thermal metamorphism. This change takes place not at very large depths but at moderate depths.

As a result of metamorphosis limestone and marl become marble Basalt and trap are converted to schist and laterite and granite becomes Gneiss.

## Stone

### Physical classification of rocks

According to general structure the rocks may be classified into following three categories.

Stratified rocks

Unstratified rocks and

Foliated or laminated rocks.

**Stratified rocks.** These are such rocks which possess planes of stratification or cleavage. These rocks can be easily split along these planes. An experienced supervisor at the quarry site can easily locate these planes. All the sedimentary rocks have distinct layers of stratification and thus are stratified rocks.

**Unstratified rocks.** The structure of these rocks is compact granular. They do not show any layers of stratification or cleavage. All the igneous rocks of volcanic origin are the examples of unstratified rocks.

**Foliated or laminated rocks.** These rocks comprise of thin laminations. They can be split in definite direction and size. Metamorphic rocks come under the category of foliated rocks.

## Chemical classification

Based upon chemical composition the rocks can be classified into following three categories

**Silicious rocks**

**Argillaceous or clayey rocks**

**Calcareous rocks.**

**Silicious rocks.** These rocks consist of silica as their predominant constituent. These rocks are very hard and durable and are not easily affected by weathering agencies.

Granite quartzite trap basalt sandstone etc. are the examples of silicious rocks. Presence of weaker materials may cause their disintegration.

**Argillaceous rocks.** Predominant constituent of these rocks is clay. The principle constituent alumina which is nothing but clay remains mixed up in varying proportion with siliceous calcareous and carbonaceous matter. These rocks are hard durable dense and brittle in nature. Laterite slate porphyry are the best example of argillaceous rocks.

**Calcareous rocks.** The predominant constituent of these rocks is calcium carbonate. The durability of these rocks is greatly dependent upon the constituents of surrounding atmosphere. Lime stone marble dolomite kankar etc. are the examples of this type of rocks.

Classification based upon hardness of the stone

According to this classification stone may be classified as soft medium hard and very hard.

**Very hard rocks.** Granite trap taconite are the very hard varieties of rocks. **Hard rocks.** Granite basalt trap gravel quartzite are the hard varieties of rocks. **Medium rocks.** Dolomite and lime stone are the medium varieties.

**Soft rocks.** Talc gypsum sand stone slate etc. are the soft varieties of stones.

Scale of hardness of various minerals starting from hardest to softest have been given as follow.

Diamond (Hardest) Corundum Topaz quartz Felspar Apatite Flouspar Calcite rocks salt Talc (softest).

## Composition of Stone (Rock forming Minerals)

Chemically the rocks are composed of mineral earths alkalies oxides or iron and manganese etc. Silica ( $\text{SiO}_2$ ) alumina ( $\text{Al}_2\text{O}_3$ ) lime ( $\text{CaO}$ ) and magnesia ( $\text{MgO}$ ) are the mineral earths which are usually found in rocks in one form or the other. Soda ( $\text{Na}_2\text{O}$ ) and Potash ( $\text{K}_2\text{O}$ ) are the usual alkalies present in the rocks. Presence of alkalies in the rocks is not preferred as it causes stone to disintegrate when exposed to weather. Generally stones comprise of more than one mineral earth.

# BRICKS AND OTHER CLAY PRODUCTS

## INTRODUCTION

This chapter deals with construction materials such as bricks tiles refractory bricks earthenwares and stonewares. All these materials are made from clay and are also known as clay products. Burning of moulded clay products makes them sufficiently strong for use as construction materials. Though tiles refractory bricks earthenwares and stonewares serve different construction purposes brick is the most commonly used building material. It is light easily available uniform in shape and size and relatively cheaper

except in hilly areas. Bricks are easily moulded from plastic clays also known as brick clay or brick earth.

## BRICK EARTH AND ITS CONSTITUENTS

### Sources of Brick Earth

Brick earth is derived by the disintegration of igneous rocks. Potash feldspars orthoclase or microcline ( $K_2O \cdot Al_2O_3 \cdot 6SiO_2$ ) is mainly responsible for yielding clay mineral in the earth. This mineral decomposes to yield kaolinite a silicate of alumina which on hydration gives a clay deposit  $Al_2O_3 \cdot 2H_2O$  known as Kaolin.

### Qualities of Brick Earth

A good brick earth should be such a mixture of pure clay and sand that when prepared with water it can be easily moulded and dried without cracking or warping. It should contain a small quantity of lime which causes the grains of sand to melt and helps bind the particles of brick clay together. It should also contain a small amount of oxide of iron which acts in the same way as lime and moreover lends the brick its peculiar red colour.

### Chemical composition of Brick Earth

According to IS 2117 1975 the clay or mixture of clay selected should preferably conform to the following mechanical composition

Clay 20-30% by weight

Silt 20-35% by weight

Sand 35-50% by weight

The total content of clay and silt may preferably be not less than 50% by weight. The total lime (CaO) and magnesia (MgO) in the case of alluvial soil should not be more than one per cent and in other cases should not be preferably more than 15%. The lime should be in finely divided form. Also the total water soluble material should not be more than one per cent by weight.

### Functions of the constituents of Brick Earth

Silica or sand in brick earth prevents shrinkage cracking and warping of bricks but too much of sand will make the bricks brittle. Clay or alumina makes brick earth plastic and lends the brick its hardness but unless mixed with sand it will shrink crack and warp in the process of drying and burning. Lime and oxides of iron both act as fluxes helping the grains of sand to melt and bind the particles of clay together. Oxides of iron also impart a red colour to the brick but excess of it makes the brick dark blue. Magnesia present in clay with oxide of iron make the brick yellow.

### Harmful Ingredients

The following materials if present in brick earth have an adverse effect on the properties of brick and should be removed or treated before undertaking the manufacture of bricks.

### Pebbles of Stones and Gravel

These do not allow the clay to be mixed uniformly and thoroughly and result in weak and porous brick. Bricks containing grits are likely to crack and cannot be readily cut or worked.

### Alkaline Salts

Alkaline salts if present act as hygroscopic substances. They absorb moisture from the atmosphere in due course of time and create damp conditions. The moisture on drying leaves behind a greyish white deposit known as efflorescence on account of which the appearance of the building is spoiled. Common salts generally present in soils are sulphates of calcium sodium and potassium. The presence of Reh or Kallar consisting of sodium sulphate with more or less of sodium carbonate and sodium chloride renders the clay utterly unsuitable for brick making. Presence of Reh or Kallar can be easily detected by the efflorescence on the sides of fresh excavation if the soil is moist but it would be appropriate in all cases to moisten the soil with water and subject it to evaporation and check for efflorescence.

### Limestone and Kankar

Presence of large quantity of lime and limestone in lumps is detrimental to brick earth as lumps of limestone if burnt in a brick slake afterwards and split the brick. Thus limestone should be present in very finely divided state.

### Vegetation and Organic Matter

Organic Matter if present in brick earth will produce porous bricks. This is due to the evolution of gas during the burning of the carbonaceous matter resulting in the formation of small pores.

## MANUFACTURE OF CLAY BRICKS

Bricks are made by treating suitable brick earth or clay moulding it to shape and size (allowing for shrinkage) drying it and then baking burning or firing it at high temperatures in order to fuse the constituents to a hard homogeneous mass. The process of manufacture can be described under the following heads.

### Selection of site

#### Preparation of clay

#### Moulding of bricks

#### Drying of bricks

#### Burning of bricks.

### Selection of Site

The site selected for the manufacture of bricks must have suitable soil available in sufficient quantity otherwise unnecessary labour and cost of digging and transportation of the soil would be involved.

Generally for making soil suitable for brick making certain materials are required to be mixed with the soil.

Therefore the availability of such materials near the site of brick making is of great importance. It is also necessary that water and fuel like coal and wood are easily available in sufficient quantities.

According to IS 2117 1975 the site should be selected after giving due consideration to the suitability of soil and location of the water table. Select a site such that the water table during burning session is at least 1 metre below the kiln floor.

### Preparation of Clay

According to IS 2117 1975 brick clay should be prepared in two stages

#### Weathering

#### Tempering

### Weathering Process

The soil is left in heaps and exposed to weather for at least one month in cases where such weathering is considered necessary for soil. The soil is turned at least twice and it is ensured that the entire soil is wet throughout the period of weathering. The purpose of weathering is to disintegrate big boulders of clay under the action of atmospheric agencies to make it a uniform mass and also to eliminate the impurities which get oxidized.

### Tempering Process

After weathering the required quantity of water should be mixed with the soil to obtain the right consistency for moulding. Addition of sand and other materials if necessary may be made at this stage to modify the composition of the soil.

The quantity of water to be added may range from  $\frac{1}{4}$  to  $\frac{1}{3}$  of the weight of soil sandy soils requiring less water and clayey soils more water. But the nature and degree of wetness of the soil at this stage should also be duly considered.

The moistened soil is kneaded with spades or other manual or mechanical equipment into a plastic mass. After the addition of water and kneading the soil may be pugged in a pug mill of suitable size corresponding to quantity of bricks to be manufactured. The pug mill may be mechanically operated or hand operated as shown in fig. 1a and 1b.

## Moulding of Bricks

Bricks may be moulded by any one of the following methods

Soft mud process (Hand moulding)

Stiff mud process (Machine moulding)

Semi dry process (Machine moulding)

### Soft Mud Process

The clay prepared by using 25 to 30% of water is pressed into the mould by hand. Some typical moulds of timber or metal are shown in fig. 2.

Fig. 1 (a) Manually operated pug mill (b) Mechanically operated pug mill.

Moulds are made either of wood or of thin steel plates. Seasoned wood should be used to prevent changes in the dimensions of the mould. The edges are protected with strips of brass or steel. Steel moulds made of plates 6 mm thick are used if the bricks are to be manufactured on large scale. Normally shrinkage allowance varying from 10% to 12% is provided. Thus the size of the mould is such that it would give the finished brick its required size.

As per IS 2117 1975 the mould should be constructed preferably of metal. The thickness of the sides of the mould shall not be less than 3 mm if made of metal and not less than 10 mm if made of wood.

The process of moulding bricks with the help of moulds is also called hand moulding. According to IS 2117 1975 handmade bricks may be either ground moulded or table moulded. In case it is ground moulded a level firm surface should be used. Typical specifications for accessories used in table moulding are given below and in Fig. 3.

**Moulding table.** The moulding table is 1.2 to 1.8 m long and 0.6 to 1.0 m wide and is made of wood or iron. It is smoothly finished at the top and supported horizontally at a height of 1m to 1.2 m. Also there are holes to accommodate accurately the bottom pins of the stock board.

**Stock board.** It is a wooden board with iron lining around the upper edge and with such dimensions as to fit accurately but loosely the interior of the mould (Fig. 3.4.). The stock board is provided with four pins one at each corner of the bottom side which when fitted into the corresponding holes on the moulding table hold the board tightly in position during moulding. The stock board should also have a projection at the top so as to form the frog of the brick being made.

**Pellet boards.** These are rectangular pieces of wood of size 30 cm x 15 cm x 10 mm thick with a smooth surface on one side. Pellet boards are used for conveying moulded bricks to the drying yard.

### Procedure

Before moulding the inside of the mould is cleaned and then sprinkled with sand or ash. If slop moulding is adopted then the mould is dipped in water and cleaned. The mould is then set firmly on the level surface. A quantity of clay slightly more than the volume of the mould is taken and rolled in sand. It is then shaped suitably into a single lump and dashed firmly into the mould with a force that is to be judged by the moulder by experience so that the clay completely occupies the mould without air pockets and with minimum surplus for removal. The surplus soil is scrapped off with a sharp straight edge known as a style or a stretched wire and the top surface is leveled.

The whole assembly of the mould is then lifted given a slight jerk and inverted to release the moulded brick on a pellet board in the case of table moulding or on a dry level surface of the ground in the case of ground moulding.

The ground may be advantageously sprinkled with sand before releasing the brick over it so that bricks do not stick to the ground. When a frog is not needed a bottomless mould may be adopted in which case inversion to release the moulded bricks from the mould will not be necessary. It may be added that each moulder can make on an average about 500 to 1000 bricks per day. Stiff Mud Process

The selected clay after proportioning of the ingredients is mixed with water up to about 12 to 18% and is thus initially prepared for being put in the machine. There are two stages in machine moulding

The final mixing when kneading and tempering is done in the pug mill.

In the second stage bricks are formed by extruding stiff clay through a mould or orifice in the extruding machine.

The pug mill and the extruding machine may be combined in one unit as shown in Fig. 5.

The upright cylinder or barrel of the machine known as the pug mill is kept filled with prepared clay which is mixed well and pressed down against the coarse threads of a horizontal spiral screw. The pressing screw is fixed near the top of the horizontal axle called `auger. At one end of the auger power is applied for driving the machine and at the other end is fixed a metal die through which the clay extrudes with the desired section. The extruding clay is a continuous mass and is received on a conveyor to be further cut into pieces of the correct sizes of bricks.

The bricks are cut either by a single wire or by a number of wires fixed on a frame as shown in Fig. 6. The bricks obtained by this method have a smooth rectangular finish.

