The Influence of Fire on the Design of Polymer Composite Pipes and Panels for Offshore Structures

Volume 2 of 2

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Also the author wishes to express his thanks to those members of the technical staff of the Department of Civil Engineering and Construction for their assistance in the preparation of experimental work.

DECLARATION

None of the material contained in this thesis has been submitted in support of an application for another degree or qualification of this or any other university or institution of learning.

David W Dewhurst

March 1997

FOREWORD

The following text contains detailed descriptions of the methodologies used and experiments performed in the development of a new, non-fibrous, highly efficient furnace lining.

The work presented here was performed as part of a multi sponsor research project in conjunction with the Energy Efficiency Office, Department of the Environment. This research is part of an ongoing project (March 1997), and should be viewed as being commercially sensitive.

David W Dewhurst

March 1997

APPENDIX A - DEVELOPMENT OF A NEW FURNACE LINING MATERIAL

A.1 Introduction

The following chapters detail the development of a new non-fibrous refractory lining material, Insuline. This lining material has been developed with the aim of replacing ceramic fibre without loss of thermal efficiency. Insuline, due to its' constituent materials is inherently free from fibre-related health hazards, and also provides an energy efficient replacement for insulating fire bricks.

Ceramic fibres have been available for high temperature applications for the last 25 years. These fibres are alumino-silicate fibres made from either naturally occurring high purity clays or from direct combination of alumina and silica[1]. Typical methods of manufacture are from melting the appropriate raw materials in a suitable furnace and either subjecting a molten stream to a blast of air, or allowing it to fall onto a spinning disk. The operating temperature of the fibres can be controlled by the percentage of alumina (the standard 1250°C fibre is approximately 50% alumina, and 94-96% alumina fibres would have an operating temperature of approximately 1600°C). The advantages of using ceramic fibres for furnace linings are that they have very low thermal conductivities and also low heat capacity. This makes them suitable for use in intermittent heating situations. Ceramic fibre installations also have a very high resistance to thermal shock related problems. Recently however the health implications of their use have been considered in detail.

A.2 Health and safety implications

Currently all fibrous materials are being viewed as potential health hazards. It is understood that, with ceramic fibres, crystobalite may be formed in certain circumstances. It is further believed that the exposure of the fibres to high temperature firing may embrittle the fibre tips causing the detachment of fibrous pieces of a respirable length. The current repercussions with regards to exposure to asbestos fibres are naturally making people more considerate of fibre-related health problems. Some countries have begun steps towards the outlawing of ceramic fibres and, due to this, a recent study in America was presented to the United States Environmental Protection Agency which considered the cost impacts of this. The conclusions of the report were that the replacement of ceramic fibres with substitute materials would reduce energy efficiency, increase maintenance costs, increase capital costs (for the rebuilding of furnaces, including the need for an increased number of furnaces due to foreseen reductions in throughput). Also in some applications it is foreseen that product quality would also be reduced. The estimated increased energy consumption in the United States for the replacement of ceramic fibre linings was 48 billion kilowatt-hours annually.

It is noted also that ceramic fibres are not the only high temperature fibres which present possible health hazards. Conversely, non-fibrous linings such as refractory concrete and firebricks have a long history of use. They are typically denser, thicker and more expensive than the comparable ceramic

linings, however there is no apparent health hazard for the use of these materials. There is, therefore, a substantial incentive to develop an alternative material which may replace ceramic fibres without the potential health hazard of fibrous materials. Candidate materials make extensive use of refractory cements.

A.3 High temperature applications of cement based materials

Refractory cements are high purity high alumina cements. Typical alumina (Al_2O_3) contents are of 50% or above, with up to 40% calcium oxide (CaO) and also less than 10% silicon dioxide (SiO_2). The cements are rapid hardening with final set times generally less than 4½ hours and the majority of the final strength attained within 24 hours from casting. Refractory cements are typically pale in colour, ranging from pale grey or cream to white. The paler the colour, generally the higher the alumina content with a corresponding increase in melt/working temperature.

The curing/firing process phase changes, and indeed strength effects are complex, and as such the mineralogical changes will not be reported in any great detail. The aim of this chapter is to give a brief overview of the physical effects of the curing and firing processes on the new furnace lining material. As there is no data available for a composition such as the new lining material data for cements and refractory concretes have been reported.

A.3.1 The curing process

Hydraulic cements develop their hydraulic bonds because of chemical reactions between the cement (calcium aluminate) and water. The mixing and curing temperature can affect the types of hydrates formed in the set cement or concrete.

