DEVELOPMENT OF A LABORATORY BENCH-TOP ELECTRIC HEATING FURNACE

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CERTIFICATION

It is hereby certified that this work was carried out by OGUNNIYI, Iyiola Olatunji, of the department of Metallurgical and Materials Engineering, Federal University of Technology, Akure, and that the work has not been submitted anywhere else for the award of a degree.

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Dr. John Ade Ajayi
Major Supervisor

EBENEZER

To Thee, my Strength for today and Hope for tomorrow maran-atha.

ACKNOWLEDGEMENT

All honour and glory must first be given to God through Jesus Christ my Lord. He has been the Glory and the Lifter of my head, my strength today and hope for tomorrow, till eternity. In this rests my confidence, that though I am weak, He is always able, and He will never leave nor forsake me.

Also towards the success of this work, there are many among men worthy of great acknowledgement. Such are: Engr. Dr. John Ade Ajayi, the major supervisor for the work; the entire staff of the Metallurgical and Materials Engineering Department, Federal University of Technology, Akure (FUTA); Mr. A. Ponle of Electrical and Electronics Department, FUTA; Mr. Kuti Abiola of Mechanical Engineering, FUTA; many other members of staff of the School of Engineering and Engineering Technology, FUTA; Mr. Johnson of Engineering Geology Laboratory, FUTA; Engr. A. O. Adeleke, Mr. Yemi Olowolafe and other staff and friends at Nigerian Metallurgical Development Centre, Jos; Prof. O. O. Adewoye, CEO, Engineering Materials Development Institute (EMDI) Akure; Mr. Odo Ayodele, Mr. O. A. Olasupo and other staff and friends at EMDI; Mr. Tosin Ijabadeniyi, Mr A. A. Badejo, Mr. Tosin Adedipe, Mr. John Isa, Miss Olubunmi Ogunniyi and other personal friends. The list can be quite extensive. Many people have been of certain impact, but God knows all of them. So unto Him, who knows them best, I commit them all, and I pray that the Lord's goodness and mercy shall manifest more in their lives. May the salvation of God be theirs, and the perfect will of God be realized in their lives in Jesus Name. Amen.

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ABSTRACT

The development of indigenous furnace technology is vital to the Nigerian engineering industry. Existing efforts have developed hydrocarbon fuel fired types such as crucible and rotary furnaces, but with concomitant problems of operational difficulties, environmental pollution, poor product qualities, and temperature control. Fortunately, these problems are usually absent in electric type furnaces. Therefore, there is the need for the development of electric furnaces. This is the thrust for this work: Development of a laboratory benchtop electric heating furnace designed to generate heat by ohmic heating, and achieve a temperature of 1200°C in one hour (on 220V ac), with electronic temperature monitoring and control.

Towards constructing the design, the required electrical components, construction steel sheet and bars were sourced from the local technical market. The refractory raw materials requirements of the design were sourced from two local deposits – Igbokoda beach silica sand and Ijapo Kaolinitic clay deposits, both in Ondo state. Process routes for using these local deposits to produce the refractories were determined by varying pretreatments, particle sizes, compositions and moulding methods for different samples to obtain the combination which will give the best of desired properties in the products.

The prototype was constructed with least design modifications. The resulting furnace was tested for performance evaluation. Although firing could not be continued to exactly 1200°C due to insufficient insulation bricks, a promising heat build up average rate of 20°C per minute was obtained. Also, the control system served for switching on/off the furnace at various set temperatures. The developed furnace has the advantages of ease of operation and control, environmental friendliness, and low cost of operation over the hydrocarbon fuel fired ones.

1.0 INTRODUCTION

1.1 GENERAL BACKGROUND

The Nigerian technological community has been classed as developing for years. Although there are efforts in keeping pace with global technology research and development both in the ivory towers and in the industry, Nigeria finds it difficult to keep pace in a sustainable form in view of the rapid pace of global technological development championed by the developed nations. There is the need to break the consumer syndrome as a nation, and evolve 'a level of productivity based on internal momentum for growth' (The Presidency, 1999).

In pursuance of this, it is imperative to recognize the need to encourage local sourcing and increased local content in her products and services, and to discourage the importation of virtually everything. No sudden boost in the production sector will survive, in a sustainable way, if all machinery and equipment are forever being imported. Though this has long been recognized, yet the result is not glaring, because there has not been much general keenness to seek/adapt applicable research findings to solve local real life problems. In this country, research is hardly accompanied by development, whereas research and development are supposed to be twin events. Since technology is all about developing solutions to life problems, a community can not develop technologically until she has started developing her own techniques of solving her own technical problems – Nigeria not an exception. This is the thrust for this work, which centers on the production of a basic tool for the production engineering industry – furnace.

1.2 FURNACES, ENGINEERING INDUSTRY AND THE CHALLENGE

Furnaces are devices used for intense heating purposes in the metallurgical industry. They are generally classified as melting or heating furnaces depending on the type of application. In heating furnaces, the charge maintains its state of aggregation, while in melting furnaces, there is a change of the state of aggregation (Krivandin and Markov, 1980). Other registers such as kilns, ovens, stoves and boilers are used to describe furnaces in the ceramics, food, and chemical industries. Furnaces directly drive the primary engineering industries – the machine tools and the materials engineering industries – as the tool for smelting, melting and diverse heating operations, to produce various technical components, machine tools, heat treated components, metal ingots, glasses, and so on. The products from these primary industries service the secondary engineering, science, food, chemical, and

textile industries, such that furnaces still one way or the other sustain all these industries. Without furnaces, it will be impossible to make metals, machines, building materials, and so on. Furnaces are therefore indispensable tools in technological development, industrialization and virile engineering industry in any nation.

Nigeria is pursuing technological development; furnaces are basic vital tools in this regard. Then the question is this: where is the technology of quality furnaces in Nigeria? Considering the quality indices of what can pass for a quality furnace such as product quality, ease, speed, efficiency and environmental friendliness of operation; and considering the level of multidisciplinary integration in such quality equipments; then, one will be cautious to supply an answer. In this country, there has been design and construction of crucible or rotary furnaces, with hydrocarbon fuels as energy sources (Oshodin, et al., 2002). These efforts have problems such as high fuel costs, temperature regulation, operational inconveniences, impurities in product, and pollution. They often fall short of the standards of quality furnaces. Since the global trend is more towards electrically powered equipment, for the inherent advantages such as ease of electronic control, cleanliness and ease of operation, and environmental friendliness, attention should be turned this way too in this country. Development of a laboratory bench-top electric heating furnace will be a good step toward development of higher capacity heating and melting furnaces.

Thus far, indigenous production of qualitative electric heating furnace has not been reported in literature. So, a fresh effort will have to be initiated in facing this challenge. There are many foreign models of such furnaces (Carbolyte Furnaces, 1986; Protherm Furnaces, 2002) constructed based on fundamental principles of ohmic heat generation, heat transfer and refractory applications; these ones can serve as good references. By giving attention to understudying and mastering the technology of this equipment, it is believed that maintenance works on the ones in operation, and production of new ones can become a domestic issue. This is the basis for this work, which is the Design and Construction of a Laboratory Benchtop Electric Heating Furnace. The targeted model, which will be for heating about 1kg samples, is hoped to have a maximum temperature of about 1200°C; a chamber size of 120 x 120 x 150 mm; use ohmic heat generation; and operate on 220 V ac.

1.3 JUSTIFICATION FOR THE WORK

i. The work will enhance better mastery of the design, construction and operation intricacies of the laboratory electric heating furnaces;

ii. It will aid maintenance of those in operation, and the repair of damaged ones; and

iii. It will serve as foundation for further development of higher capacity furnaces.

1.4 OBJECTIVES OF THE WORK

The general objective of this work is to develop a laboratory benchtop electric heating furnace. This entails the following specific objectives:

i. To design the furnace;

ii. To devise process routes for the production of the insulating and conducting refractories for the furnace from local industrial mineral deposits.

iii. To construct a prototype of the design; and

iv. To evaluate the performance of the prototype.

In pursuance of this objective, first, literature review and information gathering will be carried out on refractories – types, properties, production, and local deposits; heat generation, power rating; temperature control system technology; and heat transfer. A preliminary design of the furnace will then be made taking cognizance of refractory materials properties and dimension; evaluation of refractories' dimensions by heat transfer analysis; evaluation of total heat requirement by specific heat and heat transfer analyses; ohmic heat generation; power rating; temperature control circuitry, and specifications. Material sourcing will then be carried out based on information gathered on local resources. After this will come the aspect to determine feasible production route for the refractory requirement of the design. The process routes, will also seek to optimize the desired properties of the refractory products. The prototype construction will then be carried out as an assemblage of the refractories, heat generation and temperature control. A performance evaluation will then be carried out to appraise the whole effort.

2.0 LITERATURE REVIEW

2.1 LABORATORY ELECTRIC HEATING FURNACE

Laboratory electric heating furnaces can be described as small capacity chamber heating devices to provide controlled heating rapidly and reliably for experimental operations. The preferred choice of electricity as fuel for laboratory furnaces is obvious, since it offers very neat heating operation, with no pollution, high efficiency of energy conversion to heat, and easy electronic control. Figure 1 and Table 1 show some models and specifications of laboratory heating furnaces (Carbolyte Furnaces, 1986; Protherm Furnaces, 2002; Science Project Furnaces, 2002).

The operating principles of these furnaces vary slightly with models, but for basic models (Carbolyte Furnaces, 1986), it generally entails conversion of electricity to heat in passage through high resistance heating elements. This is called resistance or ohmic heating. The heat generated heats up a refractory tube or chamber, called the work tube, all over its exterior surface that the heat is conducted through its wall to heat up the interior space of the work tube in good time. The interior of the work tube then forms the furnace hearth or chamber. Alternatively, certain heating elements hang and heat up directly inside the chamber. Heat conservation to prevent flow in directions away from the work tube surface is done by backing/lagging the heating zone and the chamber with high insulating refractories. The temperature control is carried out using control electrical circuitry that switches the heater on/off to maintain a set temperature.

From the foregoing, the business of designing a laboratory electric (ohmic) heating furnace demands knowledge of refractories properties and production, heat transfer, electric heat generation by resistance heating, and temperature control instrumentation.

2.2 REFRACTORIES

Refractories are materials that withstand high temperatures, resist the actions of corrosive liquids, dust-laden currents of hot gases and generally harsh service environments (Chesti, 1986). Examples are graphite, silica, alumina and carbon refractory materials. The name refractory actually means hard to fuse. The leading property of this class of materials is, therefore, a high melting point. This is expressed by the term called refractoriness of the refractory material, which is the maximum temperature above which the material begins to degrade under the effect of the environment (heat). Other properties of refractories (critical to

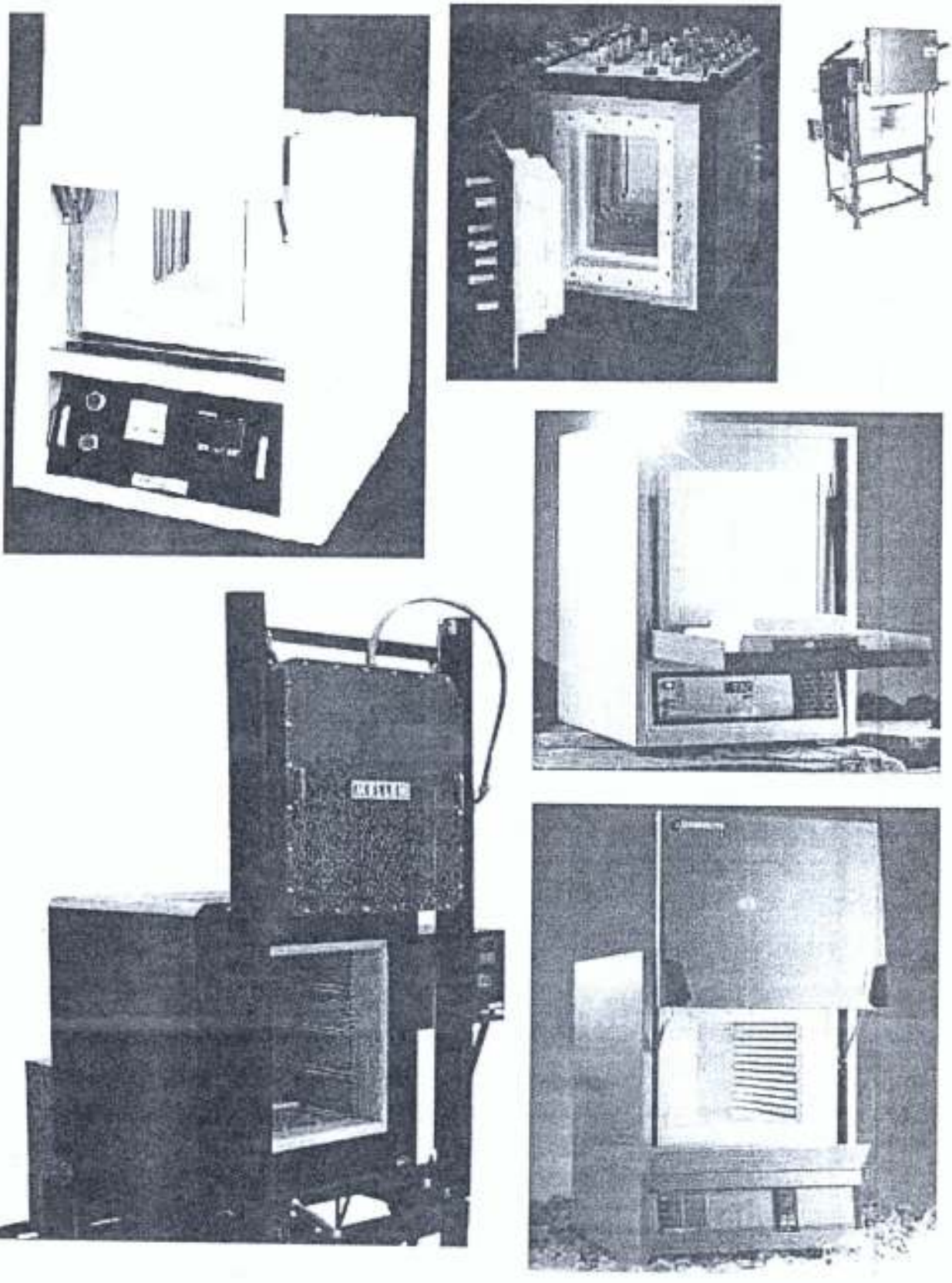


Fig. 1: Representative Laboratory Electric Heating Furnace Models

Table 1: Model Specifications of Some Carbolyte Furnaces

(Carbolyte Furnaces, 1986)

Model Code	EML – 11/2	GLM – 11/7	LMF – 12/5	ESF – 12/13
Max. Temp.	1100°C	1100°C	1200°C	1200°C
Max. Rec. Cont. Operating. Temp.	1050 °C	1050 °C	1150 °C	1150 °C
Temp. Sensor	NiCr/NiAl Thermocouple		Pt/Pt 13%Rh. Thermocouple	
Max Power Rating (kW)	1.75	3.0	6.0	6.0
Heating Time (100°C below max temp.)	65	95	125	110
Controller	PD Analogue (Standard): PID Digital (Optional)			
Chamber Size, H x W x D (mm)	90x150x185	120x175x355	140x190x560	200x250x450
External Size, H x W x D (mm)	425x335x110	625x460x610	580x475x750	740x560x700
Net Unit Weight (kg)	20	50	90	80

particular operations) include; thermal / electrical conductivities, expansivity, chemical inertness, porosity, load bearing ability, permeability, bulk density, true density, spalling resistance, and abrasion resistance.

The values of these properties vary from one refractory sample to another, as a result of method of production and constituent materials, i.e., composition and presence or not of impurities. These variations are expressed by further classifications of this class of materials based on chemical or mineralogical composition, e.g. alumina, silicates; magnetite and synthetic refractories; production method e.g. mold, cast or bonded refractories; predominant property e.g. conducting, insulating, or inert refractories; and reactivity e.g. basic neutral or acidic refractories.

2.2.1 Basis Of Variation In Refractory Properties

Variation in properties between classes of refractory materials result from:

Chemical / Mineralogical Composition – From sources, refractory materials are of basic

properties which vary from one to another. These sources, by reason of their chemical composition fall under certain refractory classes and therefore are of properties peculiar to the class. For instance, quartzite, tridymite, crystobalite and quartz are all silica refractory minerals; bauxite and corrondum are alumina refractory minerals; and coal and graphite rocks are for carbon refractories.

In achieving specific refractory properties, blending of two or more minerals is done, or some naturally occurring mixes are used. For example, aluminosilicates are natural co-occurrence of alumina and silica, with mullite, andalusite and kaolinite as examples of minerals in the aluminosilicate family. Actually, refractory minerals very often consist of two or more compounds and the relative abundance of such co-occurring compounds vary over a wide range between and within deposits. This is why there are many different minerals from the same set of chemical compounds, such as the aluminosilicate minerals cited.

This composition variation leads to production of different phases during the crystallization process of the rock deposits, which implies varying properties. For example, in the Alumina - Silica phase diagram, semi-silica quartz refractory with a fusion point of about 1700°C is on one end of the composition range, and mullite with fusion point of about 1850°C, on the other end.

Production Method The end properties of a refractory sample is a function of the alternatives adopted for various steps involved in the manufacturing process, and the actual values of the combination of variables at each of such steps. For example from the sizing, mixing and moulding stages, higher eventual porosity of the refractory sample can be ensured by ensuring coarser and narrower grain size range, to ensure large interstices and low moulding pressure to reduce compaction. A review of the general manufacturing routes will expound more on the diverse variables with which refractory properties can be manipulated.

2.2.2 Production Process of Refractories

Processing steps involved in the production of refractories, are presented in the flow sheet in Fig. 2 (Chesti, 1986). Examples of the steps in details are discussed under silica and fireclay refractories (sections 2.2.4 and 2.2.5). Mining, crushing, grinding and sizing are first done to maintain about 55:45 ratio of coarse to fine particles. Different equipment used for this purpose are crushers, pulverizers, hammer mills, ball mills, shaking tables and screens. Calcination, which is pretreatment by firing or at high temperature for considerable period of time, is carried out to bring about mineral conversion and stabilization of the material. Mixing is carried out in pug mills to facilitate the even distribution of fine and

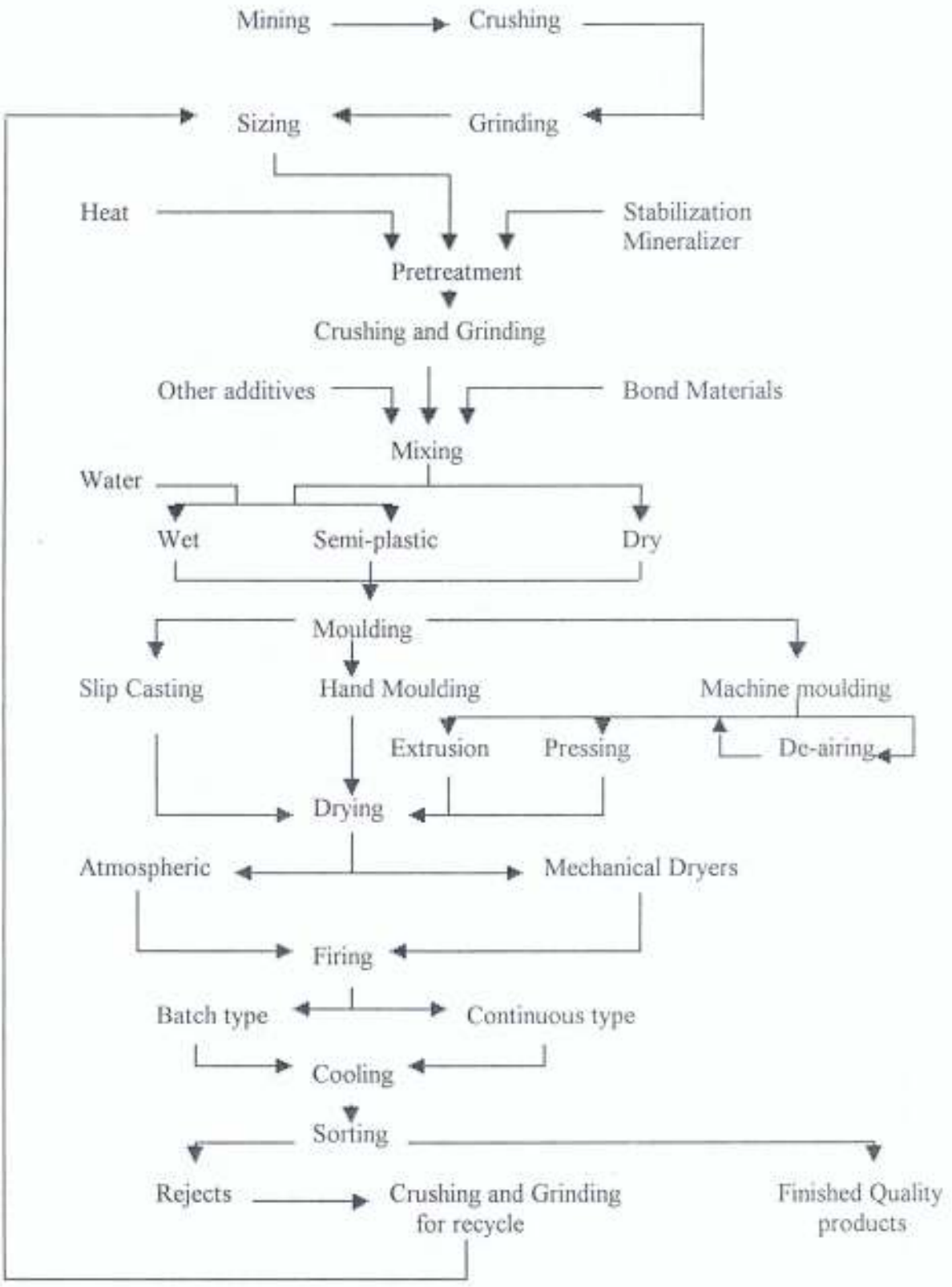


Fig. 2: Flow representation of general steps involved in refractories production (Chesti, 1986)

coarse particles in the whole mass. Mixing can be wet (14 – 20% water), semi-plastic (10 – 8% water) or dry type (< 5% water). Colloidal suspensions can also be produced for moulding by slip casting. Moulding is done to form the refractory mix into desired shape. Moulding can be by hand or some mechanical means. Wet mixes with about 14 - 20% water will be amenable to hand moulding. Semi-plastic mixes uses machine moulding by pressing or extrusion. Dry mixes require high moulding pressures of 10000N/cm^2 or more (Chesti, 1986). Slip casting, powder pressing, reactive hot pressing and isostatic pressing are other methods used for moulding some typical refractories. Drying is done on the mould shapes to remove moisture and increase green strength to make them safe for subsequent handling. Drying rate is kept at minimum to avoid abrupt shrinkages and crack formation. Drying is generally carried out under shade on large drying floors, or in tunnel kilns. Finally, firing, also known as burning is carried out, to completely dehydrate the refractory and effect vitrification, develop stable mineral forms, and cause formation of necessary ceramic bonds for high crushing strength in the finished product. Burning may involve as much as 30% shrinkage in the refractories, setting great stresses in them, which results in the distortion of refractory shapes. To avoid such bad situations, it is necessary to carry out pre-stabilization of the raw materials as mentioned earlier.

2.2.3 Refractories Performance in Service

The most primary service demand of refractories is to withstand high temperatures. For this basic need, any refractory material can serve. Examples of such common refractories are silica, alumina, aluminosilicates, carbon, dolomite and chromite refractories. The final choice will only depend on the actual refractoriness required of the refractory. Further screening criteria can include such properties as thermal conductivity, thermal shock resistance and inertness. In peculiar cases where heat conservation is paramount, it is required that refractories be thermally insulating. Refractories for such environment are specially produced as insulating refractories (see 2.2.6). In more stringent conditions such as requiring refractoriness in order of 2000^{0}C, severe thermal abuses and shocks, extra high strength, specific inertness to specific reagents, chemical abuses and other extremes of aggressive environments, such as in high temperature reactor linings, spaceship cones, electric arc furnaces, tap hole plug and glass melting, special refractories will have to be produced to such a high degree of purity to optimize specific properties. Such special refractories include synthetic silicon carbide, pure fused silica, sialons, pure alumina fusion cast as corrondum,

zirconia, zircon and cermets. Other important service forms of refractories are as refractory mortals, cements, castables and ramming masses.

Refractory Demand for the Laboratory Electric Heating Furnace

For the specific purpose of laboratory furnaces to achieve good thermal isolation, high heat efficiency and conducive operation, refractory requirement divides in to two parts:

(i) **Requirements for the work tube:**

- Refractoriness: about 100^0C excess of maximum operating temperature
- High Thermal Conductivity
- Adequate Thermal Shock Resistance

For the purpose of meeting 1200^0C, almost al common refractories will serve. Table 2 shows common pure refractory compounds and their fusion temperatures, all well in excess of 1500^0C.

Considering thermal conductivity, from Table 3 showing thermal conductivities of some refractories, silica, magnesite, carbon and graphite, in that order of increasing thermal conductivity, k, emerge as leading candidate materials. Further screening based on thermal fatigue resistance disqualifies magnesite; being of very least resistance to thermal shock (Chesti, 1986). Moreover, the thermal conductivities of magnesite refractories reduce with increasing temperature. Carbon and graphite, with best thermal shock resistance and conductivities most satisfy the requirements for the work tube, but the relative scarcity and, therefore, very high cost of graphite and carbon refractories does not justify the not so severe requirements of such a furnace. Therefore silica refractories with relative cost advantage will be a good choice compromise. In pure and fuse form, special silica refractories show very low coefficient of expansion and higher thermal shock resistance; hence silica refractories have served in applications for crucibles, tubes, and thermocouple sheaths.

(ii) **Requirements for heat conservation**

The critical refractory property for good heat conservation is low thermal conductivity. A refractoriness $\geq 1200^0$C is also necessary for a service environment of 1200^0C. To meet this specific requirement, the base refractory material must be produced as insulating refractory, see section 2.2.6.

Table 2: Common pure refractory compound and their fusion temperatures (Chesti, 1986)

Compound	Chemical formula	Fusion temperature, ^{0}C
Silica	SiO_2	1715
Mullite	$3Al_2O_3.2SiO_2$	1810
Silimanite	$Al_2O_3.SiO_2$	1816
Titania	TiO_2	1850
Alumina	Al_2O_3	2050
Spinel	$MgO.Al_2O_3$	2130
Carborundum	SiC	Solid at 2240
Chromium oxide	Cr_2O_3	2275
Beryllia	BeO	2530
Lime	CaO	2570
Zirconia	ZrO_2	2677
Magnesia	MgO	2800
Thoria	ThO_2	Solid at 3000
Carbon	C	Solid at 3600

Table 3: Thermal conductivities of some refractories (c.g.s units). (Chesti, 1986)

Refractory types	Thermal Conductivities			
	Room temperature	$300^{0}C$	$700^{0}C$	$1100^{0}C$
Silica	-	0.003	0.004	0.005
Firebricks	0.002	0.002	0.002	0.003
Silimanite	-	0.003	0.004	0.004
Magnesie	-	0.011	0.008	0.007
Chrome	-	0.004	0.004	0.005
Chrome-magnesite	-	0.005	0.005	0.004
Insulating-firebrick	0.0007	0.0006	0.0008	0.001
Kieselguhr	0.0001	0.0003	0.0004	-
Asbestos	0.00008	0.0001	0.0001	-
Slag wool	-	0.0001	0.0001	-
Carbon	0.1	-	0.005	0.008
Graphite	-	0.21	0.12	0.08
Carborundum	-	-	0.03	0.02

2.2.4 Silica Refractories Production

Quartz is the polymorphic form of silica stable at room temperature and it is obtained from quartzite rock in ~ 98% quartz with 1 – 2%Al_2O_3, titania and other impurities. Generally quartzite with 96 – 97% SiO_2 and <0.3% alkaline are acceptable for silica refractory manufacture.

Pretreatment Quartz being unstable at high temperature necessitates a pretreatment process of firing to achieve mineral conversion of quartz to the high temperature stable allotropes of silica, which are tridymite and crystobalite. Fig. 3 shows the transformations in silica with temperature and associated linear expansions. A short firing at temperature

between 870 – 1470°C will result in the transformations of quartz to tridymite with about 16% increase in volume. On heating to above 1470°C, tridymite transforms to crystobalite with little contraction in volume (Fig. 3). This physical size and structural changes makes pre-stabilization of silica very necessary.

Milling and Mixing Milling is carried out from gyratory crusher to ball mill to achieve the following typical size distribution:

Fraction	*Size (mm)*	*Percentage*
Fine	-0.2	45 – 50
Medium	0.2 – 1.0	10 – 15
Coarse	1.0 – 3.0	35 – 40

About 1 – 2% CaO is added as milk of lime. This serves as bonding agent and mineralizer to accelerate the mineral conversions. Iron oxide, borax oxides of magnesium and barium, and fluorides, phosphates and carbonates of certain metals like sodium, lithium and potassium can be used also as mineralizers, but they reduce refractoriness; hence they should be used only in minimum amounts. Lime-clay mixtures are also used for binding purposes though clay bounding is used less. Lime, too, in excess will reduce strength and resistance to abrasion. Organic binder like cellulos sulphate lye is also used and gives a product with hardened case and improved mechanical strength. Other organic minerals used to increase green strength of silica refractories include tar, gum and molasses. The graded quartzite with the additives in adequate quantities is thoroughly mixed in mixers such as the revolving bottom counter flow type, or pan mills to give wet semi plastic or dry mixes depending on the moulding practice to be adopted; about 7 – 9, 5 – 6 and 4 – 5% moisture contents for hand moulding, screw friction pressing and hydraulic pressing, respectively.

Moulding After thorough mixing, the mix is usually left for a day or more to give semi-dry mix, known as tempered mass of increased plasticity that aids easy moulding. It is to be noted that for good thermal shock resistance, high moulding pressure are not advantageous as a highly compact mass when heated without pre-stabilization, is liable to crack formation because of insufficient gap between expanding silica grains mixtures (Fig. 3). Mould dies used for shaping silica mixtures should be of undersize to the tune of allowable expansion allowance.

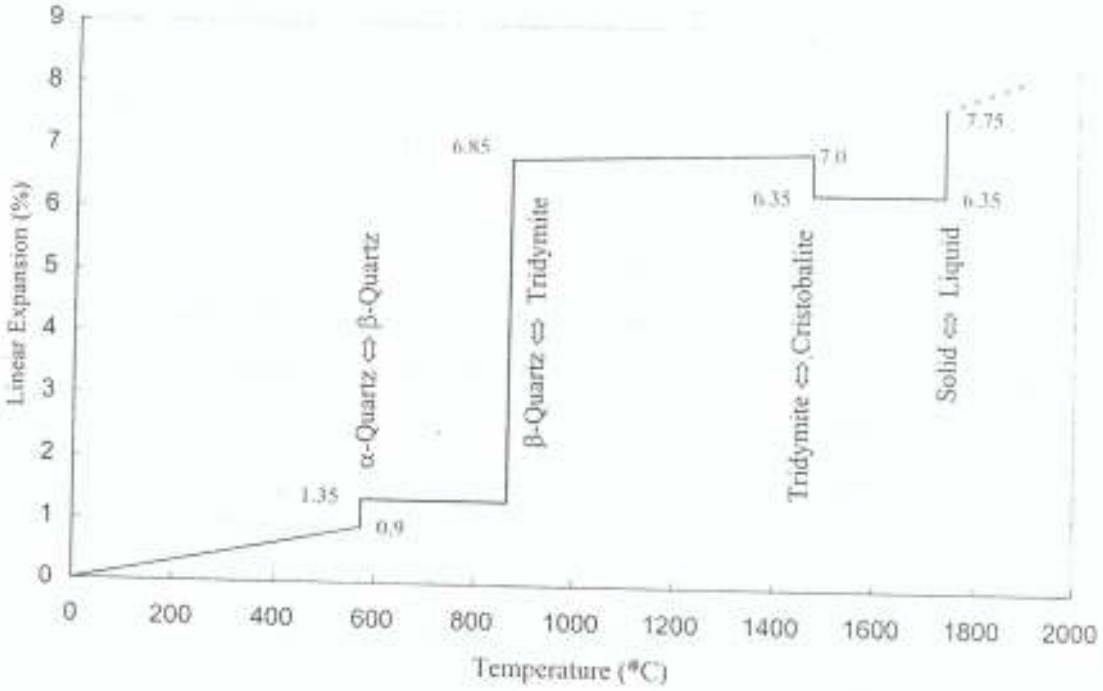


Fig. 3: Transformation in Silica with Temperature and Associated Linear Expansions (Chesti, 1986).

Drying of pressed silica refractories is carried out in well-ventilated rooms. As silica refractories are moulded out of a semi-dry mass containing a very low amount of water, they can be dried on hot floors or rooms to minimize the period of drying to less than 12 hours. Ordinary drying takes about 48 – 96 hours. Care is to be taken to eliminate uneven drying inside and outside the refractory shapes. Tunnel type dryers can be used with heating done by steam or hot gases from the firing kilns. Tunnel dryers provide uniform heating. In case of hot floor/rooms drying, refractories are turned quite frequently to maintain uniform drying rates. Non-uniform distribution of temperature in hot floor/room drying may lead to the formation of cracks in the product.

Burning Silica refractories are usually fired in batch type kilns. Careful handling of dried refractories is necessary as the refractories are still very friable. The temperature of the furnace is slowly increased to about 1450 – 1500°C and maintained for about 10 – 20 hours, depending on the extent of pre-stabilization of the raw refractory. Firing schedule for raw refractory without pretreatment is too delicate and should follows a very low heating and cooling program spanning about 240 hours to accommodate the mineral conversions and the accompanying size changes that will be occurring during the firing. (Fig.3). This makes preparation and firing of silica refractories without pretreatment unadvisable.