### Semi Dry Process

In this method only 7 to 10% of water is added so that it forms just a damp powder. It is then pressed under a pressure of 1000 to 1200 kg/cm<sup>2</sup> with the aid of a plunger machine to form the bricks. At the first plunger machine the material is automatically measured off fed into a steel mould and pressed by plungers (heated to prevent sticking) on two opposite sides. It is then expelled from the mould and transferred to another plunger machine where it is again pressed one of the beds receiving the frog at this stage. Pressed bricks do not require drying and could be put into the kiln directly for burning. They are very strong and compact and on account of the latter quality they are more durable than bricks moulded by the stiff mud process. Machine moulding can also be employed for the soft mud process to press pugged clay into moulds with the aid of the plunger. It is possible to mould 4 to 8 bricks at a time as against one by hand moulding.

# CONCRETE

## INTRODUCTION

Concrete is a construction material obtained by mixing a binder (such as cement lime mud etc.) aggregate (sand and gravel or shingle or crushed aggregate) and water in certain proportions. The mix is placed properly in moulds or forms to harden in a suitable environment. When the various ingredients are mixed these form a plastic mass which can be moulded into the desired shape and size. The moulded mass when allowed to cure in suitable environment hardens to become a solid mass capable of maintaining its shape and size and sustaining certain loads. Hardening in concrete is a result of the chemical/physical combination of the binding material water and air in a given environment. The hardened concrete so obtained serves different purposes depending on the type of binding material used quality and grade of concrete location and size of such concrete component.

The type of concrete is basically known by its binding material. It is the binding material which plays the main role in the behaviour and characteristics of the resulting concrete. Based on the binding materials the common concretes can be classified as

- Mud concrete
- Lime concrete
- Cement concrete
- Polymer concrete

These concretes are used to serve certain requirements of various concrete elements cast in different situations. Behaviour of these concretes can further be modified by the use of certain admixtures special

treatments or combination of binding materials. Properties of some of these concretes can further be improved through certain special techniques of construction (such as prestressing reinforcing impregnation etc.).

Mud concrete is made by using suitable mud as the binding material. Mud is prepared from good quality clay and water by kneading. The mud is mixed with coarse aggregate or shingle to obtain mud concrete. The mud concrete is laid in suitable layers and compacted by ramming or tamping. The mud concrete properties are mainly due to interlocking of aggregate particles and filling of voids by mud. Mud concrete can easily be affected by moisture and has poor impermeability durability and strength characteristics. This type of concrete is generally used for cheap and temporary type of constructions in foundation bases non load bearing walls with water proofing treatment on external faces etc.

Lime is very popular binding material in Civil Engineering constructions. Properly slaked lime slurry or putty is used as binding material in lime mortar and lime concrete. Lime concrete is prepared by mixing lime mortar with aggregate (shingle or gravel). Lime concrete is laid in layers and compacted suitably by ramming. Lime concrete is commonly used for foundation base layers floor base layers roof insulation layers over stone patties (slabs) etc. Lime concrete exhibits fairly good properties of durability impermeability and strength specially suitable for base courses. It has been used in many important old monumental buildings which have stood the test of time. The details of preparation properties and used shall be dealt in a subsequent section. The advent of cement as binding material in the 19th century revolutionized construction activities. Cement concrete obtained by mixing cement sand gravel or shingle or crushed aggregate and water is a versatile and popular construction material. Cement concrete is used in almost all modern structures due to its superior qualities and appropriate quality controls possible during and after construction. Cement concrete is used in various forms and grades for different purposes in construction works. Certain weak points in cement concretes for specific purposes can easily be overcome by adopting suitable techniques such as steel reinforcing prestressing fibre reinforcing Ferro cement and polymer impregnation techniques etc. The properties of cement concrete can also be modified by the use of certain admixtures during its preparation. The details of preparation properties and uses shall be discussed later.

Recently certain polymers and epoxy resins have been developed which exhibit superior binding qualities. These polymers are now being used for preparation of special polymer concretes. Polymer concrete is obtained by mixing epoxy resins or polymers with plastic aggregates. Polymer concrete exhibits very high strength. Cost of polymers and other epoxy resins used as binding material is quite high and hence the cost of polymer concrete is also high. Due to the high cost and small production of epoxy resins the use of polymer concrete is limited. In India use of such polymer concrete is yet to pick up. The future potential for the manufacture and use of polymer concrete for construction of highly sophisticated structures is very good.

1. Mud concrete. This concrete does not carry any importance. It is prepared by mixing brick bats in mud mortar. Brick bats may be made from kuchha or pucca bricks. Sometimes even crushed stone may be mixed with mud mortar to form mud concrete.

Mud concrete is used for preparing hard base. Over which lime concrete may be laid and then permanent flooring may be spread. During construction of ground floor of buildings filling of earth is first of all consolidated by sprinkling sufficient amount of water. During consolidation process broken brick bats or crushed stone pebbles lying waste at the site are also spread and rammed into the fillings by rammers. Earth filling added with water and broken brick or stone bats forms mud concrete which on setting develops a hard surface over which permanent flooring of any form may be laid. The same processes of consolidation with mud concrete may be carried out during preparation of foundation bases.

The coarse aggregate used in mud concrete is usually of broken bricks of size 4 cm 100 m<sup>3</sup> brick ballast is

mixed with 40 m<sup>3</sup> of prepared mud mortar.

## LIME CONCRETE

### Preparation of Lime Concrete

The main ingredient of this concrete is slaked lime as the binding material. The slaked lime is obtained in various forms as hydrated lime powder lime putty or slaked lime slurry prepared by grinding in suitable grinding mill (ghani etc.). Slaked lime is first mixed with sand to prepare lime mortar which is further mixed with coarse aggregate (shingle gravel crushed stone or brick aggregate) in suitable proportions.

Preparation of hydrated lime and lime putty has already been discussed in the chapter on lime and may be referred to for details.

For preparation of lime concrete a hard impervious level base is prepared by stone or brick pitching. Appropriate quantity of sand is spread as a horizontal stack (generally taking lime sand 1 1 to 1 3 by volume). Measured quantity of lime putty or slaked lime is spread over the sand stack and the whole mass is mixed thoroughly by cutting and turning the mass with shovels and simultaneously sprinkling enough quantity of water to make the mass plastic. The prepared mortar is allowed to mature for 1 to 3 days during which it is not allowed to dry. Another method of mixing is by using a small water tight trough container or tank made from stone slabs or brick masonry. Enough quantity of water and measured quantities of lime are added to the prepared trough and lime is stirred well in water.

According to the desired proportions the measured quantity of sand is added and then turned upside down a number of times till the whole mass become uniform (Fig. 1 (a) and (b)). This mortar is left for one to three days for maturing in wet conditions if the slaked lime is not already matured earlier.

The coarse aggregate of the desired type and quantity is laid in stacks on the prepared hard impervious level surface and a measured quantity of lime mortar is spread uniformly over the coarse aggregate stack. Sufficient water is sprinkled over the stack and it is cut into layers and turned upside down with the help of spades or shovel till the whole mass becomes uniform.

### Laying of Lime Concrete

The base where lime concrete is to be laid is prepared by cleaning leveling wetting and compacting by ramming properly. The material is laid on prepared foundation bases or flooring bases in layers and rammed manually with steel rammers. During the ramming process some water may be sprinkled if the concrete is dry and stiff. After ramming the lime concrete thoroughly it is moist cured by sprinkling water for 7 to 14 days.

Lime concrete can also be laid in roof terraces for providing an insulating and water proofing layer over stone slabs. Generally kankar lime is preferred for this purpose. Kankar lime is slaked and laid on the roof slab and mixed with light weight coarse aggregate (preferably brick aggregate). In case of kankar lime concrete for terracing (Dhar) no fines are used and lime is also used in much higher proportion (Lime coarse aggregate 1 1 to 1 2). After thoroughly mixing the lime and coarse aggregate some angular aggregate of 10 mm size is spread near the surface of the wet plastic lime concrete. The lime concrete terrace is compacted by light weight rammers (stone thapies of about 2 to 3 kg mass) covering the whole surface a number of times till the concrete mass becomes dense stiff and solid. While the lime concrete is wet and plastic certain admixtures such as jaggery (sugar or gur solution) powdered fenugreek seed (methi) hemp (or jute) fibres etc. are sprinkled near the surface before ramming is started. Ramming continues for 2 3 days during which the surface is kept wet by sprinkling gur water. During this process lime cream comes near the surface and forms a thin film which helps in obtaining a smooth surface. The fibrous material increases the surface resistance to shrinkage or other cracks while methi powder fills the pores and makes the surface more water proof. Jaggery solution helps in better maturing and curing of lime concrete specially near the surface.



# ASPHALT, BITUMEN AND TAR

## INTRODUCTION

The history of these materials can be traced back to ancient times. Their importance was realized by early civilizations who employed them for a variety of purposes ranging from mummification to building temples palaces and vast irrigation systems and enduring highways. A ritual pool dating back to 3000 B.C. discovered in Mohenjodaro in the valley of the Indus was water proofed with a layer of bitumen on the walls. After an apparently extinct phase through the middle ages and renaissance period bitumen was re discovered in the form of deposits of impregnated limestone in France Switzerland and Germany in the eighteenth century. Put to use for side walls and pavings in different parts of the continent it proved to be satisfactory. The utility of the material has grown ever since and today its versatility as a construction material may be well judged from its use and application for roofing road surfacing insulating varnishes acid resistant paints and cold moulded products.

For purposes of introduction asphalt bitumen and tar are referred to as Bituminous Materials which are essentially hydrocarbon materials frequently accompanied by their non metallic derivatives. They may be gaseous liquid semi solid or solid in nature and are completely soluble in carbon di sulphide (CS<sub>2</sub>). They possess some common properties as follows

1. Thermo viscosity i.e. variance of viscosity (which is roughly the opposite of fluidity) with temperature.
2. Adhesion to solid surfaces.
3. Durability.
4. Water proofing characteristics under normal circumstances.

The above desirable properties of bituminous materials render them very useful as a protective agent an adhesive and a sealant.

## TERMINOLOGY

### Asphalt/Bitumen

The Greek word for native asphalt was asphaltos and the Latin word bitumen. The latter word comes from the original Sanskrit word Gwitumen applied to native asphalts as fuels. Fundamental differences still persist between the two terms commonly used viz. bitumen and asphalt. In India as in most countries (except USA) the refinery product is officially termed bitumen and the mixture of bitumen and inert mineral matter (natural mixture) is known as asphalt. Asphalts (or bitumens) are dark brown or black solids or semi solids which are found in natural state and are also obtained by refining crude petroleum. The selected crude oil is heated in a distillation plant operating at atmospheric pressure and the volatile components such as gasoline kerosene and light gas oil are removed by distillation and fractional condensation. The residue is processed at a higher temperature and in vacuum to remove the lesser volatile products and a highly viscous material obtained is called straight run bitumen (asphalt). A range of grades from very soft to very hard consistency can be produced by varying the temperature and rate of flow during processing. The softer grades are designated by the penetration limits e.g. 80/100 whereas the harder grades are identified by their softening point e.g. 80/90.

The viscosity of the straight run bitumens has to be reduced before they can be used. There are three main groups of bitumen (asphalt) products produced from the straight run bitumens (asphalts).

1. Hot bitumens or asphalts those whose viscosity is reduced by heating.
2. Cutback bitumens or asphalts those whose viscosity is reduced by dissolving in mineral solvents.
3. Emulsion bitumens or asphalts those whose viscosity is reduced by dispersion or suspension in a water base.

## Other Allied Terms

### Cutback Asphalt

It is an asphalt liquefied with petroleum distillates used for cementing down floor coverings and for water proofing walls. Protective coatings based on asphalt cutback form economical paints for protection against salts alkalies and non oxidizing acids at temperatures up to 55°C.

Three common types of cutbacks are used depending on the type of distillate. When gasoline (petrol) is used as the solvent a rapid curing asphalt is the result kerosene produces a medium curing asphalt heavier fuel oils produce a slow curing asphalt.

### Emulsified Asphalt

It is an asphalt emulsion in water solution used for floor surfacing painting pipes and water proofing concrete walls. The addition of water decreases the viscosity and renders the emulsion easy to handle and apply. Drying involves primarily the loss of water by evaporation. They have good bonding qualities even on damp or wet surfaces.

These are divided into three groups depending upon the type of emulsifier (a) soap type in which soap is used as the emulsifier (b) clay modified soap type with a combination of clay and soap as emulsifier and (c) clay base type with a mineral matter usually clay as the emulsifier.

### Modified Asphalt

Asphalt that is combined with a rosin ester to increase the penetration tack and adhesion is known as modified asphalt. It is used for laminating paper and for impregnating flooring felts. Asphalts for paints and coatings may also be modified with synthetic resins.

### Blown Bitumen

Bitumen the properties of which have been modified by the passage of air under pressure at an elevated temperature is known as blown bitumen or oxidized bitumen. Blown bitumen is of a rubbery consistency and has a higher softening point and greater resistance to flow than straight run bitumen of the same hardness.

Artificial or mastic asphalt. It is a preparation made by using natural asphalt bitumen and sand. It is water proof fire proof and elastic to some extent. It is largely used as a D. P. C. water proofing layer over flat roofs and for making floors. It is not used much for road construction as roads paved with this become soft in summer and slippery in winter.