Set high alumina cements cured at room temperature for long durations consist of unhydrated material, CAH_{10} and some alumina gel[2]. The metastable CAH_{10} experiences a slow reaction at room temperature in the formation of C_3AH_6 and gibbite. This reaction is very rapid at temperatures above 80°C providing sufficient moisture is present. In the presence of sufficient water this reaction may occur during "drying" even at temperatures above 110°C[3]. If this conversion reaction is allowed to take place a diminuation in strength may be observed[2]. Midgley[2] reported a diminuation in compressive strength of high alumina cements cured at room temperature for 6 months of 24,000psi (165N/mm^2) to 11,000psi (76N/mm^2) after drying the samples at 110°C.

Many people[4,5,6] have investigated the effect of curing temperature of a refractory concrete. Despite these investigations the effect of curing temperature upon strength is not established conclusively. Givan et al.[4] studied the effect of curing temperature upon the flexural strength of refractory concrete (caftab 20) after curing, after drying at 110°C, and after firing to 1100°C. **Figure A.3.1** following shows a summary of the results in graphical form.

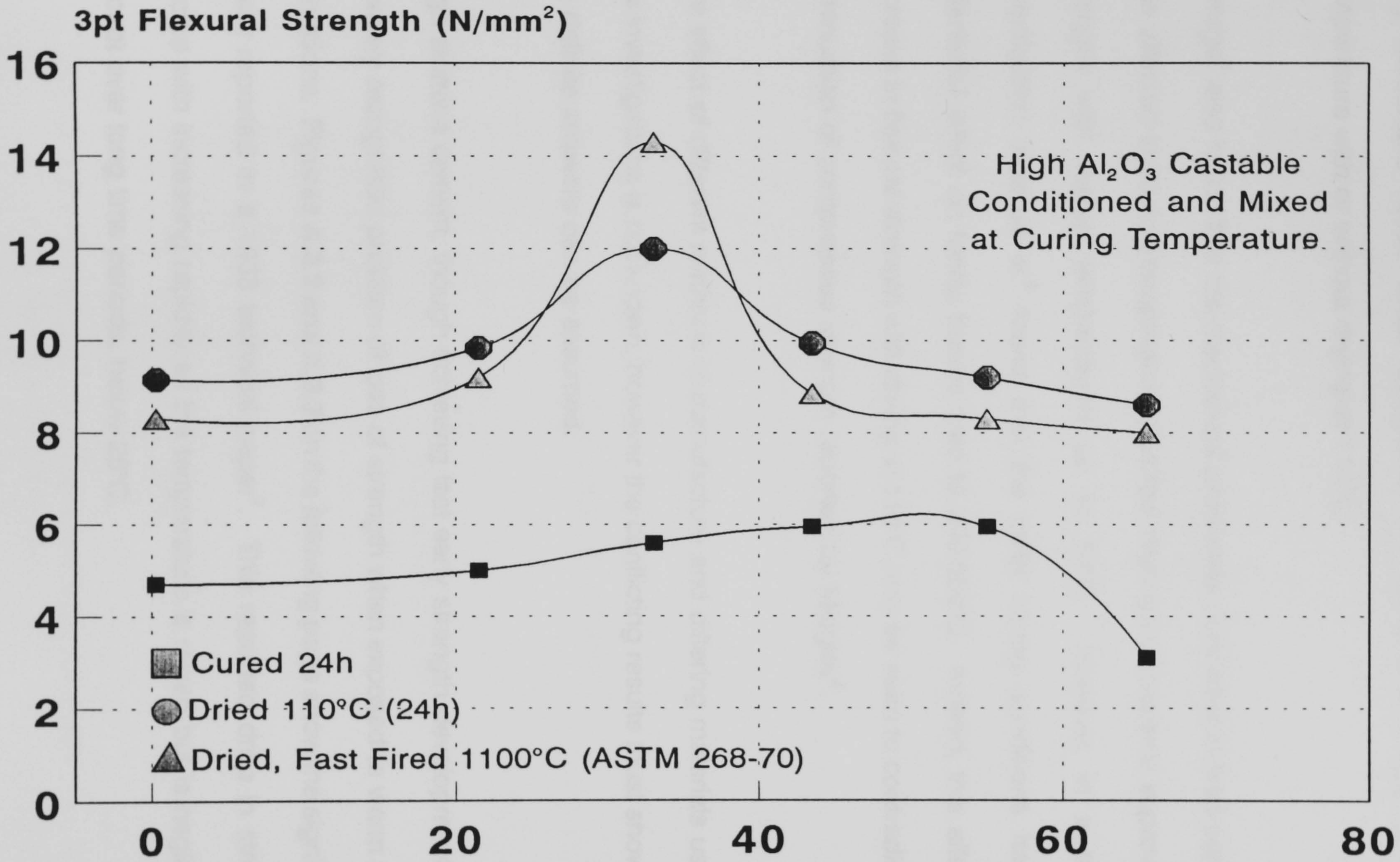


Figure A.3.1 - Flexural Strength of Tabular Alumina High Purity Cement Castable (ASTM C268)

It can be seen that mixing and curing at 30-35°C and drying at 110°C gives a very favourable result when compared to mixing and curing at room temperature with or without drying at 110°C.