Properties of silica refractories

The most prominent of silica refractories properties is that their refractoriness even under high load approaches the fusion temperature and therefore the temperatures corresponding to the their refractoriness under load (RUL) and pyrometric cone equivalent (PCE) values (see 2.2.7 for definition) are quite close; refractoriness for high quality silica refractories is about 1715°C while RUL at 20N/cm^2 ranges from 1650°C to 1690°C, depending upon their chemical composition, that is, purity. Silica refractories show a white or buff colour depending upon the amount of bonding elements and mineralizers added, with specific gravity varying between 2.3 –2.35, depending on the temperature at which it has been fired, and porosities between 22 – 26%. Within this range of porosity values, thermal conductivity varies between 0.003 – 0.004 gm cal/cm/sec at about 700 – 1100°C. Silica refractories crack under sudden temperature change, and this is worse with compact shapes. Silica refractories, being an acid refractory, are resistant to acidic environments except of hydrofluoric and phosphoric acids. Cold crushing strength of silica refractories ranges from 250 to 400 kg/cm^2 depending upon the amount and type of bonding agents present and method of production. They become brittle above 1600°C on account of further

transformations taking place therein. Table 4 summarizes properties of typical silica refractories (Chesti, 1986).

2.2.5. Aluminosilicate Refractories

Alumina (Al_2O_3) and silica (SiO_2) occurring together in the earth crust in various fractions with TiO_2, Fe_2O_3, MgO, Na_2O, K_2O as impurities are called aluminosilicates. Fireclay and high alumina refractories are obtained from this co-occurrences and minerals. Figure 4 represents the equilibrium diagram of various minerals in the $Al_2O_3 - SiO_2$ system.

Fireclay Refractories

Fireclays are naturally occurring earthy materials having the chemical compositions given by the formula $Al_2O_3.SiO_2.2H_2O$. The alumina content of fireclay generally ranges from 25 – 45% and they fuse between 1580 – 1750^{0}C. Table 5 shows the mineralogical composition of some Nigerian clay samples. Presence of alkalis, free silica, gravel, calcium carbonate, iron oxide, calcium silicate, magnesium silicate iron silicate, magnesium carbonate and iron sulphite, in fireclays seriously lower their fusion temperatures and therefore should be free from them as far as possible. Alkalis and basic oxides are the most harmful elements because they form double silicates of lower refractoriness. Clays used for refractories should not fuse at low temperatures particularly below about 1500^{0}C.

Production of fireclay refractories

Clay got from the mines, is left in the open in thin layer for weeks or months. This causes the decaying of organic matter present in the clay due to heat, cold and atmospheric moisture. Some organic acids thus produced increase the plasticity of the clay. This treatment is called weathering or souring. The weathered clays are crushed by gyratory crushers and ground in a suitable grinding mill. This finely ground clay is then mixed with grog as required and if necessary bond clay or calcined bauxite. Grog is obtained by pre-firing of clay: good refractory fireclay is hard fired in rotary kilns and crushed to desired particle sizes to achieve better interlocking of grains. Grog also increases strength of the fired refractories, decreases apparent porosity, and increases specific gravity, increase resistance to sudden changes in temperature, allow less addition of water to get workable plasticity, which implies shorter drying time and increased production rate.

Table 4: Properties of Typical Silica Refractory Compositions
(Chesti, 1986)

Type	Composition				Refractoriness (^{O}C)	RUL @20N/cm^{2}	Apparent porosity (%)	Bulk Density(g/cm^{3})	Cold Crushing Strength (kg/cm^{2})	Permanent Linear Change at1450^{O}C for 2 hrs.
	SiO_2 (%min)	CaO (%max)	Al_2O_3 (%max)	Fe_2O_3 (%max)						
General Purpose	94	2.75	1.5	1.0	1680 – 1690	1670	25	1.80 – 1.90	2.50 – 300	+ 10
Furnace roof	94	2.5	1.3	1.0	1680 – 1690	1670	24	1.70 – 1.75	250 – 300	+1.0
Steel Transfer and Bessemer converter	94	2.5	1.5	1.0	1680 - 1690	1650	24	1.70 – 1.75	250 – 300	+2.0
Coke oven	94	2.25	1.5	1.5	1680 – 1690	1650	17 –24	1.70 – 1.9	250 – 300	+0.4

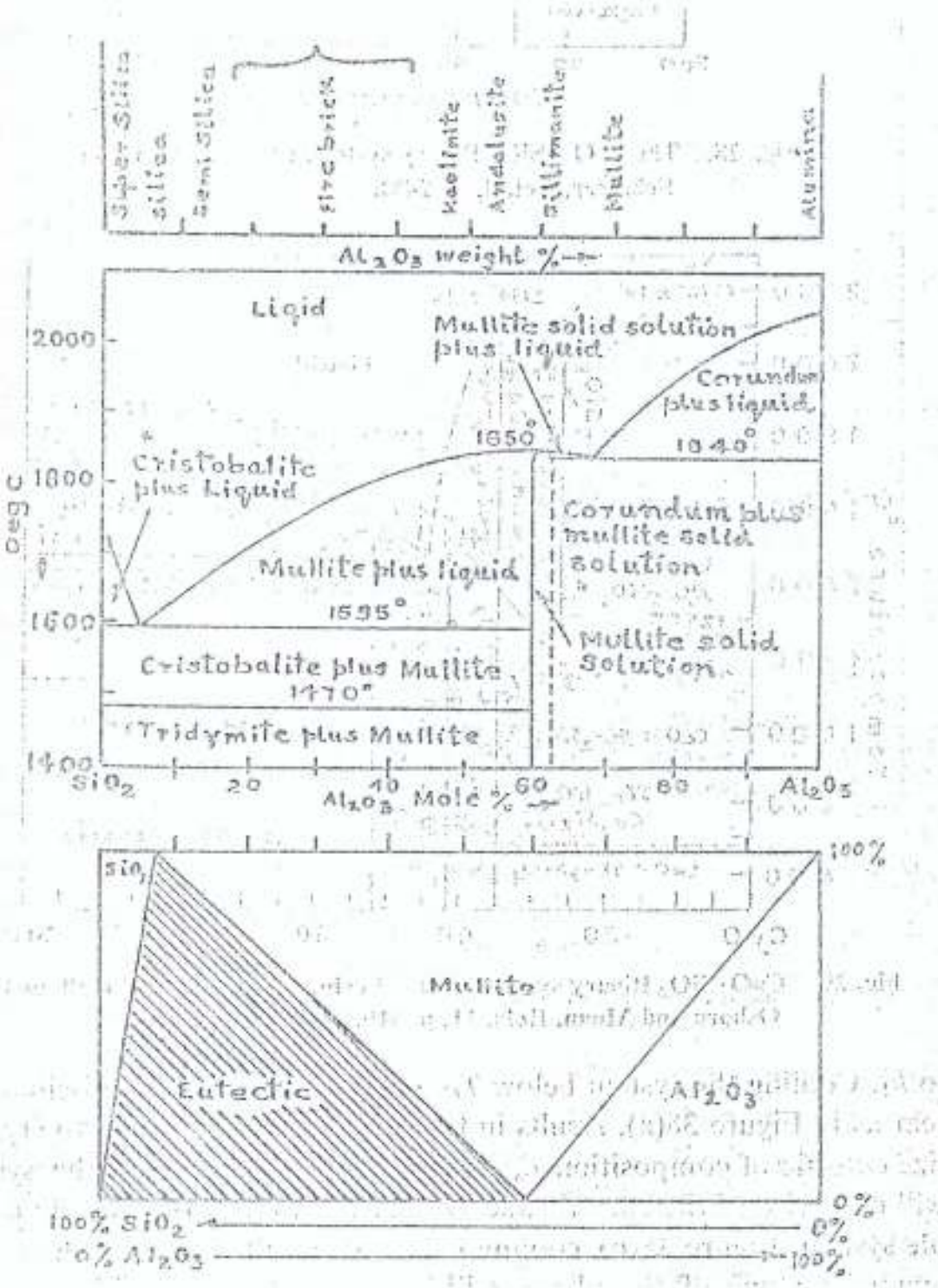


Fig. 4: Equilibrium Diagram of the Various Minerals (Phases) in the Alumina Silica Binary System

(Chesti, 1986)

Table 5: Mineralogical Composition of some Nigerian Clays.

(Afolabi and Fasuba, 1997; Borode et al, 2000, Omowumi, 2001)

Sample Location	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	Na_2O	K_2O	L.O.I
Onibode	46.10	36.07	0.76	1.32	0.14	0.09	0.02	1.2	12.80
Ara-Ekiti	56.66	28.60	1.47	1.88	0.15	0.07	0.08	0.08	10.72
Ibamajo	54.44	22.80	1.0	0.86	0.30	0.2	1.0	1.0	16.73
Ijoko	47.11	37.24	0.56	0.92	0.07	0.08	0.04	0.04	13.60
Ijapo	47.501	30.79	0.491	-	4.77	2.646	1.8911	1.917	--
Ikere-Ekiti	47.38	31.46	9.87	1.138	0.0237	0.001	0.001	0.2962	11.06
Isan	51.10	19.51	9.82	1.484	0.3296	0.7730	0.001	2.529	13.40
Ezinachi-Okigwe	45.00	34.73	1.27	4.02	Traces	0.03	0.02	0.14	13.10

Low grog fireclay refractories contain 20 – 40% grog and 60 – 80% raw clays, while high grog clay refractory contains up to 90% grog. Mixing is done wet or dry in pug mills. Minimum amount of bond clay or calcined bauxite is used as binder. About 7 – 9% or 4 – 7% water is used depending on weather hand or machine moulding, respectively, is to be used. 15 – 20% moisture is used for specials shapes to be shaped by extrusion followed by repressing. Before transferring to pug mill, the mixture may be held for 30 –36 hours to allow the water soak well and evenly. After mixing the mix may also be allowed to mature for a number of days in a cool cellar before moulding to increase its plasticity. During moulding, it is necessary to have provision to remove entrapped air while pressing, as it would otherwise not allow proper compacting. The higher the percentages of fines and moisture in the mixtures, the less effective is the process of deairation. While under-pressing results in a high porosity and low strength product, over-pressing may result in the formation of cracks. Use of refractory mixtures containing high percentages of grog; coarse particles and optimum moisture contents; a proper taper of the mould walls; simultaneous top and bottom pressing cycles of longer duration; and longer retention time at the maximum pressure may help in attaining better and improved densification of the refractory product.

Fireclay shrinks on heating and therefore the moulds and dies used for shaping fireclay refractories are slightly of oversize than the final size of the refractories by amount permissible as shrinkage allowance.

Drying This is done under shade or in well-ventilated rooms, in absence of direct sunshine as faster rate of drying will result in crack formation and non uniform drying. Steam heated floor (100^{O}C maximum) are used for final drying of green hand moulded or extruded products after 1 – 4 days of natural drying. Drying can also be done in continuous tunnel type dryers, with hot air charge at 100 – 250^{O}C and discharge at 60 – 80^{O}C flowing opposite to the motion of the refractory loaded cars. Moisture (and humidity) is carefully controlled in these dryers. Total period may vary from 14 to 200 hours depending on the composition, method of moulding and type and size of products. High grog containing shapes facilitates drying considerably.

Firing This involves five stages (Chesti, 1986):

i. Steaming: Expulsion of residual moisture after drying; temperature – 120^{0}C; 20hours

ii. Expulsion of molecular water and combustible and volatile matter; 350 – 900^{0}C; 20 hours

iii. Oxidation or full fire stage: alumina and silica combine to crystallise mullite ($3Al_2O_3.2SiO_2$) (Fig. 3) at about 1350 – 1380^{0}C. Clay shrink hardened and no shrinkage of the refractories takes place on subsequent heating and in use. About 12 – 36 hours duration.

iv. Incipient fusion: Impurities and filling up of interstices, mass become less porous (compact) and harder, clay and grog particles bond at about 1500^{0}C. The piece is then soaked for some hours at the maximum temperature.

v. Annealing and cooling in the kiln; almost the same time span as heating.

Firing rates are lower for low grog refractories as they undergo higher shrinkages.

Properties of fireclay refractories

Properties of fireclay refractories depend on the proportion and type of clay, and amounts of alumina, grog and other constituents present therein. Refractoriness of fireclay as for aluminosilicates has been related to the chemical composition as (Buduikov, 1977):

$$\text{Refractoriness, } ^{0}\text{C} = \frac{360 + 100\left(\frac{Al_2O_3 - R_2O_3}{Al_2O_3 + SiO_2}\right)}{0.228} \quad . \quad . \quad . \quad 2.1$$

where R_2O_3 is the percentage composition of any other trivalent oxide present in the sample while Al_2O_3 and SiO_2 are the percentage composition of alumina and silica respectively. Equation 2.1 is known as Shuen's formula. It can be noticed from equation 2.1 that the refractoriness decreases with increasing R_2O_3, indicating that impurities reduce refractoriness

and dictate the usefulness of a refractory deposit. The refractoriness of some Nigerian clay deposits shown in Table 6 vary between 1680 – 1760°C.

Other properties:

Colour – light buff to reddish buff depending on the iron content in the raw material; the more the iron content the darker the colour.

Thermal Fatigue – Low coefficient of thermal expansion and hence good resistance to thermal fatigue, (spalling).

Refractoriness Under Load, RUL, decreases with increasing iron contents; it does not fall below 1350°C under 20N/cm (Chesti, 1986).

Chemical activity – slightly acidic in nature, forms fusible aluminates with salts such as chlorides, sulphates and carbonates or bases like lime magnesia and iron oxide ash. The finer the grain size, the better the resistance to otherwise corrosive media.

Porosity of fireclay refractories varies form 8 to 24% depending on the grain fineness and the temperature of firing. If the temperature of firing is high, incipient fusion of the impurities thereby filling up of the voids is highly accelerated, which produces compact hard porous refractories. Amount and fineness of grog used also influences the porosity. Table 6 summarizes physical properties of some Nigerian fireclay sample.

Table 6: Physical properties of some Nigerian clay samples
(Borode et al., 2000; Omowumi, 2001)

Sample Location	Bulk Density g/cm^3	Apparent porosity, %	Permeability	Linear Shrinkage, %	Thermal Shock resistance	Cold crushing strength, kN/m^2	Refractoriness °C
Onibode	2.60	28.40	87	4	30+	17,000	1760
Ara-ekiti	1.84	18.0	75	8	26	14,000	1650
Ibamajo	1.76	16.0	78	4	30+	11,700	1630
Ijoko	2.60	22.0	88	6	28	19,000	1680
Ikere-Ekiti		43.1	-	3.59	20	7740	1450
Isan		49.7	-	3.86	32	15280	1520
Ezinachi -Okigwe		20		7.23	20	12500	1350

2.2.6 Insulating Refractories

Since refractories produced by the normal manufacturing process are dense and heat conductive, insulating refractories are therefore a special class of materials produced to have a highly porous structure with air trapped therein. Still air, being a good insulator greatly minimize the conducting capacity of refractories; and the higher the volume fraction of the pore spaces, the better the insulating characteristics. Hence the less dense and the more porous a refractory material is made to be, the better the insulating characteristics. Fireclay is the most used base material for insulating refractories (Table 3). Generally, low conductivity base material like high alumina, aluminosilicates, e.g. kaolin, will increase insulating characteristics. Anorthite light weight refractories (density – 0.5 g/cm^3, and high strength) produced form cheap raw materials like kaolin and gypsum – like masses by melting and casting are suitable for use at 1100^{0}C to 1250^{0}C and high strength demanding condition. Refractory powders of alumina or zirconia are also used, taking advantage of the loose packing of the powder. These base materials can also be made into ceramic fibres to produce highly porous wools, felts and foam with about 75 - 92% porosity.

Production of Insulating Refractory

Base material can be any refractory material whose composition gives required refractoriness. Fireclay and kaolin are commonly used, with equation 2.1 used for a check of the refractoriness, and insulating ability enhanced by pore creation. Porosity is induced by mixing the base material with a bonding agent and a combustible material like saw dust, volatile matter like naphthalene, or chemical like ammonium chloride or sulphate, and calcium phosphate. Hardened chips or crumbles of wood shavings result in larger size pores. With sawdust, about 8-20% in the mix give densities ranging from 0.85 to 1.5g/cm^3. The mix gives densities ranging from 0.85 to 1.5g/cm^3. The mix is normally moulded by hand, extrusion or dry pressing methods. Insulating refractories being highly porous and relatively weaker require very careful handling. Firing is done normally as for the base material used.

From Table 7 showing the thermo-physical properties of some solids at 300 K, it can be seen that the effect of pore creation producing reduced density of refractory bricks reduces thermal conductivity (W/m K) considerably (Gosser, 1986): Fireclay refractories normally produced to a density of 2.3 g/cm^3 have thermal conductivity of 1W/m K, while kaolin produced to a density of 0.304 g/cm^3 have thermal conductivity of 0.087 W/m K: a reduction ratio of more than 1 to10.

Table 7: Thermophysical Properties of Some Solids at 300 K

(Gosse, 1986)

Material	λ (W/(m K))	C (kJ/(kg K))	ρ (kg/m^3)	$10^7\,a$ (m^2/s)
Pure aluminium	203	0.894	2706	839
94–96 Al, 3–5 Cu, trace of Mg	160	0.882	2787	651
87 Al, 13 Si	163	0.869	2659	705
78–80 Al, 20–22 Si	158	0.853	2627	705
Silver	420	0.234	10490	1711
Bismuth	9	0.121	9800	76
Pure copper	387	0.382	8953	1132
95 Cu, 5 Al	83	0.410	8665	234
75 Cu, 25 Sn (Bronze)	26	0.343	8665	87
70 Cu, 30 Zn (Brass)	111	0.385	8520	338
60 Cu, 40 Ni (Constantan)	22	0.410	8921	60
Tin	65	0.226	7304	394
Pure iron	73	0.435	7896	213
Cast iron	57	0.460	7272	170
Magnesium	172	0.970	1746	1016
Nickel	91	0.445	8905	230
Gold	293	0.124	19250	1228
Lead	35	0.125	11380	246
Tungsten	163	0.134	19348	629
Zinc	112	0.384	7144	408
Carbon steel	45	0.460	7800	125
Tungsten steel (20% W)	44	0.389	8825	128
Invar (36% Ni)	11	0.460	8137	29
Stainless steels:				
15 Cr, 10 Ni	20	0.460	7864	55
18 Cr, 8 Ni	17	0.460	7816	47
25 Cr, 20 Ni	13	0.460	7864	36
Asbestos	0.150	1.046	575	2.5
Concrete	0.935	0.837	2305	4.8
Wood:				
Oak, normal to grain	0.209	2.385	825	1.1
Oak, parallel to grain	0.349	2.385	825	1.8
Pine, normal to grain	0.105	2.720	510	0.76
Pine, parallel to grain	0.244	2.720	510	1.8
Brick:				
mason	0.657	0.837	1700	4.6
clay refractory	1.00	0.837	2300	5.2
kaolin refractory	0.087	0.83	304	3.4
Rubber	0.15	2.009	1200	0.62
Ice (at 0° C)	2.5	1.925	912	14.2
Cork	0.043	0.167	160	16.1
Mica, normal to layers	0.0465–0.58	0.879	2600–3200	≈ 3.2
Expanded polystyrene (33 kg/m^3)	0.032	0.125	33	8
Porcelain	1.035	1.088	2400	4
Silk	0.047	1.381	80	4.3
Dry earth	0.345	1.841	720	2.6
Diatomaceous earth (powder)	0.052	0.879	224	2.6
Window glass	0.779	0.778	2550	3.9

2.2.7 Refractory Testing

The test routines for the refractory properties critical for the laboratory electric heating furnace are presented (Borode et al, 2000; Chesti, 1986; and Omowumi 2001):

Thermal Shock Resistance: 50 mm square base prism by 75 mm height test piece will be inserted in a furnace which has been maintained at 900^0C. The temperature will be maintained for 10 minutes, after which the test sample will be removed from the furnace with pre-warmed pair of tongs and then cooled for 10 minutes on firebrick, before returning into the furnace for another ten minutes. The heating and cooling cycle will continue until the specimen can be pulled apart readily in the hands. The number of heating and cooling cycles sustained serve as a measure of the thermal shock resistance.

Refractoriness: This is determined using the pyrometric cone equivalent (PCE) technique, using the standard pyrometric cone test sample. The test piece is mounted on refractory plaque along with some standard cones. Then the plaque is put inside a furnace and the temperature raised at a rate of 100^0C per minute. The heating will continue until the test piece, among other standard cones, so soften that its tip bends over and touches the base. The softening point of the standard pyrometric cone that most nearly corresponds in time of softening with the test cone is recorded as the PCE value of the test cone.

Linear Shrinkage: Standard slab test pieces will be marked along a line, in order to maintain the same position after heat treatment. The distance between the two ends of the slab will be measured with a vernier caliper. The samples will be air dried for 24 hours and then oven dried for another 24 hours. They will then be fired at 1100^0C for 6 hours. The test pieces will be cooled to room temperature and measurement taken. The firing linear shrinkage will be calculated from:

$$\text{Fired Shrinkage} = 100\% \, (D_L - F_L) / D_L \qquad 2.2$$

where D_L = Dried Length; F_L = Fired length

Bulk Density: This can be obtained either by directly measuring the dimensions of refractory block and its mass to obtain density as mass per unit volume; or by weighing the sample in air and suspended in water, to obtain bulk density, B as

$$B = D / (W - S) \qquad 2.3$$

where D is the dry weight in air, W is soaked weight, and S is weight of sample suspended in water. It is necessary to obtain true dry weight by first air-drying the sample for 24 hours, oven dry at 1100C, then cool in a desiccator.

Apparent Porosity: Apparent porosity is the percentage relationship between volume of the open pore space and the total volume of the sample. That is,

$$\text{Apparent Porosity} = \frac{\text{(Volume of open pores)}}{\text{(Volume of Sample)}} \times 100 \quad \ldots \quad 2.4$$

Since density of water is $1g/cm^3$, weight, in grams of a given volume of water is numerically equal to such volume in cm^3. Hence, using measurements for bulk density, volume value can be inferred as follow:

D = weight of sample with all pores empty

W = D + weight of H_2O to fill open pores = D + volume of open pores

$\Rightarrow$ Volume of open pores = W - D

S = D – Upthrust = D – weight of H_2O whose volume equals true volume of sample

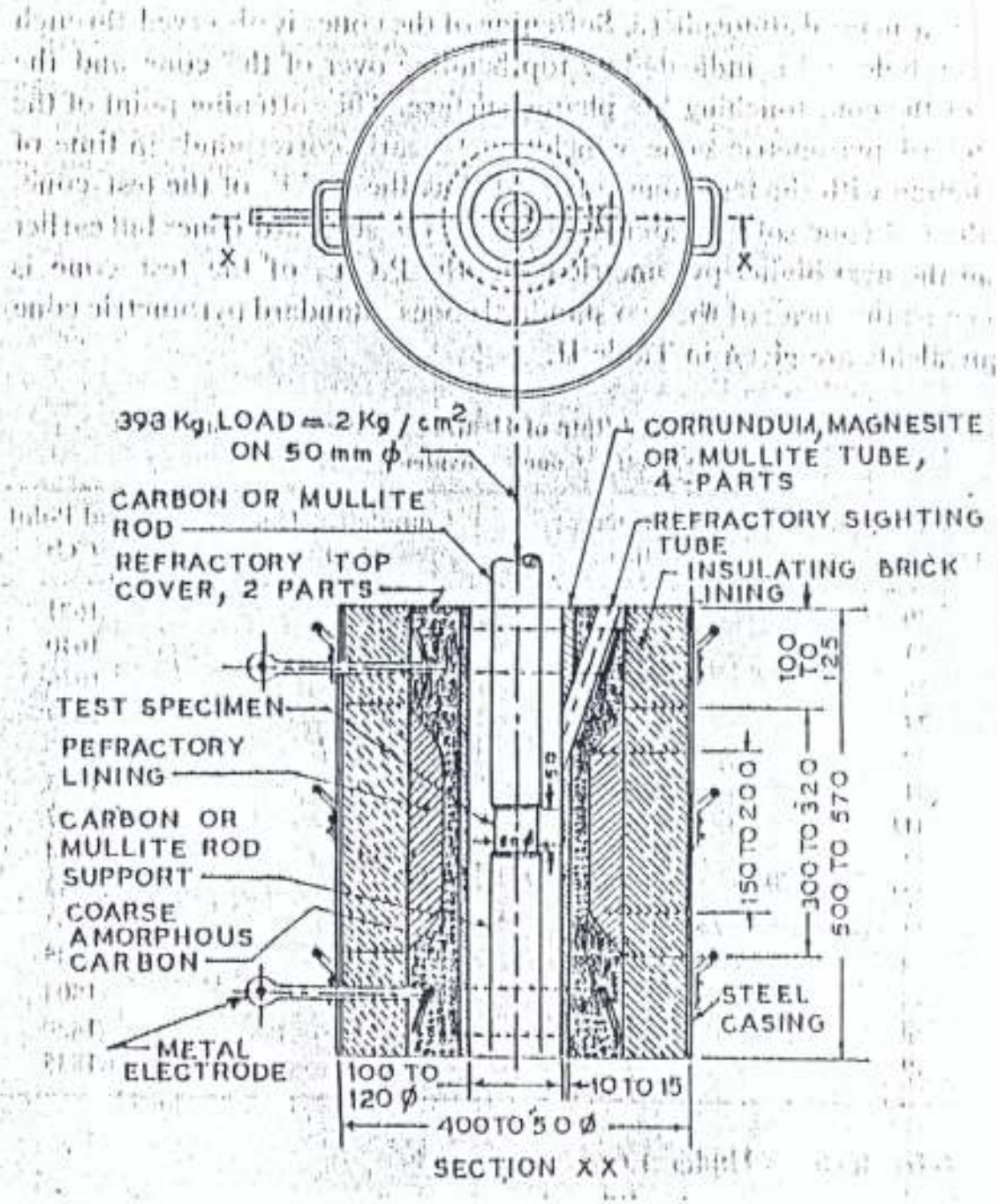
= D – (total volume of sample – volume of open pores)

= D + W – D – Volume of sample = W – Volume of sample

$\Rightarrow$ Volume of sample = W – S

$\Rightarrow$ Apparent Porosity $= \frac{W-D}{W-S} \times 100 \quad \ldots \quad 2.5$

Refractoriness under Load: A Standard test piece (50±5mmdiameter by 50±5 height cylinder) is axially loaded by refractory rods in a special cylindrical chamber electric furnace (Fig. 5 and 6). The standard load is 20 N/cm^2. Heating is then allowed at the rate of 15^0C per minute up to 1000^0C and at the rate of 8^0C per minute above 1000^0C. The heating schedule should ensure that the temperature in the horizontal plane should also not vary beyond 30^0C. The change in the height of the specimen on heating is plotted against time on a 10:1 scale to represent temperature deformation curve for the test. Temperature is measured with the help of an optical pyrometer adjusted such that the temperature recording is done about the middle of the test specimen. This is done by sighting the specimen either obliquely from above or through a 20 mm (max) ID radial tube inserted in the furnace from one of its sides such that it is sloping outward slightly outward and downward. This is tightly closed by means of a total reflecting prism interposed giving allowance for the diminution of light intensity. From the plotted curve the temperature (ta), corresponding to the point at which the curve has dropped 3 mm below its highest point denoted by the bend of the curve downwards with respect to the horizontal tangent, is noted. The temperature (te) corresponding to the point at which the height of the specimen has decreased by 20 mm from its original height is also noted. The temperature (tb) at which premature breaking of the test piece, without its actual softening, takes place denotes the breaking point of the test specimen. Typical RUL curves are shown in Figure 7.



Heating Furnace for RUL Test

sti, 1986)

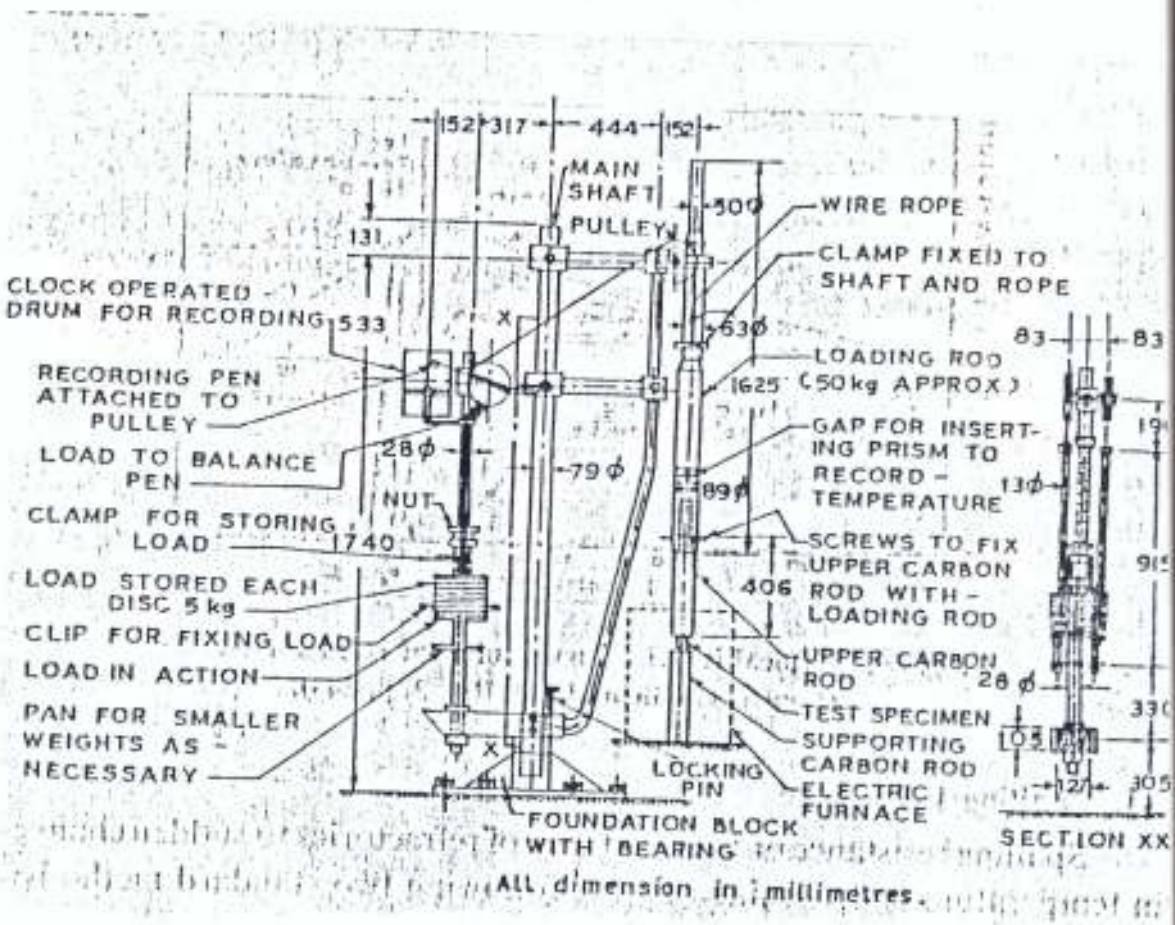


Fig. 6: Loading Arrangement for RUL Test
(Chest1, 1986)

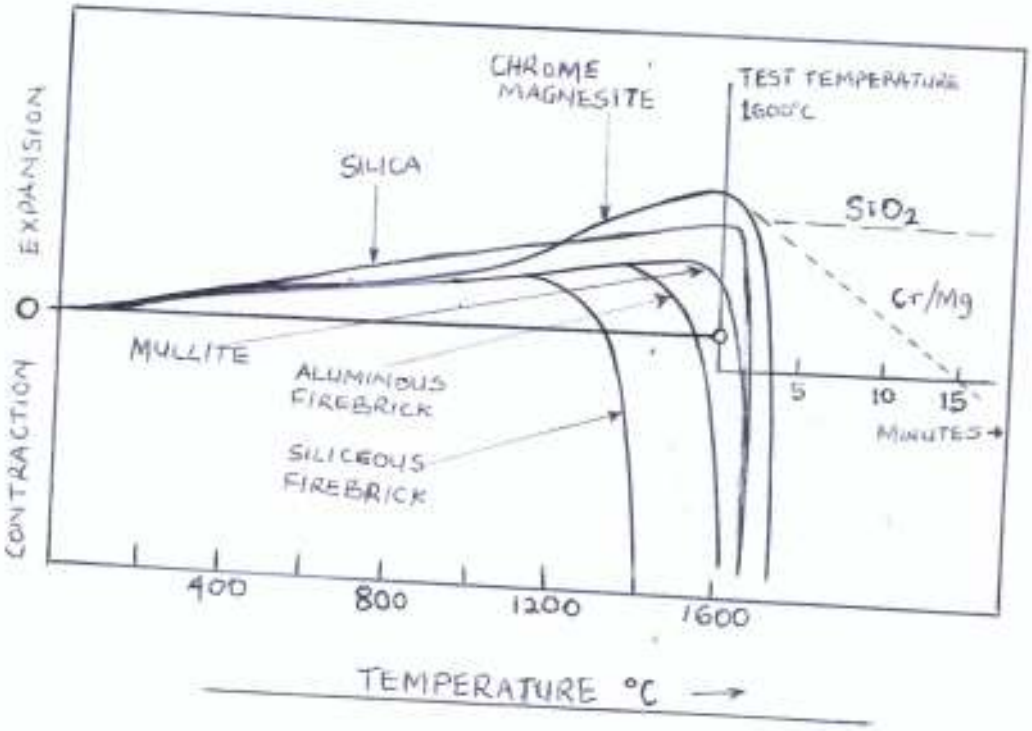


Fig. 7: Typical RUL Test Curves for Some Refractories
(Chesti, 1986)

2.3 HEAT TRANSFER ANALYSIS MODELS IN SOLIDS

Basically, heat transfer analyses in solids seek to obtain the temperature profile in a body as a result of heat been conducted through it. If the temperature difference, dT, over a distance, dx, remains constant, then, the temperature gradient dT/dx can be obtained as a constant, and the temperature – position profile gives a straight line with negative slope, -dT/dx. This simple situation is equivalent to the steady state diffusion model described as first Fick's law (Faulker et al, 1985). Hence heat flux, q, in one direction can be obtained from:

$$q = -\alpha dT/dx \quad . \quad . \quad . \quad . \quad . \quad 2.6$$

where α is $k/\rho c$ is the thermal diffusivity of the conducting material; k, ρ, and c being its thermal conductivity, density and specific heat capacity. But, for a non-steady state where dT/dx varies with time, t, and position, x, then, the temperature, $T = T(x, t)$, and it is describable by the non-steady or transient state diffusion model, called the Fick's second law, such that:

$$\frac{dT}{dt} = \alpha \frac{d^2T}{dx^2} \quad . \quad . \quad . \quad . \quad . \quad . \quad 2.7$$

The first and second Fourier laws of heat transfer follows from equations 2.6 and 2.7. Equation 2.6, is solved subject to different boundary conditions for various situations, to obtain $T(x, t)$, for a given x and t; x for a set T and t; t for a set T and x or $\left(\frac{dT}{dx}\right)_{t,}$ to obtain q_x, using eq. 2.6.