## **BITUMINOUS MATERIALS**

### **Bitumen Felt/Tar Felt**

Bitumen/tar is used to saturate felt paper and to coat craft paper to render it water proof. These saturated felts are used in built up roofing.

As per I.S. 334 1951 bitumen is defined as a non crystalline solid or viscous material having adhesive properties derived from petroleum either by natural or refinery process. It is substantially soluble in carbon disulphide.

The greatest proportion of bitumen is obtained from crude petroleum. It is obtained by fractional distillation process in which the simpler components of the crude petroleum such as white spirit kerosene fuel oil light medium and heavy lubricating oils which have lower boiling points are evaporated leaving behind the bitumen. It is black or brown in colour. Bitumen may be extracted by distillation process or cracking process. Mostly distillation process is used.

1. Straight run bitumen. The bitumen which has been distilled to a definite viscosity or penetration without further treatment is known as straight run bitumen. During processing by regulating rate of flow and temperature bitumen from very soft to a very hard consistency grade can be produced. Before this bitumen can be used it has to be processed to reduce its viscosity either by heating addition of cut or emulsifying

agent. This bitumen is mostly used for road construction.

2. Air blown bitumen. Special properties can be developed in semi solid bitumen by blowing air through the residue still in hot condition. This bitumen is sometimes called oxidized bitumen also. This bitumen is not used in paving mixes but is a useful material for roofing battery boxes water proofing etc. It is widely used as crack and joint filler material for concrete pavements.

3. Cut back bitumen. Cut back is defined as a bitumen whose viscosity has been reduced by the addition of a volatile diluent. Volatile diluents are gasoline kerosene and high boiling point light oils. Cut back is used when it is essential to have a fluid binder which can be readily poured or sprayed at relatively low temperature. The important features of a cut back are its viscosity at the temperature of its use and also the rate at which it sets. The rate of setting is the rate at which solvent evaporates from cut back.

Cut backs are commercially manufactured in three groups namely rapid curing (R.C.) medium curing (M.C.) and slow curing (S.C.). R.C. cut backs contain naphtha or gasoline M.C. cut backs contain kerosene and S.C. cut backs contain light oils as the fluxing agents. Each group of cut backs is further divided into six categories varying from 0 to 5. The six different viscosities are named by numbers from 0 to 5 in the increasing order of viscosity. Zero grade has the lowest viscosity and grade 5 the highest.

4. Emulsions. It is a combination of water bitumen and an emulsifying agent. Bitumen does not dissolve in water. But when heated bitumen and water are mixed together and agitated. The bitumen disperses in water in form of spherical globules of about 2 micron diameter. To prevent bitumen spheres from coalescing an emulsifying agent is added in the emulsion which remains dissolved in water. Soap is used mostly as an emulsifying agent. Depending upon the stability of the protective coating of emulsifying agent the emulsion may be classified as Rapid setting (R.S.) Medium setting (M.S.) and slow setting (S.S.) Emulsions are always stored in air tight drums. It is used mostly for the construction of roads. It is not required to be heated before use and as such are very useful for the places where heating of the bitumen has to be avoided. Emulsion is mixed with road metal and applied. When emulsion changes its colour from brown to black it is said that emulsion has started breaking. As the emulsion starts breaking it start binding the aggregate. Emulsion can be used for soil stabilization patch repair works of bituminous roads etc. Its main feature is that it can be used in wet conditions also.

## GRAY IRON

Although gray iron denotes a certain type of cast iron yet the chemical composition structure and properties of gray iron may vary over broad limits. The range of alloy compositions and properties produced as gray irons may be better understood by consideration of some of the principles of gray iron metallurgy. The metallurgy of cast irons depends in large measure upon the nature of the iron carbon equilibrium system.

### THE METASTABLE IRON CARBIDE SYSTEM

In the phase system iron carbide carbon in the alloys occurs as the metastable compound iron carbide ( $Fe_3C$ ). During solidification or melting and in thermal treatments in the solid state the iron carbide functions according to normal principles of phase relationships as expected from the equilibrium diagram. For example freezing of a hypoeutectic alloy less than 4.30 per cent carbon will begin with the formation of austenite dendrites and be completed by solidification of the eutectic austenite iron carbide. After solidification cooling in the solid state results in transformation of the austenite to pearlite.

### SOLIDIFICATION OF AN Fe C Si ALLOY

The presence of silicon in the alloy is the most important single composition factor promoting graphitization in gray cast irons. The effect of silicon may be visualized with the aid of vertical sections of the ternary alloy system Fe C Si. Consider the freezing processes for an Fe C Si alloy with 2% Si and about 3.50% C. Under equilibrium freezing conditions primary austenite dendrites are formed in the temperature range from the

liquids curve to the curve indicating the beginning of eutectic freezing about 2300 to 2060 F. Simultaneous solidification of the eutectic austenite plus graphite completes the freezing process. The eutectic freezing occurs in a temperature range of about 2060 to about 2010 F. When solidification is complete in the alloy under consideration the microstructure consists of about 20 per cent primary austenite dendrites and 80 per cent austenite graphite eutectic. At the solidus temperature austenite is saturated with carbon. Further decrease in temperature is accompanied by rejection of carbon from the austenite as graphite and its precipitation on the graphite flakes in the eutectic. Carbon precipitation continues until the eutectoid temperature range is reached (about 1475 to 1400F with 2 per cent silicon). At the eutectoid temperature the 2.0% Si austenite contains about 0.60% carbon. Equilibrium cooling through the range results in the transformation of austenite to ferrite and precipitation of the remaining carbon on the graphite flakes. The final microstructure then consists of isolated areas of ferrite originating in the primary austenite dendrites and other areas of mixed ferrite and flake graphite having their origin in the austenite graphite eutectic. Boyles has demonstrated the freezing processes under consideration in Fe C Si alloys and commercial cast iron alloys. The microstructural changes described above are those occurring in a ternary alloy of Fe C Si. Similar processes in commercial cast irons are much more complex since many other elements are present and a number of other factors are introduced. However the simple alloy considered does point out the three important stages of graphitization

1. Graphitization during solidification
2. Graphitization by carbon precipitation from austenite (solid state)
3. Graphitization during the eutectoid transformation (solid state)

Some Graphitization also occurs below the transformation range down to about 1000 F although this is of lesser importance unless the time spent at that temperature is very long.

These stages of graphitization and their effects on microstructure and properties will be referred to again.

## CHEMICAL COMPOSITION EFFECTS

All the elements normally present in gray iron exert some influence on the microstructure of the iron. Carbon and silicon of course are fundamental in their effect on cast irons and may be considered first.

### Carbon

Carbon in gray iron is present from about 2.5 to 4.5 per cent by weight. Two phases occur elemental carbon in the form of graphite and combined carbon as  $Fe_3C$ . The analysis reported ordinarily is the total carbon percentage in the iron. Since the two forms may be determined separately by chemical analysis the degree of graphitization may be assessed by the following relationship % total carbon = % graphitic carbon + % combined carbon.

If graphitization is complete the percentage of total carbon and the percentage of graphitic carbon are equal. If no graphitization has occurred the percentage of graphitic carbon is zero. If about 0.5 to 0.80 per cent combined carbon exists in a gray iron it generally indicates that the microstructure is largely pearlitic since pearlite in gray iron having about 2 per cent silicon forms from the austenite eutectoid containing about 0.60 per cent carbon. Thus the relationship above offers a chemical criterion of the degree of graphitization in a gray iron. For sufficient graphitization to develop during solidification of a true gray iron a certain minimum total carbon content is necessary which is probably about 2.20 per cent but this value depends on silicon percentage in the iron.

If the carbon equivalent of a particular iron is calculated to be 4.3 then that iron corresponds approximately to a eutectic alloy (even though it is not a true eutectic in the sense of the ternary phase diagram). If the carbon equivalent of an iron is less than 4.30 the alloy is a hypoeutectic alloy. The carbon equivalent is a useful expression because many properties of gray iron have been found related to it. If the combination of carbon and silicon exceeds 4.30 according to the carbon equivalent equation the iron is a hypereutectic

one. In this case the freezing process begins with the formation of graphite. When graphite precipitates first during solidification the melt is said to form kish. Because of its buoyancy kish pops out of the melt into the air and can be observed as sparkly graphite flakes floating on the surface of the iron or in the air above the iron.

Not only is the eutectic point shifted by silicon in cast irons but it also shifts the eutectoid point and the solubility limits of carbon in austenite to the left of equivalent points in the Fe C system. For this reason pearlite in a 2.0% Si gray iron may contain only about 0.60% carbon rather than the 0.76% C value on the Fe C diagram

Micro structurally silicon occurs dissolved in the ferrite of gray iron. As such it hardens and strengthens the ferrite as pointed out in Chap. 18. Ferrite in pure iron will measure 80 to 90 Bhn whereas 2.0 per cent silicon in a ferritic iron raises the hardness to about 120 to 130 Bhn.

### Silicon Content and Graphitization

Silicon promotes graphitization. Low percentages are not sufficient to cause graphitization during solidification but will cause nucleation and graphitization in the solid state at high temperature as for example during malleabilizing heat treatment. Certain silicon percentages will cause limited graphitization during solidification and a mottled iron partly white and partly gray results.

A certain minimum silicon (and carbon) concentration is necessary for graphitization to proceed sufficiently during solidification to develop a satisfactory gray iron. More accurate diagrams have as their purpose a limiting description of the silicon and carbon percentages which will cause an iron to freeze gray in the section sizes of commercial castings poured into green sand molds. Although these diagrams are useful as a guide successful metallurgical performance in the type of castings made in particular foundries remains the ultimate criterion for the carbon and silicon content. Hence foundries producing certain sizes of castings and types of gray irons will ultimately develop silicon and carbon combinations suitable to their work.

### Sulfur and Manganese

Sulfur which may be present up to about 0.25 per cent is one of the important modifying elements present in gray irons. A low sulfur iron silicon carbon alloy under 0.010% S will graphitize most completely. Boyles has shown that higher sulfur percentages favor the retention of a completely pearlitic microstructure in a gray iron. The latter effect causes sulfur to be known as an element restricting graphitization (carbide stabilizing). Above about 0.25 per cent sulfur is considered to contribute undesirable hardness and decreased machinability because of its retardation of graphitization.

The influence of sulfur needs to be considered relative to its reaction with the manganese in the iron. Alone sulfur will form FeS in cast irons. The latter compound segregates into grain boundaries during freezing and precipitates during the final stages of freezing. When manganese is present MnS or complex manganese iron sulfides are found depending on the manganese content. The manganese sulfides begin to precipitate early and continue to do so during the entire freezing process and are therefore usually randomly distributed. As MnS the effect of sulfur in causing a pearlitic microstructure to be retained is lost to a major extent. The effect of Mn alone as an alloying element is to promote resistance to graphitization. Therefore manganese above that necessary to react with the sulfur will assist in retaining the pearlitic microstructure.

### Phosphorus

Segregation of phosphorus may result in lowering of the temperature of final solidification to about 1800 F. The percentage of steadite present in the final structure may amount to ten times the percentage of phosphorus in the iron. Because of segregation the steadite usually adopts a cellular pattern characteristic of the eutectic cell size developed during solidification. In certain conditions of melting and chilling iron carbide is associated with the phosphide in a ternary iron iron phosphide iron carbide eutectic. Then an amount of the latter constituent considerably in excess of ten times the per cent phosphorus may be

formed. If the ternary eutectic is accompanied by graphitization of its carbide during solidification expansion of the liquid occurs and beads of eutectic exude from the iron. These are often found at the surface of sprues and risers.

Because it forms a eutectic as it segregates phosphorus is often looked upon as increasing the tendency for a particular iron composition to be a eutectic type alloy.

The phosphide of iron is hard and brittle as is the carbide. Increasing phosphorus percentage in the iron causes a proportional increase of the hard constituent and therefore increasing hardness and brittleness of the iron especially above about 0.30% P. To a limited degree improved fluidity of the molten iron is a desirable property contributed by phosphorus through its influence on carbon equivalent.

### Gray Iron Specifications

Because gray iron is used in so many different engineering applications numerous specifications covering its use in special fields have been developed.

## CAST IRON

Cast irons are the tonnage product of the foundry industry. Cast iron foundries produce over a million tons of castings monthly and thus supply more than twice as much casting weight as all other foundries combined. Iron foundries are found everywhere that manufacturing occurs. Of the 5674 foundries in India 2068 produce gray iron 350 nodular iron and 116 malleable iron castings. These foundries send a steady stream of iron castings into every conceivable industry. The demand for iron castings is based on the nature of cast irons as engineering materials and their economic cost advantages. Cast irons offer a tremendous range of the metallic properties of strength hardness machinability wear resistance abrasion resistance and corrosion resistance and other properties. Furthermore the foundry properties of cast irons in terms of yield fluidity shrinkage casting soundness ease of production and others make the material highly desirable for casting purposes. From all standpoints the cast iron family offers a variety of engineering properties which ensure its continued and widespread use. Since many cast irons of different properties are employed it is desirable that a student engineer obtain an over all picture of the entire field. This chapter offers such a picture and presents some of the simpler and more fundamental differences between members of the cast iron family.