George[5] also found that the mechanical properties of dried and fired cements was affected by curing temperature, but that high purity cements improved in strength with curing temperatures up to 54°C. However in a similar investigation Kula et al.[6] found that the initial curing conditions had no differential effect on fondu pastes fired to 300-500°C. Indeed, this effect of increase in flexural strength with drying at 110°C may be seen to contradict the diminuation of compressive strength reported by Midgley[2].

The effect of different methods of manufacture and differing materials used in the investigations is not known, however the conflicting results must show that no definite answers can be assumed.

High alumina cement, though achieving fast early strength development, has a widely recognised problem of loss of strength when exposed to warm moist conditions. **Figures A.3.2 and A.3.3** on the following page show the significant effect reported in a 1933 technical paper[7]. This reported drop in strength occurs with increasing rapidity as the temperature is raised but is negligible, except over long time periods, below 25°C.

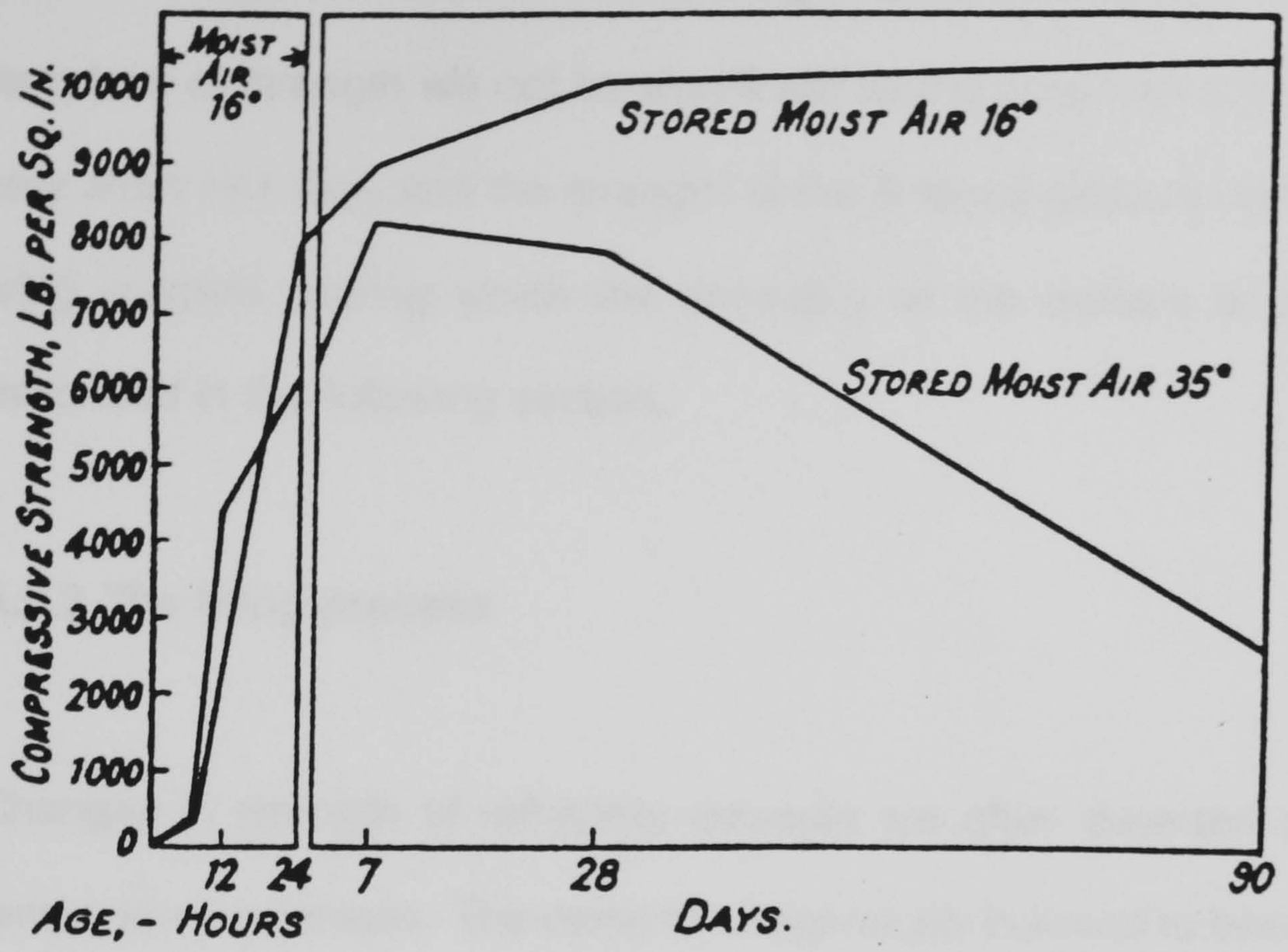


Figure A.3.2 Effect of Air Storage Conditions on Strength of High Alumina Cements