2.4 HEAT BALANCE FOR THE LABORATORY ELECTRIC HEATING FURNACE

To raise the chamber temperature from, say, T_1 to T_2, in a target time, the total quantity of heat required can be divided as:

i. Heat to heat up chamber wall,

ii. Heat to heat up chamber space,

iii. Specific Heat in Insulation,

iv. Heat loss through Insulation

The ohmic heat generation source will then have to supply the resulting total heat requirement to achieve a heat balance for the furnace operation.

2.4.1 Chamber Wall Heat

The quantity of heat to raise the temperature of the chamber wall through θ can be evaluated from the basic equation of specific heating as:

$$Q = mc\theta \quad . \quad . \quad . \quad . \quad . \quad . \quad 2.8$$

where Q is the heat required to raise the temperature of a body of mass, m and specific heat capacity, c, through θ°C. This equation can be reexpressed for chamber wall heat, Qw, as

$$Qw = m\,C_{pw}\,dT = \rho_w V C_{pw} dT \quad . \quad . \quad 2.9$$

where C_{pw} is the specific heat capacity of the wall material at constant pressure; ρ_w is the density of the wall material; V is the volume of the chamber material.

2.4.2 Chamber Space Heat, Hs

This can be evaluated from a modification of equ. 2.9 as:

$$H_s = \int_{T1}^{T2} mC_p dT = \int_{T1}^{T2} \rho V C_{pa} dT \quad . \quad . \quad . \quad 2.10$$

where Cpa is the specific heat capacity of air at constant pressure. The chamber volume being constant, implies:

$$H_s = V\int_{T1}^{T2} \rho C_{pa} dT \quad . \quad . \quad . \quad . \quad . \quad 2.11$$

Since density and heat capacity of air, ρ and C_{pa}, are functions of temperature, they will have to be expressed as such. ρ is related to T as (Gosser, 1986):

$$\rho = {}^{P}\!/_{287T} \quad . \quad . \quad . \quad . \quad . \quad 2.12$$

where T is temperature in K, and P in N/m^2 (Pa) is the pressure. At constant pressure, atmospheric, of 1.013×10^5 Pa, it follows that

$$\rho = 352.962/T \quad . \quad . \quad . \quad . \quad . \quad 2.13$$

C_{pa}: Considering air as a mixture of N_2 and O_2 in 0.79/0.21 proportions, and C_p being physical properties, C_{pa} can be obtained as:

$$Cpa = 0.21Cpo + 0.79\,Cpn \quad . \quad . \quad . \quad . \quad 2.14$$

where C_{po} and C_{pn} are the specific heat capacities, in kJ/kg K, of oxygen and nitrogen as a function of temperature, respectively.

Between 300 – 780 K (Gosser, 1986):

$$C_{pn} = (1.093943 - 3.771278 \times 10^{-4} T + 7.547419 \times 10^{-7} T^2 - 2.967465 \times 10^{-10} T^3) \quad 2.15$$

$$= C_{pnl} \quad \text{(low temperature value of } C_{pn})$$

$$C_{po} = 0.951021 - 4.453037 \times 10^{-4} + 1.395253 \times 10^{-6} T^2 - 8.464839 \times 10^{-10} T^3 \quad 2.16$$

$$= C_{pol} \quad \text{(low temperature value of } C_{po})$$

Between 780 – 1500 K:

$$C_{pn} = 0.839063 + 4.61217 \times 10\text{-}4\ T - 1.412246 \times 10^{-7} T^2 + 9.257405 \times 10^{-12} T^3 \quad 2.17$$

$$= C_{pnh} \quad \text{(high temperature value of } C_{pn})$$

$$C_{po} = 0.671706 + 8.237775 \times 10^{-4} T - 5.376317 \times 10^{-7} T^2 + 1.330776 \times 10^{-10} T^3 \quad 2.18$$

$$= C_{poh} \quad \text{(high temperature value of } C_{po})$$

Hence, C_{pa} can be taken as C_{pal}, between 300 – 780 K, and as C_{pah} between 780 – 1500 K.

This implies that:

$C_{pal} = 0.79\ C_{pnl} + 0.21\ C_{pol}$

$= 1.0639244 - 3.914454 \times 10^{-4} T + 0.88924913 \times 10^{-6} T^2 - 4.12191619 \times 10^{-10} T^3$

$C_{pah} = 0.79\ C_{pnh} + 0.21\ C_{poh}$

$= 0.80391803 + 5.37396575 \times 10^{-4} T - 2.2446743 \times 10^{-7} T^2 + 0.3525935 \times 10^{-10} T^3$

Where $T_2 > 780°C$, and $T_1 < 780°C$, 2.13 substituted in 2.11 gives,

$$Hs = 352.962V \left(\int_{T_1}^{780} C_{pal} \frac{dT}{T} + \int_{780}^{T_2} C_{pah} \frac{dT}{T} \right) \quad 2.19$$

2.4.3 Specific Heat in Insulation

The equation of specific heating still applies. But for an insulation body, temperature gradient exists. The temperature rise θ will have to be estimated as an average over its surface. If such a body is initially at temperature To, and the temperature of one face was increased to T_2, such that heat transfer occurs normally until the temp of the opposite face rises to T_1, after which steady flow was maintained (Fig. 8a), then the average body temperature rise can be expressed as:

$$\theta = (T_1 - To) + (T_2 - T_1)/2 \quad . \quad . \quad . \quad 2.20$$

For a composite insulation (8b), temperature at interface, Ti, will be determined by first evaluating the heat flow per unit area,Q/A , through the composite layers using an adaptation of Ficks first law:

$$Q/A = \frac{-(T_1 - T_2)}{\sum \Delta x/k} \quad . \quad . \quad . \quad . \quad . \quad . \quad 2.21$$

where Δx and k are the thickness and thermal conductivity per layer respectively. At this flow rate, temperature Ti at the interface can be obtained from:

$$Q/A = \frac{-(T_1 - T_2)}{\Delta x/k} \quad . \quad . \quad . \quad . \quad . \quad . \quad 2.22$$

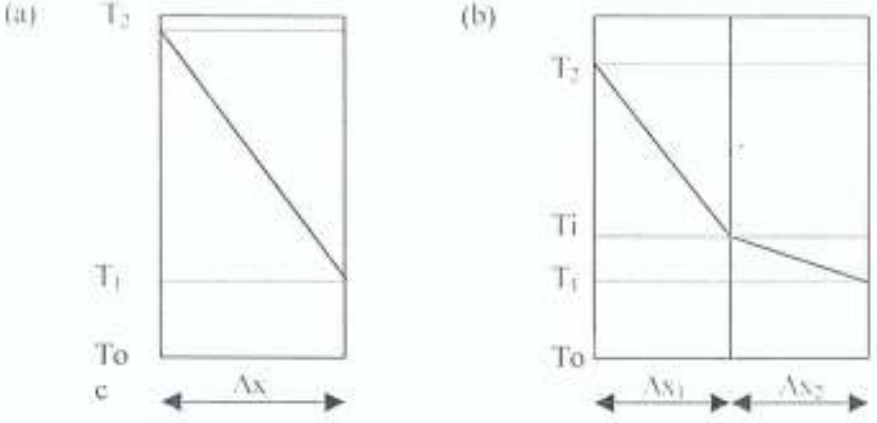


Fig 8: (a) Temperature Profile in an Insulation Thickness, (b) Temperature Profile in a Composite Insulation

2.4.4 Heat Loss Through Insulation

The maximum heat loss rate will occur when the maximum chamber temperature is achieved. At this condition, the temperature gradient, dx/dT is constant and maximum, such that the Fick's first law gives the heat loss rate as:

$$Q = -k A (T_1 - T_2)/dx$$

The total heat loss over a time t, in seconds, will follow as:

$$H = -k A t (T_1 - T_2)/dx \quad . \quad . \quad . \quad . \quad . \quad 2.22$$

2.5 OHMIC HEAT GENERATION

The heat energy, E, generated by an ohmic conductor is related to the current, *i*, in amperes (A), passing through the ohmic conductor as (Bel and Witherland, 1987):

$$E = i^2Rt \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad 2.23$$

where R = the resistance in ohms (Ω) of the ohmic conductor; and

t = the time in seconds (s) for which *i* flows.

Eq. 2.23 can be rewritten, substituting $i = V/R$ (ohms law), as

$$E = V^2t/R \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad 2.24$$

The rate of power discharge then follows as

$$P = E/t = i^2R = V^2/R \quad . \quad . \quad 2.25$$

Further, R relates with the length, l, in meters (m), the cross-sectional area (A) in square meters (m^2), and the resistivity of the ohmic conductor, ρ, in ohm-meters (Ωm), as

$$R = \rho l / a \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad 2.26$$

Hence basic design variable for E include *i*, l, A and ρ. ρ being a material constant, its variation therefore implies making choice between high ρ materials specially made as heating elements. This choice determines the heat release rate from the heating element.

2.5.1 Heating Elements

Several types of heating element materials are commonly used in electric furnaces, including metallic and ceramic type. Examples are the ferritic steel, austenitic steel, molybdenum disilicide, and silicon carbide types (Grahl, 2002; Hyndman Inc, 2002; Kanthal Handbook, 1987; Majumdar et al, 2002).

These elements, by reason of their specific properties (Table 8) vary in heating temperature range, longevity and cost. Metallic elements are the least expensive in terms of upfront cost, but they also have the lowest temperature rating and, in some cases, the shortest lifespan. Additionally, the resistance of metallic elements increases with age due to the

reduction in cross section by oxidation, as well as elongation of the loops. This will result in decreased power to the furnace and the ultimate failure of the element.

Table 8: Material and Operational Properties of some Heating Elements (Majumdar et al. 2002)

	Nickel Chrome (NiCr)	Iron-Chrome-Aluminium (Fe-Cr-Al)	Silicon Carbide	Molybdenum Disilicide ($MoSi_2$)	Zirconia (ZrO_2)
Max. Element Temp. (air)	1200°C	1350°C	1600°C	1900°C	2000°C
Max. Furnace Temp.	1100°C	1300°C	1550°C	1800°C	1850°C
Element Material Class	Metallic	Metallic	Ceramic (Synthetic)	Intermetallic (Cermet)	Ceramic
Major Limitation	Limited to 1100°C	Poor hot strength; needs element support	Elements age; brittleness	Brittle	Slow; must be preheated to 1200°C
Major advantage		Ductile		High surface loads	

However, there are many furnaces that use metallic elements—mainly because they cost less, they operate on less expensive line voltage, and they are ideal for processes that require temperatures lower than 1250°C. Additionally, not all metallic elements feature a short lifespan. Many furnaces, like those in the ceramic field, are very clean, and metallic elements can last many years in clean operating environments. The oldest and most common metallic heating wire is the nickel – chromium alloy, 80% nickel, 20% chromium, (Hyndman, 2002), with very low temperature coefficient of resistance. SiC elements fall within the middle range in both price and operating temperature. They can be used between 650 and 1650°C. Under optimum operating conditions, they can last up to five years. The lifespan of a SiC element primarily depends on the maximum operating furnace temperature.

The temperature of the element depends on the furnace temperature and the amount of power per unit area on the hot zone. At a low watt loading, the element operates cooler and therefore lasts longer.

$MoSi_2$ elements are on the top of the list in terms of temperature, longevity and cost. Although the base cermet is consumed in the formation of a thin silica glass skin when exposed to oxygen at elevated temperatures, making the element decompose with time, increasing element resistance and reducing the diameter of the heating shank, yet the decomposition rate is so minimal that $MoSi_2$ elements do not exhibit the rapid aging of SiC and metallic elements. $MoSi_2$ being brittle, the elements are available in preformed shapes. Some support about $20W/cm^2$ of element surface area at an element temperature of 1700^0C, $14\ W/cm^2$ at 1800^0C and $5W/cm^2$ at 1900^0C (Majumdar et al, 2002; ZIRCAR Inc, 2001). Tables 9a and 9b show some characteristics of $MoSi_2$ elements. With all the advantages, $MoSi_2$ however exhibits increase in its resistance with temperature, necessitating

Table 9a: $MoSi_2$ Heating Element Properties
(ZIRCAR Inc., 2001)

Tensile Strength, N mm^2, 1800 K	> 190
Bending Strength, N mm^2, 300 K	> 350
Impact Strength, N/mm^2, 300 K	0.7
Hardness, mohs, 300 K	7.5 – 7.8
Density, g/cm^3	5.5 – 5.6
Porosity, %	< 2
Emmisivity,	0.74 – 0.82
Resistivity, ohm mm^2/m	3.53

Table 9b: $MoSi_2$ Heating Element Prices
(ZIRCAR Inc., 2001)

ITEM	DESCRIPTION**	PRICE*
D2001	MOSI2, 18-3/6-5.50-5.00-0.98in.	$ 223.00
D2004	MOSI2,18-6/12-9.50-8.00-1.97in	$530.00
D2005	MOSI2,19-6/12-6.88-7.87-1.57in	$830.00

*UK Prices ** $MoSi_2$ Heating Elements are described by:
Grade: 1700, 1800 or 1900 Diameter: D_1/D_2, mm/mm Heated Length: Le, in. or mm
Terminal length: Lu, in. or mm Shank Spacing: A, in. or mm
(Le, Lu and A specified with the same units of in. or mm)
Examples: Grade 1800, D_1=3mm, D_2=6mm, Le=5.5in., Lu=5in., A=.98in.: Specify as - MOSI2, 18-3/6-5.5-5-0.98in. Grade 1700, D_1=6mm, D_2=12mm, Le=300mm, Lu=250mm, A=50m - Specify as: MOSI2, 17-6/12-300-250-50mm.

phase angle fired silicon controlled rectifier (SCR) with current limiting capability to control the high currents established when the element is first fired. After warming up, its resistance increases and limits the flow of current by itself. Lastly, $MoSi_2$ heating elements are relatively expensive (Table 9b). Hence, except for such high temperature demand, use of $MoSi_2$ element may be unjustified.

In the choice of heating element it must be noted that the furnace cost increases with increase in their rated temperature because of the added cost of the more expensive heating elements, refractories and other systems. However, the efficiency and returns from a production process also increase considerably when using a higher temperature rated furnace.

2.6 TEMPERATURE CONTROL INSTRUMENTATION

The control system requires that the process temperature closely track a set of desired temperature. This requires that there be a means of sensing the process temperature, setting the desired temperature, and generating corresponding electrical signals. This is done using transducers: the feedback and the reference transducers for sensing the process temperature and setting a desired temperature, respectively. The resulting signal after adequate conditioning serves as control signal that actuates the final control elements and switch on the chamber heater on/off after a specific control law. Fig. 9 is a block diagram representation of single loop continuous feedback temperature control system. The various stages and components are hereby reviewed (Chesmond, 1988).

2.6.1 Feedback Temperature Transducers

Generally, a transducer is a sensory device that is capable of converting the value of a data variable into a signal whose magnitude and sense are representative of the magnitude and sense of the data variable (Chesmond, 1988). There are many methods by which temperature may be measured. Thermometers (temperature meters) may be classified as (i) Expansion thermometers such as solid, liquid and gas expansion types; (ii) change of state thermometers; and (iii) electrical transduction thermometers such as variable resistance, semiconductor, thermoelectric, and radiant energy types. The most suitable for indication and control application are those most capable of remote electrical transmission without appreciable transmission loss, which would create inaccuracy. An example of this is the thermocouple.

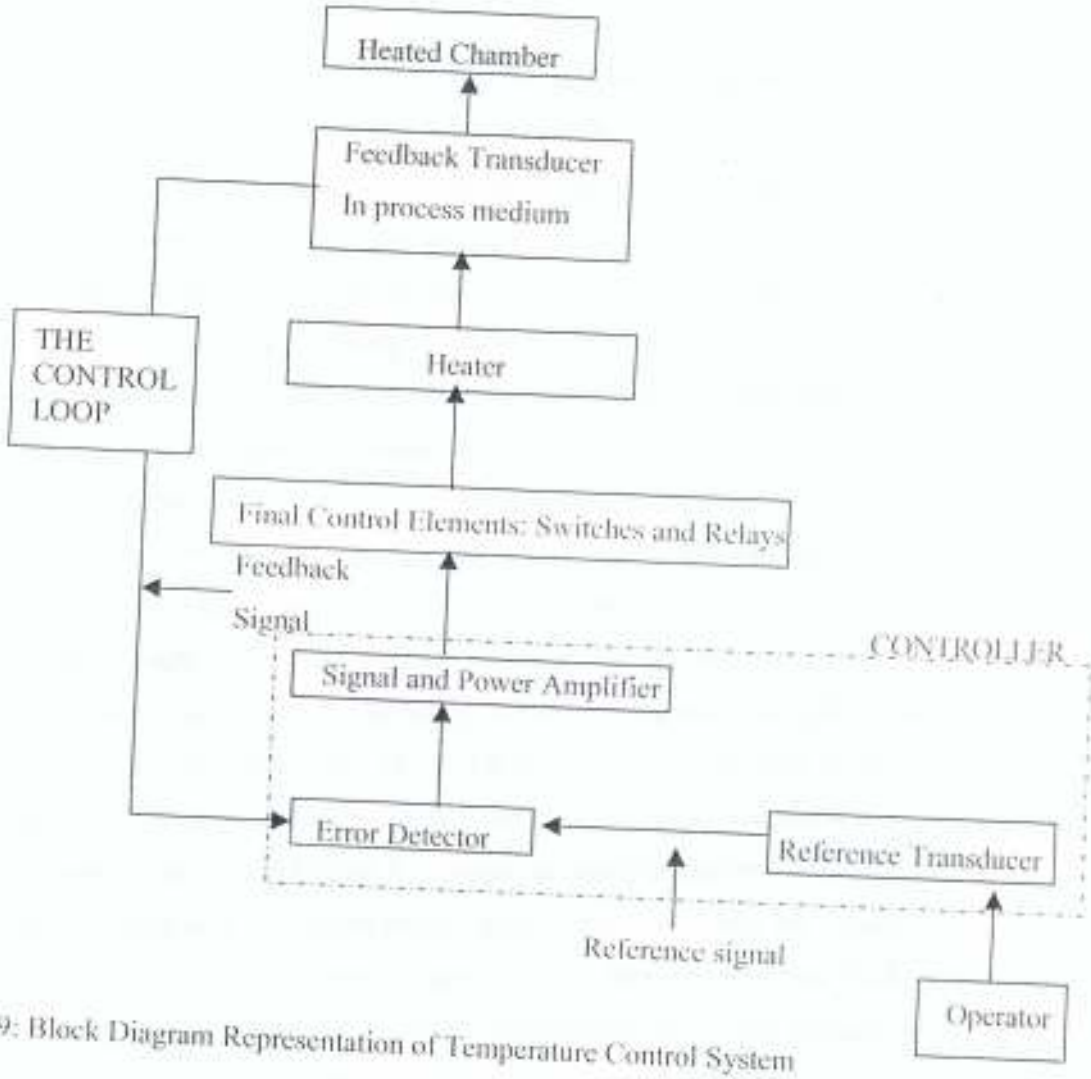


Fig. 9: Block Diagram Representation of Temperature Control System

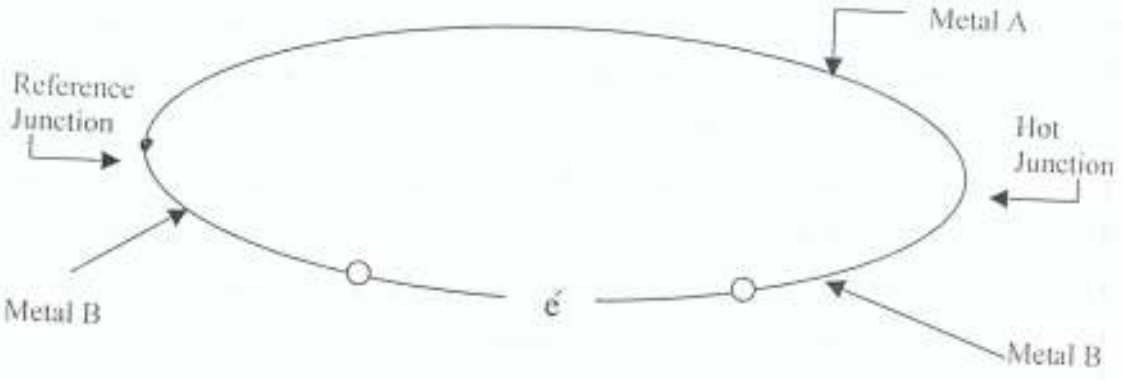


Fig 10: Simple Thermocouple Connection

Thermocouples are classified as a thermoelectric temperature transducer. The principle of operation is based upon the Seebeck effect, which states that, whenever two dissimilar metals are connected together as shown in Fig. 10 and the junctions are subjected to different temperatures, an emf e is generated, and this emf is proportional to the difference between the temperatures of the two junctions. The wire of a thermocouple may be connected together, to form a junction, by twisting, clamping, soldering, or electric/gas welding, with the last two methods being the preferred alternatives for good industrial practice. The choice of wire materials depends upon the temperature range to be sensed by the hot junction, among other considerations, and tends to be limited to the combinations listed in Table 10. The wires are manufactured in a range of gauges, and usually are supplied as sleeved pairs, with the sleeve frequently colour coded for ready identification. Other selection factors include physical strength, corrosion resistance, electrical resistance and cost.

Figure 11 shows some typical calibration curves for the thermocouple types listed in Table 10. It will be seen that some of the curves depart significantly from straight-line relationships, with the departure being most prominent in certain temperature ranges. EMF versus temperature tables are published by various standard institutions, and the data therein, together with the published calibration curves, assume that calibration has occurred with the reference junction held at 0^{O}C. Tolerance figure for such data vary between -0.25 to +0.25 % and -2 to +2% of measured temperatures. Some temperature controllers include provision for analog or digital computation of correction laws for the most common types of base metal thermocouple.

As a result of the small magnitude of the emf generated, care must be taken to preserve a good signal/noise ratio. Wherever feasible, it is preferable that the voltage should be suitably amplified at a point as close as possible to the reference indicator, or recorder. The amplifier often would be of the instrumentation amplifier variety. For signal preservation and for technical and cost factors, it is often impracticable to use extensive lengths of thermocouple wire. For this reason, specified types of extension wires (with alternative insulation materials), together with copper interconnections, may be interposed between the thermocouple and its load. Fig. 12 shows some alternatives of connection configurations.

Table 10: Commonly Used Thermocouple Materials

(Chesmond, 1988)

Thermocouple Type	Wire Materials	Typical Useful Temperature Range, ^{o}C
B	Platinum 6% - platinum rhodium 30%	38 – 1800
C	Tungsten – tungsten rhenium 26%	0 – 2300
E	Chromel – constantan	0 – 982
J	Iron – Constantan	-184 – 760
K	Chromel – Alumel	-184 – 1260
R	Platinum – platinum rhodium 13%	0 – 1593
S	Platinum – platinum rhodium 10%	0 – 1538
T	Copper – constantan	-184 – 400
-	Platinum 30% rhodium – platinum 6% rhodium	0 – 1780
-	Iridium 40% rhodium – iridium	0 – 2000
-	Tungsten – rhenium	0 – 2220
-	Tungsten – tungsten 26% rhenium	0 - 2330

Note: Alumel Nickel-aluminium alloy
Constantan copper-nickel alloy
Chromel nickel-chromium alloy

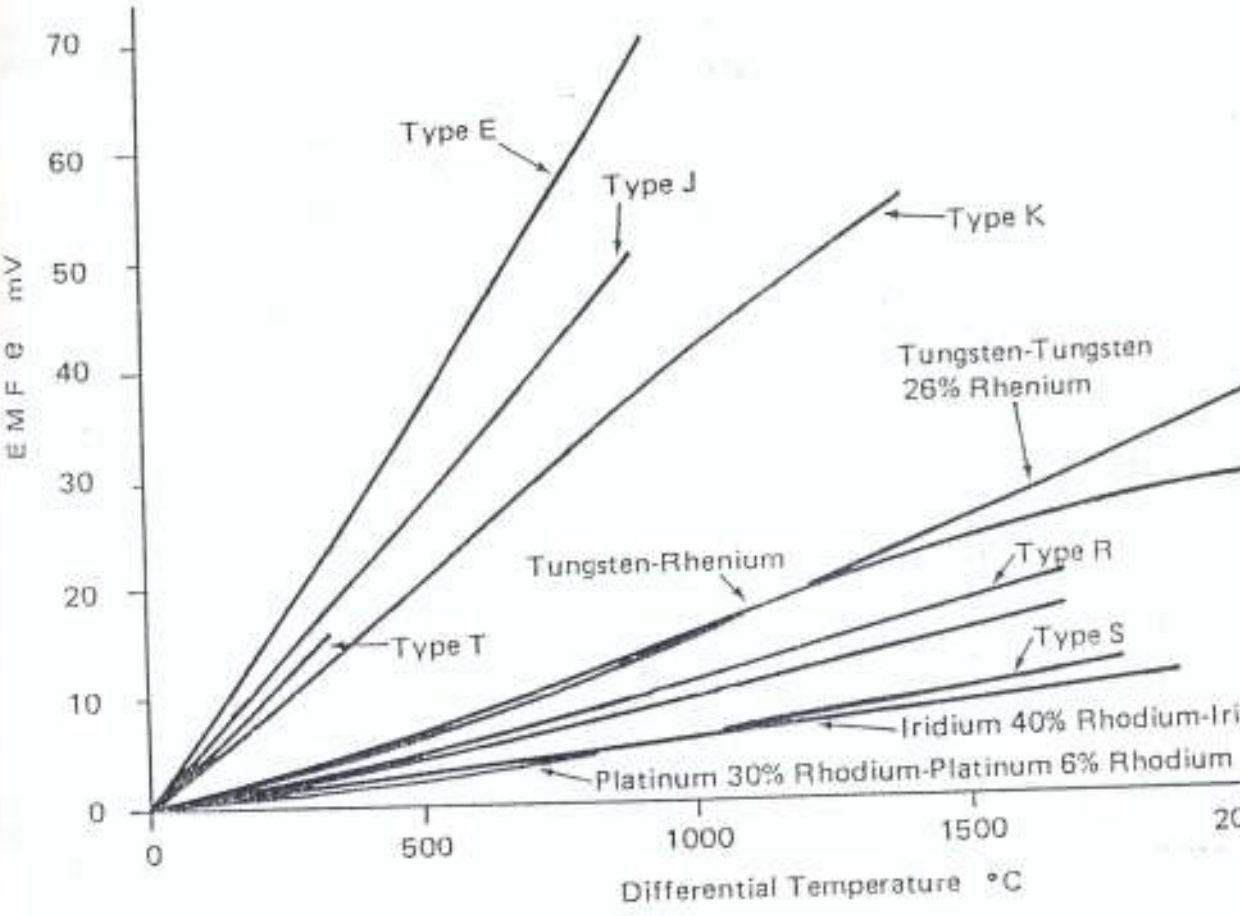
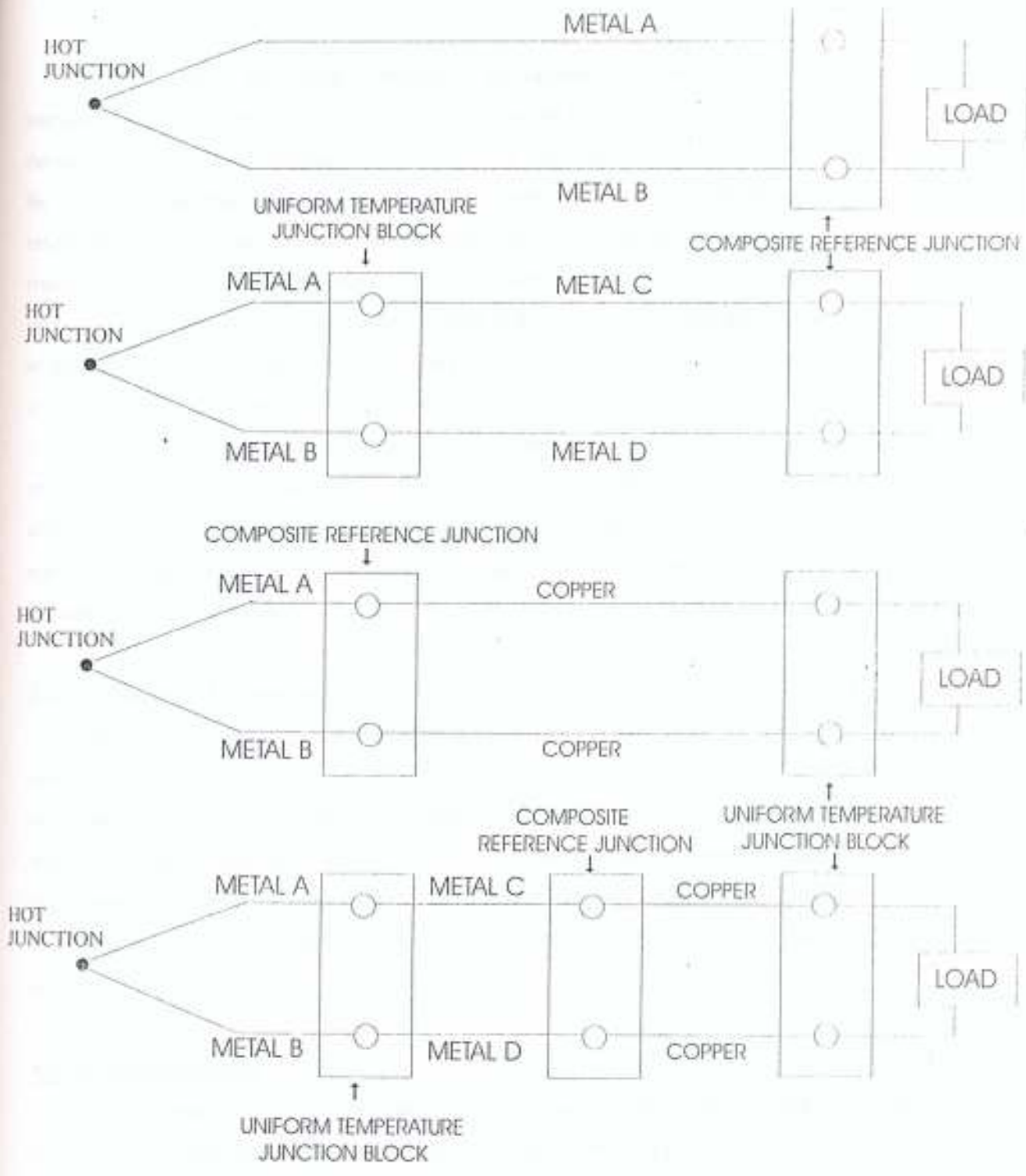


Fig. 11: Calibration Curves of Thermocouple Wires

(Chesmond, 1988)



NOTE: Metals A and C are chosen to have comparative thermoelectic properties over a specified temperature range. Metals B and D are chosen to have comparable thermoelectric properties over a specified temperature range.

Fig. 12: Thermocouple Connection Configurations
(Chesmond, 1988)

The thermoelectric emf being a function of the temperature differential between the two junctions, the temperature of the measuring junction can be interpreted from the emf, only if the temperature of the reference junction is known and preferably held constant. Typically, the composite reference junction would be in thermal contact with a metal heat sink, but electrically isolated from it. The heat sink would have its temperature measured by, say, a resistance thermometer and the signal developed would either be used in temperature feed back loop to control the temperature of the heat sink, using a small integral electrical heater, or would be used for compensation purposes to correct the thermocouple-generated data for sensed changes in reference junction temperature.

In construction, for physical and chemical protection, the hot junction often has to be enclosed in a rigid tube, sometimes called a thermowell, which usually is made either from metal (typically, stainless steel) or ceramic. This tube will add considerably to the thermal mass of this composite temperature sensor, so that the overall response time is likely to be in the range of many seconds.

2.6.2 Reference Transducers

Reference transducers are usually built using such devices as are used in displacement transducers. These include servo potentiometers, differential inductors and transformers, synchros, resolvers, and inductosyns. Synchros, inductosyns and resolvers are normally used where they have been used in the feedback transducers, hence, they are applicable in displacement controls. Using servo potentiometers and differential inductors and transformers, reference transducers can be built as potentiometric reference or rotary switches.

Servo potentiometers

Servo potentiometers differ from conventional potentiometers in that they have superior manufacturing qualities, and these include: close linearity of the wiper voltage versus wiper displacement characteristics; close tolerance on the resistance of the track; use of high grade bearings to minimize friction; and, use of a high quality precious metal brush for the wiper to minimize friction wear and contact resistance.

In servo potentiometers, the track may be wire wound or it may be manufactured from homogeneous materials such as carbon composition, high conductivity, metal or metal composition film, or conductive plastic. Wire wound servo potentiometers have high degree of linearity, and fixed tapping can be added. But it also suffers granularity effect, that is,

voltage must increase in fixed amounts equal to the voltage dropped across one turn of the winding as the wiper moves. Wiper voltage versus wiper displacement can be made non-linear by graduating the cross-sectional area of the track, or by using wires of different diameter in different section of the track.

Servo potentiometers are made as rectilinear or rotary. Rectilinear potentiometers are constructed with lengths of travel ranging typically from 1mm to 6 meters (with 1cm to 15cm common), track resistances ranging from 20Ω to 200 kΩ (with 100Ω to 10 kΩ common), and linearity errors ranging from less than 0.1% (with 0.1, 0.5, and 1% common). Typical resolution for wire wound potentiometers ranges from 70 to 200 steps per centimeter. More than one track may be mounted within the case of the device, in order to provide multiple data channels. Rectilinear potentiometers will be destroyed if overdriven mechanically, so that mechanical limits, must be placed externally on the input displacement. Life of these potentiometers may be short, due to problems experienced with trying to maintain a good contact between the wiper and the track.

Rotary potentiometers are manufactured in single turn and multi-turn versions. Single turn types use a toroidal former for the track and the usual need for a dead-space, to provide electrical isolation between the two ends of the track, means that the useful input travel is less than 320° to 335°, and whenever the wiper is driven into the dead-space, the load connected to the wiper will be presented with an open circuit signal source. Typical track resistance is from 50 Ω to 200 kΩ, with linearity errors extending down to 0.01% and resolution (for wire-wound types, and dependent upon the diameter of the toroidal former) extending down to 0.04%. Toroidal potentiometers may be ganged together, on a common shaft, to provide multiple data channels. Multi-turn potentiometers use a helical former for the track, and the wiper assembly is designed so that the brush simultaneously rotates and advances axially, as the input shaft is rotated.