## DEFINITIONS

The term cast iron is a generic one referring to a family of materials differing widely in their properties. In general a cast iron is an alloy of iron carbon (up to about 4.0 per cent) and silicon (up to about 3.50 per cent) which ordinarily is not usefully malleable as cast. Definitions of specific types of cast irons are given below.

**Gray cast iron.** An iron having a chemical composition such that after solidification a large portion of its carbon is distributed throughout the casting as free or graphic carbon in flake form. Gray cast iron always presents a gray sooty surface when fractured.

**White cast iron.** An iron having a composition such that after solidification its carbon is present in a chemically combined form as cementite (iron carbide). White iron presents a white crystalline surface when fractured.

**Mottled iron.** An iron of intermediate composition which freezes partly as a white iron and partly as a gray iron under prevailing cooling conditions.

**Chilled cast iron.** An iron of such composition that it would normally freeze as a gray iron but which is caused to freeze white in some locations by rapid cooling during solidification i.e. chilling. Fractured surfaces of chilled irons show areas of white iron where freezing was rapid and other areas of gray iron where the cooling rate was normal.

Malleable iron. An iron with ductility or malleability produced by heat treating (malleableizing) a white iron casting of suitable chemical composition. The carbon in malleable iron is present as nodular shaped aggregates of graphite.

Nodular cast iron (also known as ductile cast iron or spheroidal graphite cast iron). A specially prepared iron treated in the molten condition with a small percentage of magnesium cerium or other agent that will cause a large proportion of its carbon to occur as spheroids of graphite rather than as flakes. Ductility is obtained in the iron as a result of the spheroidal type of graphite formed. This type of cast iron presents a bright steely surface when fractured.

The definitions given above suggest certain factors of major importance controlling the nature of cast irons. These are chemical composition solidification process cooling rate and microstructure. A number of other factors are involved but the aforementioned ones are of prime importance.

## CHEMICAL COMPOSITION

The broad limits of chemical composition of some cast irons are given in Table 1. The Table shows that even the terms gray iron and white iron are general ones in that they refer to a number of alloys falling within broad composition limits. Within the broad limits occur a number of irons with narrower composition limits and different properties. Typical Chemical compositions specifications and uses of a few commercial cast irons are given in Table 2.

### Composition and Graphitization

The influence of chemical composition on the properties and uses of cast irons is largely related to the two alloying elements carbon and silicon and their effects on the process of graphitization. Both elements promote the formation of graphite as their percentage increases in the iron. Carbon may occur in cast irons as iron carbide (cementite) and is then referred to as combined carbon. It may also occur in free form as graphite. Graphitization is the process whereby free carbon is precipitated in the iron or chemically combined carbon  $Fe_3C$  is changed to free carbon (or graphite). Increasing the percentage of carbon in an iron especially above 2.00% C increases the likelihood of graphitization. Furthermore the presence of certain other elements in the iron such as silicon causes iron carbide to become less stable and thus promotes the formation of graphite these are said to be graphitising elements. Probably the simplest picture of the combined effects of carbon and silicon on graphitization is that presented by the diagrams in Fig. 2a and b. In Fig. 2 it can be seen that if carbon and silicon are both below certain percentages a white iron is formed during solidification. If either carbon or silicon is held at a constant percentage and the other is increased the iron changes from white to mottled to gray. Carbon and silicon thus may be varied to produce a white or gray iron as desired. It must be recognized that the diagrams of Fig. 2 do not consider the variable of cooling rate or section size in castings. This is another variable affecting graphitization. Slower cooling rates (heavy casting sections) shift the lines on the diagram to the left and rapid cooling (thin casting sections) shifts them to the right. Thus in practical situations gray iron piston rings are high in carbon and silicon percentages whereas heavy machine tool casting gray irons are low in carbon and silicon percentages. White irons for making malleable castings are even lower in carbon and silicon content so that the carbon will be in combined form as cast. The carbides in this white cast iron however are still sufficiently unstable so that they can be graphitized slowly in the solid state by a malleableizing heat treatment.

Carbon and silicon are not the only elements which influence graphitization and the structure of the iron. At this point however it is obvious that chemical composition is a prime factor in causing the differences in the various types of cast irons.

## SOLIDIFICATION PROCESS

The differences between gray mottled and chilled irons are largely established during the freezing process.

The fundamentals of the freezing process are related to the nature of the iron carbide silicon ternary equilibrium system (Fig. 2). However a simplified schematic diagram presenting the essential ideas is given in Fig. 3. With reference to the diagram the freezing and cooling of an iron composition A may be described by the following steps

A. Liquid melt cools until freezing begins at point 1. At this point solid austenite dendrites begin to form and grow until the temperature at point 2 is reached. This step is omitted when the composition is eutectic at B on the diagram.

B. Eutectic (a liquid saturated with respect to two solids) freezing begins as the area at point 2 is entered with decreasing temperature. The eutectic solids which form may be a mixture of austenite and carbide or of austenite and graphite. If the former occurs the iron is freezing as white iron. If the latter occurs the iron is freezing as a gray or a nodular iron. Graphite will prevail if graphitizing factors such as high silicon content and slow cooling rate are operative. Low silicon content and rapid cooling will cause the eutectic to freeze as a mixture of carbide and austenite (white). When the temperature has dropped to point 3 freezing is completed. Thus an iron freezes as white gray or nodular iron. Actually the solidification of nodular cast iron is somewhat more complex than this. If the iron freezes as gray or nodular the nature of the graphite is established during freezing mottled irons are borderline cases where both graphite and carbide have formed.

C. At the end of freezing the structure consists of the solids developed during steps A and B. In gray and nodular irons these are austenite and graphite and in white irons austenite and carbide.

D. Further cooling between points 3 and 4 results in the precipitation of carbon from the austenite present since the austenite may contain as much as 2.0% C at the end of freezing but only about 0.60 to 0.80% as the temperature decreases to point 4. The excess of carbon in the austenite is precipitated as carbide in white irons and as graphite in gray and nodular irons.

E. Between points 4 and 5 the final change occurs in the solid state during cooling. Austenite transforms over the temperature range of points 4 to 5. Because this change is quite complex only a few generalizations are offered. With the most favourable of graphitizing conditions only ferrite is formed in gray and nodular irons. With less severe graphitizing conditions ferrite and pearlite or only pearlite is formed. In nodular cast iron mixed structures of ferrite and pearlite form as bull s eyes of ferrite around the graphite spheroid (Fig. 1). In white irons only pearlite is formed. The final microstructure of white iron such as is used to produce malleable castings.

F. Cooling below point 5 to room temperature produces little change in the iron.

From the foregoing it can be seen that the type of iron whether white mottled chilled or gray is largely established during the freezing process. Furthermore the room temperature microstructure reflects the entire freezing and cooling process of the iron. Thus the properties of cast irons are greatly influenced by the thermal and chemical changes occurring during its entire history from liquid melt to cooled casting.

# STEEL CASTINGS

## INTRODUCTION

Many of the advantages that make wrought steel such an outstanding material of construction can also be assigned to steel castings. In addition the casting process confers special advantages not obtainable otherwise and by the same token is accountable for certain disadvantages.

Steel is strong with tensile strengths ranging from 60 000 to about 280 000 psi. Steel is also ductile and the combination of strength and ductility adds up to give steel great toughness and resistance to shock. The properties of steel can be controlled within rather wide limits by controlling its composition specifically its carbon content. Steel is essentially an alloy of iron and carbon and its remarkable properties and the ability



to control its properties stem from the presence of carbon. For example when carbon is absent iron is quite soft and weak. If carbon is added in as little as 0.2 to 0.3 per cent the strength is raised appreciably and the ductility although reduced is still appreciable. The result is that steel exhibits a versatility found in no other metal. Figure 1a. shows this effect of carbon on the tensile strength and percentage reduction of area of plain carbon cast steel. Curves for yield strength and percentage elongation showing similar trends are also available.

Favored as it is with this means of controlling properties steel is further favored by another control of its properties namely heat treatment. Iron and steel undergo a change in their crystal lattice structure (i.e. the arrangement of the atoms in the solid state) that makes it possible to control properties by controlling the cooling rate from an elevated temperature (1500 to 1650 F). Further control is also obtained by reheating (tempering or drawing) after rapid cooling (quenching). See Fig. 1b.

A special attribute of steel castings in comparison with wrought products is the fact that steel castings have a uniformity of properties regardless of the direction in which they are tested. This so called isotropic behaviour is absent in steel that has been worked down into structural shapes from ingots or billets because the working operation introduces a directionality in properties. Thus steel so worked is tough and strong when tested in the direction of greatest elongation but is weaker and more brittle if tested in a transverse direction. Cast steel does not possess this directionality and is therefore better suited to applications where this effect might prove harmful.

A distinct advantage of steel castings not readily realizable in other ferrous foundry products is ease of welding. The fact that steel can be readily welded with no serious loss of properties means that this valuable tool can be used in fabrication and in the repair and salvage of castings. Of perhaps greater potential importance is the opportunity to combine by welding steel castings with shapes fabricated by other means to produce a composite structure composed partly of castings and partly of wrought steel parts.

Rather ironically one of the major advantages of steel namely its strength and ductility becomes a definite handicap in the foundry. Steel castings require (as do certain other castings and alloys) extensive risering to compensate for a rather large shrinkage that occurs during freezing. After casting removal of these sometimes quite massive gates and risers presents a definite problem since the ductility and strength of the metal preclude their being merely hammered off as in the case of brittle alloys like cast iron. Saws abrasive cutoff wheels torches etc. are required for this purpose leading to high finishing costs in many cases.

The excellent combination of properties found in steel has already been mentioned. From a foundry practice standpoint however it taxes the ingenuity of the designer and metallurgist because of its casting properties and the close limits of its composition. The high pouring temperature of steel also demands that special attention be given to refractories ladles molding sands metal transfer in the shop filling the mold with no misruns and related problems. The high solidification shrinkage of steel also introduces design and molding problems seldom exceeded in other alloys. In the melting of this alloy there are also special problems more or less unique to steel. The nature of the alloy and its reactivity with oxygen and other impurities require that a rather intricate procedure of melting and refining be established to ensure the production of good quality metal.

## MOLDING PROCESSES AND SANDS

Molding for steel castings is no different from that for other casting alloys. However because of certain characteristics of steel certain methods cannot be used and others are not used to the extent that they are employed in other metals.

Steel can be cast into molds made by any of the sand molding processes. Dry sand molds core sand molds skin dried molds and cement bonded molds are used to a greater extent in steel foundries than for most of the other casting alloys. The reason for this is the severe conditions imposed by steel. The problems

associated with various molding methods should become more apparent as these methods are discussed. With reference to molding methods other than those using sand the high pouring temperature required for steel prevents its being made by the permanent mold process except in certain special cases or by die casting or plaster molding. Steel can be poured in investment molds because the investment materials are sufficiently refractory. Graphite molds can be used for steel if precautions are taken to avoid carbon pickup. Ceramic molds can be and are being used.

## Green sand Molding

Many steel castings are made using green sand molds. The general practice is no different from that for other alloys. However steel foundry sands differ from others chiefly in the following characteristics.

### Refractoriness

Because sand in contact with steel may be heated to an excessively high temperature the molding sand must be of sufficient purity so that it will not fuse together or deteriorate. Figure 15.6 illustrates that the sand at a metal mold interface may reach high temperatures but a short distance away the sand does not get so hot nor does it heat up so rapidly as at the interface. As a consequence of the demand for high thermal stability most green sand molding for steel is done with compounded sand mixtures the bond is usually bentonite. Associated with refractoriness of the sand is the problem of durability. The high temperature exposure to which the sand is subjected alters the sand and its bond both physically and chemically leading to a gradual change in its properties unless it is amply replenished with new sand. Unfortunately there is no simple test to indicate the occurrence of these gradual changes. In one investigation it was observed that the rate of deterioration of the sand could be linked with the development of relatively high hot strength and sensitivity to thermal shock with progressive build up of cokey coatings on the sand grains.

### High Permeability and Low Moisture Content

These two requirements are linked together because they are inter related. When sand is heated part of the moisture in the sand is changed to steam. The air in the mold is heated and increases in volume and organic additions may decompose to gaseous products. These gases must be vented away from the mold cavity. Steel heats the mold to higher temperatures than do other alloys hence a greater gas volume may develop and more venting is needed. The necessary conditions can be achieved for steel by increasing the permeability above that required for other alloys and restricting the moisture content to a relatively low value (around 3 per cent). Much of the gas can escape through risers and other openings in the mold.

### Organic and Other Additions

The use of synthetic sands with a relatively low binder content for steel is accompanied by a tendency toward certain casting defects such as scabs buckling and rattails that result from the expansion of the sand as it is heated. The addition of certain materials to the sand may reduce the tendency to form these defects.

The net effect of these special conditions imposed by steel on green sand properties results in establishing a range of properties that differ rather markedly from those for molding sand mixtures used for other alloys. These differences are demonstrated by the data in Table 5.9 which lists typical sand compositions and properties for various alloys including steel.