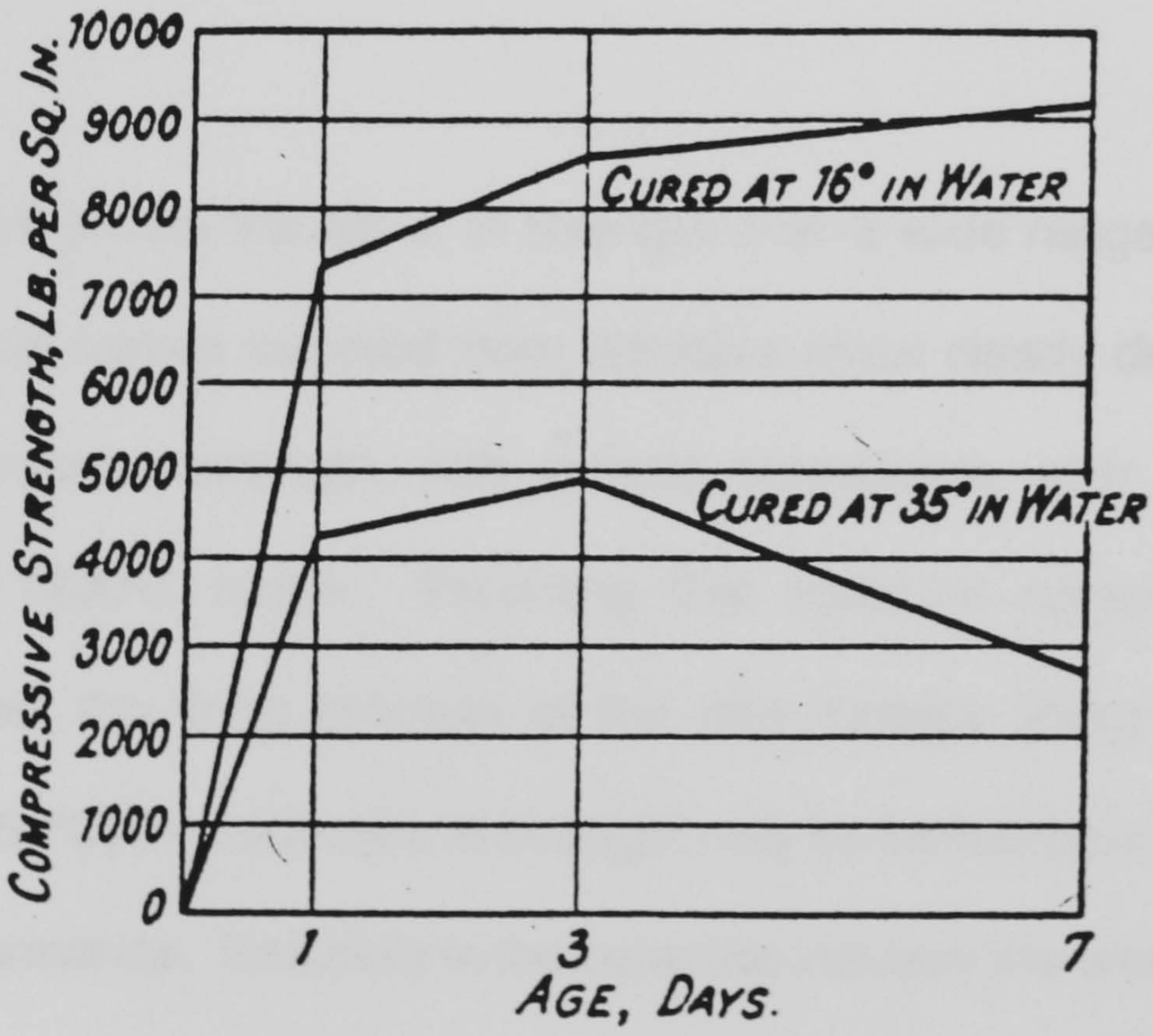


Figure A.3.3 Effect of Curing Conditions on Strength of High Alumina Cement

Due to the nature of the new furnace lining material it is envisaged that this long term loss of strength will not be significant as the cure time of the material is very short (<1 day) and the strength of the finished product comes from the firing process (during which the chemistry of the cement is changed) as described in the following section.

A.3.2 The firing process

Changes in strength of refractory cements are often detected as the firing temperature increases. The cements are generally believed to become weaker between 100°C and 900°C, during the dehydration of the hydraulic bonds, but recover their strength at higher temperatures when "ceramic" bonds begin to form[3].