Figure 13 shows the most common method of connection of servo potentiometers, which is connected as a potential divider supplied from a stable voltage reference source, which usually is DC but occasionally may be AC. In some applications, the voltage supply will be distributed symmetrically about a signal common and, in this case, it is preferable to use a center tap on the potentiometer track and to connect this tap to signal common, thus establishing a rigid electrical datum at the mid-point of the shaft travel: this mid-point would then also be used as the mechanical datum for calibration purposes. An important characteristic of servo-potential is the distorting effect that the presence of significant load current can have on the calibration of the potentiometer, Fig. 13, with the deviation between

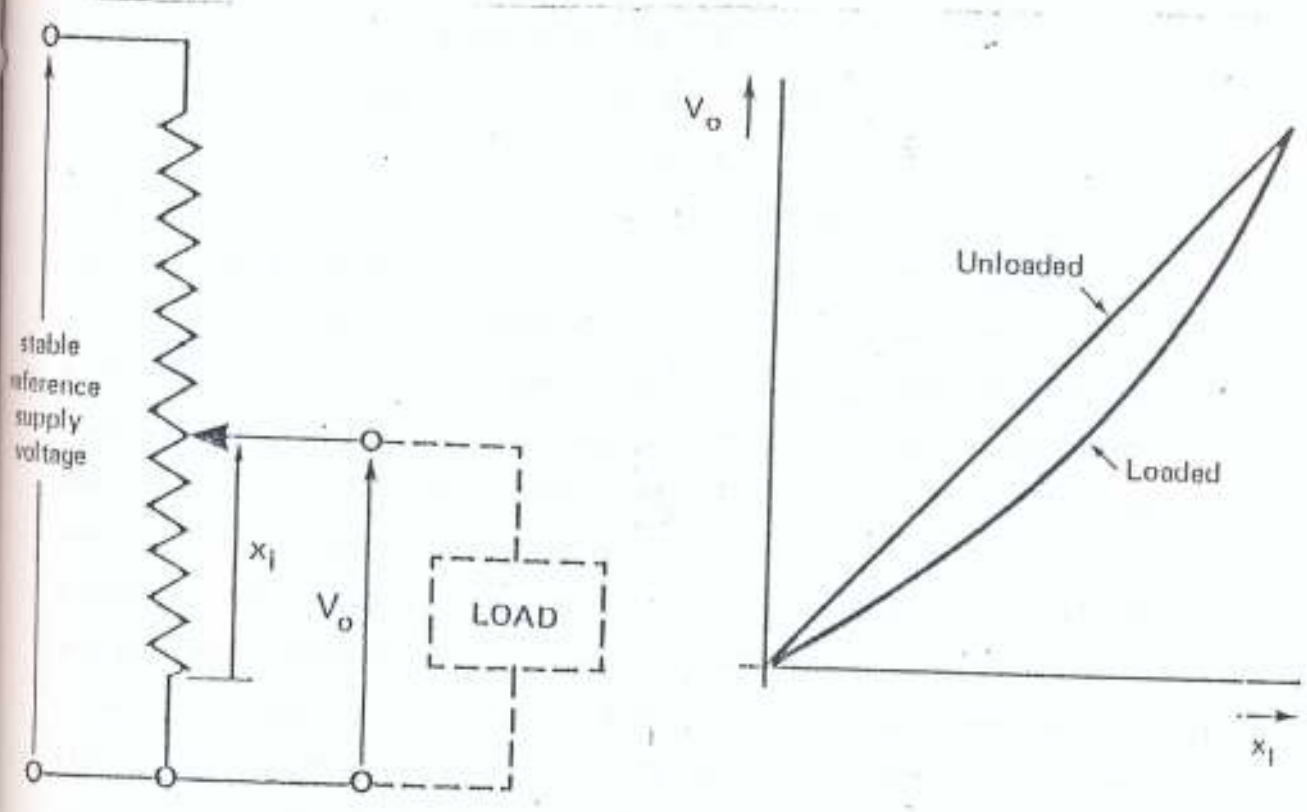


Fig. 13: Simple Servo-potentiometer Connection and Wiper Voltage Versus Load Current plot

the loaded and unloaded characteristics being greatest at mid-travel: loading can readily degrade the linearity of a potentiometer, so that, where necessary, a precision buffer amplifier should be inserted between the wiper and the load, in order that negligible current will be drawn from the wiper.

Differential Inductors and Transformers

Increasingly, theses devices are replacing potentiometers, mainly because of the superior reliability associated with the former. They can be subdivided into four categories:

- LVDI – Linear Variable Differential Inductor
- LVDT – Linear Variable Differential Transformer
- RVDI – Rotary Variable Differential Inductor
- RVDT – Rotary Variable Differential Transformer

The LVDI and LVDT are rectilinear transducers, whereas the RVDI and RVDT are rotary. The rotary versions have a very limited travel (typically $\pm 60^{\circ}$) and sometimes are referred to as 'Rotary Pick-Offs'. The rectilinear versions are by far the more extensively used, mainly because they are available with a wide range of travel. The inherent reliability associated with these inductors and transformers arises because they do not involve the use of any moving electrical contacts: in fact, there need be no physical contact whatsoever between their moving and stationary component parts. Fig 14 shows the electrical circuits for half bridge and full bridge differential inductors and for differential transformers. In each case, the applied AC voltage is sinusoidal and has a frequency which may lie between 50 Hz and 20 kHz but which usually is in the 1 kHz to 5 kHz range. In the case of differential inductor, the device is connected in an AC Wheatstone bridge arrangement, which is balanced electrically at the null position for the core(s), so that Vo = 0; as the cores are offset, by input displacement x, the magnetic coupling with the primary increases for one secondary and decreases for the other, so that Vo changes.

Figure 15 indicate the type of construction for the LVDT and RVDT, and comparable configurations are used for the LVDI and RVDI respectively. As can be seen, the moving member need make no physical contact with the fixed member, since magnetic flux linkages form the transducing medium. Fig. 16 shows the type of output voltage versus input displacement characteristic obtained with all of these devices. Typically, Vo will nominally be in phase with V_{AC} for positive values of x. In practice, due to the effects of magnetizing current, core losses, and winding impedances, there will be small parasitic phase shifts present, so that the phase of Vo will not be precisely 0° or 180° relative to V_{AC}. In addition, the presence of ferrous material in the magnetic circuit will cause a small amount of

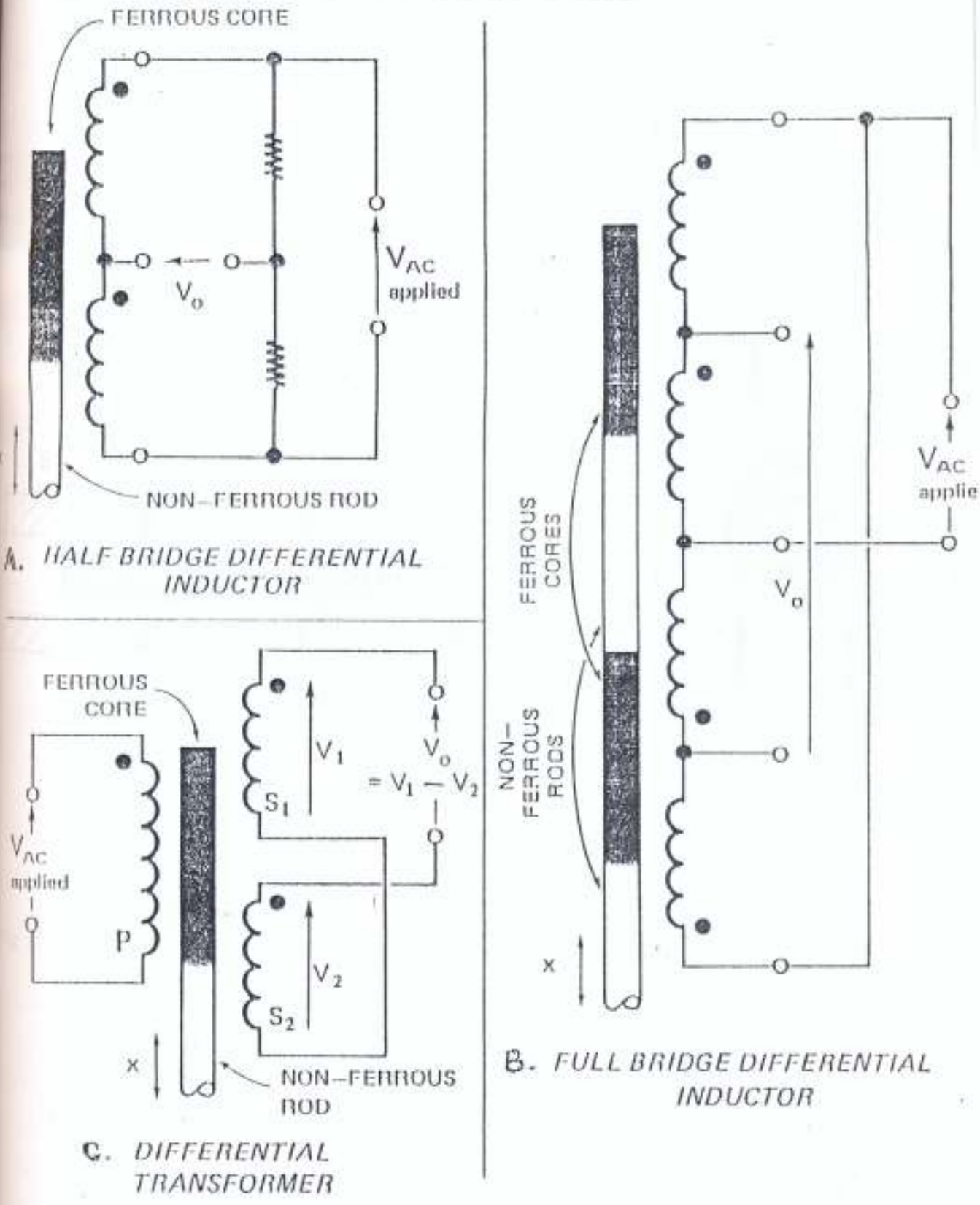


Fig. 14: (a) Half Bridge Differential Inductor (b) Full Bridge Differential Inductor (c) Differential Transformer (Chesmond, 1988)

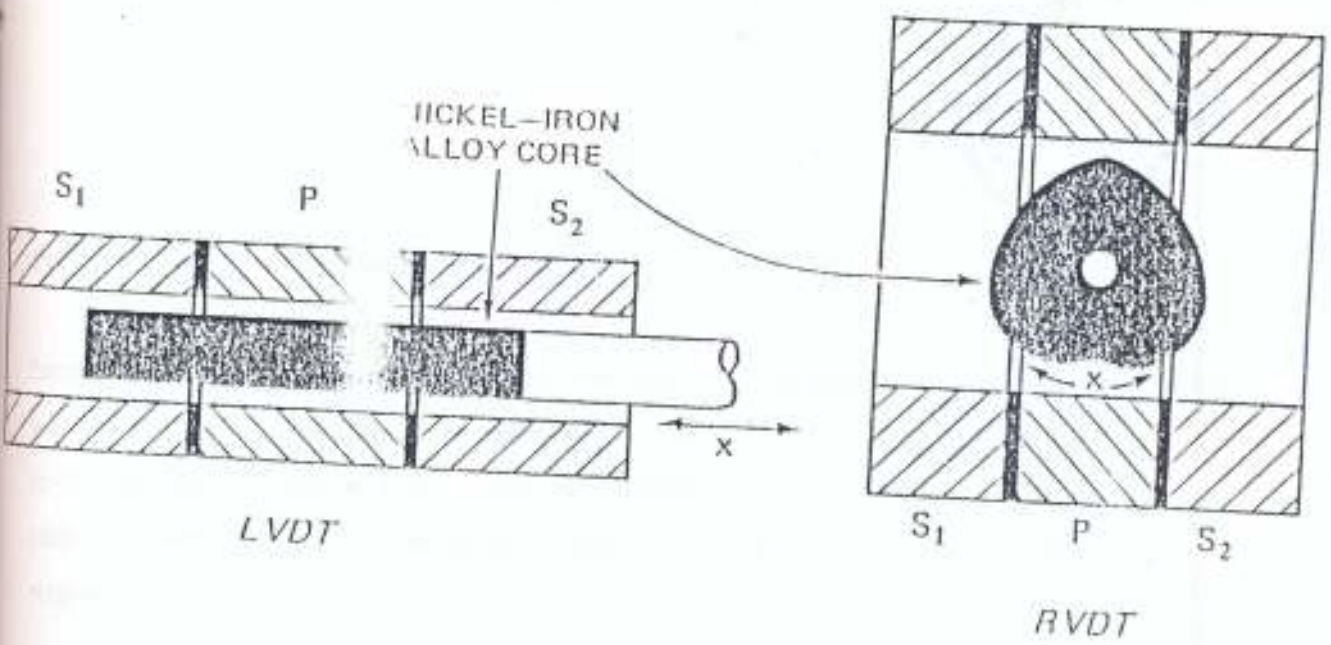


Fig. 15: Typical Constructions of LVDT and RVDT

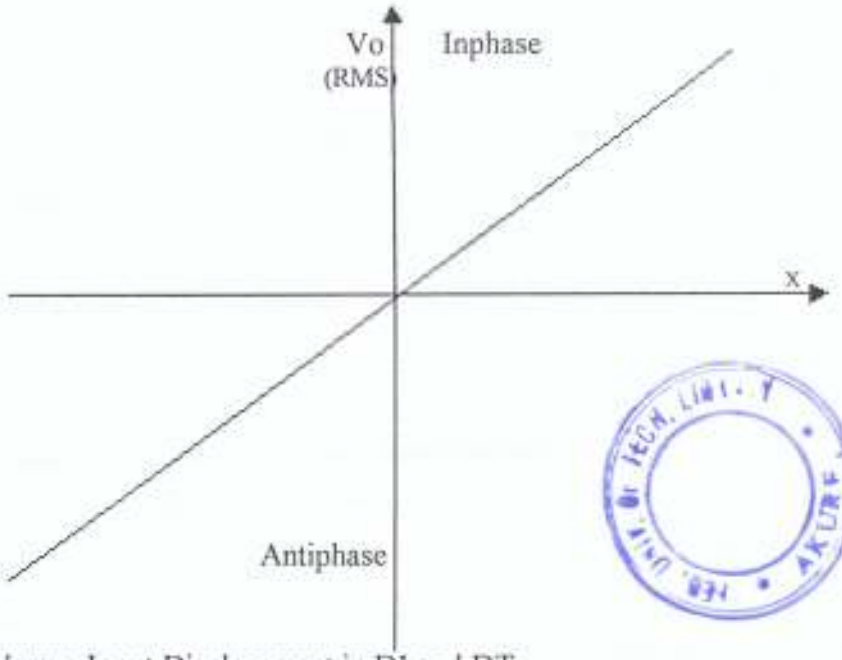


Fig. 16: Output Voltage Versus Input Displacement in DI and DT

harmonic distortion in the output voltage waveform. The rectilinear types are constructed with travels ranging typically from 1 mm to 30 cm, although this range has been extended to 0.1 mm to 250 cm in certain devices. Linearity errors as low as 0.1 % are achieved relatively easily, whilst the resolution is infinite, because the Vo versus x characteristics is stepless.

Potentiometric References

When servo potentiometers are used as reference transducers they are referred to as reference potentiometers. For this purpose, the existence of dead space and / or limits to travel no longer presents a problem, however, and a reasonably high level of friction often can be a definite advantage. There is no requirement for the voltage supply for the reference potentiometer to have a high value, whatever maximum value of the feedback signal, since proportional data combination can be done for error signal generation (2.5.3). Theoretically, almost any voltage supply level can be used; in practice, the level should be sufficiently high to maintain high signal/noise ratios but not so high as to generate significant heating of the potentiometre circuit or to render the supply hardware unnecessarily expensive. There is a definite requirement for the supply voltage to be stable, because the stability of the feedback transducer sensitivity depends upon this.

Rotary Switch Networks

With the use of multi tapped transformer or resistive potential divider, reference

setting can be done by switches settings. Fig. 17 shows two of such arrangements for generation of analog reference data. It is normal for each of these switches to have ten positions (representing 0 – 9), so that one switch would be allocated to each decimal digit of the desired value. When the requirement is for digital data, the usual procedure is to encode each decade separately into natural binary code, so that the overall format for the reference data becomes binary-coded-decimal (BCD). Fig. 18 shows two commonly used alternatives for generating natural binary code for one decade of the BCD word. Some off the shelve switches are supplied with integral logic hardware.

2.6.3 Error Signal Generation: Differential Data Combination

The error signal is the difference between the feedback and reference transducer signals, and it is generated by adequately wired components, for signal subtraction. Since it is more convenient to add, rather than subtract signals, the summation of data can be arranged to implement the subtraction of data by firstly inverting the sign of the data to be subtracted, Fig. 19. The inversion required would be implemented by the insertion of sign inverting hardware, or alternatively by modifying either the input/output connection of the transducer generating one of the signals.

It is important to note that the combination is usually not done in one-to-one proportion, due to the different sensitivities of the reference and the feedback transducers. Suppose, for instance, that when the maximum temperature desired is achieved, the reference transducer generates a 10 V signal (full- scale deflection signal), while the feedback generates 100 V, then it implies that the sensitivities and, therefore, the calibration in volts per Kelvin (V/K) on the reference and feedback transducers would have to be in ratio 1 to 10, since the same maximum temperature (say, $1200^{O}C$) generated 10 and 100V in the reference and feedback transducer respectively. Hence when a set temperature is achieved, to achieve an error signal, V_e, of zero the reference and feedback transducers signals, V_r and V_f, should be combined in the proportions of the reference and feedback transducers sensitivities, K_r and K_f. Suppose Fig 20 represents such signal combination instrumentation, when the set temperature is achieved, the error e (= r – c), is zero, and the error signal V_e must be zero. It follows that:

$$V_e = K_r K_1 r - K_f K_2 c \quad \ldots \quad 2.27$$

$$= K_1 V_r - K_2 V_f = 0$$

$$\Rightarrow \quad 10K_1 - 100K_2 = 0 \quad \Rightarrow \quad K_1/K_2 = 10$$

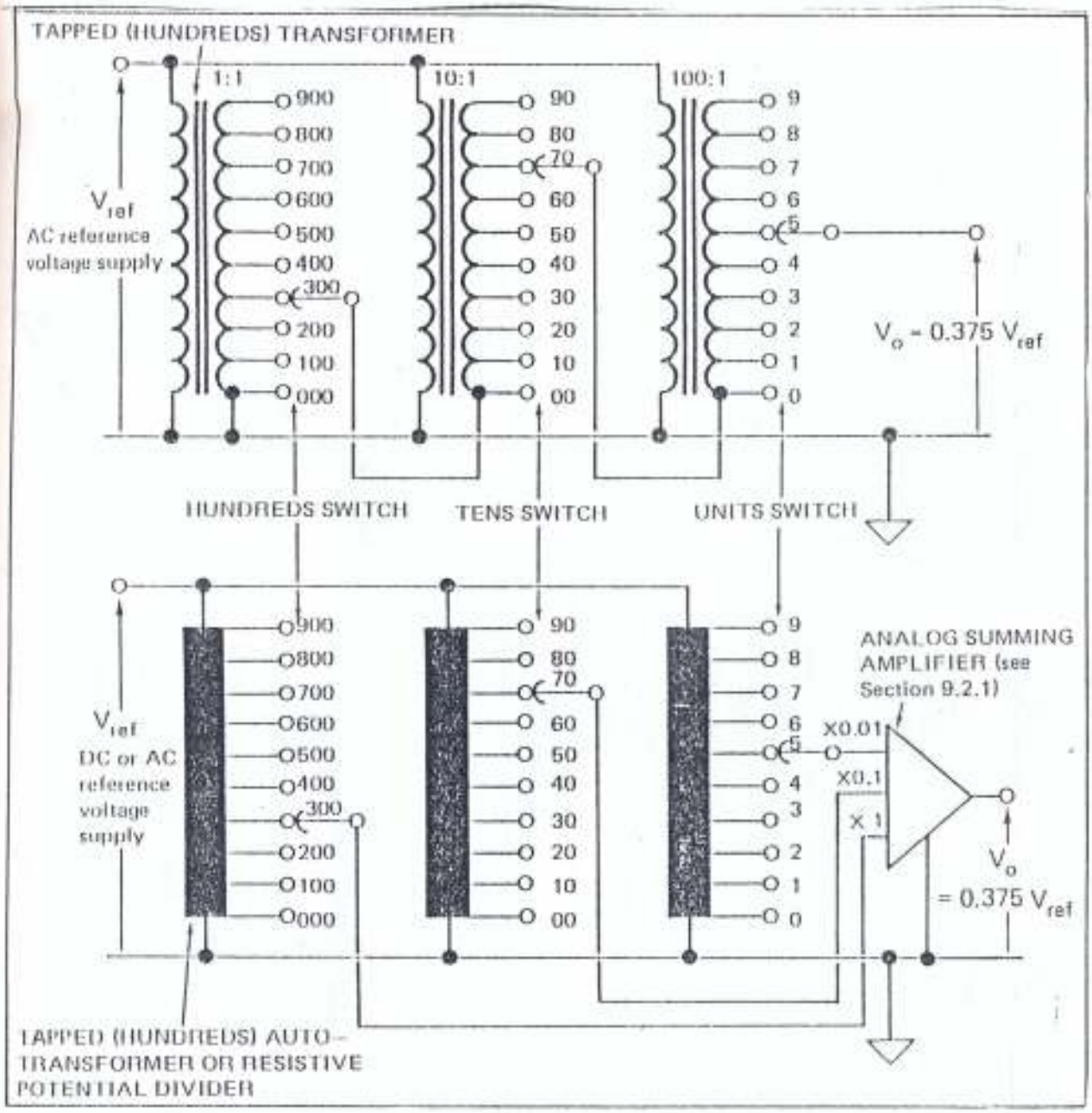
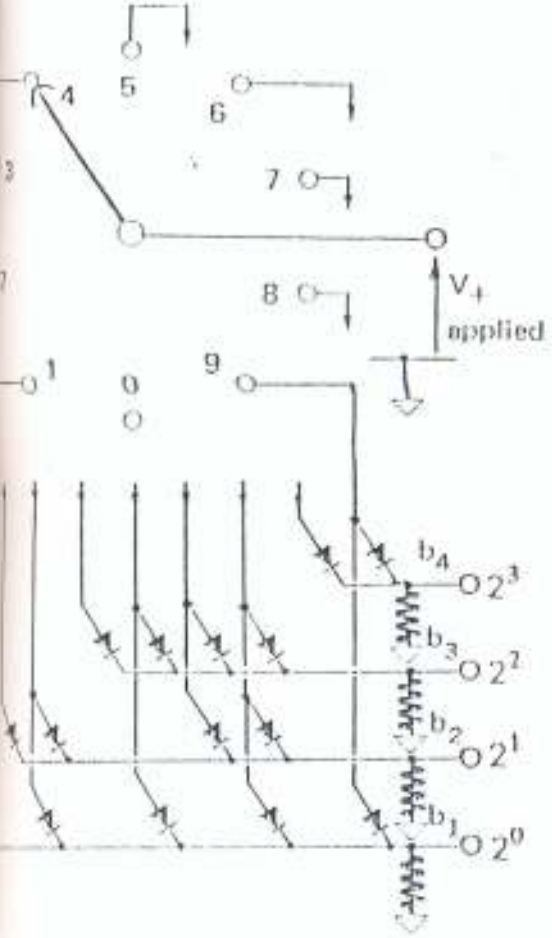
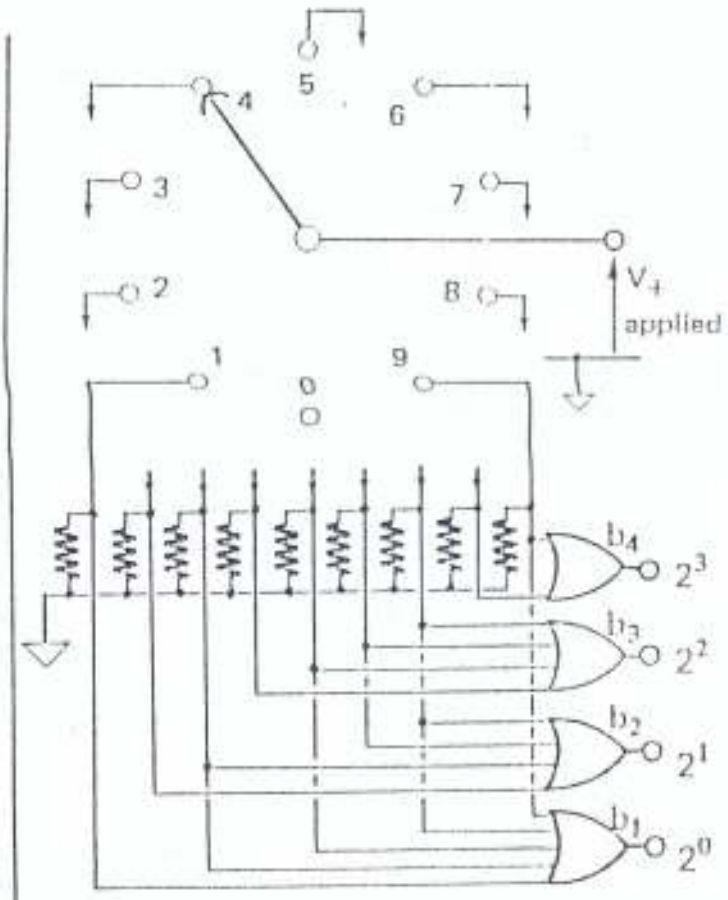


Fig. 17: Arrangement of Tapped Transformer for Switch Network Reference Transducer System (Chesmond, 1988)



IODE MATRIX ENCODER



LOGIC GATE ENCODER

Alternative Configurations of Rotary Switch Networks for Digital Reference Data

end, 1988)

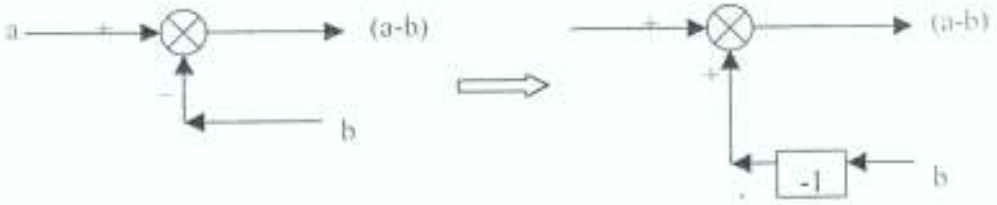


Fig. 19: Signal Inversion before Combination

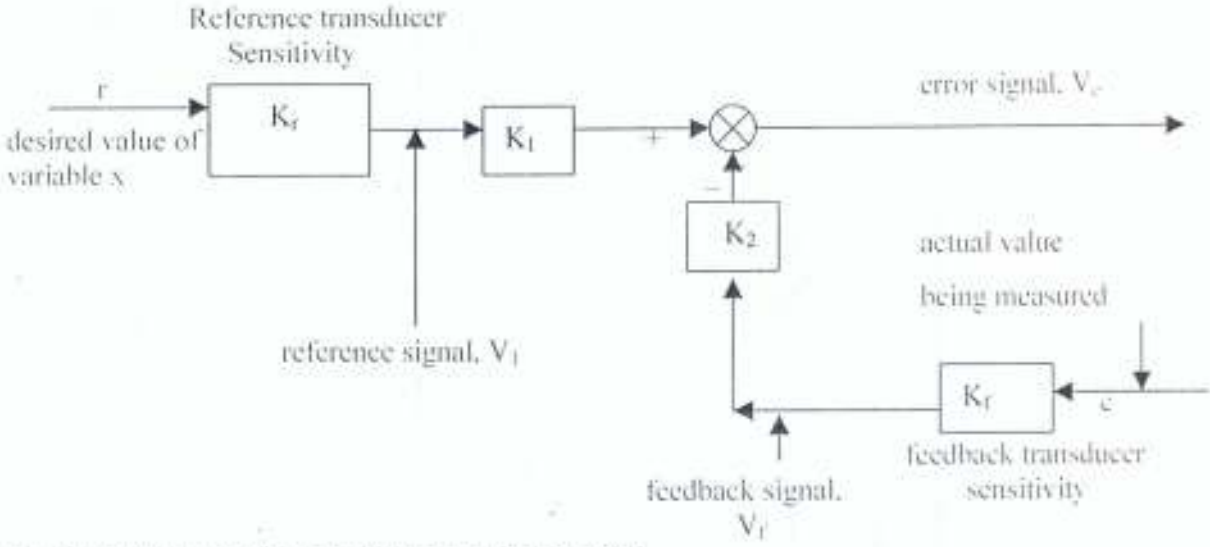


Fig.20: Proportional Signal Differential Combination

Thus the two signals must be combined in inverse proportion to the sensitivities of their transducer sources:

$K_r = 10/1200$; $K_1 = 100/1200$; $K_r/K_1 = 1/10 = K_2/K_1$

A simple circuitry to effect signal differential is shown in Fig. 21, where it can be shown that:

$$Vo = -\frac{R_3}{R_1}V_1 + \frac{R_4}{R_2+R_4}\cdot\frac{R_3+R_1}{R_1}V_2 \quad = \quad -K_1 V_1 + K_2 V_2 \qquad 2.28$$

To effect proportional control (2.5.5), Fig. 21 may be configured as an analog comparator by making $R_3 = \infty$, for which $Vo = V_{sat-}$ when $\frac{R_4}{R_2+R_4}V_2 - V_1 < 0$ and $Vo = V_{sat+}$ when $\frac{R_4}{R_2+R_4}V_2 - V_1 > 0$. Vsat+ and Vsat- are the output saturation levels of the operational amplifier, the values of which will depend upon the values of the amplifier supply rail voltages. For high speed switching, it is not good practice to drive the output voltage into saturation, but to limit its excursion by means of appropriate components: Figure 22 shows two typical arrangements.

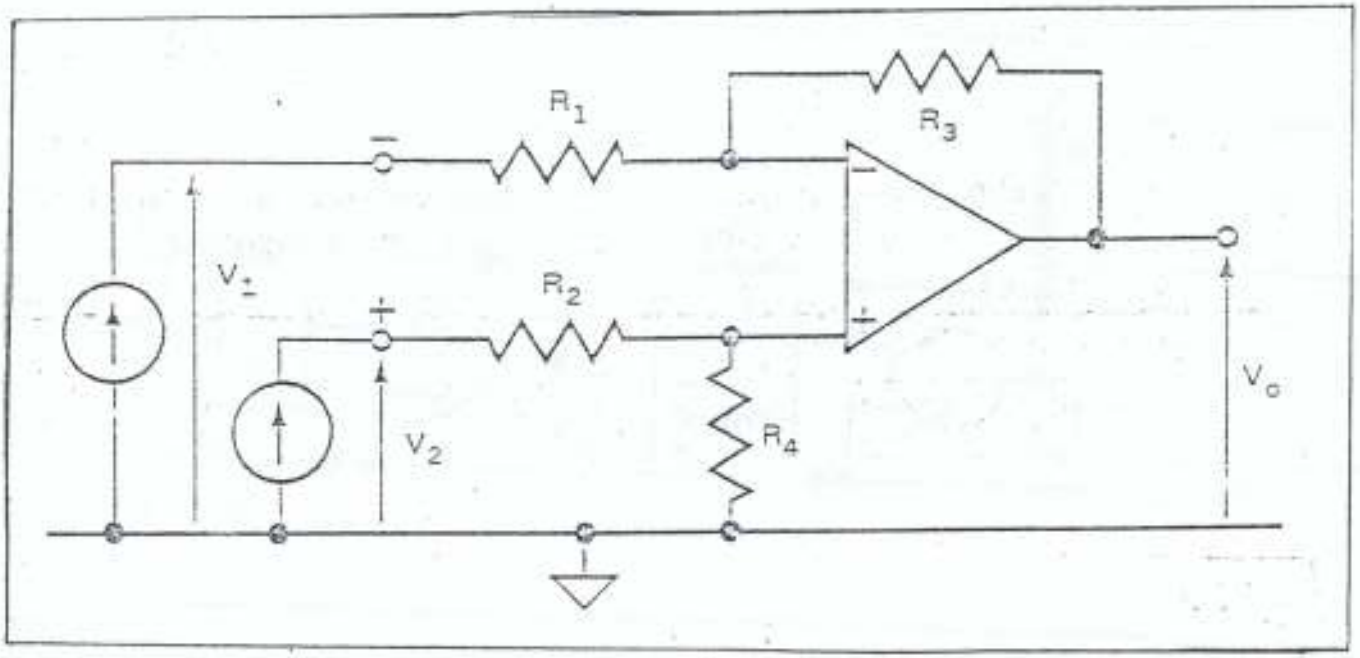


Fig. 21: Differential Amplifier Configuration
(Chesmond, 1988)

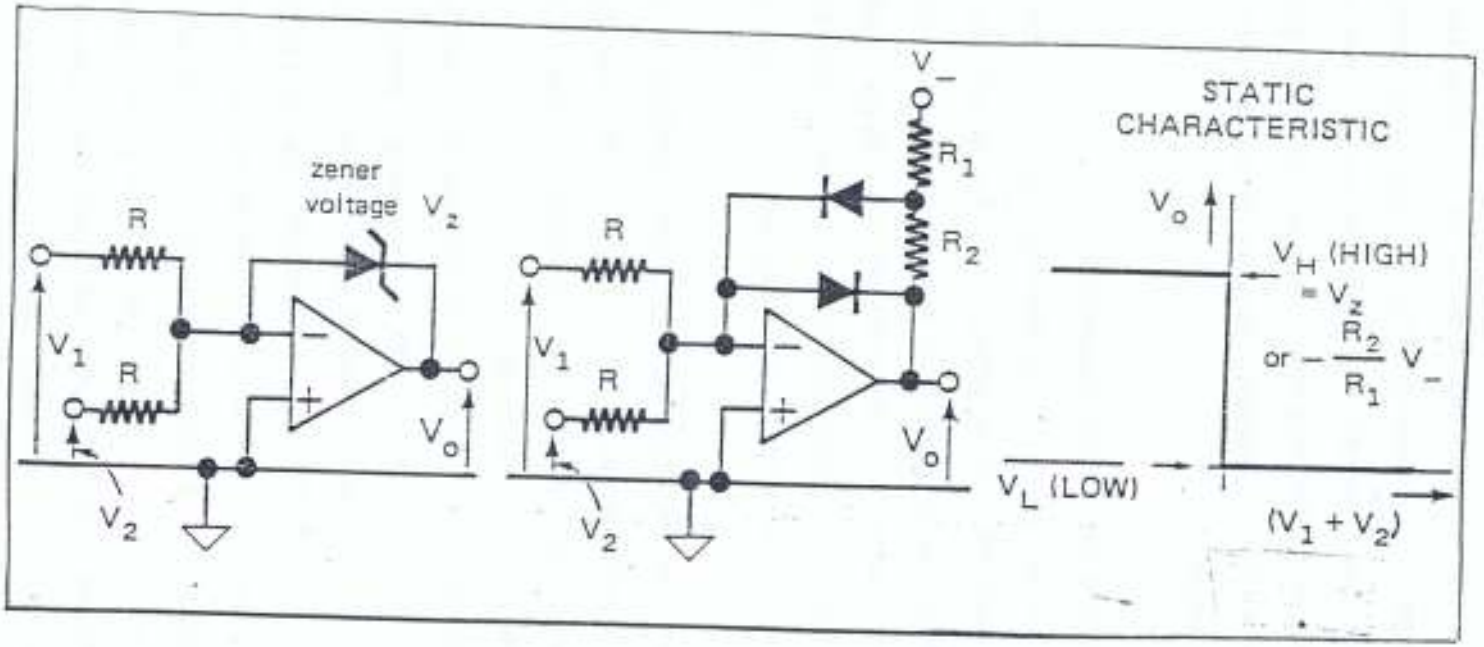


Fig. 22: Limiting Configurarion for Vsat for Analog Comparism
(Chesmond, 1988)

2.6.4 Control Laws

The simplest law involves switching on and off a heat supply, with switching in both directions occurring at the same (preset) temperature. This results in a cycling of the load temperature, as shown by Fig. 23. The magnitude and frequency of the temperature cycling will depend upon the magnitude of the heat source and the dynamics of the thermal load: in many installations, the cooling interval is much longer than the heating interval, especially where cooling occurs entirely as a result of heat transmission to the atmosphere.