Much green sand work is done with a facing sand which is especially compounded to produce the desired properties and a backing sand which being essentially reused facing sand is also controlled as to properties and grain size. This practice although it adds to the complexity of molding since it involves delivery of both facing and backing sand to the molder has the advantage of cutting down the quantity of sand that must be treated with additives and ensures sand properties at the metal mold interface that are always under close control.

## Green sand molding Casting Defects

In addition to such defects as ratters buckles scabs hot tears etc. which are discussed elsewhere in this book and also treated thoroughly in reference material another defect that can develop is pinhole porosity. It is characterized by small smooth walled holes elongated in a direction perpendicular to the mold wall and occurring immediately below the casting skin. The exact cause of the defect is still a matter of debate but it is generally agreed that the formation of either CO or H<sub>2</sub>O or both by a reaction at the metal surface or slightly below is responsible. The fact that the defect occurs more frequently in green sand molds suggests that it is at least aggravated by certain conditions existing at the metal sand interface and since the only major difference between green sand and dry sand molds would be in moisture content the formation of H<sub>2</sub>O by reaction between hydrogen and oxygen in the steel is strongly suspected as at least a contributing factor. Moisture in the sand could aggravate the condition by being dissociated to hydrogen which could then diffuse into the steel and react with dissolved oxygen. This would explain why pinhole porosity can be prevented by deoxidizing the steel with aluminum before pouring since the oxygen would react with the aluminum instead of the hydrogen.

### Dry sand Molds and Skin dried Molds

Green sand molding is preferable to other methods of molding because it is more economical and gives maximum production rates. There are times however when because of the need to increase the strength of the mold or to avoid pinholes or for other reasons drying of the mold before pouring is desirable. Superficial drying can be accomplished by heating the surface with torches infrared lamps or hot air or the molds can be dried in large car type ovens at temperatures up to 500F.

The moisture content of the green sand used for skin dried or dry sand molds may be somewhat higher than for ordinary green sand work for greater moldability and also because a higher moisture content leads to greater dry strength.

### Other Types of Molds

A few foundries have used cement as a sand binder but the practice has not been very popular in this country.

One field where investment molding has proved effective is in the castings of the special alloys and shapes used for gas turbine blades and other parts subject to high temperature service that cannot be readily formed by other methods.

Shell molds have been used with some success but there is a tendency to form surface defects. These can be eliminated by use of hill type shell molds. Ceramic molds are also feasible. These permit pouring thinner sections than with conventional sand molds. A special process combining graphite molds and air pressure pouring has been used to produce steel car wheels and other shapes.

### Molding Methods

The usual methods of molding such as hand ramming jolt ramming squeezing and sand slinger ramming are used on steel sands no difference exists in the ramming methods used for steel in comparison with other casting alloys.

## ALUMINIUM AND MAGNESIUM ALLOYS

Pure aluminium and magnesium being relatively poor casting materials aluminium and magnesium castings are actually produced from alloys. The casting alloys used are those having properties peculiarly suited to casting purposes. Since a large number of aluminum and magnesium base casting alloys are available it is evident that quite widely different properties may be obtained from the various alloys. For all these alloys two types of properties should be considered the casting properties those characteristics of the alloy which determine the ease or difficulty of producing acceptable castings and the engineering properties those properties which are of interest to the designer or user of the castings. These two sets of properties can be

used as a basis for studying the similarities and differences of the large number of aluminium and magnesium casting alloys.

## ALUMINUM ALLOYING PRINCIPLES

The aluminium base alloys may in general be characterized as eutectic systems containing intermetallic compounds or elements as the excess phases. Because of the relatively low solubilities of most of the alloying elements in aluminium and the complexity of the alloys that are produced any one aluminium base alloy may contain several metallic phases which sometimes are quite complex in composition. These phases usually are appreciably more soluble near the eutectic temperatures than at room temperature making it possible to heat treat some of the alloys by solution and aging heat treatments. Specific instances of the application of these heat treatments are given in subsequent paragraphs.

All the properties of interest are of course influenced by the effects of the various elements with which aluminium is alloyed. The principal alloying elements in aluminium base casting alloys are copper silicon magnesium zinc chromium manganese tin and titanium. Iron is an element normally present and usually considered as an impurity. Some of the simpler effects of alloying can be considered.

### Copper

The diagram shows solubility of copper in aluminium increasing in the solid state from less than 0.50 per cent at room temperature to 5.65 per cent at 1018 F. Copper above the solubility limit at any temperature appears micro structurally as the phase. The latter phase has a composition approximating the formula  $\text{CuAl}_2$  (46.5% Al 53.5% Cu) and is a hard brittle constituent. By comparison the solid solution phase is relatively soft and ductile. Structurally then increasing copper content in Cu Al base alloys result in an increasing percentage of the hard phase. The mechanical properties of hardness and strength can then be expected to increase as copper content increases while the ductility decreases. A limited percentage of copper thus has a beneficial effect of strengthening and hardening in Cu Al base alloys. Furthermore ductility is reduced to a very low level and brittleness results in alloys of high copper content. Therefore copper percentages do not exceed 12 per cent in most aluminium casting alloys. Actually the copper percentages in aluminium casting alloys are adjusted so that the lower contents 2 to 5 per cent are used in alloys required to have optimum ductility (or toughness) whereas the higher percentages are used when greater hardness and strength are desired.

### Heat treatment of Cu Al Alloys

The mechanical property curves of Cu Al alloys are shown to be markedly shifted by solution heat treatment and age hardening. In fact the degree of strengthening obtainable by heat treatment is greater than that gained by alloying alone. A few elements namely Cu Mg Zn and combinations of Mg and Si confer heat treating potentialities to Al base alloys in which they are present. These are referred to as heat treatable grades of aluminium alloys and they greatly extend the range of properties available in aluminium castings.

**Solution heat treatment.** Solution heat treatment of aluminium casting alloys consists of a thermal cycle of heating a suitable period of holding the metal at some elevated temperature and then rapid cooling of the castings usually by quenching in water. The temperature and time of holding are exceedingly important factors in the treatment. The temperature must be high enough to cause a substantially large amount of the alloying elements (usually present as intermetallic compound phases) to dissolve in the aluminium rich solid solution phase.

After sand casting and slow cooling to room temperature this alloy consists micro structurally of the aluminium rich phase  $\alpha$  and the hard phase copper being concentrated mainly in the latter phase. Reheating the alloy to a temperature of about 900 to 950 F causes the phase to disappear from the microstructure since the higher temperature permits all the copper in the alloy to be dissolved by the

aluminium hence the name solution heat treating. Of course adequate time for dissolving of the phase into the K phase must be allowed. Thus emphasis is placed on the time at temperature of the solution heat treatment. A sufficient holding period at the solution heat treating temperature is one which results in the aluminium rich phase having reached a uniformly high percentage of dissolved alloying elements. When this condition exists rapid cooling from the elevated temperature will retain the enriched solid solution phase 4% Cu 96% Al in the present case down to room temperature. The end microstructure after solution heat treating then is a supersaturated Al rich solid solution phase. In this case the k phase contains 4 per cent dissolved copper rather than the normal amount of less than 0.50 per cent for the slow or equilibrium cooled condition. Since solution heat treating results in a more uniform distribution of soluble alloying elements it also assists in minimizing the harmful effects of segregation developed during solidification. Accompanying the microstructural effects of solution heat treatment are improvements in mechanical properties. A marked increase in tensile and yield strengths and an improvement in ductility are revealed in Fig. 3 as a consequence of this treatment. Most important is the fact that solution heat treatment is the necessary step in preparing the alloys for age or precipitation hardening from which further benefits may be obtained.

Solution heat treatment by chill casting. Rapid cooling from any elevated temperature particularly above 700 to 800 F will cause retention of a supersaturated Al rich phase down to room temperature. Hence casting processes such as permanent mold or die casting which are inherently rapid in their cooling effect have this possibility. Sand casting by contrast is a slow cooling process. Therefore if a given alloy Cu Al for example is cast in a metal mold it will usually show higher hardness strength and ductility than if the same alloy is cast in a sand mold. This point will be considered again later.

Age hardening or precipitation hardening. Natural age hardening is a gradual increase in hardness (and strength) which occurs with the lapse of time at atmospheric temperatures. The increased hardness may reach a maximum value in a few days but may require several years in some alloys. More rapid aging can be caused to occur at elevated temperatures 300 to 400 F. Heat treating to cause aging is called artificial age hardening or precipitation hardening. Aging effects by either method are obtained only from alloys which have been previously solution heat treated. Or the alloy can be aged if it has been processed so that effects similar to solution heat treatment are retained as for example by chill casting. The metallurgical changes associated with aging are exceedingly complex so that only the more simple details are considered here.

Aging or precipitation hardening temperatures are such as to promote precipitation from the supersaturated solid solution remaining from solution heat treatment. In the case of the 4% Cu 96% Al alloy considered earlier the direction of microstructural changes during aging is toward reprecipitation of the phase from the supersaturated k phase developed by solution heat treatment. However the most beneficial aging effects are obtained before microstructural evidence of precipitation is revealed. In fact when the precipitating phase is metallographically visible overaging has occurred. Overaging results in a substantial decrease in hardness strength and other properties.

Temperature and time of aging are exceedingly important factors determining the end effect of aging. High temperatures are to cause rapid aging or overaging at extended times. Low temperatures can prevent aging. Thus it is evident that a proper temperature and time interval will produce the most desirable properties. Aging treatments for specific alloys will be considered later.

## DUCTILE IRON

Ductile iron requires foundry operations which are similar to those for other cast metals. Process control is critical however and the conventional foundry operation must be adapted to the requirements of ductile iron. Since the development of graphite as spheroids is of principal concern in this material factors affecting this

part to the structure are considered first.

## SOLIDIFICATION OF DUCTILE IRON

Although the base chemistry of gray and ductile iron is essentially the same (with the exception of sulfur and magnesium) these alloys solidify according to quite different modes. These dissimilarities are especially pronounced in the solidification of the eutectic and are responsible for many of the processing variations experienced in gray and ductile iron production.

### Development of Graphite Spheroids

This eutectic solidifies in a more or less conventional manner with both the austenite and the graphite in contact with the eutectic liquid. Solidification proceeds by the growth of cells of austenite and flake graphite at the expense of the liquid. Since gray iron is essentially an Fe C Si alloy the eutectic solidifies over a temperature range usually about 60 F. Further cooling of the completely solidified alloy results in the rejection of carbon from the solid austenite and the precipitation of graphite on the preexisting graphite flakes. This process continues until the eutectoid temperature range is attained. Cooling through the eutectoid range will result in a variety of matrix structures from all ferrite to all pearlite depending on the rate of cooling and /or the influence of alloying elements.

In comparison solidification of the spheroidal graphite eutectic in ductile iron starts at temperatures above those of the flake graphite eutectic for similar carbon equivalents. In this case the graphite spheroid is enveloped by a shell of austenite so that only one phase austenite is in contact with the eutectic liquid. Solidification of this type has been termed neoeutectic. Each unit of a graphite spheroid and austenite shell may be considered a cell where carbon must diffuse through the shell of austenite in order for the spheroid to grow. The result is that this process is slower than that of gray iron eutectic solidification and the neoeutectic freezing range is extended to about 120 F. Liquid metal is then present over a wider temperature range and to lower temperatures for ductile iron than for gray iron.

No nucleation of spheroidal graphite occurs once growth of the neoeutectic starts. The number of graphite spheroids is therefore determined at an early stage of solidification. Subsequent cooling of the solidified ductile iron is accompanied by graphite precipitation on the existing spheroids at temperatures down to the eutectoid range. As with gray cast iron the cooling rate through the eutectoid range and/or alloying treatment determines the matrix structure.

The importance of an adequate number of spheroids in obtaining fully spheroidal graphite structures must be stressed. When the number of spheroids is low there are an inadequate number of sites to which the carbon of the liquid may diffuse. Depending on the composition and processing variables either flake graphite or iron carbide will form from the liquid during further cooling. Both alternatives result in properties inferior to fully spheroidal graphite structure.

### Role of Magnesium

As mentioned earlier a magnesium addition is the most commonly accepted method of obtaining spheroidal graphite in either hypo or hypereutectic analyses. Other elements have been suggested but all these have proved inadequate. Among them are cerium calcium and yttrium. The mechanism by which magnesium causes graphite spheroids to occur is unknown however the function of magnesium additions is well known. First magnesium serves as a deoxidizer and desulfurizer of the molten metal. If the oxygen and/or sulfur content of the melt is too high a substantial amount of magnesium will be consumed in the formation of magnesium oxides and sulfides. Second magnesium promotes the development of graphite as spheroids by a mechanism not yet defined. Finally magnesium prevents the nucleation of flake graphite during the solidification process and thereby promotes the growth of graphite spheroids. Generally only 0.05 per cent residual magnesium is necessary to achieve spheroid formation in most ductile irons. Methods of magnesium addition are discussed in a later section.

## **Control of the Common Elements**

### **Carbon**

The carbon content for commercial ductile iron is from 3.0 to 4.0 per cent although much narrower limits are usually desired. Nodule counts are directly affected by the carbon content greater numbers of spheroids formed at the higher carbon contents. Increasing the carbon content also increases castability by improving fluidity and feeding.