Figure A.3.4 shows the fall of in strength over a wide range of temperature. These typical curves selected from literature show clearly defined minima of cold compressive strength, with various aggregates, after pre-firing in the 1000°C to 1200°C range. Providing that sufficient compressive strength remains after the firing process of the new furnace lining material (at the moment 1150°C) the reduction in strength may be beneficial in terms of thermal shock performance. Generally in the ceramics industry it is well recognised that by keeping the microstructure poorly bonded a high degree of thermal shock resistance may be achieved.

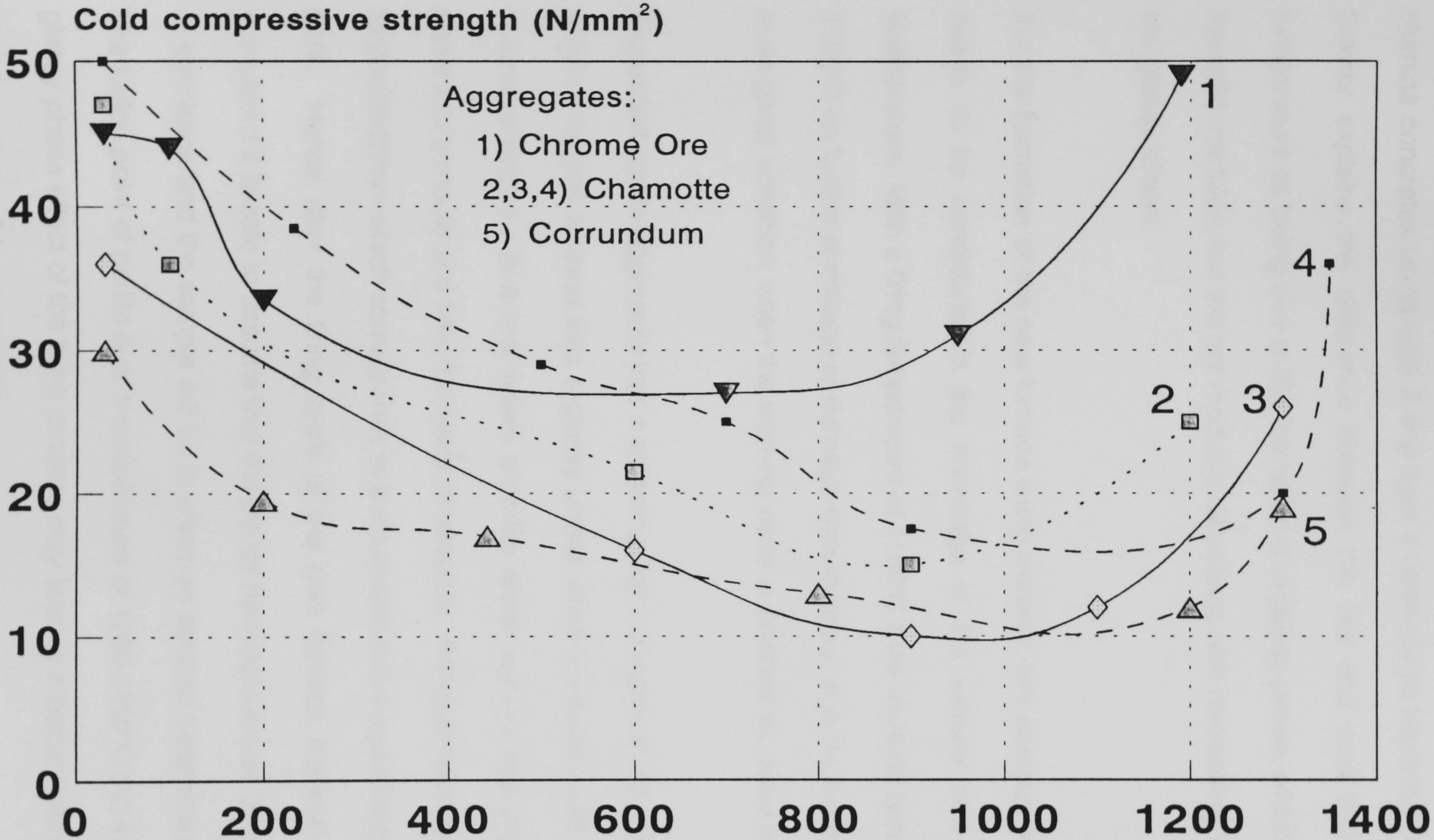


Figure A.3.4 - Variation with temperature of pre-heat of the cold compressive strength of aluminous concretes with various aggregates.

Figures A.3.5 and A.3.6 show the hot and cold moduli of rupture of 40-50% alumina concretes using type 3 and type 4 cements as reported by Bakker[8]. Bakker explains the difference between the hot and cold MOR at high temperature as being due to the formation of a glassy phase, which increases the cold modulus, but the hot modulus decreases with decreasing viscosity of the glassy phase.