A greater degree of control over the temperature cycling can be effected by designing the control law such that the switching point during heating occurs at a higher temperature compared with the switching point during cooling, as shown in Fig. 24. This type of control law property sometimes is referred to as "overlap" or "hysteresis". Increasing the overlap zone will result in a reduction in the frequency of the temperature cycling, but this will be accompanied by an increase in the temperature excursion.

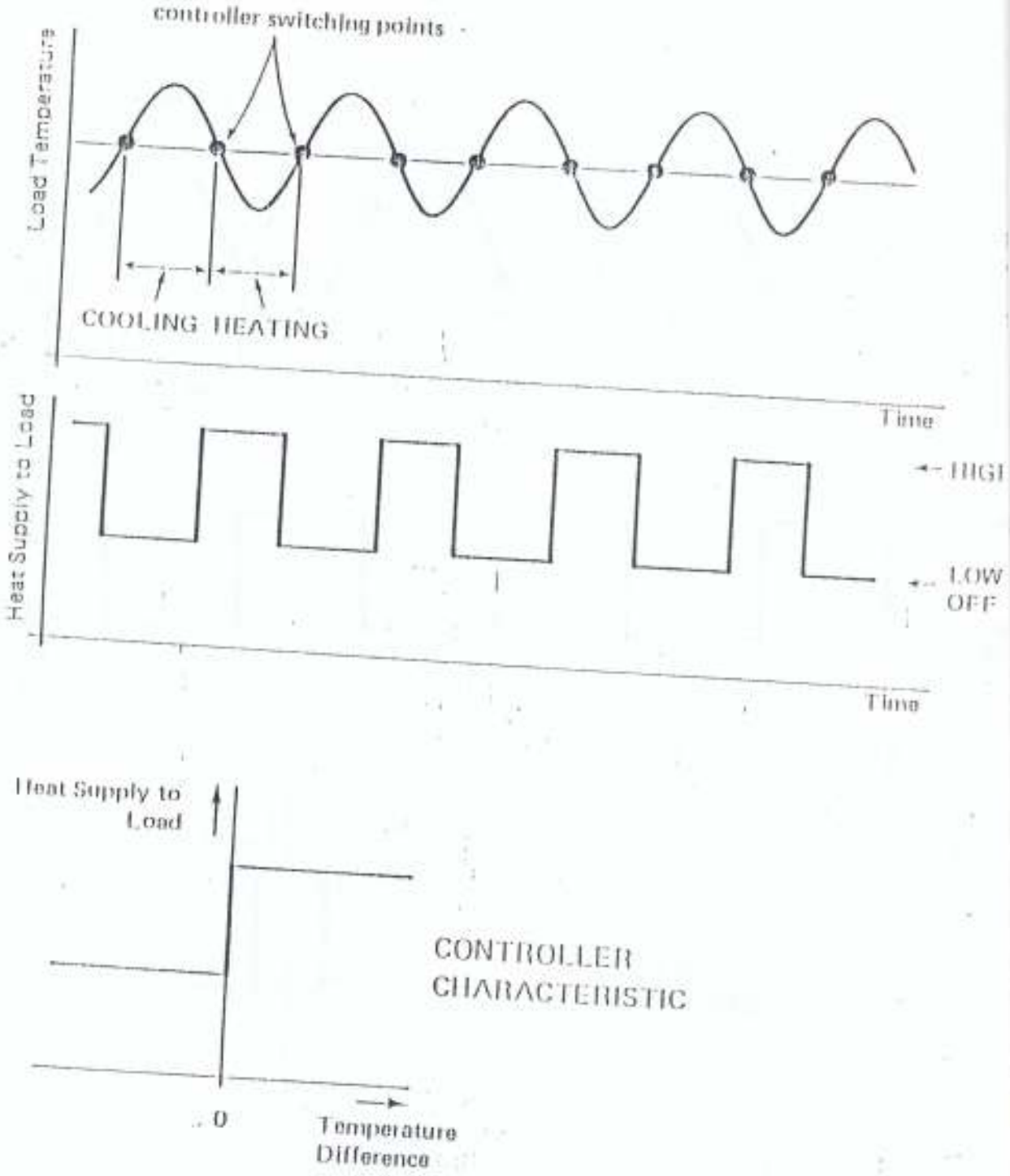
With multi-step control, the hardware makes provision for three or more switching temperatures, resulting in an equal number of different applied heat levels. Such a controller inevitably is more complex but can result in a significant reduction in the temperature excursion.

Proportional control may be effected, using on-off switching hardware, with the type of arrangement shown in Fig. 25. The mark space of the controlled heat supply changes as a function of the value of the temperature error voltage, v_e, such that the average value of the heat delivered varies linearly with the temperature error. In this manner, proportional action is achieved, although the hardware does not require a linear power amplifier. If provision is made for offsetting the sawtooth waveform, the quiescent value of average heat, which is generated when the temperature error is zero, may be adjusted.

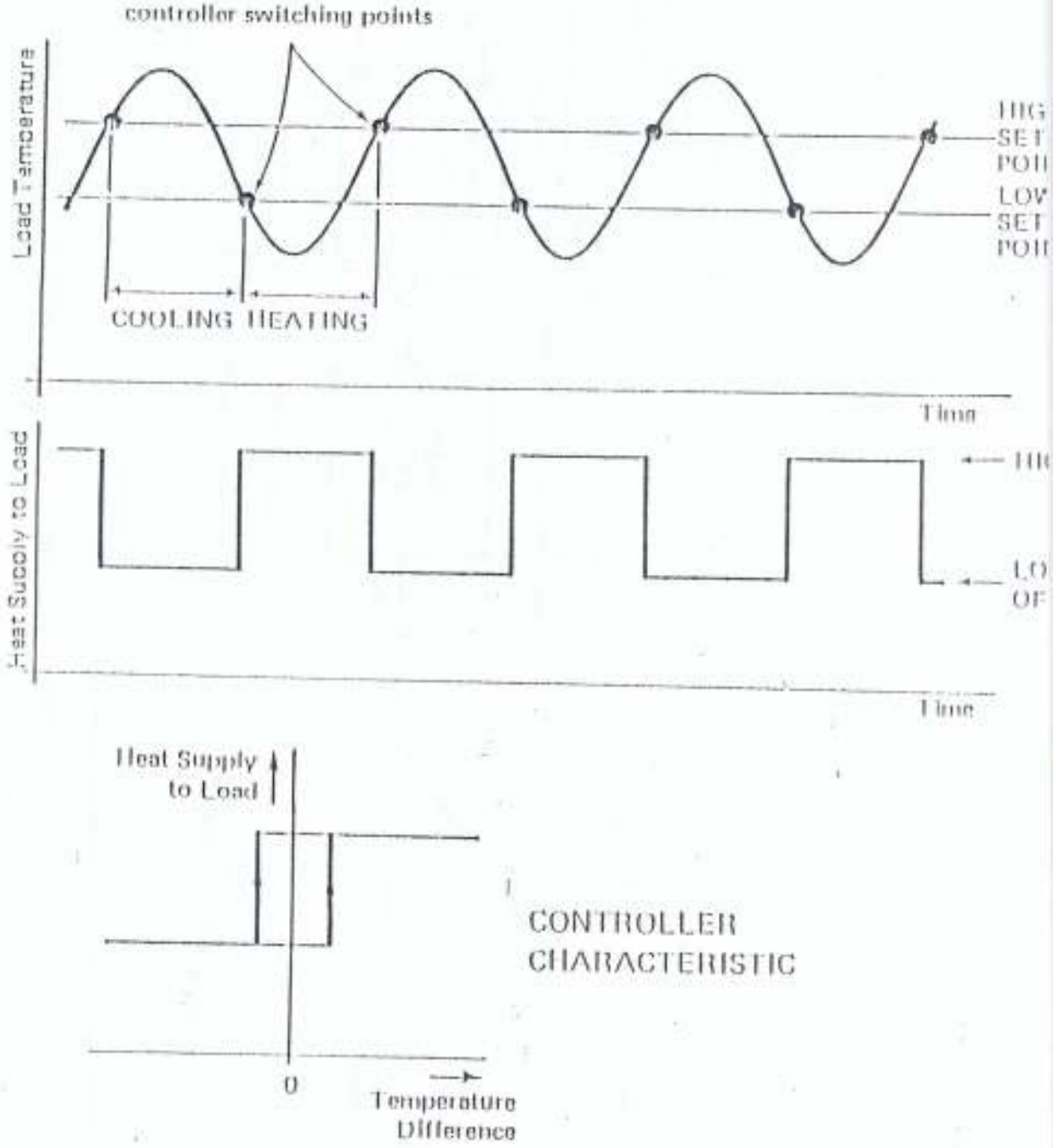
2.6.5 Final Control Elements

The function of a temperature controller is to switch on and off a source of heating or cooling. This is undertaken most readily with electromagnetic relays or contactors, in the case of electric heaters; however, the simplicity of the hardware must be traded against reduced reliability and maintainability. An alternative is to use solid state relay consisting of Triac or SCR networks, with the thyristors being gated on and off by a suitable drive network: since the source of electrical energy usually is AC and there is no advantage to be gained from rectification, it follows that Triacs would be preferable to SCRs. Reliability and maintainability will be high with power semiconductors, provided that they are protected

against excessive transients: the fact that a heater is almost purely resistive assists, in this respect.



g. 23: Temperature – Load Cycling for a Simple On/Off Control
hesmond, 1988)



g. 24: Temperature – Load Cycling for Overlap or Hystresis Control
Chesmond, 1988)

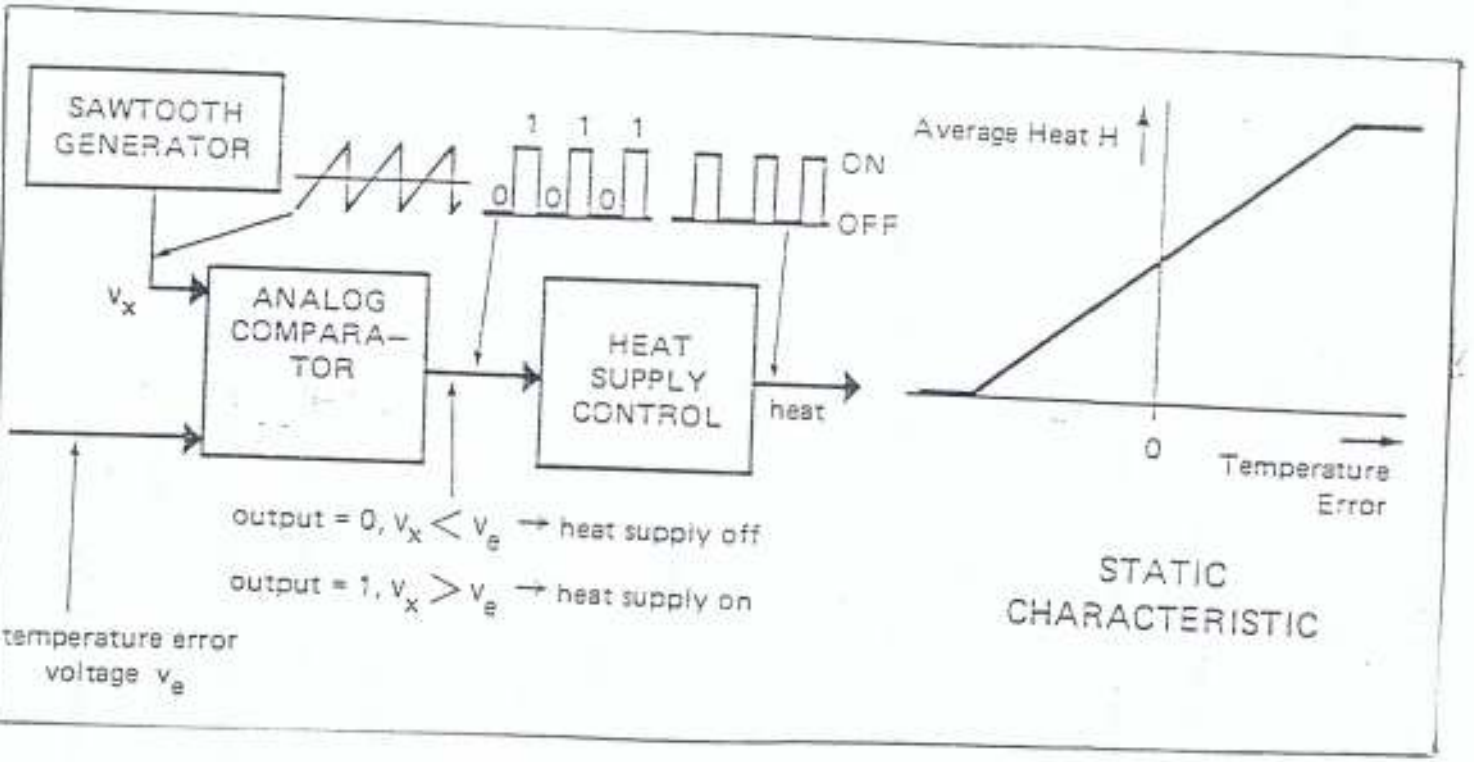


Fig. 25: On – Off Switching Hardware for Proportional Control
(Chesmond, 1988)

3.0 THE DESIGN

3.1 INTRODUCTION

The furnace to be designed is intended to serve for laboratory heating purposes of small test samples and objects of about 1 kg weight. It will be of a benchtop size. The target specifications for the design are as follows:

Chamber size	120 x 120 x 150 mm
Atmosphere	Air
Fuel type	Electricity: 220 V, 50 Hz
Heat up time	3000 - 3600s
Maximum Temperature	1200^0C
Load Capacity	1 kg

The design will entail:

i. material selection and design of the furnace chamber;

ii. refractory materials selection for the insulation and determination of their dimmensions;

iii. layout of process steps and variables for the production of required refractory, and devising of optimum process route;

iv. determination of total heat requirement for the temperature build up;

v. determiation of heat rating (power) to achieve the total heat requirement within the heat up time;

vi. design of the heating element (material and forming) to generate the thermal power; and

vii. selection of the control system specification.

3.2 MATERIAL SELECTION AND DESIGN OF THE FURNACE CHAMBER

From comparison of thermal conductivity values of common refractories (section 2.2.3.)), silica refractory material will be appropriate for the chamber material. The thickness of the lining will be fixed as an optimum between very thin dimensions (for best heat transfer, reduced waste specific heat in the wall and reduced overall unit weight), and thicker sections (for higher flexural rigidity and reduced fragility during refractory production). A compromise thickness of one centimeter can be used as a first estimate subject to corresponding heating rating requirement. This will dictate the maximum charge load density in the consideration for statical stability. Fig. 26 and 27 show the design drawing for the work tube. The work tube is designed as four interlocking slabs with the following advantages over slip extrusion as a single tube:

(i) the tube is collapsible and therefore safer for preinstallation handling;

(ii) less fragile handling stages during production;

(iii) tube failure cannot be total as any failed side can be separately replaced.

3.3 INSULATION MATERIALS AND DIMENSIONS

3.3.1. Heating Zone Insulation

This insulation is closest to the hottest zone in the furnace. To bring the temperature down on a very steep gradient in direction opposite to the work chamber the material of the insulation must be of very low thermal conductivity. For this purpose, standard made glass wool, with k = 0.038 W/m K will be an adequate insulation material.

The heat loss for varying thickness of insulation can be obtained from using the Fick's first law:

$$Q = -kA\frac{dT}{dx} \qquad 3.1$$

The temperature difference dT will be the difference between maximum target cold surface temperature, T_2 and the heating element's maximum temperature, T_1, say 1200 ^{o}C, the

normal area A will be the effective surface area of the work tube (Fig. 27 and 37); and k for glass wool. Then

$$A = 0.14 \times 0.11 \times 4 = 0.062\ m^2$$

Taking $T_2 = 40^{\circ}C$, an insulation thickness of 0.05m gives

$$Q = Q1 = -0.038 \times 0.062 \times (40 - 1200) / 0.05$$

$$= 54.66\ W$$

Any larger thickness than this will further reduce this value of heat loss. However, this will be unnecessary. Glass wool being structurally non supporting, with tendency for fibre deterioration under surface air current and certain health considerations, strong **backing** refractory will still be necessary, and this will further reduce q. Since 0.05m thickness of glass wool gives a bearable heat loss of 74.0544 W, this can be used as a target to obtain the effective serial thermal resistance, R_{TH} for the backing insulation. At a constant cross sectional area A, R_{TH} can be expressed as

$$R_{TH} = \frac{1}{A}\sum_i \frac{x_i}{k_i} \quad . \quad . \quad . \quad . \quad 3.2$$

Combining 3.1 and 3.2 gives,

$$R_{TH} = \frac{1}{A}\sum_i \frac{x_i}{k_i} = \Delta T/Q \quad . \quad . \quad . \quad 3.3$$

With $\Delta T = 1160$ and $Q1 = 54.66$ W,

$$R_{TH} = 1160/54.66 = 21.22\ K/W$$

A good and cheap insulation material at less severe thermal gradient is fireclay, and particularly kaolinitic clay with $k = 0.087$ W/ m K. At $A = 0.062\ m^2$, if a glass wool thickness of 0.025 is used, the corresponding kaolinitic clay backing for the target thermal resistance will be obtained using equ. 3.3:

$$1/0.062\ (0.025/0.038 + x/0.087) = 21.2224$$

$$\Rightarrow \quad x = 0.0572\ m$$

This implies that the total insulation thickness normal to each side of the work chamber is 0.0572 + 0.025 = 0.0822m. This rather bulky thickness can be reduced by a slight compromise on R_{TH} with resulting higher value of Q_1. At a backing thickness of 0.04m, using equ.3.2,

$$R_{TH} = 1/0.062\ (0.025/0.038 + 0.04/0.087) = 18.0267\ K/W$$

$$\Rightarrow Q_1 = 1160/18.0267 = 64\ W$$

3.3.2. Open Ends Insulation

The two open ends of the work tube will have to be insulated using porous insulating silica bricks. This is to ensure material continuity, prevent differential thermal expansion of mating surfaces at the end of the work tube, and offer structural support to suspend the work tube on either ends (Fig. 37). Taking thermal conductivity for the silica insulating bricks as 0.29 W/m°K, and starting from an initial value of 0.05m thickness, heat loss rate at chamber maximum temperature of 1200°C will follow from equ 2.2. With $A = 0.12^2\ m^2$, the heat loss from the charging end, Q2, will be

$$Q_2 = -(0.29)(0.12^2)(-1160)/0.05 = 96.8832$$

This value can be reduced by increasing the insulation thickness. Considering thicknesses of 0.06, 0.07 and 0.08m will give 80.736, 69.2 and 60.552 W as values of Q_2, respectively. The final choice is subject ot the maximum bearable heating inefficiency.

The foregoing analysis will guide the refractory materials dimmesioning and the entire furnace architecture. Figs. 28 – 43 show the design dimmensions of the refractory bricks. The charging end insulation bricks (Figs. 28 – 34) will be made to have a step to accomodete and suspend the work tube below and guide it in position above and on each side. The effective insulation thickness normal to the open ends will be a minimum of 0.075 m. The work tube in position (Fig. 37) will be wrapped with the glass wool thickness 0.025m. The kaolinitic clay will provide an insulation thickness of 0.04m on the four sides of the

and the rear end. The bricks will then be made as slabs (Fig. 35 – 46) to line a box panel. The box panel will be folded from a sheet mild steel to have an open front and appropriate provisions for connection at the back . Since silica bricks shows linear change of +1 at 1350°C, generous expansion allowances are reflected in the bricks dimmensions. The refractory dimmesion is also done to prevent the work tube from carrying any mechanical load; bricks 4 and 8 do not rest on the work tube (Fig. 37).

The resulting thermal resistance and heat losses on every side will be evaluated under estimation of total heat requirement.

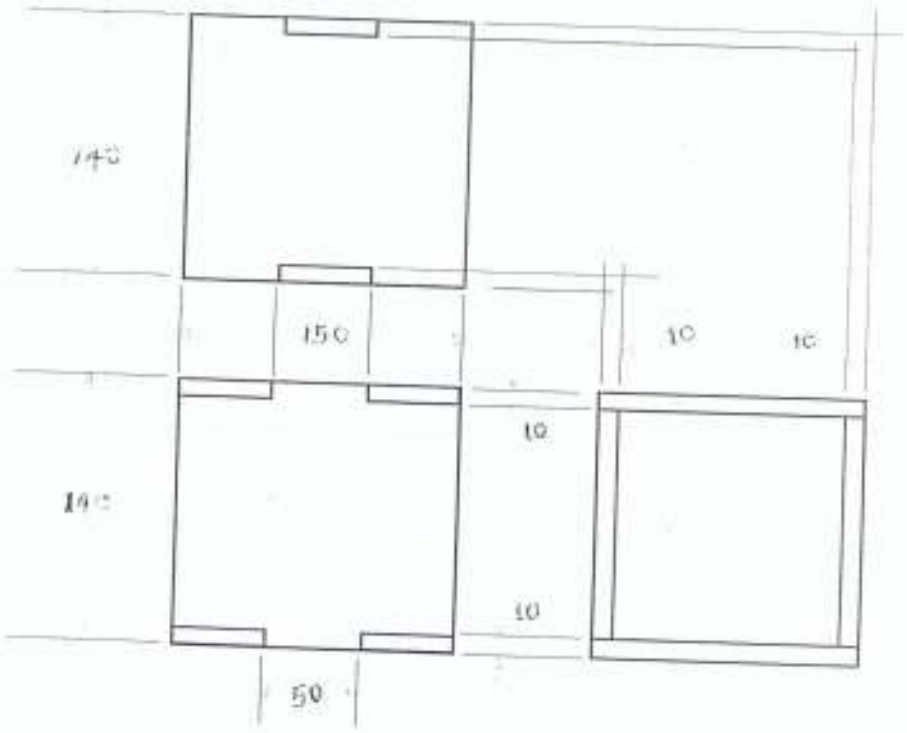


Fig. 26: Work Tube Assembly (Third Angle Projection, x 0.25)

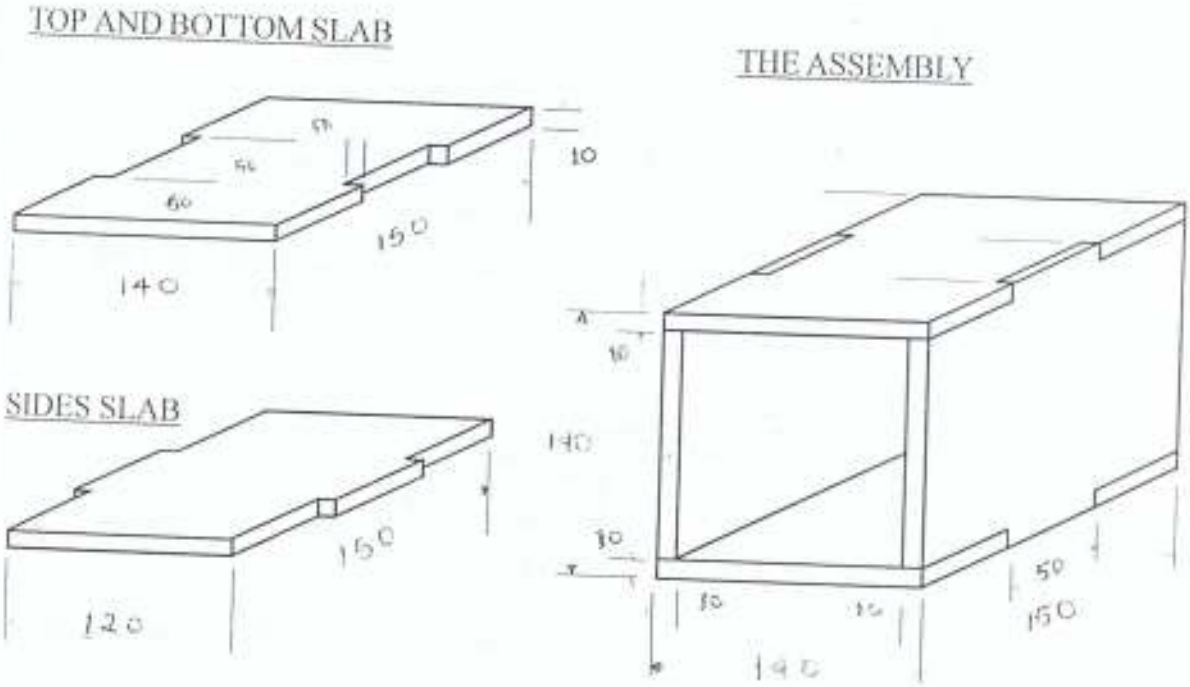


Fig. 27: Isometric View of the Work Tube Slilica Refractory Slabs and the Assembly (x 0.25, Dimm: mm)

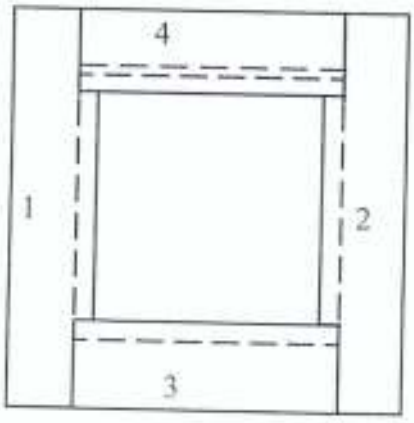


Fig 28: Front View of the Charging End of the Work T u be and the Supporting Silica Bricks (Dimm: mm)

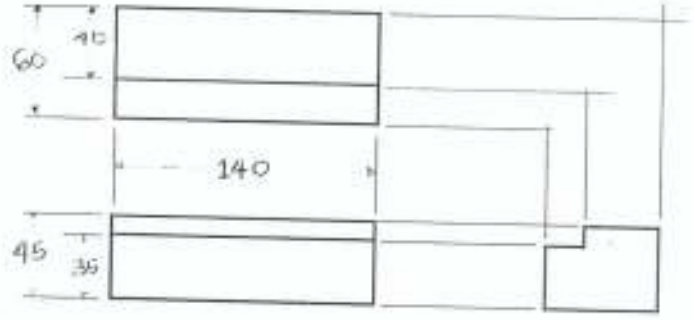


Fig. 29: Third Angle Projection of Brick 3 (Dimm: mm)

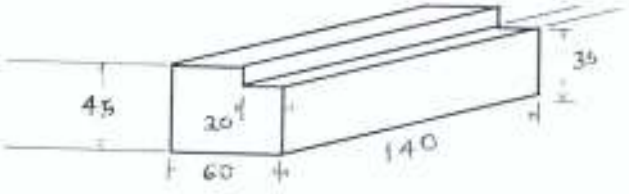


Fig. 30: Isometric View of 3 (Dimm: mm)

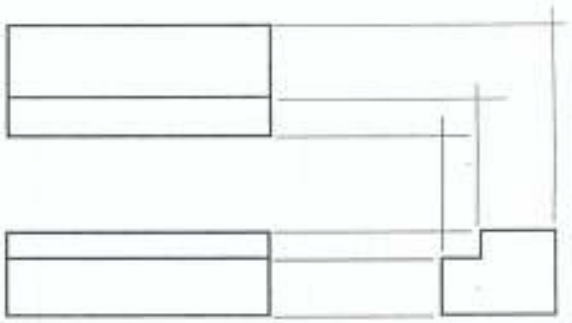
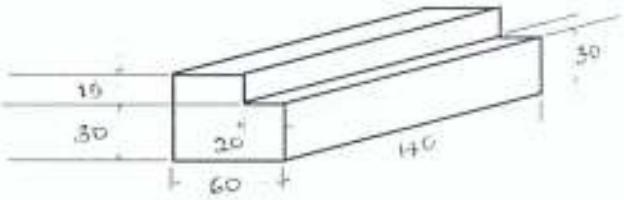


Fig. 31: Third Angle Projection of Brick 4 (x 0.25, Dimm: mm)



ig. 32: Isometric View of Brick 4 (Dimm: mm)

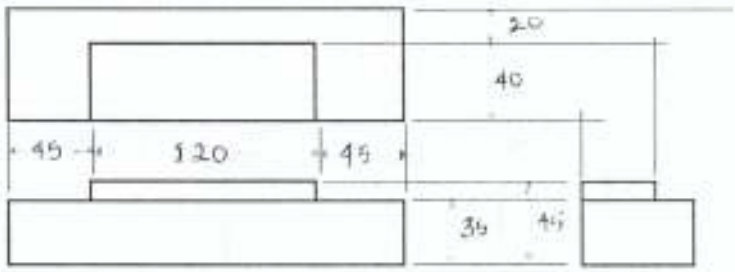


Fig. 32: Third Angle Projection of Brick 1 & 2 (0.25, Dimm: mm)

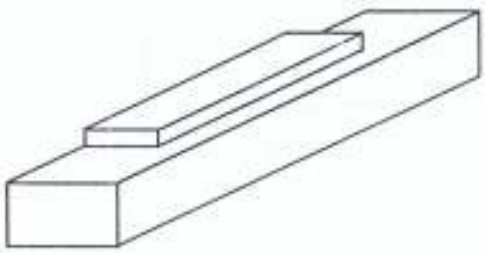


Fig. 34: Isometric View of 1 & 2 (x 0.25, Dimm: mm)

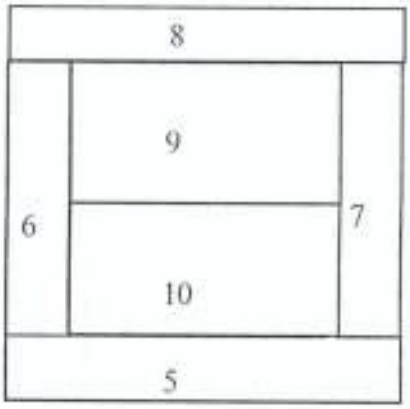
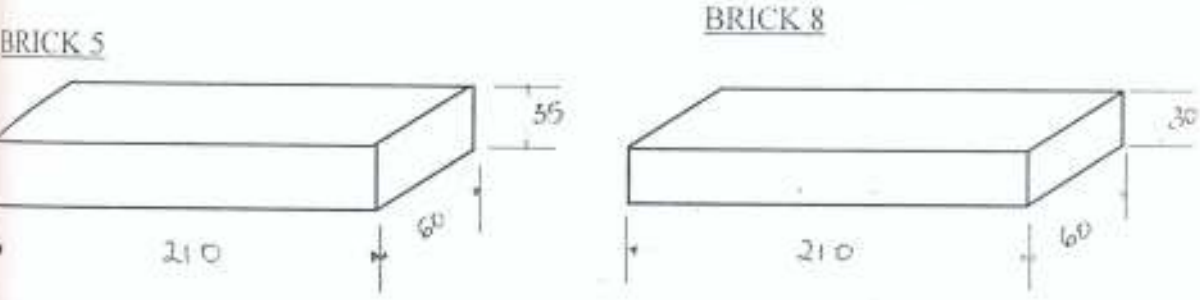
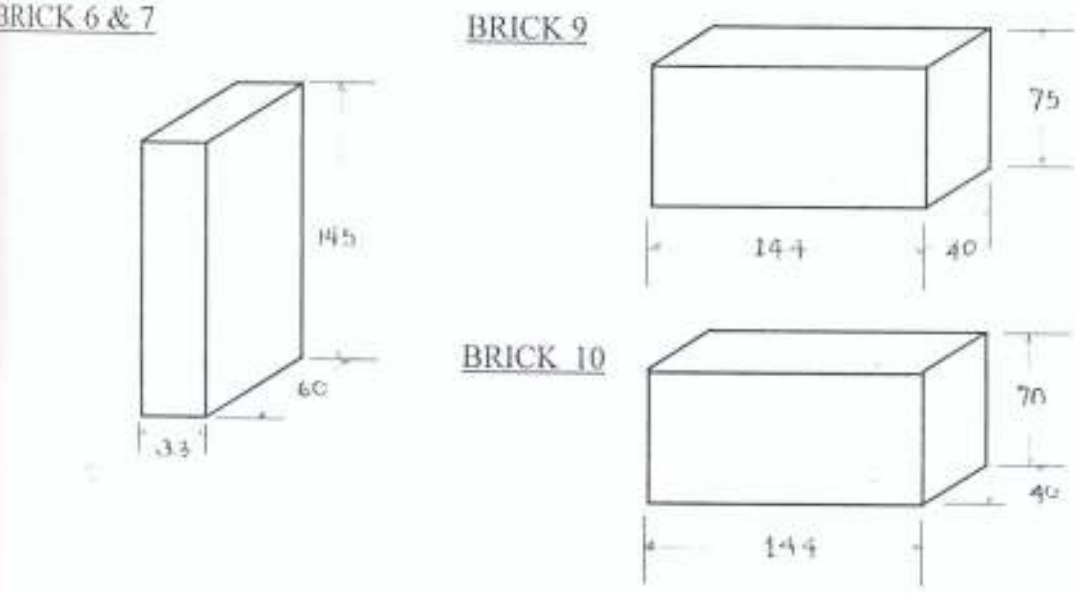