Carbon equivalents greatly in excess of 4.3 promote the development and growth of graphite spheroids. Since graphite is far less dense than molten iron these spheroids may become buoyant and float toward the cope surface of a casting resulting in gross carbon segregation. Flotation as this phenomenon is called is prevalent in analyses having carbon equivalents greater than 4.60 and in section sizes greater than 1 in.

### **Silicon**

The normal range for silicon in ductile irons is 1.80 to 2.80 per cent. Since silicon affects the carbon equivalent value it also affects the number of spheroids and the occurrence of flotation. Silicon increases the amount of ferrite formed during the eutectoid transformation and also strengthens the iron by strengthening the ferrite. Additions of silicon are more influential in spheroidal graphite control when the additions are made late (inoculation). This operation is described in a later section.

### **Sulfur**

The most important effect of sulfur in ductile iron is to increase the amount of magnesium required to achieve spheroidal graphite. The level of sulfur in the iron prior to magnesium treatment is a function of the melting practice used. Sulfur content after treatment is usually 0.015 per cent.

### **Phosphorus**

Phosphorus forms the very brittle structure known as steadite in ductile iron as well as in gray cast iron. Since phosphorus adversely affects toughness and ductility a maximum of 0.05 per cent is usually specified.

### **Other Elements**

In addition to the elements carbon silicon sulfur and phosphorus discussed above a number of other elements may be present in ductile iron. Most alloying of ductile iron makes use of manganese nickel molybdenum and copper. Alloys involving these elements may be designed for higher strengths greater toughness or increased high temperature or corrosion resistant properties. Other elements however even in trace amounts may be avoided because of their deleterious effect on the development of the ductile iron structure. Lead titanium aluminium antimony and zirconium for instance have been cited as promoting the development of flake graphite. On the other hand arsenic boron chromium tin and vanadium are known to promote the formation of pearlite and/or iron carbide. Accordingly close control over the quantities of these elements is usually exercised.

## **MELTING PRACTICES**

The relationship of melting practice to the type and amount of spheroidizing alloy used is important to casting quality and physical properties. Considerable reduction of the amount of spheroidizing alloy and the percentage of defective or inferior castings can be realized by paying close attention to charge materials melting methods and control and iron composition. Ductile iron producers have therefore found it necessary to improve normal melting practices and to exercise a greater degree of control than that used for gray iron. Cupola melting is the most common method of melting for ductile iron however electric induction furnaces are in use in a number of foundries. About 75 per cent of the ductile iron producers employ the acid cupola. In nearly all these instances the cupola is used for both gray and ductile iron production. Among those foundries which have provided separate melting facilities for ductile iron the basic cupola is preferred. Approximately 70 to 85 per cent of the tonnage of ductile iron produced is melted in basic cupolas.

## Acid Cupola Melting

Since many ductile iron producers also produce gray iron and are generally limited to using a common cupola for melting both materials the acid cupola has been adopted. Acid melting is much less costly than basic melting. Estimates of the cost differential in lining and maintenance of refractories for the basic cupola have been as high as four to five times as much as for acid refractories. Using acid cupolas necessitates close control over charge materials and coke since the acid slags produced are not capable of reducing the sulfur content of the iron. This results in sulfur contents of 0.06 to 0.12 per cent which if not lowered necessitates the use of increased amounts of spheroidizing alloy. Acid cupola melting however is capable of controlling the more readily oxidizable elements in the charge such as chromium and manganese since it is a more oxidizing process than basic cupola operation. Because of the moderate carbon pickup in acid cupola melting and the desired base iron chemistry the use of pig iron in the charge is required and the use of returns is somewhat limited. Close composition control and high metal temperatures however can be produced without the need of a hot blast.

## Desulfurization

If the high sulfur content of acid cupola iron is not reduced prior to treatment with the spheroidizing agent an appreciable amount of the high cost magnesium alloy will be consumed before graphite spheroidization can occur. A reduction of 0.01 per cent sulfur requires approximately 0.01 per cent magnesium by this technique. It is therefore desirable to desulfurize the iron by one of a number of commonly used methods. Desulfurization from 0.12 to 0.02 per cent has been reported from the injection of calcium carbide into the melt. These injections are commonly made either in the forehearth or in the ladle and have an efficiency of approximately 15 per cent. The fine calcium carbide is injected through a refractory tube using dry nitrogen gas as the carrying agent. The calcium sulfide formed floats to the surface of the melt as a readily removable dross.

Soda ash additions are also used to reduce the sulfur level of the melt and can cause desulfurization from 0.14 to about 0.06 per cent. A second treatment with soda ash may lower the sulfur to between 0.030 and 0.025 per cent. Desulfurization with lime is also used by some producers either alone or in conjunction with other materials.

A recent innovation of desulfurization has been the development of the shaking ladle. In this process desulfurization occurs by the reaction of lime with the sulfur of the melt. Shaking the ladle increases the contact of the iron with the lime resulting in sulfur levels as low as 0.02 per cent at a 70 to 75 per cent efficiency.

## Basic Cupola Melting

Basic cupola melting is characterized by the definite advantage of sulfur control. Average sulfur content of the basic melt before spheroidizing ranges from 0.025 to 0.035 per cent. This decreased sulfur level in the melt is obtained at the expense of higher operating costs higher silicon losses during melting less effective temperature and composition control and a greater carbon pickup during melting. Attempts to reduce the refractory cost and to provide greater operating control have resulted in the widespread use of water cooled cupolas and the incorporation of hot blast equipment. When operated on a steady and continuous basis however basic cupolas are capable of producing a high carbon low sulfur content melt at a lower cost than acid cupola melting.

## Induction Furnace Melting

The most widely used induction furnaces for ductile iron production are the low frequency 60 cycle type of unit. These furnaces can be operated either for cold melting or for duplexing i.e. using the induction furnace to superheat an existing melt. Very close control must be exercised over raw materials in these furnaces since the rust on scrap and other slag forming ingredients rapidly attacks furnace linings. Extremely close



control of composition and of metal temperature is possible in these furnaces so that quality ductile iron can be produced. Future trends in the ductile iron industry indicate that an increased use of low frequency induction furnaces is to be expected.

The use of melting units other than those mentioned for ductile iron production is not widespread because of either their cost of operation lack of versatility or the degree of control which can be exercised over metal composition and temperature.

## MALLEABLE IRON

American malleable iron occupies the unusual position of being truly a product born of the American foundry man's inventiveness. The first blackheart malleable iron castings were developed by Seth Boyden at Newark N.J. starting in 1826. Boyden's work eventually resulted in the growth of the American or blackheart malleable iron industry until it has become the third largest tonnage producer in the castings field.

Malleable iron is an important engineering material largely because its properties offer certain special advantages among the family of cast irons. Desirable properties include ease of machinability toughness and ductility corrosion resistance in certain applications strength adequate for wide usage magnetic properties and uniformity resulting from 100 per cent heat treatment of all castings produced. Applications of malleable castings usually reflect a need for one or more of the foregoing properties. Principal users of the castings are the automotive and truck industries construction machinery producers and agricultural equipment makers.

The properties of malleable iron are mainly related to its metallographic structure. Malleable iron may be defined micro structurally as a ferrous alloy composed of temper carbon in a matrix of ferrite containing dissolved silicon. The structure is the result of heat treatment applied to white iron castings. The chemical composition of the common grades of white iron which may be heat treated to malleable iron is given in Table 1.

Heat treatment converts the massive carbides and pearlite of the white iron to ferrite and temper carbon. Chemically heat treatment causes a change from combined carbon to graphite or temper carbon the combined carbon generally being less than 0.15 per cent by weight after heat treatment. The ferrite structure with interspersed graphite gives malleable iron mechanical properties in the range of those specified in Table 2 under standard malleable iron. The tensile properties and Bhn are characteristic of ferrite alloyed with 1 per cent silicon.

Except for annealing or malleableizing the manufacture of malleable iron castings involves the same basic foundry processes used with other alloys. Molding core making cleaning melting pouring etc. are adapted to the special casting properties of malleable iron which are primarily related to its metallurgical nature. This area will therefore be considered first.

### MELTING

Melting iron for malleable casting is generally performed in the air furnace the cupola induction or direct arc electric furnaces or a combination of these furnaces when duplexing is employed.

#### Batch melting Process

The cold melt air furnace shown in Fig. 3 is used for batch melting. The air furnace is a reverberatory type furnace fired with pulverized coal or oil. Common furnace capacities range from 15 to 40 tons. The furnace hearth is rectangular and provides a molten bath depth of generally less than 12.0 in. Tapholes are provided on the side of the furnace. The side walls are made of firebrick supported by steel and the bottom is either silica sand or firebrick. The furnace top consists of a series of removable firebrick arches known as bungs. By removing some of the bungs the furnace may be charged with cold metal through the top. A

typical furnace charge is given below. Smaller size charge materials are usually placed on the bottom of the furnace. Both charges listed above contain about 50 per cent sprue because this is the usual percentage of remelt in a malleable foundry. The balance of the air furnace charge is selected so that the iron will melt down at about 2.65 to 2.85 C phosphorus and sulfur percentages below the maximum permitted and silicon and manganese within or slightly below the desired analysis range. Less than 0.07 per cent and preferably less than 0.03 per cent chromium should be in the charge since this element interferes with annealing. Melting down is performed with a fuel air mixture which will produce flame temperatures of about 3080 to 3150 F and hold oxidation of the metal to a minimum. A slag forms during melting down from metal oxidation products and refractory attrition. During melting down and as the bath reaches a temperature of about 2600 F the slag is skimmed. The bath temperature is then raised to the desired pouring temperature usually 2800 to 2900 F. Losses of silicon and manganese occur during melting down and until the metal has reached a temperature of about 2700 F. At higher temperatures carbon losses can occur rapidly under oxidizing atmospheres but there may be a silicon pickup from the refractories and slag. The iron gains about 0.05 to 0.15 per cent silicon per hour at 2800 to 2900 F from reduction of silica by carbon in the iron. Typical composition changes during a heat are given in Table 3. Carbon losses are counteracted by melting with a higher fuel to air ratio (reducing) by adding graphite petroleum coke or proprietary recarburizer or by dropping powdered coal on the metal surface from the burners.

The analysis changes occurring in the course of an air furnace heat are accompanied by structural changes in the solidified iron. Early in the heat iron cast into a bar about 1¾ to 2 in. in diameter and 8 to 10 in. long will freeze gray or mottled. Mottling results from the formation of flake graphite during freezing the iron then not being a completely white iron. As the temperature increases above 2600 F and the carbon percentage in the iron drops mottling gradually disappears. Finally before tapping the test bar will cast white and will have a completely white fracture as illustrated in Fig. 5. Generally the objective of quality malleable iron melting is to produce a completely white iron with no free flake graphite in the castings since flake graphite lowers the properties of malleable iron. Melting may be conducted to favor white iron by using high temperatures oxidizing conditions low carbon and silicon percentage in the iron additional steel in the charge moisture in the air and a number of other practices. When the iron has reached the necessary composition limits and is known to freeze white it is tapped from the furnace. Furnace addition of ferrosilicon and ferro manganese may be employed if it is necessary to adjust the analysis of the iron. Tapping is usually done at 2800 to 2900 F and pouring occurs at 2600 to 2800 F depending on casting section thickness. Tapping in air furnace heat may require from 30 min to over an hour depending on the furnace size and pouring facilities.

The strength of malleable iron combined with its ductility makes it suitable for many applications. Probably its greatest engineering value rests in the combination of its mechanical properties service life cost and suitability to many fabricating and processing operations. Among these advantages are

1. Machinability. Malleable iron is among the most machinable of ferrous alloys. Especially desirable is the fact that a high degree of uniformity of machinability in large numbers of castings can be maintained because every casting has been heat treated.
2. Ductility in processing. Many processing operations such as coining crimping press fits punching and straightening can utilize or require ductility.
3. Ductility or toughness in service. Many applications are best served when the casting is capable of deforming rather than fracturing when overstressed. Clamps pipe fitting threads chain links tractor bolster posts and many other cases may be cited.
4. Surface coatings. Corrosion resistance of malleable iron may be greatly increased by coatings of zinc cadmium aluminium and lead. Hot dip galvanizing may be applied to clean malleable castings to provide good corrosion resistance to exposure in a wide variety of outdoor conditions which may be encountered by

electrical conduct boxes and fittings fence fixtures playground equipment castings and numerous other applications.

5. Wear resistance. Malleable iron with a ferritic structure does not have inherent wear resistance other than that normal to soft ferrous alloys. It may be hardened however. If the metal is heated to the austenitic temperature range carbon goes back into solution and permits a hard martensitic structure to be obtained by quenching. Caster wheels cams rollers and other items may be flame or induction hardened to give wears resistance.