For the formation of the new furnace lining material one obvious effect which needs to be considered is the shrinkage of the sample with increased temperature. With a firing temperature of 1150°C if the working temperature is 1100°C no further shrinkage or damage should occur, and the brick should be in as good condition when the working cycle is finished as when it started.

Expanded perlite aggregate has a softening point of approximately 870°C after which it begins to pass into a glassy phase which coincides with very large volume changes. In a test where a perlite alone sample was pressed with water into a mould and fired the linear dimensions changed from 54x52x51mm to 26x26x20mm which corresponds to a volumetric shrinkage of approximately 90%. Hence after the firing cycle of the new furnace material has been completed it is safe to assume that the perlite has experienced a high degree of shrinkage and the sample will be in effect an aerated ceramicised cement. The fusion point of perlite is at a temperature of 1280-1350°C but even so, the glassy phase effect of the firing process may lead to a better finished sample than for a lower firing temperature.

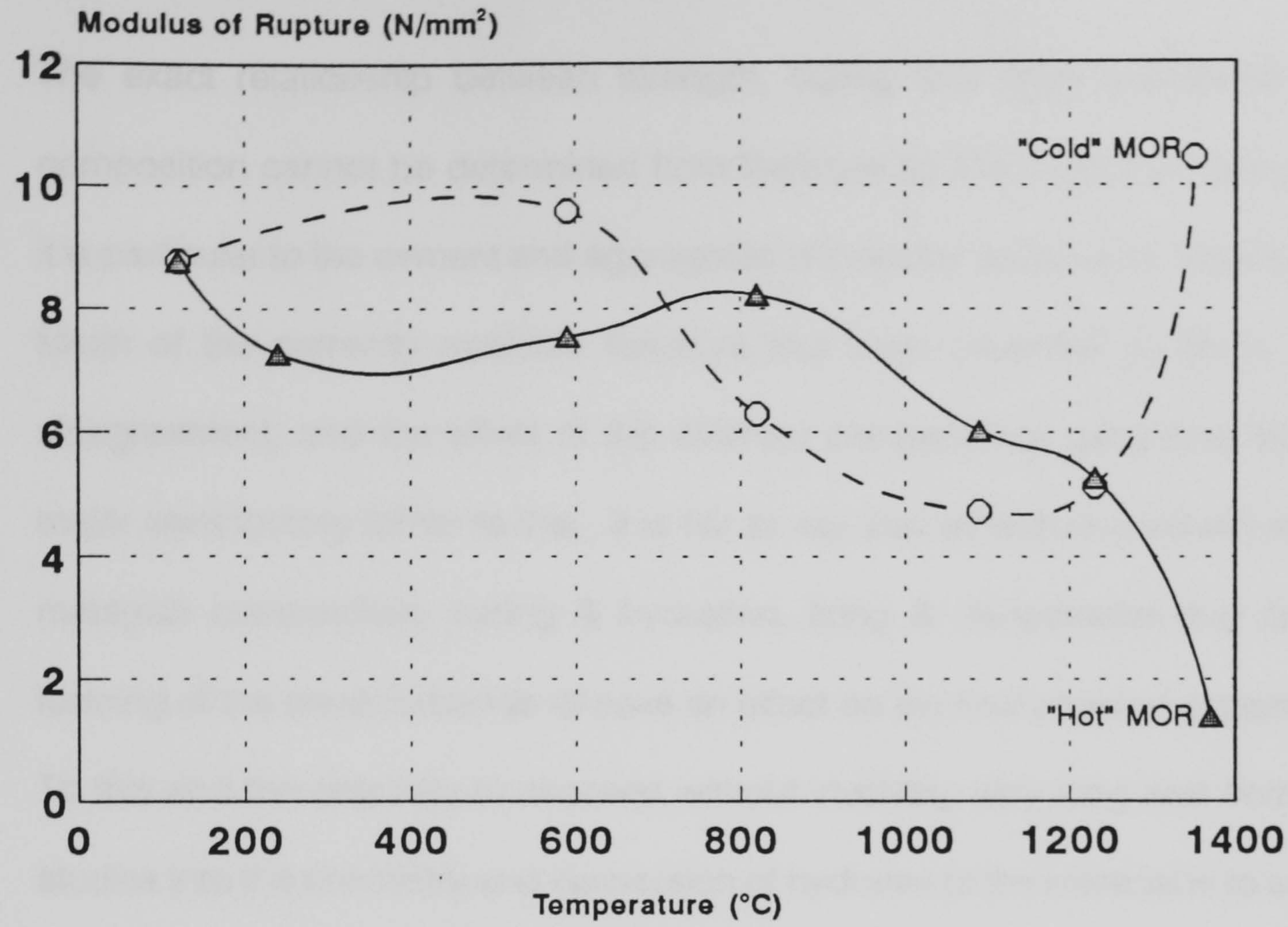


Figure A.3.5 - 45% Al_2O_3 - 55% type 3 refractory cement
Hot and Cold Moduli of Rupture against temperature