Fig. 35: Rear End Insualtion and and Suspension Silica Bricks Assembly: End View (Dimm: mm)





36: Isometric Views of Bricks 5 - 10 (Dimm: mm)

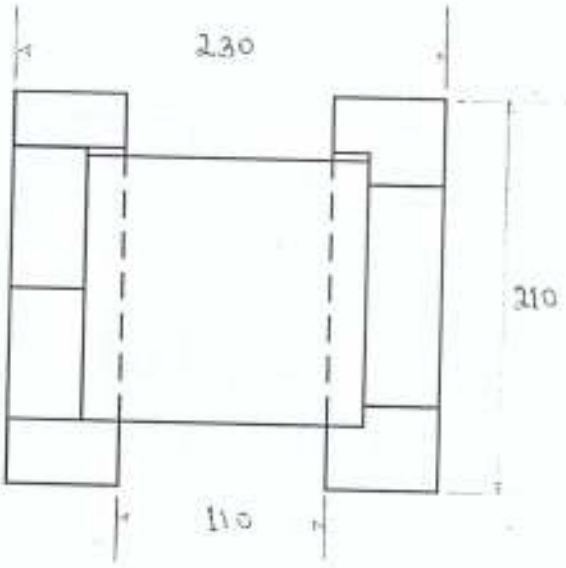


Fig. 37: Vertical Section of the Side View of the Work Tube and the Silica Bricks Assemmbly (x 0.25, Dimm: mm)

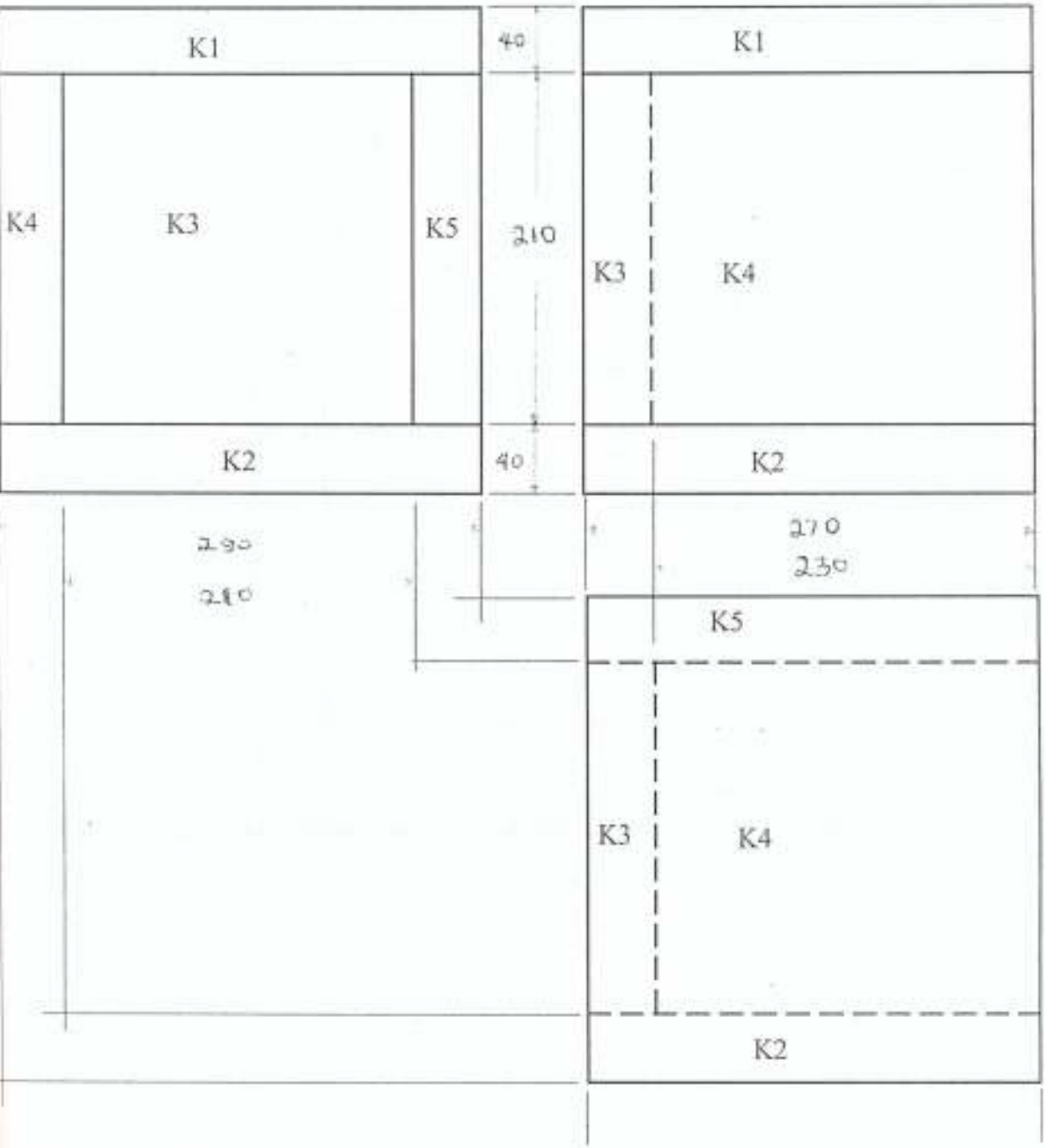
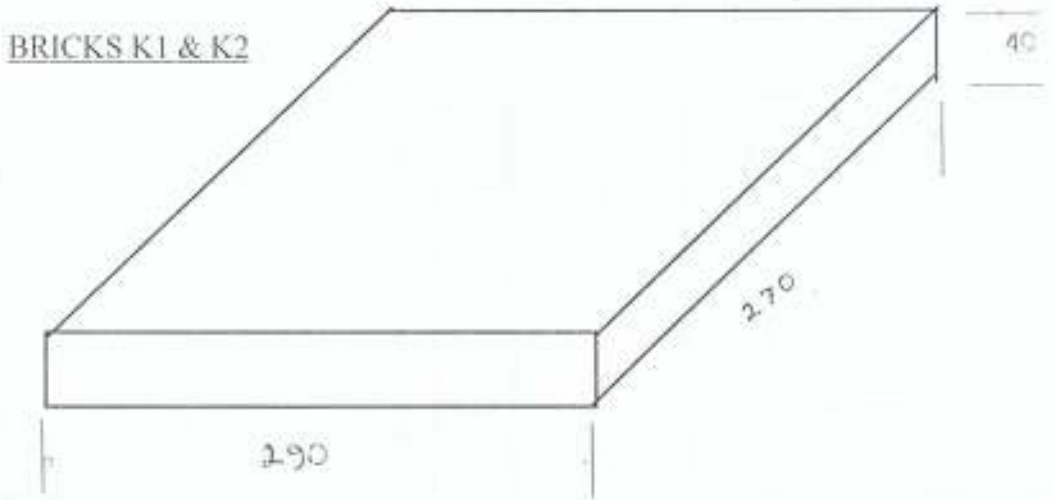


Fig. 38: Kaolin Backing Slabs Assembly (First Angle, x 0.25)



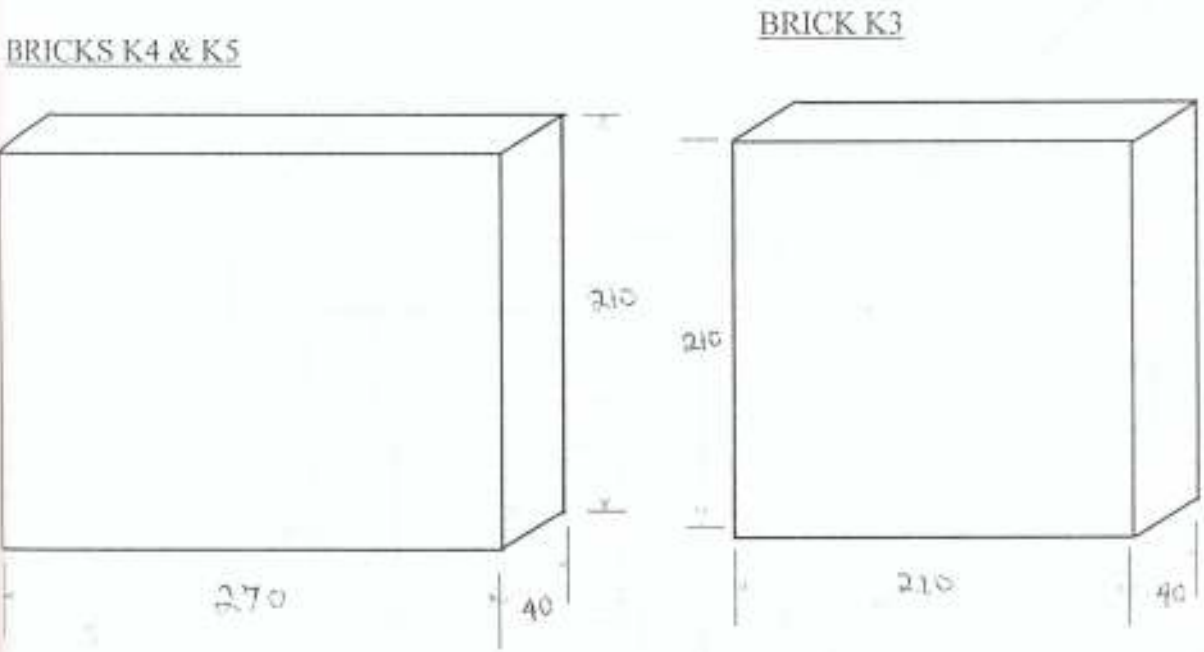


Fig. 40: Isomteric View of Kaolin Backing Bricks (Dimm: mm)

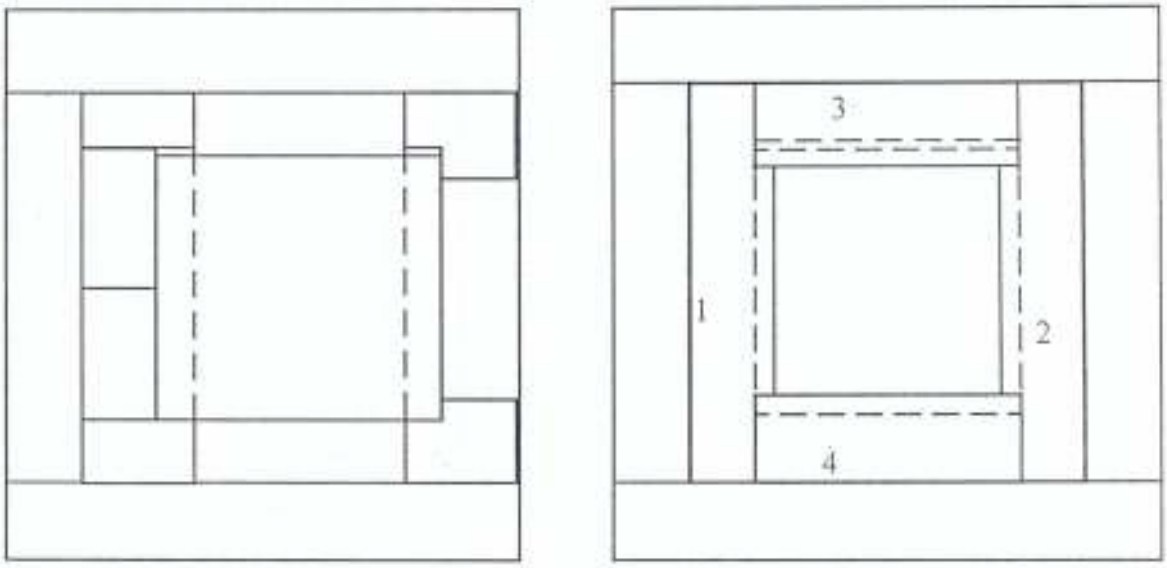


Fig. 41: Side and End View of the Final Refractory Assembly (Dimm: mm)

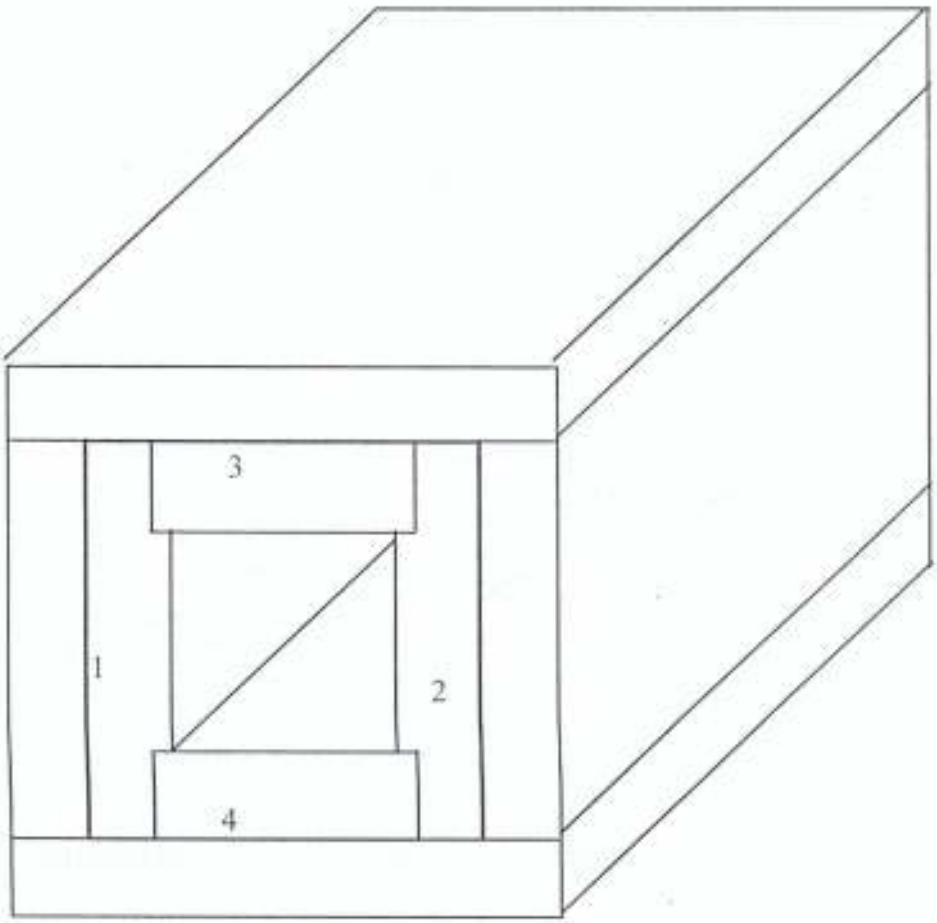
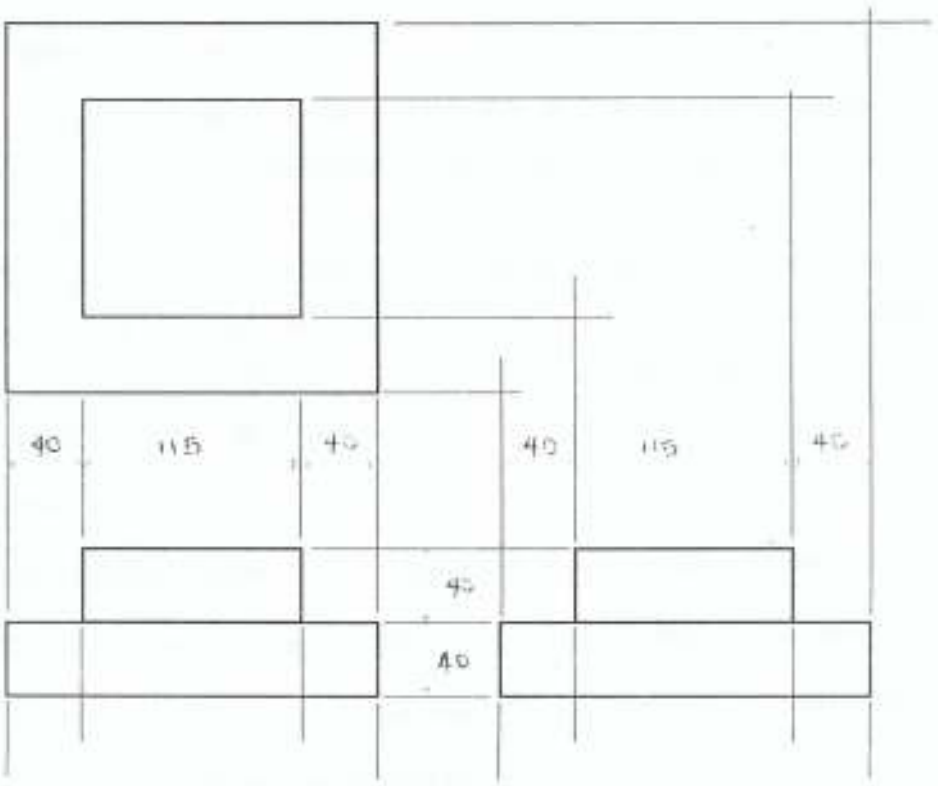
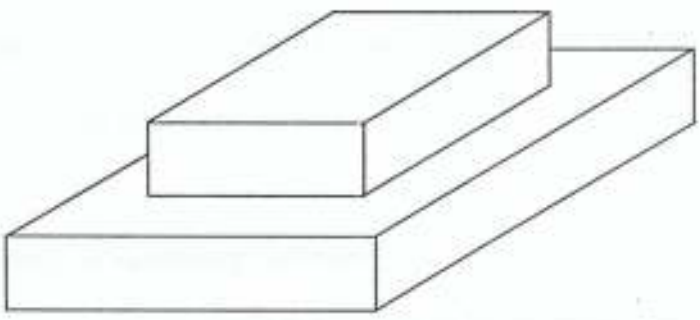


Fig. 42: Final Refractory Assembly - Isometric View (x 0.25, Dimm: mm)



43A: Charging End Cover Brick 11 (Third Angle, Dimm: mm)



43B: Charging End Cover Brick, Brick 11 (Isometric View, Dimm: mm)

3.4 REFRACTORY PRODUCTION

3.4.1 Process Route Design

As part of the set objectives of this work, it is desired to evolve process routes for producing the refractory requirement of the furnace from local refractory deposits. The process routes, which will seek to optimize the desired properties of the refractory products, should be feasible within the realities of equipment limitations to this kind of production in this environment. Towards this objective, after sourcing for appropriate clay and sand deposits as required by the design, the following steps will be taken:

a. Preliminary treatments: Washing of silica sand; and removal of free silica from clay by pulping and sieving.

b. Determination of mineralogical composition of the deposits to be used.

c. Variation of calcination pre-treatment, to portions of refractory raw materials.

d. Variation of particle size and proportion of wood flour additive in the refractory mix.

e. Variation of moulding process, hand or machine moulding.

f. Variation of firing rate to obtain shortest firing time.

g. Inspection and testing of products from all the combinations of process variables used to establish firing cracking, dimensional stability, and cold crushing strength.

h. Testing of test samples from the deposits used, to establish guide values of their porosity, bulk density and thermal shock resistance, in case of specific quantitative refractory work on these deposits later.

3.4.2 Composition of Insulating Refractory

The volume of the combustible materials to be introduced into the composition of a refractory mix to achieve a porous insulating structure can be obtained from the ratio of the normal density to the reduced density of the refractory material. For the silica refractory, normally the density of silica as quartzite is 2300 kg/m^3, to achieve a brick with a density of 900 kg/m^3, is to reduce density by a factor of 2.3/0.9. Since

Density = Mass / Volume

To reduce density by a factor of 2.3/ 0.9 implies increasing volume by the same factor (or reducing mass by the same factor). Working with volume, volume should be increased to 255%. Hence 155.5% (1.5 times) by volume of the silica mix will be added to the mix as the combustible material. On firing this will burn out leaving its volume.

3.5 HEAT REQUIREMENT

The estimation of the total heat requirement involves evaluation of the heat to heat up the chamber wall and space, HC; specific heat in insulation bricks, HS; and cumulative heat loss over a time interval, QL.

3.5.1 Evaluation of Heat for Chamber Wall, Hw

Using the basic equation for specific heating:

$$Q = mc\theta \quad . \quad . \quad . \quad . \quad . \quad . \quad 3.4$$

where Q is the heat required to raise the temperature of a body of mass, m and specific heat capacity, c, through $\theta^{o}C$, H_w can be estimated as

$$Hw = m\,C_{pw}\,dT = \rho_w V C_{pw} dT \quad . \quad . \quad 3.5$$

where C_{pw} is the specific heat capacity of the wall material at constant pressure; ρ_w is the density of the wall material; V is the volume of the chamber material. For a silica refractory work tube, a working approximate of ρ_w and C_{pw} can be taken as 2300 kg/m^3 and 837 J/kg K respectively (Faulker et al., 1985; Gosser, 1986; Holman, 1992); for a room temperature of $25^{O}C$, dT = 1200 – 25 = 1175; and V can be evaluated from Fig. 26 as

$$V = (0.14^2 - 0.12^2)\,0.15 = 0.00078\ m^3$$

$$\Rightarrow \quad Hw = 2300 \times 0.00078 \times 837 \times 1175 = 1764354.15\ J$$

3.5.2 Quantity Of Heat To Heat Up Chamber Space, Hs

This can be evaluated from a modification of equ. 3.4 which gives

$$H_s = \int_{298}^{1500} mC_p dT = \int_{298}^{1500} \rho V C_{pa} dT \qquad 3.6$$

The chamber volume being constant, implies

$$H_s = V \int_{298}^{1500} \rho C_{pa} dT \qquad 3.7$$

Since density and heat capacity of air, ρ and C_{pa}, are functions of temperature, they will have to be expressed as such. ρ is related to T as (Gosse, 1986):

$$\rho = P/287T \qquad 3.8$$

where T is temperature in K, and P in N/m^2 (Pa) is the pressure. At constant pressure, atmospheric, of 1.013×10^5 Pa, it follows that

$$\rho = 352.962/T \qquad 3.9$$

C_{pa}: Considering air as a mixture of N_2 and O_2 in 0.79/0.21 proportions, and C_p being physical properties, C_{pa} can be obtained as:

$$Cpa = 0.21Cpo + 0.79\ Cpn \qquad 3.10$$

where C_{po} and C_{pn} are the specific heat capacities, in kJ/kg K, of oxygen and nitrogen as a function of temperature, respectively.

Between 300 – 780 K (Gosse, 1986):

$C_{pn} = (1.093943 - 3.771278 \times 10^{-4} T + 7.547419 \times 10^{-7} T^2 - 2.967465 \times 10^{-10} T^3)$

$= C_{pnl}$ (low temperature value of C_{pn})

$C_{po} = 0.951021 - 4.453037 \times 10^{-4} + 1.395253 \times 10^{-6} T^2 - 8.464839 \times 10^{-10} T^3$

$= C_{pol}$ (low temperature value of C_{pn})

Between 780 – 1500 K:

$C_{pn} = 0.839063 + 4.61217 \times 10\text{-}4\ T - 1.412246 \times 10^{-7} T^2 + 9.257405 \times 10^{-12} T^3$

$= C_{pnh}$ (high temperature value of C_{pn})

$C_{po} = 0.671706 + 8.237775 \times 10^{-4} T - 5.376317 \times 10^{-7} T^2 + 1.330776 \times 10^{-10} T^3$

$= C_{poh}$ (high temperature value of C_{po})

Hence, C_{pa} can be taken as C_{pal}, between 300 – 780 K, and as C_{pah} between 780 – 1500 K.

This implies that:

$C_{pal} = 0.79\ C_{pnl} + 0.21\ C_{pol}$

$= 1.0639244 - 3.914454 \times 10^{-4} T + 0.88924913 \times 10^{-6} T^2 - 4.12191619 \times 10^{-10} T^3$

$C_{pah} = 0.79\ C_{pnh} + 0.21\ C_{poh}$

$= 0.80391803 + 5.37396575 \times 10^{-4} T - 2.2446743 \times 10^{-7} T^2 + 0.3525935 \times 10^{-10} T^3$

Substituting 3.9 in 3.7,

$$Hs = 352.962V \left(\int_{298}^{780} C_{pal}\, dT/T + \int_{780}^{1500} C_{pah}\, dT/T \right)$$

$$= (352.962)(0.00216) \left(\int_{298}^{780} C_{pal}\, dT/T + \int_{780}^{1500} C_{pah}\, dT/T \right)$$

$$= 0.7624 \left(\int_{298}^{780} C_{pal}\, dT/T + \int_{780}^{1500} C_{pah}\, dT/T \right) \quad 3.12$$

Then with the expressions of Cpal and Cpah, the definite integral can be estimated in steps as:

$\int_{298}^{780} C_{pal}\, dT/T =$

$= (1.0639244/T - 3.914454 \times 10^{-4} + 0.88924913 \times 10^{-6} T - 4.12191619 \times 10^{-10} T^2)\, dT$

$= (1.0639244 \ln T - 3.914454 \times 10^{-4} T + (0.88924913 \times 10^{-6}/2) T^2$

$- (4.12191619 \times 10^{10}/3) T^3) \Big|_{298}^{780}$

$= (1.0639244 \ln T - 3.914454 \times 10^{-4} T + 0.444624 T^2 + 1.373972 T^3) \Big|_{298}^{780}$

$= 1.0639244 \ln (780/298) - 3.914454 \times 10^{-4} (482) + 0.444624 (780^2 - 298^2)$

$- 1.373972 (780^3 - 298^3)$

$= 1.00445$ kJ

$\int_{780}^{1500} C_{pah}\, dT/T$

$= (0.80391803/T + 5.37396575 \times 10^{-4} - 2.2446743 \times 10^{-7} T + 0.3525935 \times 10^{-10} T^2)dT$

$= 0.80391803 \ln T + 5.37396575 \times 10^{-4} T - (2.2446743 \times 10^{-7}/2)T^2$

$+ (0.3525935 \times 10^{-10}/3) T^3 \Big|_{780}^{1500}$

$= 0.80391803 \ln(1500/780) + 5.37396575 \times 10^{-4} (720)$

$- (1.12233715 \times 10^{-7})(1500^2 - 780^2) + (0.117531166 \times 10^{-10})(1500^3 - 780^3) \Big|_{780}^{1500}$

$= 0.525703277 + 0.386925534 - (1.12233715 \times 10^{-7})1641600 +$

$(0.117531166 \times 10^{-10}) (2900448000) \Big|_{780}^{1500}$

$= 0.525703277 + 0.386925534 - 0.184242866 + 0.034089303$

$= 0.762475$ kJ

Hence,

$$Hs = 0.7624 \left(\int_{298}^{780} C_{pst} \, dT/T + \int_{780}^{1500} C_{psh} \, dT/T \right) \quad . \quad . \quad 3.12$$

$$= 0.7642 (1.0445 + 0.764275) = 1.347 \text{ kJ}$$

Then,

$HC = Hs + Hw = 1764354.15 + 1347 = 1765701.25$ J

3.5.3 Specific Heat In Insulation

The heat to effect body temperature rise, the specific heat, follows from equ. 3.4. as:

$$H = mc\theta \quad . \quad . \quad . \quad . \quad . \quad . \quad 3.4$$

$$= \rho vc\theta \quad . \quad . \quad . \quad . \quad . \quad . \quad 3.13$$

where m is the mass (kg), c the specific heat capacity (J/kg K), θ is the average body temperature rise, ρ the density and v the volume of the body. Hence to evaluate H in the bricks, the volume of the individual bricks will be evaluated and the θ will be obtained by first evaluating the facial temperatures of the insulation.

Volumes of Insulation Materials: From the design drawings (Figs. 26 – 43), Volume, in m^3, of

Brick 1 & 2, V12 = 2 (0.035 x 0.06 x 0.21) + (0.01 x 0.12 x 0.04) = 0.888×10^{-3}

Brick 3, V3 = (0.06 x 0.14 x 0.03) + (0.04 x 0.015 x 0.14) = 0.333×10^{-3}

Brick 4, V4 = (0.06 x 0.14 x 0.035) + (0.04 x 0.01 x 0.14) = 0.35×10^{-3}

Brick 5,V5 = 0.06 x 0.21 x 0.035 = 0.441×10^{-3}

Brick 6 & 7, V67 = 2 (0.033 x 0.06 x 0.145) = 0.2871×10^{-3}

Brick 8, V8 = 0.03 x 0.21 x 0.06 = 0.378×10^{-3}

Brick 9, V9 = 0.075 x 0.144 x 0.04 = 0.004032

Brick 10, V10 = 0.07 x 0.144 x 0.04 = 0.0004032

Brick 11, V11 = $(0.195^2 \times 0.04) + (0.115^2 \times 0.04)$ = 0.00205

Brick K1 & K2, VK12 = 2 (0.29 x 0.27 x 0.04) = 6.264×10^{-3}

Brick K4 & K5, VK 45 = 2 (0.27 x 0.21 x 0.04) = 4.872×10^{-3}

Brick K3, VK3 = $0.21^2 + 0.04 = 8.4 \times 10^{-3}$

Glass Wool, VG = 0.14 x 0.11 x 0.025 x 4 = 8.4×10^{-3}

Face Temperatures and Average Body Temperature Rise of Insulation: The charging end silica bricks (Figs. 28, 37, & 43) are in contact with hottest zones of the furnace (the heating element's surface and the chamber interior at maximum operating temperature) over one face and in contact with the room atmosphere over the other face. If the maximum projected cold surface temperature is 40°C, and the room temperature taken as 25°C, then the average body temperature rise for end silica bricks 1 – 4 and 11, ΔT_1, is

$$\Delta T_1 = (40 - 25) + (1200 - 40) / 2 = 595 \text{ K}$$

For the rear end silica bricks, bricks 5 – 10, there is a backing insulation of 40 mm kaolinitic clay slab, K3, (Fig. 38, 40 & 41). The effective heat flux through this thickness follows using eq. 3.3 and rearranging as:

$$= \frac{1160}{\frac{0.04}{0.29} + \frac{0.04}{0.087}} = 1940.77 \text{ W/m}^2$$

At this value of Q/A, the temperature at the colder face 0.04 m thickness of the silica bricks will be obtained from

$$1940.77 = \frac{-(T - 1200)}{\frac{0.04}{0.27}}$$

$\Rightarrow$ T = 1200 – 1940.77(0.04/0.29) = 932.3°C

This will correspond to the hot face temperature for brick K3. The average body temperature rise from room temperature (25°C) for silica bricks 5 – 10,

$$\Delta T_2 = (932.3 - 25) + (1200 - 932.3)/2 = 1041.15 \text{ K}$$

For the Kaolin Brick K3,

$$\Delta T_3 = (40 - 25) + (932.3 - 40)/2 = 461.15 \text{ K}$$

For the kaolinitic clay brikes K1, K2, K3 and K5 (Fig. 38 & 39), they are not in contact with the hottest zones. Their hot surfaces temperature will be evaluated from the effective heat flux through its thickness and the glass wool. From the heating zone insulation analysis (3.3.1), heat flux through the 2.5mm glass wool and 40mm slab is 64W. From equ. 3.1, it follows that:

$$Q1/A = -k\frac{dT}{dx} \quad . \quad . \quad . \quad . \quad . \quad . \quad 3.15$$

$\Rightarrow$ 64/ 0.62 = (0.087)(40 - T) /0.04

$\Rightarrow$ $-0.087T = \frac{(0.04)(74.7)}{0.062} + (0.087)(40) = 48019 + 3.48 = 51.6735$

$\Rightarrow$ T = 51.6735/0.087 = 594°C

From a room temperature of 25°C, aveage body temperature rise of the kaolinitic clay bricks, ΔT_4, will be

$$\Delta T_4 = (594 - 40)/2 + (40 - 25) = 302 \text{ K}$$

This will correspond to the cold surface temperature of the slag wool. Hence average body temperature rise for the slag wool, ΔT_S, will be,

ΔT_S = (594 – 25) + (1200 – 594)/2 = 569 + 315.5 = 442.25K

From these evaluations of the volume and temperature of rise of each insulation material the consequent specific heat in the insulation will follow from equation 3.3. Taking production densities of slag wool, kaolinitic clay and silica as 24, 304, and 900 kg/m^3, and their specific heat capacities as 700, 830 and 850 J/kg K respectively (Chesmond, 1988, Chesti, 1986; Gosse, 1986) then the specific heat in

Bricks 1 – 4 and 11 = 3.621 x 10^{-3} x 900 x 850 x 595 = 1648188.68 J

Bricks 5 – 10 = 1.9413 x 10^{-3} x 900 x 850 x 1041.5 = 1546725.922 J

Bricks K3 = 8.4 x 10^{-3} x 304 x 830 x 461.15 = 977401.88 J

Bricks K1, K2, K4 and K5 = 11.138 x 10^{-3} x 304 x 830 x 302 = 712421.23 J

Glass Wool = 1.54 x 10^{-3} x 24 x 700 x 442.25 = 11441.892 J

$\Rightarrow$ Total Heat In Insulation, HS = 4896178.924 J

3.5.4 Insulation Losses

From the charging end, using equ. 3.1:

$$Q = -kA\frac{dT}{dx}$$

the heat loss is through area A = 0.12^2 of a thickness of 0.08m silica insulation will be estimated as:

Q = (1160 x 0.12^2 x 0.29) / 0.08 = 60.552 W

From the rear end, A = 0.12^2, through composite silica and clay brick layers of 0.04m thicknesses each; then

Q = 1160 x 0.12^2 / (0.04 x (1/0.29 + 1/0.087)) = 27.9 W

Through the composite glass wool and clay backing layers on the sides of the chamer. A = 0.14 x 0.11 = 0.0154 m^2 on each of the sides, then

Q = (1160 x 0.0154 x 4) / (0.025/0.038 + 0.04/0.087))

= 71.456/1.117665 = 64 W

Hence, Total Heat Loss Rate through insulation, QL = 152.452 W

⇒ Over time **t** at maximum temperature in the chamber, total heat loss through insulation, HL = 152.452 **t** J

Then the overall heat requirement , HO, will be

HO = HC + HS + HL = (1765701.25 + 4896178.924 + 152.425t) J

= (6661880.174 + 152.452t) J

3.6 HEAT GENERATION

3.6.1 Heat Generation Rate / Power Requirement

For a target heat up time of t seconds, the corresponding heat generation rate to meet the estimated total heat requirement (HO), will be obtained from:

Power = Energy / Time = HO / t

= (6661880.174+ 152.452t) / t

= (6661880.174/ t + 152.452) W

With the target range of 3000 < t < 3600, the boundary power value will be

P1 (at t = 3000s) = (6661880.174/ 3000 + 152.452) W

= (2220.627 + 152.452) = 2373.079 W

P2 (at 3600s) = (6661880.174/ 3600 + 152.452) W

= 1850.52 + 152.452 = 2002.972 W

If heating efficiency is projected to about 90%, then,

P1 = 2636.75W ; P2 = 2225.52 W

The heat generation design will target this range of power values for the heat up time range.