6. Magnetic properties.

# RESIN CHARACTERIZATION

## INTRODUCTION

Optimum conditions for curing thermosetting resins used in particleboard and other composite wood products differ between manufacturing sites and quite often change in any one operation due to planned or unplanned alterations of the process variables. Composite manufacturers rely almost entirely on adhesive suppliers to provide them with the most appropriate resin to bind their product. Necessary changes in resin formulation or synthesis modification are based on empirical experience and often require extensive laboratory and plant trials. Although the performance of a resin depends on its reaction with numerous variable environmental conditions adhesive characterization is presently restricted to physical and chemical features such as viscosity solids content pH and molecular distribution and reaction in simple gel tests. The purpose of the research outlined in this paper was to develop a means to characterize a resin based on its response to the common process variables of time temperature and moisture.

A number of methods have been used to quantify the extent of reaction in an adhesive as it ages or cures. Gel permeation chromatography (GPC) is useful in the early stages of cure while the resin is still in a liquid stage. Fourier transform infrared (FTIR) spectroscopy can characterize the reactions into the solid stages but represents major difficulties in making quantitative determinations. Nuclear magnetic resonance (NMR) spectroscopy has been used to characterize chemical structural changes in phenolics as curing progresses. Differential scanning calorimetry (DSC) measures the cure of resins by sensing the exothermic heat output of a small sample during controlled heating. However FTIR NMR and DSC do not differentiate chain extension from cross linking reactions and the data are not directly informative about mechanical property buildup.

Because the objective of the research was to characterize resin response to changes in environmental conditions during the pressing process a method was required for measuring degree of cure in samples that had been exposed previously to different combinations of those variables. Prior research showed that the dynamic mechanical analyzer (DMA) had good potential for meeting these needs. The DMA is sensitive to changes in the mechanical stiffness of a curing resin sample and therefore gives a direct indication of the molecular cross linking that occurs in the late stages of resin cure.

The research was motivated in part by problems associated with steam injection pressing of phenolic resin bonded composites. During this newly developed process saturated steam was injected into the mat to enhance transfer of heat to the core. Use of phenolics with this pressing system has produced sporadic results. Good bonding was obtained with Douglas fir ring flakes. However bonding was poor when oak disk flakes were used in conjunction with phenolic resins to make structural flakeboard.

Poor bond formation has been attributed to various factors including precure moisture retardation starved glue lines caused by excess penetration and type of wood particle. The steam injection system was appropriate to this study in that environmental conditions are severe and can be finely controlled in the pressing operation. However techniques developed to characterize the curing and bonding of phenolic

resins used in steam injection pressing are also applicable to conventional pressing operations and can be expanded to include other resin types. MECHANICAL PROPERTIES OF A PARTIALLY CURED RESIN DMA CHARACTERIZATION A Du Pont 983 DMA was used for all mechanical measurements of resin samples. In tests using this DMA the sample a special 0.2 by 12.5 by 35 mm nonwoven glass filter cloth impregnated with resin is clamped horizontally between the ends of two parallel arms (Figure 2). One arm and the sample are driven to oscillation at a prescribed amplitude by an electromagnetic drive. Energy dissipation by the sample causes the actual sample strain to be out of phase with the driver signal. The instrument detects this time shift as a phase angle and calculations are made to determine the storage modulus  $E'$  a measure of the material's stiffness and the loss modulus  $E''$  a measure of the material's viscosity. The ratio of these two properties  $E''/E'$  is designated  $\tan \delta$  which is useful in determining the extent of resin mechanical cure.

In practice the resin impregnated glass cloth sample is first conditioned at room temperature in a humidity chamber (for the rest of the paper called precure conditioning). The sample is then precured for a period of time in an atmosphere of controlled temperature and humidity. After reconditioning (for the rest of the paper called pre DMA conditioning) at 91% relative humidity (RH) over a barium chloride solution the sample is tested in the DMA to determine initial stiffness and its reaction to further heating while in the DMA.

There were numerous difficulties encountered in developing the testing technique. Substrate selection resin application resin shrinkage clamping mode clamping torque oscillation amplitude fixed and resonant frequency modes of DMA operation and isothermal and isochronal heating have been discussed in detail elsewhere. The precure conditioning is necessary to prevent sudden resin expansion or shrinkage with resultant crazing when the sample is exposed for a short time to the desired environmental conditions. For a dry exposure the sample is conditioned over phosphorous pentoxide to bring it close to 0% moisture content. The pre DMA conditioning at 91% RH plasticizes the sample enhancing the response of the partially cured resin to DMA conditions and accentuating differences caused by precure treatments.

Storage modulus  $E'$  curves for two samples precured for two minutes at two temperatures are shown in Figure 3. Typically a sample precured at the higher temperature (160°C) showed a higher initial modulus. However after exposure to further heat in the DMA in this case at a constant temperature of 150°C the sample cured at the lower temperature (130°C) attained a higher ultimate modulus. It was hypothesized that this phenomenon was caused partially by the lengthening of molecular chains in the precure exposure which reduced their mobility and their subsequent ability to cross link during the final curing process in the DMA.

Heat induced softening counteracted by a loss of moisture determined the shape of the storage modulus curve prior to the point where the glass transition and storage modulus rise due to rapid molecular cross linking. Previously the counteracting effects of heat softening and moisture loss have hindered the expansion of DMA techniques to solvent based adhesives. Considerable progress in following resin cure has been made due to the refinement of specific procedural techniques such as substrate selection and pre DMA conditioning at high RH. Additionally in the pursuit of data interpretation the focus has been shifted from the storage modulus  $E'$  to  $\tan \delta$ .

The area under the  $\tan \delta$  curve is related to the development of mechanical stiffness.  $\tan \delta$  curves for a resin exposed to 115°C for three precure times are shown in Figure 4. The areas under the curves indicate the extent to Fig. 3 Storage modulus Resin A precured at two temperatures temperature in the dynamic mechanical analyzer was 150°C.

In Figure. 5 the  $\tan \delta$  area has been plotted as a function of precure time at 115°C for two different phenolic resins. Compared with resin A resin B had a higher molecular weight and contained less free formaldehyde and more NaOH at 115°C resin B reacted at a much lower rate than did resin A. However after precuring at 140°C both resins developed mechanical properties at relatively fast rates indicated by a

rapid decrease in the tan delta areas (Figure 6).

Chemical Advancement of a Partially Cured Resin Differential Scanning Calorimeter Characterization To determine how resins differ in their chemical response to environmental conditions a Perkin Elmer DSC 2 differential scanning calorimeter (DSC) was used to test a partially cured sample trimmed from the DMA specimen immediately following precure exposure and reconditioned to 91% RH. A 10 mg sample was placed in a stainless steel capsule that had been fitted with an O ring to prevent release of volatile components. The partially cured sample was compared to an empty control capsule. The difference in energy supplied to maintain a constant temperature rise of 10°C/min in each capsule was a measure of the exothermic reaction remaining in the partially cured resin and thus the chemical response of the resin to heat. The weight corrected area under the exothermic heat curve decreased with precure exposure time.

The chemical responses of two resins precured at 115°C and 140°C are shown respectively in Figures 7 and 8. Unlike their mechanical responses (Figure 5 and 6) the chemical responses of these two resins maintained the same general relationship at both temperatures.

# FLAME RETARDANT TREATMENT OF WOOD

## INTRODUCTION

Wood has many inherently good properties which make it a preferred building material for many applications. However the flammability of wood can be a problem when wood is used to build permanent structures. The tendency to burn can be greatly reduced by adding fire retardant chemicals to wood. However in many instances these chemicals introduce other problems including increased hygroscopicity and corrosivity and reduced adhesive bonding. These problems occur especially when inorganic salts are used as fire retardants.

A great deal of research has been done to develop treatments that make wood flame retardant but free of the problems associated with leachable inorganic salts. One such treatment is the amino resin system which uses chemicals such as dicyandiamide melamine urea formaldehyde and phosphoric acid. The amino resin fire retardants greatly reduce or eliminate hygroscopicity corrosivity chemical blooming and leaching. Leach resistance is attributable to polymerization of the components within the wood and possibly to some reaction with the cellulose in wood. Despite the reported leach resistance of the amino resin treatment Juneja reports that water leaching can remove as much as 91% of the phosphorus from shingles treated with dicyandiamide phosphoric acid formaldehyde and 71% of the phosphorus from shingles treated with melamine dicyandiamide phosphoric acid formaldehyde.

Urethane foams are commonly made flame retardant by reaction of a phosphorus polyol with an isocyanate. Malz prepared flame retardant compounds by reacting chemicals having free hydroxyl groups with cross linking agents such as isocyanates. Von Bonin propose flame proofing absorbent substrates used in roofing and packaging with a mixture of a polyisocyanate and a condensate that contains phosphorus and has two or more hydroxyl groups. Isocyanates have been reacted with wood to increase its dimensional stability and decay resistance.

Chemicals that contain phosphorus change the thermal degradation processes in wood. They are effective as flame retardants because they reduce the temperature at which pyrolysis occurs and increase the amount of residual char. Phosphorus compounds are acid precursors during combustion or pyrolysis and the acids formed cause selective decomposition of the carbohydrate materials. Dehydration and char formation are enhanced and combustible volatile formation is suppressed.

Objectives of our research were to develop new fire retardant treatments for wood that are leach resistant and at the same time not hygroscopic or corrosive. To accomplish these objectives an aliphatic

diisocyanate and an oligomer phosphonate were mixed in an appropriate solvent impregnated into the wood and cured with heat. Reaction of the diisocyanate with the oligomer phosphonate and the wood should enhance leach resistance.

## MATERIALS AND METHODS

An aliphatic diisocyanate was selected for this study because it is less reactive than aromatic diisocyanates which allows the reaction to be controlled. Reaction does not occur at room temperature in most cases but can be initiated by increasing the temperature. Aliphatic diisocyanates are less likely to yellow than aromatic diisocyanates when exposed to sunlight. Isophorone diisocyanate (IPDI) was used in this study. It has a relatively high molecular weight (222) and a low vapor pressure which make its use and handling relatively safe.

Fyrol 51 used in this study is an oligomer phosphonate containing 20.5% phosphorus and having hydroxyl groups that will react with an isocyanate. Fyrol 51 is soluble in water methanol ethanol. 2 propanol chloroform and dichloromethane. Water and alcohols are not appropriate as reaction solvents for isocyanates and none of the solvents known to swell wood dissolve both Fyrol 51 and IPDI. Chloroform and dichloromethane were selected as the most appropriate solvents for making solutions of Fyrol 51 and IPDI. The ratio of the fyrol diisocyanate solvent solutions was 1 2 20 (by volume). The amount of solvent was increased or decreased to control chemical weight gains in the reacted wood.

### Preparation of Specimens

Specimens of ponderosa pine and southern pine were cut into 25 by 25 by 6 mm (radial by tangential by longitudinal) pieces for dimensional stability tests. Specimens 140 by 7 by 3 mm were cut for leaching thermogravimetric analysis and elemental analysis. All specimens were oven dried at 105 C before weighings and reactions.

### Treatment of Specimens

Specimens were placed in a container inside a desiccator and a vacuum was drawn for 1 h using a water aspirator. Specimens were then covered with solution and the pressure returned to atmospheric. After a 1 h soak the solution was drained and a vacuum drawn for 10 min to remove excess chemicals and solvent. The specimens were then cured overnight at 105 C reweighed and the weight percent gain (WPG) calculated based on the original oven dry weight. Ten specimens of each size were treated at each level of treatment.

### Leaching

Three different methods of leaching were used. The method used depended on what was to be done later with the specimens. Method 1 was used with specimens milled to pass a 40 mesh screen. One half of the milled specimens were soaked 24 h in distilled water followed by several rinses with distilled water. Next these specimens were rinsed with several 50 ml portions of acetone then oven dried at 105 C. Each half of the milled specimens was subjected to thermogravimetric phosphorus and nitrogen analysis and unleached and leached portions were compared.

Method 2 a combination of water and solvent leachings was done using solid specimens (140 by 7 by 3 mm). Leaching data represent the average of four specimens in each group. The specimens were (1) leached 4 days in running water (2) extracted 18 h with toluene ethanol (2 1 v v) in a Soxhlet extractor (3) extracted 18 h with acetone in a Soxhlet extractor and (4) leached 4 days with running water. Before and after each cycle of leaching or extraction the specimens were oven dried and weighed and the WPG was calculated based on the original untreated weight.

Method 3 consisted of 4 day cycles of leaching in running water. Solid specimens (140 by 7 by 3 mm) were subjected to three cycles of leaching with oven drying and weighing before and after each cycle. The WPG based on the original untreated weight was calculated after each cycle. Data reported are the average of

four specimens per group.

## Dimensional Stability Tests

Dimensional stability was determined using a flat bed micrometer by measuring the increase in volume (swelling) of treated and untreated specimens. Specimen volume was determined oven dry and after specimens were soaked in water. The volume was measured after each hour for the first 5 h then after 24 h. The percentage of swelling was calculated from the wet volume of the specimen compared to its treated oven dry volume.

## Thermogravimetric Analysis

Thermogravimetric analysis (TGAs) was done using a Perkin Elmer TGS 2 system. Specimens were pyrolyzed in a flow of nitrogen (40 ml/min). Pyrolysis temperatures were programmed from 30 to 600 C at 20 C per minute. The specimen weight remaining at 600 C was used to calculate the percentage of residual char. The temperature at maximum rate of pyrolysis was recorded.