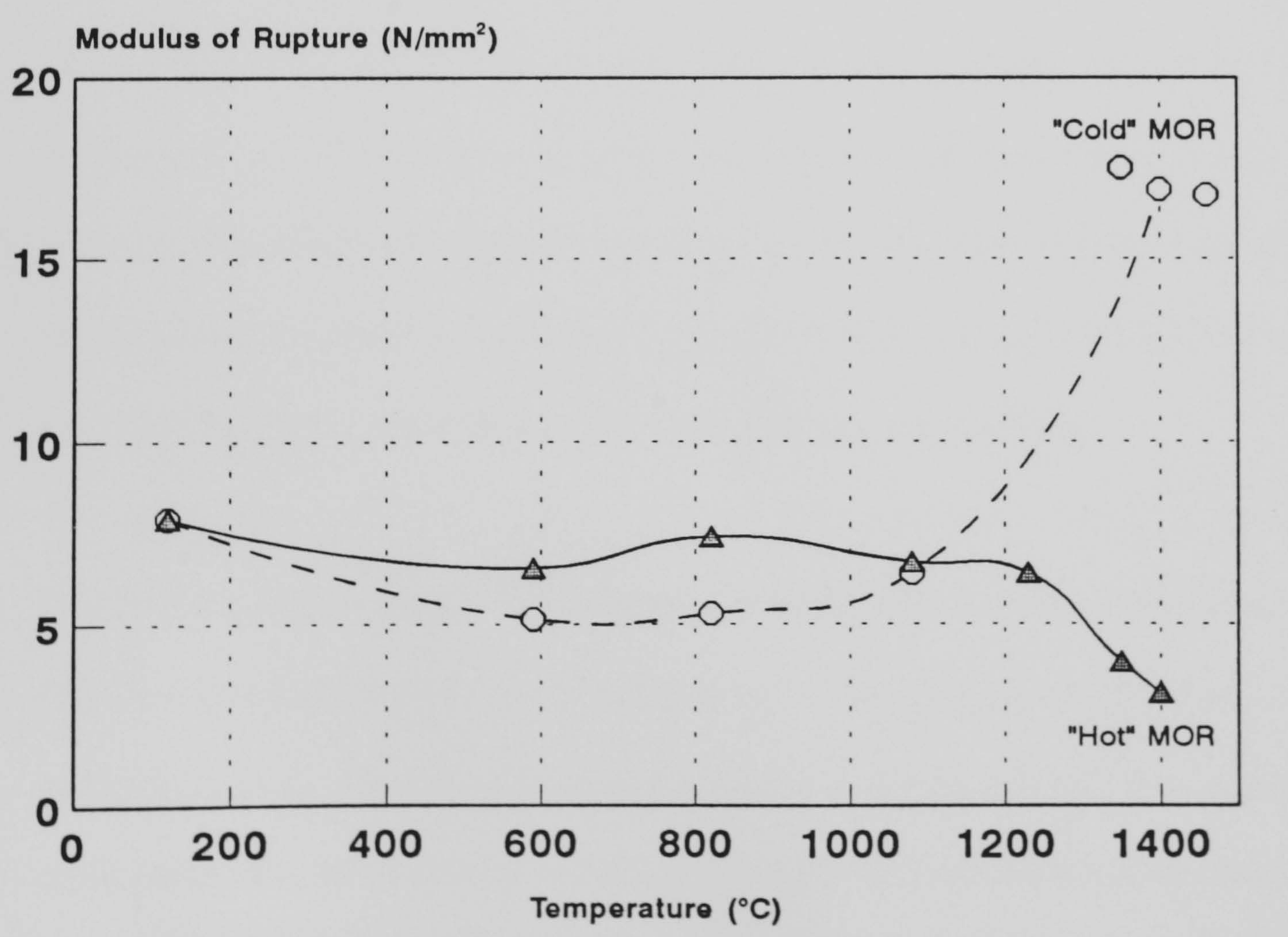


Figure A.3.6 - 50% Al_2O_3 - 50% type 4 refractory cement
Hot and Cold Moduli of Rupture against temperature

The exact relationship between strength, curing and firing conditions and composition cannot be determined from literature as it is more than likely that it is particular to the cement and aggregates chosen for a particular application. Much of the currently available literature has been reported[3] to be in wide disagreement, and the effect of the different compositions used may be the major contributory factor to this. It is fair to say that all factors involved with a materials composition, curing & hydration, firing & dehydration and hence forming of the ceramic bonds all have an effect on the final physical properties. To this end the only way to proceed without involving very long and complex studies into the chemistry and conversion of hydrates of the material is to adopt the long standing research method of "trial and error".

A.4 A new approach to furnace linings.