3.6.2 Heating Element: Material and Forming

From alternatives of heating elements, metallic heating element is considered suitable for this work, specifically the alloy '875PM' (Appendix II). This alloy, also known as Kanthal APM, is a ferritic Iron-Chrome-Aluminum alloy from heating element manufacurers with the trade name Kanthal (Kanthal, 1987; Hyndman, 2002). Its specific qualities are good form stability, high temperature strength, and self supporting ability at ~1300^{0}C. It has an excellent surface oxide, which gives good protection in corrosive atmospheres. The chemical composition is 22% Cr, 5.8% Al and Fe in the balance. Appendix II gives more detailed technical information.

Determination of Heating Element length: For a fixed line voltage of 220V (rms) and power requirement of 3 kW, (considering bearable electricity cost, heating uncertainties, voltage fluctuations, it is safe to round up the power requirement from 2636.75 W to 3 kW):

$$P = V^2/R \quad \Rightarrow \quad R = V^2/P$$

where, P is the power (the heating rate), V is the voltage, and R is the resitance of the heating element. Taking,

$$R = rL$$

where r is the resistance per unit length in Ω/m (a function of the crossectional area), and Ls is the length (m), and, considering the temperature coefficient of resistance, C_T, gives:

$$R = C_T r L$$

then,

$$C_T r L = V^2 / P$$

$$\Rightarrow \quad L = V^2 / P C_T r \qquad . \quad . \quad . \quad . \quad . \quad 3.16$$

Considering three option of KAPM resistance diameters 1.0, 1.2 and 1.5 mm with r = 1.8457, 1.2818 and 0.8203 Ω/m respectively (appendix I), eq. 3.16 gives the respective required lengths L1, L2 and L3 as:

L1 $= 220^2 / (3000 \times 1.8457 \times 1.04) = 8.405$ m

L2 $= 220^2 / (3000 \times 1.2818 \times 1.04) = 12.102$ m

L3 $= 220^2 / (3000 \times 0.8203 \times 1.04) = 18.911$ m

The final choice of the wire length/diameter will have to consider the resulting surface load for a giving power rating. From the alloy table data (appendix I), the surface area per meter for the three diameters of wire 1.0, 1.2 and 1.5 are repectively 31.4159, 37.6991 and 47.1239 cm^2/m. Evaluating surface load p as

p = power required / (total length x surface area per unit length) . 3.17

surface loads, p, at a power requirement of 3000 W, for the different diameters of the wires gives:

p1 $= 3000/ (31.4159 \times 8.405) = 11.36$ W/cm^2

p2 $= 3000/(37.6991 \times 12.102) = 6.58$ W/cm^2

p3 $= 3000/(47.1239 \times 18.911) = 3.37$ W/cm^2

subscripts 1, 2 and 3 representing wires of diameters 1.0, 1.2 and 1.5, repectively.

Alternatively, using the alloy table formula and values for surface load, with consideration for C_T at the maximum operating temperature,

$$p = \frac{I^2 C_T}{cm^2/} = \frac{C_T (P/rL)}{cm^2/} \quad . \quad . \quad . \quad . \quad . \quad 3.18$$

p1 $= (1.04 \times 3000) / (1.8457 \times 8.405 \times 17.0208) = 11.82$ W/cm^2

p2 $= (1.04 \times 3000) / (1.2818 \times 12.102 \times 29.4120) = 6.83$ W/cm^2

p3 $= (1.04 \times 3000) / (0.8203 \times 18.911 \times 57.4453) = 3.5$ W/cm^2

Since the element life is longer at lower surface loads, a 1.5 mm wire will be chosen, with corresponding element length of 18.911 (~ 19 m); at a higher initial cost compromise.

Forming - For a work tube depth of 300 mm, with cross-sectional perimetr of 2 (L x B) = 600 mm, and length of wire, *l*, the winding pitch for uniform heat distribution can be inffered from:

Length of wire = length per turn x no. of turns

= cross-sectional perimetre x Depth of tube / Winding pitch . 3.19

Hence,

Winding Pitch = crossectional perimeter x depth of tube / length of wire

For, *l* = 18.911 m

Winding Pitch = 0.6 x 0.3 / 18.911 = 0.0095 m = 1 cm (approx.)

No. of turns = 300 / 9.5 = 31.58 turns = 31 turns

The approximation was round down because rounding up will imply longer length of wire which will reduce the power output (eq. 2.14); the already very low surface load of the 1.5 mm wire and the winding pitch rounded up will allow and compesate for this.

3.7 CONTROL SYSTEM SPECIFICATION

The general aspects of control include the feed back transducer type, the reference transducer, error detection, the signal and power amplification circuitry, and the final control element (Fig. 9). Various circuitry and components are available to achieve these tasks in stages (section 2.5). Design decisions between the alternatives will be made based on their different levels of accuracy, justifiable cost compromises and other relative advantages (component availability, ruggedness).

The Feedback Transducer: With the target maximum temperature of 1200°C, the operating temperature of the thermocouple wire should be about 1300°C; 100°C above the maximum. This implies, the type-S thermocouple, Platinum – Platinum Rhodium 10% (Table 10) will be adequate for the control system.

The Reference Transducer: The range of options here spans servo-potentiometer, differential inductor and differential transformer based reference transducers. The choice deciders actually are projected cost limit for the control unit and the desired input graduation. Since the cost of the control unit is an integral part of the expenses of the whole furnace, cost minimization choices will be favoured at the expenses of fineness in the graduation. Rotary potentiometer will be good for the reference transducer specification.

The Controller Module and the Final Control Element: By adopting the proportional control law, the final temperature control will be achieved by a kind of electronic circuitry shown in Fig. 25. The temperature error signal for the comparator will be generated by either of the two circuitries in Fig. 22.

This set of specification will be sought for on the various off-shelf controller modules. The final control signal from the controller module will be fed to SCR as the final control element, which will switch the heater on or off proportionally. The wiring diagram for the furnace is shown in Fig. 44. Appendix IV shows the entire control circuit diagram.

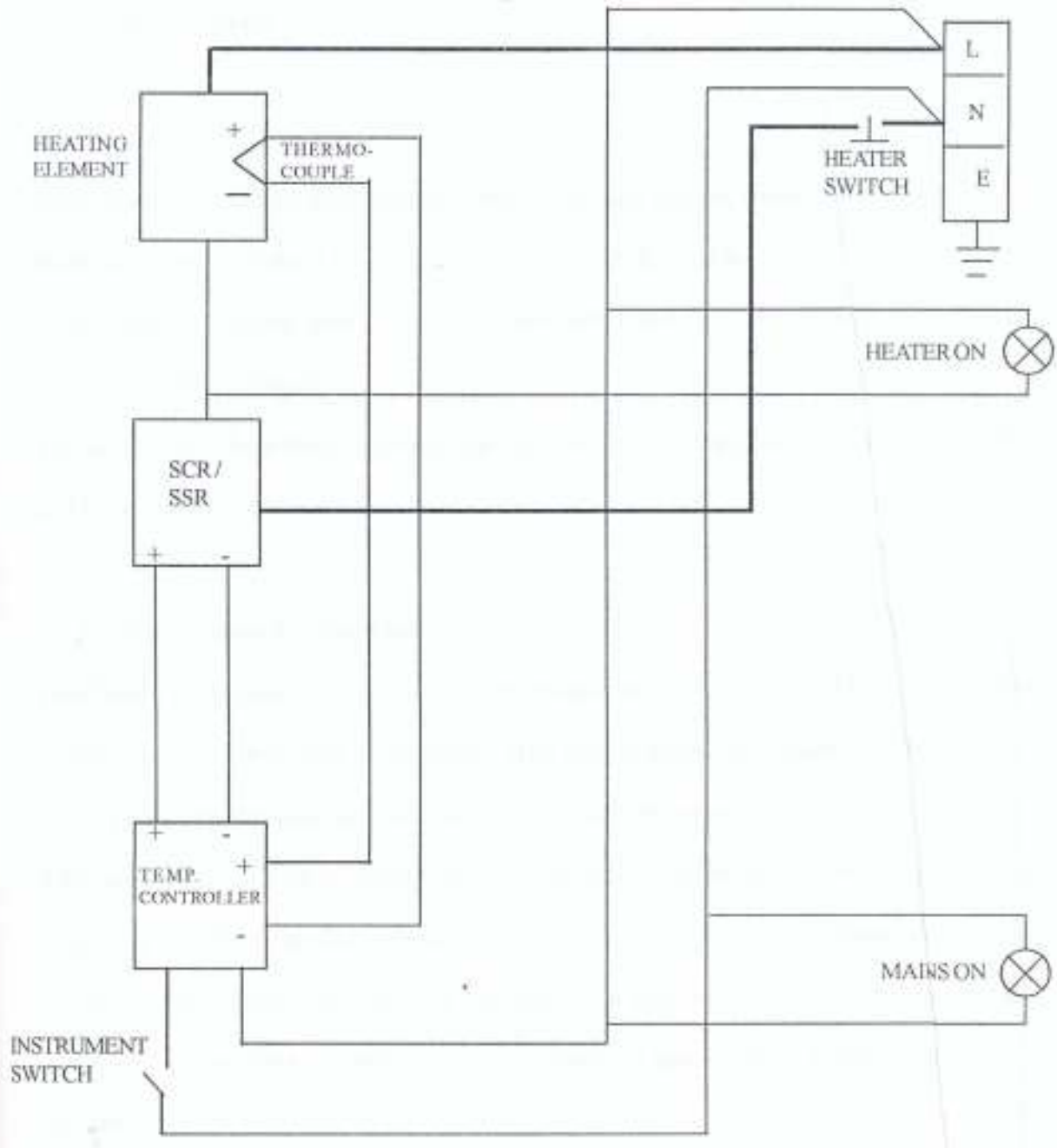


Fig. 44: The Furnace Wiring Diagram

4.0 THE FURNACE CONSTRUCTION

4.1 MATERIALS

4.1.1 Refractory Materials

Silica Sand - obtained from Igbokoda beach silica sand deposit, Ondo State, Nigeria.

Kaolinitic Clay - obtained from Ijapo, Akure, Ondo State, Nigeria.

Wood Flour - obtained from wood planer machines, wood workshop, Federal University of Technology, Akure, Nigeria.

The samples for the refractory production experiment were designated $S_1 - S_7$, S_T, C_1 - C_3, and C_T, defined as in Table 4.1.

4.1.2 Temperature Control Materials

Feedback Transducer A type-S thermocouple was obtained from Alaba International Market, Lagos Nigeria. The thermocouple wire type is platinum – platinum rhodium 10%, with a ceramic thermowell material; temperature sensitivity range is $0 - 1538^{\circ}C$.

The Controller An off-shelf controller was obtained from Alaba International Market, Lagos Nigeria. The controller operates by on-off control law; required feedback transducer is a type-S thermocouple. The reference transducer is a system of four rotary potentiometers. Temperature sensitivity, tolerance: $\pm$ 5°C. Display: Light Emitting Diode (LED) digital display.

Final Control Element An on-off electromagnetic 20-amps switching relay. Source: Alaba International Market, Lagos, Nigeria.

Table 4.1: Designations of Refractory Samples and Variations in Their Compositions and Processing Treatments

Label	Composition	Moulding Method
S_1	From uncalcined sand, 40% of -1mm – 750μm, 15% of -750 – 500μm, and 45% of -500μm sized sand was mixed by weight proportions. 5% bentonite was added. The batch was thoroughly mixed with 15% water.	Hand Moulding
S_2	Same as composition S_1 but from calcined Sand	As S_1
S_3	Raw rod milled sand, mixed with 5% bentonite and 15% water, by weight.	As S_1
S_4	Calcined sand: 40 % by weight of -2.5 – 2.0mm size range and 20% by weight of -2.0 – 1.5mm, and 40% by weight -1.5 – 1.0mm, thoroughly mixed by hand. 1.5 times by volume of the sand was added as -1200 – 1000μm wood granules. After thorough mixing, 5% by weight of the total mix is added as bentonite, then 20% by weight of total mix is added as water.	Hand Moulding
S_5	Same as S_4 but with raw sand and 15% water.	Machine pressed at 250N/cm^2
S_6	Rod milled raw sand, mixed with 1.5 times by volume of 1000 - 1200μm wood granules and 5% by weight bentonite, and after thorough manual mixing, 15 % by weight water was added and thoroughly mixed manually.	As S_5
S_7	Raw milled sand mixed with equal amount by volume of –850 μm size wood flour. 5% by weight of the mix is then added as bentonite. After thorough mixing, 15% by weight of water was then added and thoroughly mixed together manually.	As S_5
S_8	Same as S_1	As S_5
C_1	Treated Calcined clay and 5% bentonite by weight was thoroughly mixed manually, then 10% by volume of 600μm wood flour and 15% by total weight was added as water and the final mix was thoroughly mixed manually to very even mix.	Machine Pressed at 250 N/cm^2
C_2	Uncalcined clay, 10% by volume of 600μm wood flour. This was thoroughly mixed and 15% by weight of the mix was added as water.	As C_1
C_3	Same as C_2 but with 20% by volume of 600μm wood flour.	As C_1
C_4	100% clay with treatment A.	As C_1

4.1.3 The Heating Element

Metallic heating element was obtained from Alaba International Market, Lagos, Nigeria. It is of the wire type with a cross sectional diameter of 1.7mm. The physical properties of the heating element such as its dull gray colour, shearing difficulty, magnetic attraction conforms to that of the ferritic Al-Cr-Fe heating element alloys (Appendix I) (Hyndman Ind., 2002). The resistance was measured to be 0.88Ω/m using digital multimeter.

4.1.4 Construction Materials

A mild steel sheet was obtained from a steel product store in Akure. The thickness of

the sheet was measured to be 1.32 mm. This sheet will serve for the construction of the housing for the refractory assembly and open-end insulation brick (Figs. 42 and 43). A right-angled profile bar, 1" x 1" section, was also obtained. The bar is to serve for the construction of the chassis that will carry the furnace.

4.2 REFRACTORY EXPERIMENT AND PRODUCTION

As outlined in the refractory production process route design (3.4.1), the following steps were taken for the production of the different refractories specified by the design, which are the conducting silica refractory, the insulating silica refractory, and the insulating kaolinitic clay refractory.

4.2.1 Conducting Silica Refractory

Sample Analysis: After washing to remove physical dirt, and free drying on concrete floor, a sample of the Igbokoda beach silica sand was taken for determination of its mineralogical composition by classical chemical analysis. The analysis was carried out by the wet chemical analysis section of the Nigerian Metallurgical Development Center, Jos, Nigeria. Appendix-II outline the standard procedures for the key compounds. The result is shown in Table 5.1.

Pretreatments: Three portions of the raw sand were given separate treatments as below:

A. Classification to four particle size ranges: -2.5 – 2.0mm; -2.0 – 1.5mm; -1.5mm – 1.0mm; -1.0mm – 750μm; and -750μm – 500μm.

B. Calcination at 1400°C for 30 minutes, and cooling, before classification as in A.

C. Grinding in rod mill to 45% -500 – 250μm particle size. Complete particle size analysis of the rod-milled sand is shown in Table 4.2.

Table 4.2: Complete Particle Size Analysis of the Rod Milled Sand:

Total weight of sample = 1000g; Sieving time = 15 minutes

Sieve Size (μm)	Weight (g)	Sample Weight Percent	Cumulative weight percent	Cumulative passage weight
+1000	11.86	1.19	1.19	98.81
-1000 + 710	20.73	2.07	3.26	96.74
-710 + 500	55.09	5.51	8.77	91.23
-500 + 250	470.85	47.10	55.88	44.12
-250 + 130	311.85	31.20	87.08	12.92
-130	129.05	12.91	99.99	0.01
	999.43*	99.98		

*sieving error = 0.057% (negligible)

Composition and Mixing:

Composition S_1 – From portion with treatment A above, (not calcined), 40% of -1mm – 750μm, 15% of -750 – 500μm, and 45% of -500μm sized sand was mixed by weight proportions. This corresponds to a weight ratio of 4 kg : 1.5 kg : 4.5 kg respectively. 5% bentonite was added. The batch was thoroughly mixed with 15% water.

Composition S_2 - Same as composition S_1 but from calcined sample with treatment B above.

Composition S_3 – Raw rod milled sample, mixed with 5% bentonite and 15% water, by weight.

Moulding: Composition S_1, S_2 and S_3 were hand-moulded to slabs for the furnace chamber (Fig. 26 & 27) *in custom made wooden moulds. The mix* was manually rammed into the mould and excess material scrapped off. Silica flour obtained by pulverizing the raw sand to 10μm was rubbed into the surface of the slabs to seal open pores and improve densification. A **test sample, S_T** was machine pressed on a table moulding machine at 250N/cm^2 from

composition S_1 to test for bulk density, porosity and thermal shock resistance of the silica sand as a refractory material.

Firing Kiln temperature kept at 200°C for 30 minutes, and then uniformly increased to 1400°C in six hours, soaked for 30 minutes, and allowed to cool to room temperature inside the furnace for about 18 hours (overnight).

4.2.2 Insulating Silica Refractories

Pre-treatment: The silica sand used was given similar pretreatment as for the conducting silica refractory. The wood shaving was classified to -1200 – 1000μm granules, -1000 – 850μm, -850 – 600μm and -600μm flour.

Composition:

Composition S_4: Calcined sand: 40 % by weight of -2.5 – 2.0mm size range and 20% by weight of -2.0 – 1.5mm, and 40% by weight -1.5 – 1.0mm, thoroughly mixed by hand. 1.5 times by volume of the sand was added as -1200 – 1000μm wood granules. After thorough mixing, 5% by weight of the total mix is added as bentonite, and then 20% by weight of total mix is added as water.

Composition S_5: Similar composition as in S_4 but with raw (uncalcined) sand and 15% water.

Composition S_6: Sample from treatment C (rod milled raw sand), mixed with 1.5 times by volume of 1000 - 1200μm wood granules and 5% by weight bentonite, and after thorough manual mixing, 15 % by weight water was added and thoroughly mixed manually.

Composition S_7: Raw rod milled sand (treatment c) mixed with equal amount by volume of – 850 μm size wood flour. 5% by weight of the mix is then added as bentonite. After thorough mixing, 15% by weight of water was then added and thoroughly mixed together manually.

Moulding: Composition S_4 was manually rammed by hand into custom-made moulds and the mould carefully removed. Composition S_5, S_6 and S_7 were machine pressed at 250 N/cm^2.

Drying: Free air-drying for 24 hours, followed by oven drying at 200°C for one hour for all the compositions.

Firing: Same as for the conducting silica refractory.

4.2.3 Insulating Kaolinitic Clay Refractory

Pre-treatments:

A. Weathering of raw clay on open floor under shade for two weeks, followed by water leaching of trace alkali oxides present; pulping, sieving with improvised cloth sieve, with sieve size estimated under the microscope to about 150μm, to remove free silica. Settling and decantation of the clay suspension followed by slip pressing in porous packing to drain off water; atmospheric drying; and pulverizing.

B. All of the above treatment followed by calcination at 1400 °C for 30 minutes.

Sample Analysis: The mineralogical composition of the clay was carried out by classical chemical analysis, after pretreatment A. The analysis was carried out by the wet chemical analysis section of the Nigerian Metallurgical Development Centre, Jos, Nigeria. Appendix-II outline the procedures for the key compounds. The result is shown in Table 5.1.

Composition:

Composition C_1: Treated Calcined clay and 5% bentonite by weight was thoroughly mixed manually, then 10% by volume of 600μm wood flour and 15% by total weight was added as water and the final mix was thoroughly mixed manually to very even mix.

Composition C_2: Uncalcined clay (treatment 1), 10% by volume of 600μm wood flour. This was thoroughly mixed and 15% by weight of the mix was added as water.

Composition C_3: Same as C_2 but with 20% by volume of 600μm wood flour.

Composition C_T: 100% clay with treatment A. This is a test composition like S_T for the silica sand. It is to test for bulk density, porosity and thermal shock resistance of the clay as a refractory material. The results are shown in Table 5.5.

design modifications in the heating element material and forming became necessary since the design specification of the heating element could not be obtained. 21m length of 1.7mm wire was used instead of 18.911m of 1.5mm wire specified by the design.

4.4 HOUSING CONSTRUCTION AND FURNACE ASSEMBLY

A net was cut from the mild steel sheet with dimensions such that on folding it conforms to the shape of Fig. 42. The gives the box panel to house the insulation assembly. A supporting frame was also made from one-inch angle bars to carry the box panel and provide for a door to hold the charging end insulation brick (Fig. 43B). The panel was then lined with the insulating kaolinitic clay bricks produced. Whole bricks were sawn to fit in spaces where necessary. The conducting silica slabs were assembled to form the chamber. The formed heating element was then carefully positioned around the silica slabs chamber unit. The chamber with the heating element in place was then wrapped up with the insulating refractory glass wool. This chamber assembly (the silica slabs, the heating element and the glass wool insulation) was gently seated on silica bricks, linearly centered within the clay-bricks-lined box, with the free ends of the heating elements passed through the lining, in spaces provided, to allow for power connection. The chamber assembly was then carefully supported with insulating bricks to fix it in place. The insulation brick of the charging end of the chamber (Fig. 43B) was inserted into the space provided in the door. The external view and a sectional drawing of the furnace are shown in Plate I and Fig 46 respectively. Then the electrical connection was done, guided by the wiring design (Fig. 44). The thermocouple was inserted into the chamber through the clay bricks lining from the backside of the box panel. With the temperature controller and the final control element (the electromagnetic relay) in place, and all electrical connections intact, the furnace was powered for a performance evaluation.

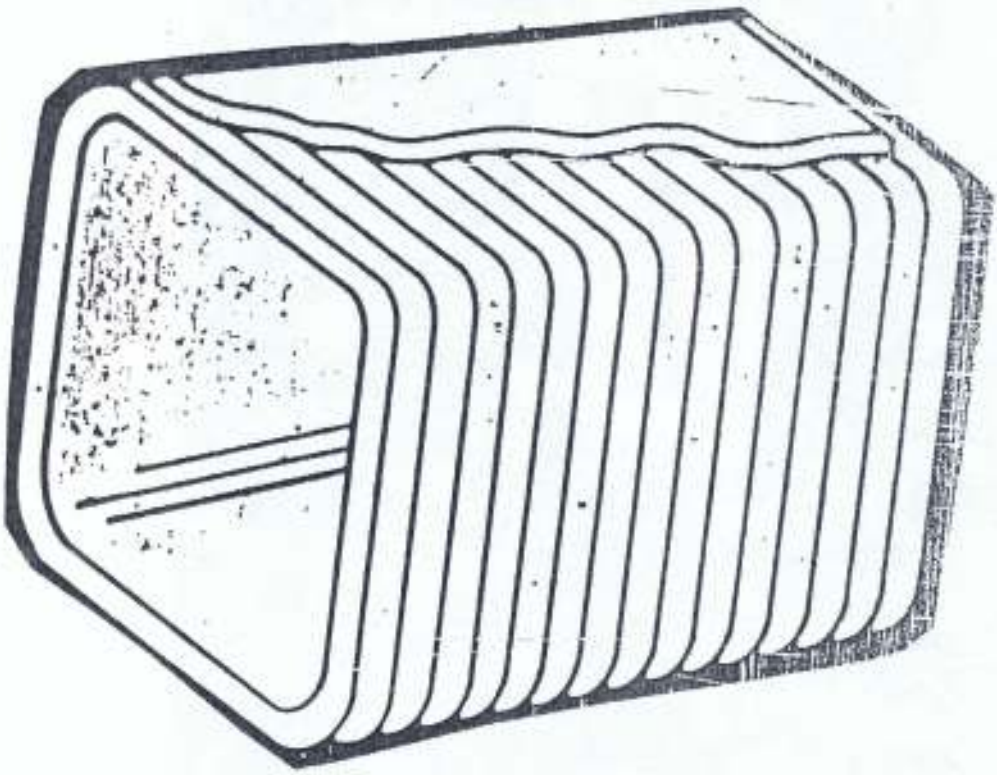


Fig. 45: Final Form of the Heating Element

Plate I: External View of the Furnace

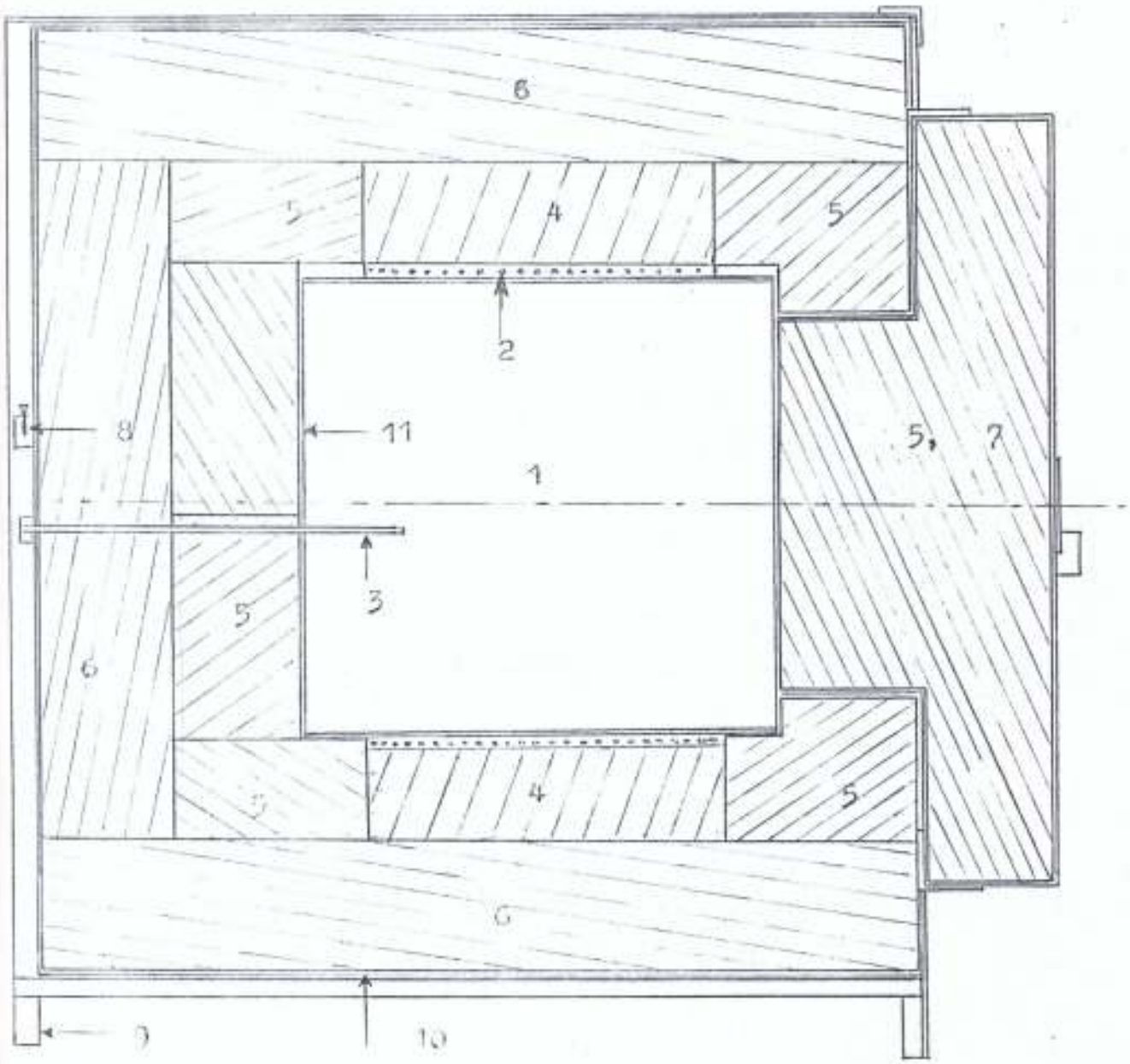


Fig. 46: A Sectional View of the Furnace (x 0.5)
1 – Chamber; 2 – Heating Element; 3 – Thermocouple; 4 – Glass Wool; 5 – Insulating Silica Bricks; 6 – Kaolinitic Clay Bricks; 7 – Door Insulation Brick; 8 – Power Connection Terminal; 9 – Chassis; 10 – Mild Steel Box; 11 – Conducting Silica Work Tube

5.0 RESULTS, PERFORMANCE EVALUATION AND DISCUSSION

5.1 RESULTS

5.1.1 Compositions Of Refractory Deposits

The result of the chemical composition analysis of the Ijapo clay was shown in Table 5.1.

Table 5.1: Chemical Composition of the Ijapo Kaolinitic Clay

Constituent Compounds	SiO_2	Al_2O_3	FeO	Fe_2O_3	CaO	MgO	Na_2O	TiO_2	L.O.I.
Percentage Composition	52.02	28. 83	3.23	3.58	N.D.	N.D.	--	--	11.22

The result of the chemical composition analysis of the Igbokoda beach sand is as shown in Table 5.2.

Table 5.2: Chemical Composition of the Igbokoda Beach Sand

Constituent Compounds	SiO_2	Al_2O_3	FeO	Fe_2O_3	CaO	MgO	Na_2O	TiO_2	L.O.I.
Percentage Composition	92.54	1.13	1.07	1.19	N.D.	N.D.	--	--	0.05

5.1.2 Properties Of Refractories Produced

Firing Crack The result of the firing crack inspections is as shown in Table 5.3.

Cold Crushing Strength The result of qualitative destructive testing carried out on the different bricks compositions are summarized in Table 5.3.

Table 5.3: Firing Crack and Cold Crushing Strength of the Refractories Produced

COMPOSITIONS	FIRING CRACK	COLD CRUSHING STRENGTH (kN/m^2)
S_1	None	12265
S_1	None	9732
S_2	None	9486
S3	None	16184
S_4	None	1192
S_5	None	4753
S_6	None	8150
C_1	None	12058
C_1	None	9240
C_2	Decompression Cracks	11560
C_3	More Severe Cracks	11281

Linear Dimensional Changes The dimensional measurements taken on samples from composition S_1, S_2, C_2, C_3 and the resulting linear expansivities are shown in Table 5.4.

Thermal Shock Resistance The test clay and silica samples, C_1 and S_1, were both tested for thermal shock resistance and both withstood twenty cycles of firing and cooling without any crack, Table 5.5 (copy of the NMDC test certificate in AppendixIII).

Table 5.4: Linear Expansivity Measurements on Some Composition

Composition	Length before Firing, L1 (mm)	Length after Firing, L2 (mm)	Linear Expansivity, $\lambda = (L2 - L1)/L1$
S_1	140.50	141.60	0.0078
S_2	140.50	141.28	0.0111
C_1	234.82	224.18	-0.0453
C_3	234.82	209.72	-0.1069

Other Properties Porosity and bulk density were established for reference composition C_T and S_T and this are shown in Table 5.5. Since the composition and process routes of C_1 and S_1 do not represent any of the ones for the actual service in this work, these properties were determined just to serve as a guide for more quantitative later work on these refractory raw materials.

Table 5.5: Other Properties of the Refractory Raw Materials Used

Material	Bulk Density, g/cm^3	Porosity, %	Thermal Shock Resistance, Cycles endured
Clay	1.38	50.15	20
Silica	1.77	28.18	

5.2 PERFORMANCE EVALUATION OF THE FURNACE

5.2.1 Temperature Build Up And Heating Rate

The temperature build up with time for the first 18 minutes of powering the furnace was recorded in Table 5.6, and represented by the plot of Fig. 5.1. From the Table, temperature rise picked up from the first minute of heating, with temperature rising to 293°C in the first 15 minute. This is an average temperature rise of about 20°C per minute.

Table 5.6: Temperature Build Up on Powering The Furnace

Time, min	Temperature, °C
1.0	28
2.0	36
3.0	46
4.0	60
5.0	71
6.0	89
7.0	110
8.0	128
9.0	151
10.0	169
11.0	194
12.0	217
13.0	240
14.0	264
15.0	293
16.0	321
17.0	345
18.0	369

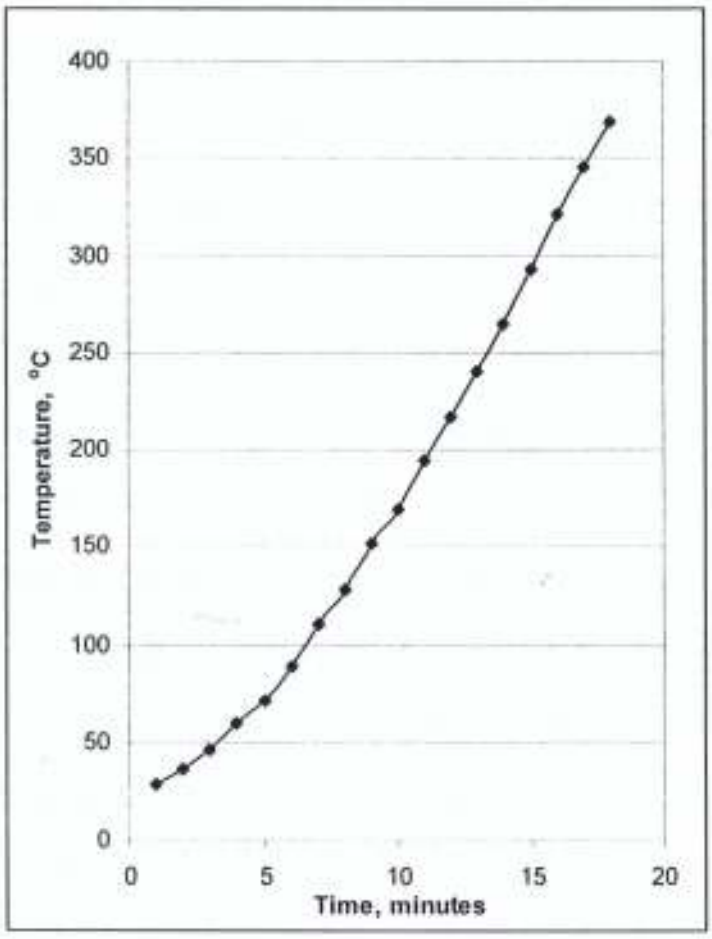


Fig. 5.1: Plot of Temperature Build Up with Time on Powering the Furnace

5.2.2. The Control System

Different set temperatures were used to check the effectiveness of the control system. The control switched off the power supply to the heating element at the different set temperatures, on cooling and on heating, with a tolerance of about 5°C. However on heating, after the power supply to the heating element has been switched off by the controls system, about 40°C temperature offshoot was noticed before the temperature started to drop, fluctuate and hold about a set temperature.