# FUNGAL AND TERMITE RESISTANCE OF WOOD

## INTRODUCTION

Protecting wood against attack by fungi and termites by methods based on modification of the cell wall polymers has been investigated at the Forest Products laboratory for several years. Most of this research has dealt with the bonding of reactive organic monomers to the hydroxyl groups on lignin hemicelluloses and cellulose. More recently we have investigated chemicals that form stable complexes with cell wall hydroxyl groups and are resistant to water leaching.

Two chemicals that form stable complexes with wood are periodic acid and sodium periodate. Periodic acid is known to react with diols in carbohydrates with cleavage and oxidation taking place between the two diols. This method has been used for many years to determine carbohydrate structure. Even though these chemicals have been reacted with isolated carbohydrate polymers and monomers their reactions with whole wood have not been reported.

In preliminary investigations we found that both periodic acid and sodium periodate reacted with wood to form leach resistant complexes. Little oxidation of the cell wall polymers took place as evidenced by infrared spectroscopy.

The purpose of this research was (1) to determine the resistance of reacted wood to brown and white rot fungi and subterranean termites in standard laboratory tests and (2) to study the reactions of periodic acid and sodium periodate with wood.

## MATERIALS AND METHODS

### Fungal evaluations

Loblolly pine or sweet gum sapwood blocks 1.9 X 1.9 X 1.9 cm (radial X tangential X longitudinal) in size were selected according to the American Society for Testing and Materials (ASTM 1976) standards with three to four annual rings per centimeter. Blocks were reacted with aqueous solutions of either periodic acid or sodium periodate at six concentration levels 1% 0.5% 0.25% 0.1% 0.05% and 0.01. % (w/w).

For each test wood blocks were placed in a vacuum chamber for 1 h at 16 to 22 mm Hg. They were then impregnated with one of the six aqueous solutions. Blocks treated with periodic acid were soaked for 2 h those treated with sodium periodate for 4 h. After soaking seven blocks per treatment were dried under a hood for 1 day and then conditioned at 27 C and 30% relative humidity (RH) for 3 weeks. Another seven blocks per treatment were leached in 350 ml of distilled water daily for 2 weeks. After leaching the leached blocks were also conditioned at 27 C and 30% RH for 3 weeks.

Soil block fungal decay tests were run according to the ASTM standard. *Gloeophyllum trabeum* a brown rot

fungus was used with loblolly pine blocks and *Coriolus versicolor* a white rot fungus was used with sweetgum blocks. Five replicate blocks from each treatment and five control blocks were tested for decay resistance over a period of 12 weeks. The extent of fungal attack was determined by weight loss. Solution retention concentration that resulted in weight loss by decay of less than 2% was generally considered as the threshold retention.

## Termite Evaluations

Loblolly pine sapwood blocks 0.4 x 2.5 x 2.5 cm (tangential x radial x longitudinal) in size were selected according to the ASTM standards with four to six annual rings. Blocks were reacted with aqueous solutions of either periodic acid or sodium periodate at five concentration levels 5% 1% 0.5% 0.25% and 0.1% (w/w) in addition 10% concentration was included for the periodic acid solution.

For each test eight blocks were placed in a vacuum chamber for 20 min at 16 to 20 mm Hg. They were then impregnated with one of the six aqueous periodic acid or five sodium periodate solutions and soaked in the treating solution for 24 h. After soaking four blocks per treatment were dried under a hood for 1 day and then conditioned at 27 C and 30% RH for 3 weeks. Another four blocks per treatment were leached in 200 ml of distilled water daily for 2 weeks. After leaching the leached blocks were also conditioned at 27 C and 30% RH for 3 weeks.

*Reticulitermes flavipes* for this study were freshly collected at Janesville WI. Each treated and control block was exposed to 1.0 g of termites (natural caste mixture averaging 270 undifferentiated functional workers 1 soldier and 0.3 nymph). Termite resistance of the treated wood was evaluated for a period of 4 weeks. The extent of termite attack and mortality were determined by weight loss of the blocks and final live weight of the termites. Threshold retentions based on weight loss and termite mortality were determined.

## Reaction Time and Chemical Analysis

Fourteen loblolly pine blocks (1.9 X 1.9 X 19 cm) were placed in a vacuum chamber for 1 h at 16 to 22 mm Hg. They were then impregnated with 0.1% periodic acid solution and soaked at room temperature for 2 8 and 24 h. After soaking seven blocks per treatment were dried under a hood for 1 day and then conditioned at 27 C and 30% RH for 3 weeks. Another seven blocks per treatment were leached in 350 ml of distilled water daily for 2 weeks. After leaching the leached blocks were also conditioned at 27 C and 30% RH for 3 weeks. Five replicate blocks from each treatment and five control blocks were tested for fungal decay resistance by *G. trabeum* over a period of 12 weeks. The extent of fungal attack was determined by weight loss.

# WEATHERING OF WOOD

## INTRODUCTION

Wood is an extremely durable material even under adverse conditions but the durability depends upon the environment (Fig. 1). Buried deep under ground fully exposed to the weather submerged under water or hidden in an ancient tomb it can last for tens of centuries. There are many examples of structures ships and other wooden objects which have survived centuries of use. The same type of wood exposed to an unfavourable environment however may vanish almost without a trace within a year or two. In the tropics a wooden house may under certain conditions disintegrate in a few years.

Like other biological materials wood consists of organic substances primarily polysaccharides and polyphenolics cellulose hemicelluloses and lignin. Extractives are also present in relatively small quantities and their concentration determines color odor and other properties of a wood species.

The degradation of wood by any biological or physical agent modifies some of its organic molecules. The cause of the change may for example be an enzyme a chemical or electromagnetic radiation but invariably the net result is a change in molecular structure through some chemical reaction. Stalker conveniently



divided the environmental agencies that bring about wood degradation into categories. Physical forms of energy were used to describe all factors other than fungi insects or animals. In Table 1 the importance of the various destructive agents on wood can best be considered by comparing two situations inside and outside wood structures. The most serious risk to wood indoors comes from the intense heat of an accidental fire. Outdoors the factor most deserving of attention is weathering a complex combination of chemical mechanical and light energies.

Fig. 1 Old Fairbanks house at Dedham Massachusetts. Built in 1637 most of the white pine clapboard siding was replaced in 1903 and has stood 75 years without paint.

Weathering is not to be confused with decay which results from the presence of excess moisture for an extended period of time. The condition of decay can lead to rapid deterioration of the wood and result in a phenomenon far different than that observed for natural outdoor weathering.

## EARLY HISTORY

Apparently Berzelius (1827) was the first to comment on the changes in wood caused by weathering (Browne 1957). Studies in considerable detail were made by Wiesner (1864) Schramm (1906a 1906b) Wislicenus (1910) Browne (1925) and Frey Wyssling (1950). Browne (1957) concluded that there was not a sufficiently thorough going analysis of weathered wood to establish conclusively the changes that take place in the proportions and composition of the lignin and of the cellulose as normal wood changes to weathered wood. An excellent annotated bibliography was compiled by the California Redwood Association which provides a source of references which pertain to wood weathering and applied surface treatments and/or coatings as well as to wood substrate and/or extractive modification materials and methods the CRA bibliography covers references to significant published material and to pertinent unpublished material as well.

## THE WEATHERING PROCESS

In outdoor weathering of smooth wood original surfaces become rough as grain raises and the wood checks and the checks grow into large cracks grain may loosen and boards cup and warp and pull away from fasteners. The roughened surface changes color gathers dirt and mildew and may become unsightly the wood loses its surface coherence and becomes friable splinters and fragments can come off. All these effects brought about by a combination of light water and heats are comprehended in one word weathering.

### Weathering Factors

Action of water. The principal cause of weathering is frequent exposure of the wood surface to rapid changes in moisture content. The action of water on wood has been thoroughly described. Rain or dew falling upon unprotected wood is quickly absorbed by capillary action in the surface layer of the wood then adsorbed within wood cell walls. Water vapor is taken up directly by adsorption under increased relative humidities. Adsorbed water has been shown to virtually add its volume to that of the cell walls resulting in swelling. Stresses are set up in the wood as it swells and shrinks as a result of moisture gradients between the surface and the interior. These induced stresses are greater the steeper the moisture gradient and are usually concentrated near the surface of the wood. When unbalanced they may result in warping cupping and face checking. Grain raising results from differential swelling and shrinking of summerwood and springwood.

Fig. 2 White oak log cabin near Middleton Wisconsin constructed about 1845 and never painted or finished.

Fig. 3 Close up view of weathered white oak logs in Fig. 2.

Action of light. The photochemical degradation of wood or wood related materials has been reviewed in several publications. It was recognized quite early that the initial color change of wood exposed to sunlight was a yellowing or browning. The graying of wood occurs after browning and was thought to be related to

iron salts. Sunlight particularly the ultraviolet (UV) end of the spectrum degrades the organic materials in wood lignin decomposes preferentially to a relatively shallow depth of 0.05 to 0.5 mm. Photo degradation by UV light induces changes in chemical composition particularly in the lignin.

Browne found that infrared light penetrated deeper than visible light while the penetration of UV light was negligible. Stout using reflectance curves showed that absorption of UV light is primarily due to lignin and lignin like substances. Pine cellulose exhibited a high reflectance whereas the reflectance curve for lignin substances closely approximated that for wood. Sandermann and Schlumbom made a comprehensive study of color changes of numerous wood species. In another study decomposition by UV light as indicated by the coloring process of wood during the first several hours of exposure appeared to be independent of ambient atmosphere exposed wood samples darkened with or without oxygen in the environment. Desai described the photo degradation process for cellulose.

It is important to note here that the two most significant elements of weathering light irradiation and water tend to operate at different times. Exposed wood can be irradiated after having been wet by rain or when surface moisture content is high from overnight high humidity or from dew. Time of wetness therefore is an important parameter in relating climatic conditions to exterior degradation. The action of the combined elements can follow different degradation paths with irradiation accelerating the effect of water or the converse.

Action of heat. The role of temperature in the natural weathering process is generally felt to be of less importance than those of light and water.

## Property Changes

Chemical changes. Over a century ago Wiesner reported that intercellular substance of wood had been lost because of weathering and concluded that the remaining gray layer consists of cells that leached by atmospheric precipitation have been robbed entirely or in large part of their infiltrated products so much that the remaining membranes consist of chemically pure or nearly chemically pure cellulose.

Browne in work reported by Kalnins compiled some analytical data on white pine wood that had been weathered outdoors for 20 years. Results showed that weathering degraded and solubilized lignin. Cellulose appeared to be affected considerably less except for the top surface layer of the wood. Similar results were obtained with various kinds of wood exposed on a test fence for 30 years. The top gray layer consistently exhibited very low lignin content. The brown layer immediately under the outer gray layer had a lignin content varying from 40% to 60% of that normally found for fresh unexposed wood. The interior wood layers only a few millimetres under the outer gray surface had a wood composition similar to that of normal unweathered wood. Analysis of wood sugars from hydrolysis of a water extract of the weathered wood showed that xylan and araban were solubilized more rapidly than was glucosan. It was also observed that glucose did not predominate in the hydrolysed water extract during analysis although glucose units do predominate in unaltered wood polysaccharides.

The UV degradation process is initiated by the formation of free radicals and begins presumably with oxidation of phenolic hydroxyl groups. This results in a decrease in methoxyl and lignin content and an increase in acidity and carboxyl concentration of wood substance. These photochemical changes are enhanced more by moisture than by heat. The products of decomposition of weathered wood in addition to gases and water are mainly organic acids vanillin syringaldehyde and higher molecular weight compounds which are all leachable. Cellulose is also to some degree attacked photochemically by UV irradiation and is bleached however it is weathered less rapidly than other wood components.

It can be concluded from all the work on chemical changes of weathered wood that absorption of UV light by lignin on the wood surface results in preferential lignin degradation. In the graying of wood most of the solubilized lignin degradation products are washed out by rain. Fibers high in cellulose content and whitish

to gray in color remain on the wood surface and are resistant to UV degradation.

Color changes. The color of wood exposed outdoors is affected very rapidly (Fig.4). Generally all woods change toward a yellow to brown color which is due to the breakdown of lignin and extractives. This yellowing or browning occurs after only several months of exposure in sunny warm climates. Woods rich in extractives like redwood and cedar may first become bleached before the browning becomes observable. In the absence of microorganisms wood weathers to a soft silver gray as a result of the leaching of decomposition products of wood lignin.

Fig. 4 Artist's rendition of the changes (including surface character and color) that occur during the outdoor weathering process of a typical softwood.

Changes in wood color reveal chemical changes in wood during weathering as described in the preceding section. Only those parts of the wood close to the exposed surface are affected. Initial browning penetrates only 0.01 to 2 mm into the wood. As rain leaches the brown decomposition products of lignin a silver gray layer 0.08 to 0.2 mm thick consisting of a disorderly arrangement of loosely matted fibers develops over the brown layer. The gray layer as indicated previously is composed chiefly of the most leach resistant parts of the wood cellulose. This surface color change to gray is observed when the wood is exposed to intense radiation of the sun in cooler climates with little rain. This would also occur on structures with large roof overhang which protects the wood underneath from rain and sun. However another mechanism of graying of weathered wood usually predominates particularly in the presence of moisture.

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