5.2.3 Ergonomics

Ergonomics considerations in evaluation of any tool include considerations of factors that will enhance most conducive and comfortable use and handling of such tool. In the performance evaluation of the furnace, the following ergonomic features were observed.

Operational Ease: When the power plug is connected to the mains, heating commences. The controller automatically starts with a display to monitor the chamber temperature. The control can be set to a target temperature without interrupting the heating. At the set temperature, the controller switches off heating. This is a relatively easy operation compared with most indigenous hydrocarbon fuel fired models where: fuel has to be ignited manually with a flame; heating temperature depends on ingenious manual flame color control; heating control entails manual control of fuel supply valve; and, there is no temperature monitoring device.

Operational Hazard: The furnace operates almost with no operational hazard: no noise, no gas pollution, no vibration, and no naked flame with attendant high stray thermal radiation to operators.

Weight: Although furnace units are installed in fixed position, minimal unit weight is always preferred for freight ease. Compared to standard imported model of similar size, the furnace is still relatively heavier - about 1.5 times (30 : 20kg). This weight extra is contributed solely by the insulating refractories that are still quite heavier than those in imported models. With further works on the insulating refractory, the overall unit weight can be reduced.

5.2.4. Economics

Operating Cost: The unit operates on electricity with a target to achieve 1200°C in one hour. Since

Energy, kWh = Power Rating in kW x Time in hr

and

Energy Cost, ₦ = Energy, kWh x Cost per kWh in ₦,

then, at a power consumption of about 3.5kW (3437.5W), and electricity cost of ₦ 4/kWh, this translates to ₦14.00 for one hour of firing. A similar size butane-fired muffle chamber will use about 5kg of gas in such time interval, costing about ₦500.00, excluding freight costs during gas refill.

Overall Cost of the Furnace Unit: The present model is only a preliminary design model. Since product cost evaluation is done on perfected design model, overall product cost estimation cannot be presented at this stage of the work. Rather what applies at this stage is the project work cost evaluation, an estimate of which is about ₦65, 000.00. It can be projected that the perfected model may not cost more than this, since all experimental costs will not be involved, but only the actual materials and the standardized process costs will be accounted. However, if a perfected model should cost up to about ₦70,000.00, to account for all possible finishing intricacies, it will still be a worthwhile foreign exchange saving for the Nigerian technological community. Similar models sell in excess of $1000.00 (Science Projects Ltd., 2002), excluding freight costs and delivery delay. Apart from this foreign exchange savings advantage, the technological slavery, and other odds attached to consumption of foreign technology are more facts to consider. Nevertheless, if the cost implication of perfecting this model should be more demanding and final unit cost higher than projected, it is still worth the while. Nigerians should not think that there is hope in technological independence; there is no alternative to technological development for any nation to survive in this present world. So, for what there is no alternative, economics itself recommends that it is better to suspend economic consideration.

5.3 DISCUSSION

5.3.1 The Refractory Deposits

From the result of the analysis (Table 5.1), the Ijapo clay with 28.18% Al_2O_3 is suitable for consideration as a kaolinitic fireclay deposit, since the alumina percentage of fireclay accepted for refractory applications is usually 25 to 45 (Chesti, 1986; Borode et al, 2000; Omowumi, 2001). With no traces of the alkali and basic oxides, it can be inferred that the extensive pulping and sieving pretreatments have assisted in leaching off this common impurity oxides, which do form double silicates and lower refractoriness (Chesti, 1986). With this composition, using equation 2.1:

$$\text{Refractoriness, } ^{\circ}\text{C} = \frac{360 + 100\left(\frac{Al_2O_3 - R_2O_3}{Al_2O_3 + SiO_2}\right)}{0.228} \quad . \quad . \quad . \quad 2.1$$

where Al_2O_3 and SiO_2, are the percentage compositions of alumina and silica, respectively, while R_2O_3 represents total percentage composition of Fe_2O_3 and other trivalent oxides present, the refractoriness of this clay can be estimated as:

$$\text{Refractoriness, } ^{\circ}\text{C} = \frac{360 + 100\left(\frac{28.183 - 3.58}{28.183 + 52.02}\right)}{0.228} = 1713.5^{\circ}\text{C}$$

This is an adequate figure, the refractoriness demand of the work at hand being 1300°C: 100°C above the target maximum operating temperature. With this composition analysis and the kind of pretreatment given this clay, the Ijapo kaolinitic clay deposit could have been recommended greatly for refractory work, but the deposit suffer certain limitations. Ijapo clay deposit is more of the reddish clay type, with higher iron content and more thermally conducting. The white kaolinitic portion of the Ijapo clay has a very low reserve. Buildings are also closing up on the deposit. Hence, the deposit may not be regarded for kaolinitic clay on economic scale in a near future.

With 92.54% Silica, this beach sand is a bit on the low side for typical silica refractory compositions with percentage silica of about 94% (Table 5.2). Although, with a total of 2.26% iron bearing impurities that can be eliminated by roasting and magnetic separation, the Igbokoda beach sand is still a valuable asset around for silica refractory production.

5.3.2 Refractories Production

From Table 5.3, it can be seen that none of the compositions of the silica refractories gave firing crack at the firing rate used. This is noteworthy, because the firing rate of 0 -1400°C in seven hours is relatively very rapid compared to recommended firing time of about 160 hours for silica refractories (Chesti, 1986). This firing rate was initially used as first trial after which slower rate was to be tried. With this observation, it can be inferred that changing beach temperatures and weathering must have effected the stabilization of the beach sand compared to freshly crushed quartzite. Hence, in using the Igbokoda silica sand:

i. Precalcination is unnecessary, and the attendant cost is avoidable;

ii. Firing and total production cost will be highly reduced with firing spanning only seven hours.

With the clay compositions, C_1 gave no firing cracks, while cracks were seen in C_2 and C_3. The cracking was initiated by a stress relieving decompression in the axial direction of pressing. The firing effects then acted on this crack sources. This analysis is supported by the fact that the cracking is more pronounced in C_3 that was more compressed, having 20% wood flour. Normally, wood flour will not compress compactly. With this tendency to decompress, it follows that the larger the wood flour proportions in the mix, the larger the decompression crack initiation factor/effect. Contrasting C_1 and C_2, with C_1 from calcined clay and C_2 from raw clay, it can be concluded that calcination has helped the firing stability

of C_1. This is an expected result, as precalcination normally effects pre-stabilization (Chesti, 1986). Contrasting C_T with C_1, C_2 and C_3, C_2 and C_3 showed firing cracks, while C_1 and C_T gave neither firing nor decompression cracks. Since C_T has no wood flour content, it follows that the decompression effect will be absent. The regulated firing programme for C_T would also have assisted in preventing firing crack in it. Since the presence of wood flour in the mix is desired for better insulation properties, C_1 is best recommended for firing stability in using this clay, at this stage of the work. The firing programme could also be a bit relaxed to span eight hours instead of the six hours used for 200 –1400°C.

For the conducting silica refractories, S_1, S_2, S_3, from the cold crushing strength result (Table 5.4), it can be inferred that hand moulding can give a good strength in the refractories. With the poor strength of S_4, it can be inferred that hand pressing is not good for moulding such insulating composition. The significant proportion and unfavourable shapes of wood granules in the composition will not make the mix compress compactly with the low pressure in hand moulding. This inference agrees with the fair strength of S_5, having the same composition as S_4, but machine pressed. Comparing S_6, rated to have a good strength, with S_5, it can be inferred that a much finer size grading of the mix (-500μm + 250μm modal sieve size) and not only the size distribution assists in compaction and improves the resulting strength. S_7, composed from the rod-milled sand and not with wood granules, but with lesser proportion (50% by volume) of wood flour, gave the best green strength of the insulating silica compositions. Unfortunately, S_7 could not be fired due to certain limitations this work suffered (6.2). S_T, with no wood content and machine pressed, gave better strength compared to S_1 of similar composition, and this agrees with the analysis made that wood flour reduces strength while machine pressing improves it.

With the best strength of 16184 kN/m^2, recorded for S3, being low compared to usual minimum of 24000 kN/m^2 for silica refractory (Chesti, 1986), it shows that there is need for overall strength improvement of the silica refractories. Apart from moulding method and

particle size, the cold crushing strength of the silica refractories could also be increased by firing to about 1500°C. The higher temperature will increase the extent of particle sintering, and thus increase crushing strength of the sample (Borode, et al, 2000). Heating to this higher temperature can now take a longer time of seven hours, to avoid rapid heating.

The clay compositions, although all gave low strength compared to the standard minimum of 15000kN/m^2 clay refractories (De Bussy, 1972, Omowumi, 2001), yet this strength figures are all satisfactory for porous insulating compositions like these, and they are also better than those for the insulating silica refractories. This should follow from the fact that the compositions were machine pressed; have much lesser proportions of wood flour compared to the silica refractories; and of finer clay particle size. A portion of the C_1 that was hand pressed gave a very poor green strength and even, after firing was too weak to be handled.

From Table 5.4, the linear expansivities noted for composition S_1 (from raw sand) and S_2 (from calcined sand) are approximately equal. If the calcination will have effect, then S_2 (calcined) should show lesser value of expansivity than S_1. Rather, S_1 (from raw sand) even showed lesser value of expansivity. Then it can be inferred that precalcination does not (at least significantly) affect dimensional changes of silica bricks made from this beach sand. The difference in expansivities values for S_1 and S_2 could therefore be attributed to inconsistency in material distribution caused by uneven pressure in hand moulding; S_1 and S_2 being hand pressed.

For the clay composition, C_1 (calcined clay) showed a linear shrinkage of 4.52% while C_3 (raw clay) showed 10.69%. For the lesser volume of clay in C_3 (80% clay, 20% wood flour) to show shrinkage than C_1 (90% clay, 10% wood flour), it implies that the precalcination really has stabilization effect on the clay, as expected (Chesti, 1986). Hence, toward dimensional stability, precalcination will be advantageous in making use of this clay.

Although about 25 -30 cycles is usually preferred before concluding on the thermal shock resistance (De Bussy, 1972; Omowumi, 2001) this result at least gave a guide of the minimum this materials can offer. Since the composition and process routes of C_1 and S_1 do not represent any of the ones for the actual requirement in this work, these properties have being determined just to serve as a guide for more critical quantitative work on the actual compositions to be recommended. In a more detailed quantitative work exclusively on the refractory aspects of this work, this test could be extended to failure of the test pieces, and the actual endurance limits established for the recommended compositions.

From the foregoing discussion, the two flow charts of Fig. 5.1 and 5.2 have been drawn, summarizing the recommended process variables for production of refractories from the two deposits used.

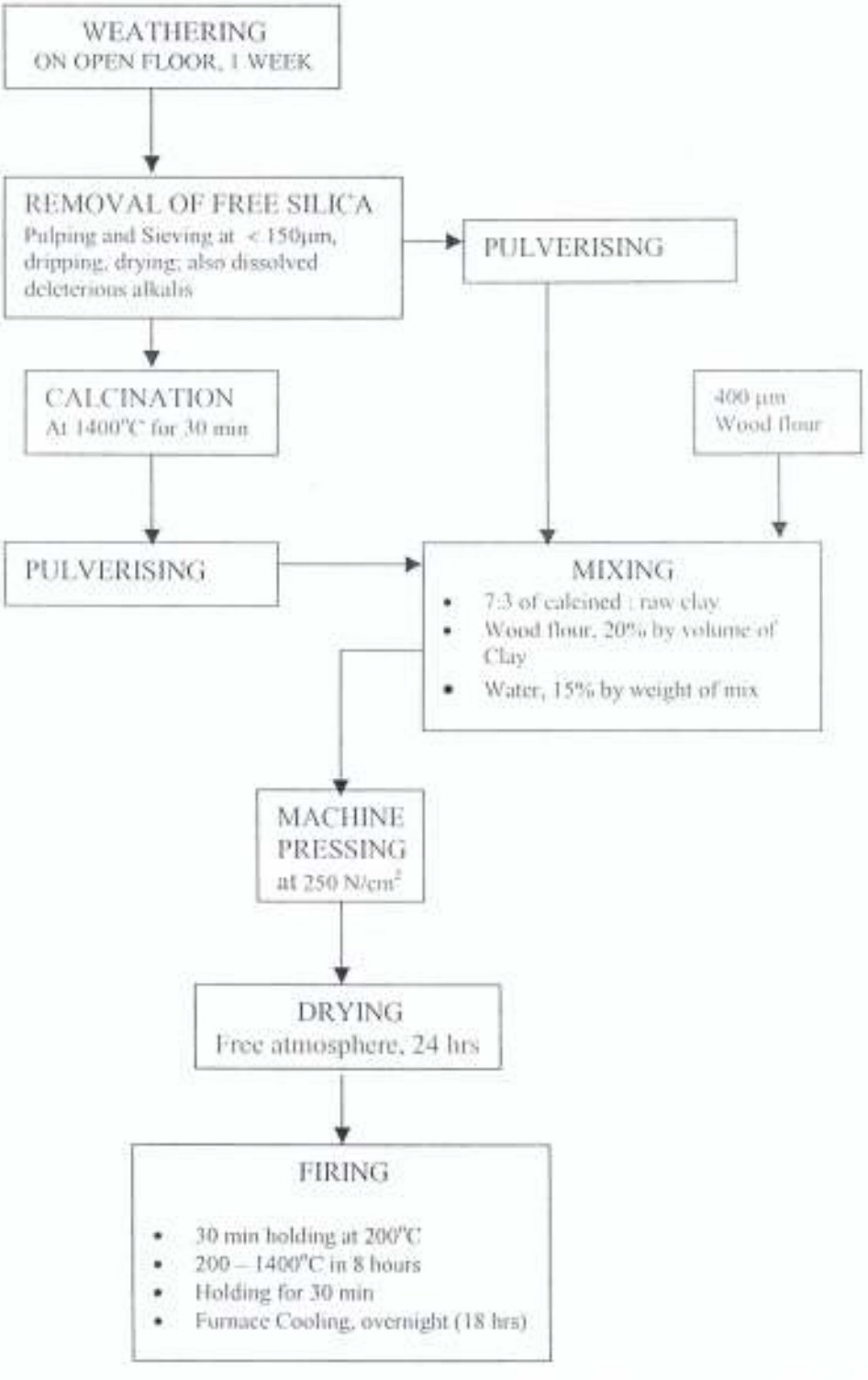


Fig 5.2: Recommended Process Route for Producing Insulating Refractories from Ijapo Kaolinitic Clay

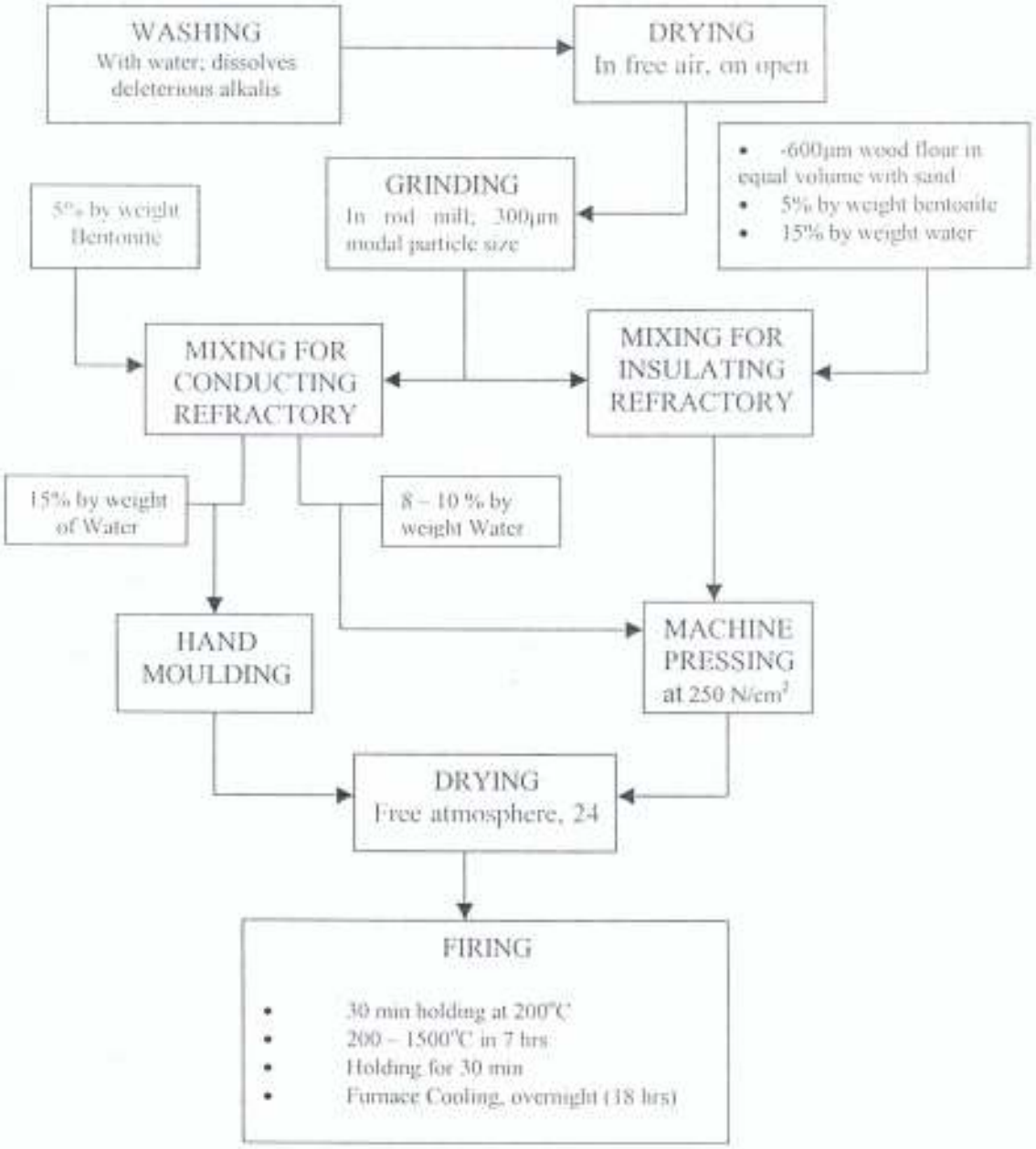


Fig. 5.3: Recommended Process Route for Producing Conducting and Insulating Refractories from Igbokoda Beach Sand

5.3.3 Furnace Performance

From the rate of temperature rise (Table 5.6), it can be inferred that:

i. The chamber material conducts heat adequately into the chamber;

ii. The insulation effectively contains the heat generated so that heat loss rate is low enough to ensure net conduction into the chamber and heat build up therein;

iii. The heat generation rate is adequate to meet the furnace design target of 1200°C in one hour.

About the control system, the temperature off-shoots is a process-sensor time lag effect (Chesmond, 1988), resulting from the fact that the heat generated and the temperature rise by the element take time to be transferred to the chamber where the thermocouple senses it. This implies that during heating, the surface temperature of the element will be higher than that of the chamber. Hence, heat transfer from the element to the chamber still continues for a while after the controller switches off the heating. Since the element's surface temperature must be higher than that of the chamber for heat transfer into chamber, the process-sensor time lag will always exist, but the effect, which is the off-period temperature rise, can be checked. If by a more sensitive control circuitry, the current supply to the heating element during heating is reduced in proportion to the closeness to a set temperature, heating rate will proportionally reduce toward zero at the set temperature. In this manner the off-period temperature rise will be minimal. Also, if during cooling, the control increases the current supply in proportion to the closeness to a set point, heating rate will proportionally increase to a maximum at the low set temperature; temperature drop below this set point will be minimal. With this proportional control, temperature excursions before settling at a set point, and stabilization time will be reduced. This is the type of control in proportional and differential controller (2.6.4). By using such controller instead of the on-off controller, a better control can be achieved.

6.0 CONCLUSIONS AND RECOMMENDATIONS

6.1 CONCLUSIONS

- A prototype electric heating furnace was constructed using refractories produced from local deposits, ohmic heating and electronic temperature monitoring and control. The average temperature build up rate of 20°C per minute was noted on testing the furnace: this is adequate to meet the target of 1200°C in one hour. The operating cost of the furnace is estimated to ₦14.00k in one hour. The furnace also has better operational convenience compared to hydrocsarbon fuel fired types. The prototype is worth improving on.

- The Igbokoda beach silica sand is a good source of refractory raw materials, judging from its large reserve and satisfactory properties. A processing route for producing insulating and conducting refractories using this beach sand deposit was devised. This beach sand does not require precalcination. Also, it is amenable to a quite rapid firing programme, thus reducing production cost.

- A processing route for producing insulating refractories from the Ijapo kaolinitic clay deposit was evolved.

6.2 RECOMMENDATIONS

The on-off temperature controller system suffered from a process-sensor time lag, which causes temperature offshoots before settling at a set temperature. Besides, sufficient refractories for the furnace insulation could not be made using the recommended optimum

process routes due to non-availability of refractory firing kiln at the project base. Hence, the following envisaged further work are recommended:

- Sufficient refractories for the furnace construction should be made following the recommended process routes as reflected in Figs 5.1 and 5.2.
- The On/Off Controller should be substituted with a Proportional and Derivative (PD) controller type for the temperature control system.
- The present design could be used as a basis for development of larger capacity heating furnaces.
- Melting furnace can also be developed with certain modifications in design of the hearth, the feedback transducer, heating element and general architecture of the present work.

REFERENCES

Afolabi, A.S. And Fasuba, O. A. (1997): "Study of the Suitability of the Ijapo Clay for Refractory Application", Undergraduate Thesis, Met. and Matls. Eng'g. Dept., FUT Akure.

Bel, E. C. & Witherland R. W.: Basic Electrical Engineering and Instrumentation for Engineers, 3rd ed., Collins Books, London, 1987.

Borode J. O., Onyemaobi O. O., And Omotoyinbo J. A. (2000): "Suitability of Some Nigeria Clays as Refractory Raw Materials", Nigerian Journal of Engineering Management, Vol. 1, No. 2.

Buduikov, R.: "The Technology of Ceramics and Refractories", Edward Arnord Metals Society London, 1977.

Carbolite Furnaces Ltd.: Carbolite Furnace Model GLM 11/2 Product Manual, Sheffield, 1986.

Chesmond, C. J.: "Control System Technology"; ELBS, 1 ed., 1988.

Chesti, A. R.: "Refractories – Manufacture, Properties and Applications"; Prentice Hall, New Delhi, 1986.

De Bussy, J. H.: "Materials and Technology", Vol. 2, Non-Metallic Ores, Silicates Industries and Solid Minerals Fuels, Longman Group Ltd, 1972, pp 267-290.

Faulker, R. G., Fray, D. J., And Jones, R. D.: "Worked Examples in Mass and Heat Transfer in Material Engineering"; 1st ed.; The Institution of Metallurgist, Carlton House, London, 1985.

Gosser, J.: "Technical Guide to Thermal Processes", English translation, Cambridge University Press, 1986.

Grahl, C. L. (2002): "An Elemental Analysis"; Ceramic Industry Magazine. Feb. 2002. pp 33 – 35.
http://www.ceramicindustry.com/CDA/ArticleInformation/features/BNP__Features__Item/0,2710,71514,00.html

Holman J. P.: "Heat Transfer"; International ed., Mc-Graw Hill Book Company, Singapore. 1992.

Hyndman Industrial Products Inc. (2002): "Resistance Heating Wire Iron-Chromium-Aluminium (Fe-Cr-Al) Alloy – KAPM"; Hyndman Alloy Data Tables. http://www.resistancewire.com/Html/Services.html

Kanthal Industries Handbook: "Resistance Heating Element for Industrial Furnaces". Kanthal Ltd., Ljungforetagen, Orebor Publishers, Sweden. 1987.

Krivandin, V And Markov B: "Metallurgical Furnaces"; 1st ed. Mir Publishers, Moscow. 1980.

Majumdar, S., Singh, A. K., Basu, B. And Sekhar, J. A. (2002): "Heating Elements for Higher Productivity"; Ceramics Ind., May 2002, pp 18 – 22.
http://www.ceramicindustry.com/CDA/ArticleInformation/features/BNP__Features__Item/0,2710,71514,00.html

Omowumi O. J. (2001):
"Characterization of Some Nigerian Clays as Refractory Material for Furnace Lining", Nigerian Journal of Engineering Management, Vol. 2, No.3, pp. 1.

Oshodin T. E., Olunlade B. A., Odo E. A., Olasupo O. A., Azeez O., Olaniyan O.O., Idowu A.S., And Adewoye O. O. (2002): "Design and Optimisation of a 100 kg Rotary Furnace"; NIMACON 2002 Abstracts, pg 46.

Protherm Electrical Furnaces Alser Teknik Co. Inc., Ankara Turkey. www.prothermfurnaces.com

The Presidency, Office Of The Honourable Minister, Economic Matters. "Obasanjo's Economic Direction, 1999 - 2003", FGN Press, 1999, Section 2.2, pg. 8.

Science Projects – 817-453-5555: New Jesey Ind. Research Center, Clifton, New Jersey. Sales Site: www.scienceproject.com

Zircar Ceramics Inc., 2001: "Molybdenum Disilicide Heating Element Type MOSI2"; Zircar Ceramics, Florida, N.Y. 10921 – 0519. http://www.zircar.com/pages/elements/index/.htm

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nce Heating Wire Iron-Chrome-Aluminum (Fe-Cr-Al) Alloy - KAPM

$m^2/ = \frac{I^2 C_t}{p}$

I = Current
C_t = Temperature factor
p = Surface load W/in

Names: KAPM, Alloy 875 PM, Kanthal APM

es: Typical applications are in furnaces for firing of high temperature ceramics, heat treatment furnaces, laboratory furnaces, diffusion furnaces, and high temperature kilns. Produced from powder metallurgy technology this alloy has demonstrated significant improvements in both physical and mechanical properties. Its advantages are better form stability, high temperature strength, and the ability to be self-supporting at temperatures above 1300° C. It has an excellent surface oxide, which gives good protection in corrosive atmospheres as well as in atmospheres with high carbon potential, and no scaling.

sition

Ni	Cr	Fe	Al	Si	Mn	Cu	C	Ti	Mo	W
None/Trace	22%	Balance	5.8%	None/Trace	None/Trace	None/Trace	None/Trace	None/Trace	None/Trace	None/Trace

nical Data

Resistivity (/cmf)	872	Resistivity (/sqmf)	684
Resistivity (/cm)	147.47	Nom. Temp. Coeff. of Resistance (TCR)	0.00002
Std. Res. Tol. <.020"	3%	Std. Res. Tol. >.020"	5%
Thermal EMF vs. Cu	-6.4	Specific Heat (20°C)	0.11 cal/g
Density (g/cm)	7.10	Density (lb/in)	0.256
Thermal Conductivity	0.187 W/cm/°C	Coeff. of Linear Expansion (X 10)	15.00 in/in/°C
Approx. Melting Point	1500°C	Max. Continuous Operating Temp.	1400°C
UTS – Hard (KPSI)	200	YTS Tensile – Hard (KPSI)	
UTS – Stress Relieved (KPSI)	175	YTS Tensile – Stress Relieved (KPSI)	
UTS – Annealed (KPSI)	115	YTS Tensile – Annealed (KPSI)	
Magnetic Attraction	Strong	Emissivity – fully oxidized	0.70
Designations/Specifications	ASTM = B603	Forms Available	Wire, Ribbon, Insul.

perature Factor – To obtain resistance at working temperature multiply by the factor C_t in the following table:

	20	100	200	300	400	500	600	700	800	900	1000	1100	1200	1300	1400
C_t	1.00	1.00	1.00	1.00	1.00	1.01	1.02	1.02	1.03	1.03	1.04	1.04	1.04	1.04	1.05

y Data

Diameter mm	Resistance at 20° C /m	Resistance at 20° C /kg	Weight kg/1000 m	Surface area cm /m	cm /m at 20°C
10.0000	0.0185	0.0332	556.5375	314.1593	17020.8372
9.5000	0.0205	0.0407	502.2751	298.4513	14593.2403
8.2500	0.0271	0.0716	378.7933	259.1814	9557.4660
8.0000	0.0288	0.0810	356.1840	251.3274	8714.6686
7.3500	0.0342	0.1136	300.6555	230.9071	6758.3851
7.0000	0.0377	0.1381	272.7034	219.9115	5838.1472
6.5000	0.0437	0.1858	235.1371	204.2035	4674.3474
6.0000	0.0513	0.2559	200.3535	188.4956	3676.5008
5.5000	0.0610	0.3624	168.3526	172.7876	2831.8418
5.0000	0.0738	0.5306	139.1344	157.0796	2127.6046
4.7500	0.0818	0.6515	125.5688	149.2257	1824.1550
4.5000	0.0911	0.8088	112.6988	141.3717	1551.0238
4.2500	0.1022	1.0165	100.5246	133.5177	1306.6152
4.0000	0.1154	1.2955	89.0460	125.6637	1089.3336
3.7500	0.1313	1.6771	78.2631	117.8097	897.5832
3.5000	0.1507	2.2101	68.1758	109.9557	729.7684
3.2500	0.1747	2.9726	58.7843	102.1018	584.2934
3.0000	0.2051	4.0944	50.0884	94.2478	459.5626
2.8000	0.2354	5.3956	43.6329	87.9646	373.6414

Diameter mm	Resistance at 20° C Ω/m	Resistance at 20° C Ω/kg	Weight kg/1000 m	Surface area cm²/m	Ω/m at 20°C
2.6000	0.2730	7.2574	37.6219	81.6814	299.1582
2.5000	0.2953	8.4901	34.7836	78.5398	265.9506
2.3000	0.3489	11.8512	29.4408	72.2566	207.0925
2.2000	0.3813	14.1574	26.9364	69.1150	181.2379
2.0000	0.4614	20.7279	22.2615	62.8319	136.1667
1.7000	0.6387	39.7081	16.0839	53.4071	83.6234
1.5000	0.8203	65.5103	12.5221	47.1239	57.4453
1.2000	1.2818	159.9372	8.0141	37.6991	29.4120
1.0000	1.8457	331.6458	5.5654	31.4159	17.0208

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APPENDIX II

COMBINED DETERMINATION OF SILICA AND ALUMINA

1. Weigh exactly 1g, of the sample in a Platinum crucible.
2. Mix thoroughly with mixture of $Na_2CO_3 + K_2CO_3$ (1:1) and cover up with some of the fusion mixture.
3. Fuse at a temperature of 900 – 950°C in a muffle furnace for 15 to 20 minutes.
4. Cool and extract the fused mass into a 400ml beaker with 1: 1 HCl 50ml. Dehydrate and bake.
5. Cool, add 10ml of concentrated HCl and add hot distilled water up to 150ml in a beaker. Allow to boil.
6. Filter through No. 41 filter paper. Wash with (1:4) HCl 2-3 times, and with hot water several times until the residue is free from HCl. Keep the filterate for the Al_2O_3 determination.

Silica Determination

7. Ignite the residue in the Platinum Crucible at 950°C to constant weight, cool in the desiccator and weigh (W_1).
8. Add 5-8 drops of 1:1 H_2SO_4 to the residue and then 10 – 15ml HF. Evaporate slowly over a low heat (at the end, copious fumes are seen leaving in a dry mass).
9. Ignite the residue in a muffle furnace to a constant weight cool in desiccator and weigh the crucible (W_2).

$$\% SiO_2 = \frac{(W_1 - W_2 \times 100)}{\text{Wt. of the Sample}}$$

where W_1 = wt. of the Platinum Crucible with the material before hydrofluorisation.

W_2 = wt. of the Platinum Crucible after hydrofluorisation.

Alumina Determination

10. Take the filtrate from Silica determination in a 250ml. Volumetric flask and make up to the volume and mix well (if the filtrate is bulky, reduce the volume by boiling).
11. Take 100ml of the main solution in a beaker, add nearly 2gm, of Ammonium Chloride add few drops of methyl add indicator and neutralize with 1:1 ammonium solution till a yellow colour persists. Avoid excess (a faint smell of ammonium should persist).
12. Boil to coagulate the precipitate and filter through No. 41 filter paper. Wash the residue with hot 0.1% Ammonium nitrate solution till free from Chloride ions - (Test the washings with silver nitrate and nitric acids).
13. Take the residue along with the filtrate paper into a weighted platinum crucible, burn off the filter paper at a low temperature and finally ignite the residue at

($1100^{O}C$ for one hour. Cool in a desiccator and take the weight. Find out the weight of R_2O_3 (W).

$$R_2O_3\% = \frac{W \times 250}{\text{wt. of Sample}}$$

$$Al_2O_3\% = R_2O_3\% - (Fe_2O_3 + P_2O_5 + TiO_2)$$

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LETTER REF. NO. NMDC/TECH/10/762

DATE 30th Nov., 2002

CERTIFICATE OF ANALYSIS

1. NAME:- Ogunniyi I.O.,
Department of Metallurgical and Materials Engineering,
Federal University of Technology, Akure.

2. JOB:- (a) Refractories Analysis
(b) Chemical Analysis

3. TEST RESULT:-

(a) Refractories Analysis

S/NO	NAME OF SAMPLE	BULK DENSITY (g/cm^3)	% POROSITY
1.	Clay	1.38	50.15
2.	Silica	1.77	28.18

(b) Chemical Analysis

	FeO	Fe_2O_3	Al_2O_3	SiO_2	CaO	MgO	LOI
Ijapo Clay	3.23	3.58	28.83	52.2	ND	ND	11.22
Igbokoda Sand	1.07	1.19	1.13	92.54	ND	ND	0.05

O. O. Popoola

NAME/SIGNATURE OF VERIFYING OFFICER

APPENDIX IV: DIAGRAM TO SHOW THE CIRCUITRY OF THE FINAL CONTROL ELEMENT