The objective of the experimental programme was to develop a modular system for furnace linings which had beneficial properties when compared to traditional lining materials such as refractory concrete, fire bricks and ceramic wool. For the purpose of research the manufactured units were brick shaped for ease of production and use. It was proposed that the brick developed would show energy savings at the same thickness as ceramic wool and be inherently free from fibre related problems. The majority of fire bricks are medium density and formed from a fired mixture of fire clay and crushed fire brick as an aggregate. The use of crushed fire brick as an aggregate helps minimise shrinkage during the firing process. Refractory concretes are high density castables which are, as a rule, fired in-situ during the first run of the furnace. The major disadvantages of both these materials with respect to ceramic fibre systems are both their thermal conductivities and density. The new material developed would have the advantages of being non-fibrous and easy to install, but would be relatively light weight and have a low thermal conductivity.

Due to the excellent performance of the cement-perlite panels manufactured for fire testing within the research programme it was decided that this type of composite may provide an excellent furnace lining material if it could be developed with other criteria in mind. In particular the new material should be able to withstand high temperatures without fracturing, distorting or disintegrating. Four cements produced by Lafarge Specialist Cements[9] were

investigated . These were ciment fondu, Secar 51, Secar 71 and Secar 80. The pyrometric cone equivalent (melting point) temperatures of neat cement pastes of these cements are 1270°C, 1440°C, 1680°C and 1750°C respectively. It was anticipated that Secar 51 would be the most suitable material with a maximum continuous operating temperature of about 1350°C.

A.4.1 Initial research

The new material to be developed will be referred to as **Insuline** for the remainder of this appendix. Upon further investigation into the capabilities of the cements it was decided that although Ciment Fondu is the cheapest of the cements, and cost being an important consideration (with respect to capital costs of re-lining a furnace), its iron content would render it unsuitable for reducing atmospheres. It was hence decided that the prime material for investigation should be based around Secar 51, although the advantages of using higher grade cements would also be investigated.

Compositions made from similar raw materials as Insuline have been investigated in great detail as a fire resistant core for sandwich panels. In that particular use (Voidfill) the materials were prone to crazing during the fire test (indicating shrinkage of the sample) and were relatively brittle with low bending strengths. These limitations in strength do not restrict the use of similar compositions for furnace linings. The indicated shrinkage of the materials on exposure to high temperatures would restrict the use of the compositions. It

was first important to devise a method of manufacture which would produce samples free from flaws and defects which would not crack on firing. It was envisaged that the perlite content of the bricks would shrink to a small glassy bead during firing leaving a very finely aerated cement-ceramic body.

Preliminary investigations were made on brick sized samples (≈220 x 110 x 70mm). The materials for the initial specimens were Tilcon perlite pack 3, cements Secar 51, 71 and 80, and water. Pack 3 perlite was a non-graded material typically used for non-demanding applications such as filling timber floor voids. The samples were mixed with care by turning in a polythene bag before mechanical mixing with water to form the pressable material. The mechanical mixing time was kept to a minimum to retain as much volume of material as possible. The brick shaped samples were compressed in a mould to the finished size, although a small degree of "recovery" was noticed when the bricks were released from the mould.

The first samples to be manufactured (mix A) were made to the following mix proportions:

Perlite (729g), Cement (594g), Water (954g)

giving an aggregate-cement ratio of 1.23, and a water-cement ratio of 1.61. The samples were then placed in polythene bags and cured at high humidity for 5 days after which they were air dried for three weeks. The samples were then dried at 110°C in a fan assisted oven to constant temperature. A programmable electric kiln was used for the firing process. The kiln

temperature was programmed to climb at 60°C per hour to a temperature of 1150°C, followed by a dwell of 10 hours, and finally natural cooling to room temperature. The total firing regime is shown in **figure A.4.1** below.

The samples were mounted to a furnace door as shown in **figure A.4.2** and the test results are shown in **figure A.4.3 and A.4.4**. The results from this test were used in a finite difference numerical analysis program was used to predict that the cold face temperatures at 210 and 250mm thick would be 70.6 and 64.2°C respectively. These predictions were very encouraging for the possible use of Insuline as a furnace lining material. Crude measurements taken before and after the firing process indicated that a linear shrinkage of 4-6% could be expected. After the samples were tested they were removed from the furnace door and exposed to thermal cycling between 1100°C and 300°C. The face temperature was monitored with a contact thermocouple. Twenty five heating and cooling cycles were applied, with a typical heating time of six seconds, and a cooling time of twenty four seconds. In the test series there was no apparent damage to the bricks indicating that the thermal shock characteristics were good.

Subsequent samples manufactured by a similar manner (but no air-drying) showed signs of drying shrinkage with very faint cracks being observed on the bricks surfaces. The firing process opened up these cracks to the point where the bricks were rendered useless. Several samples were marked with gauge points and measured carefully during the whole of the manufacturing period.

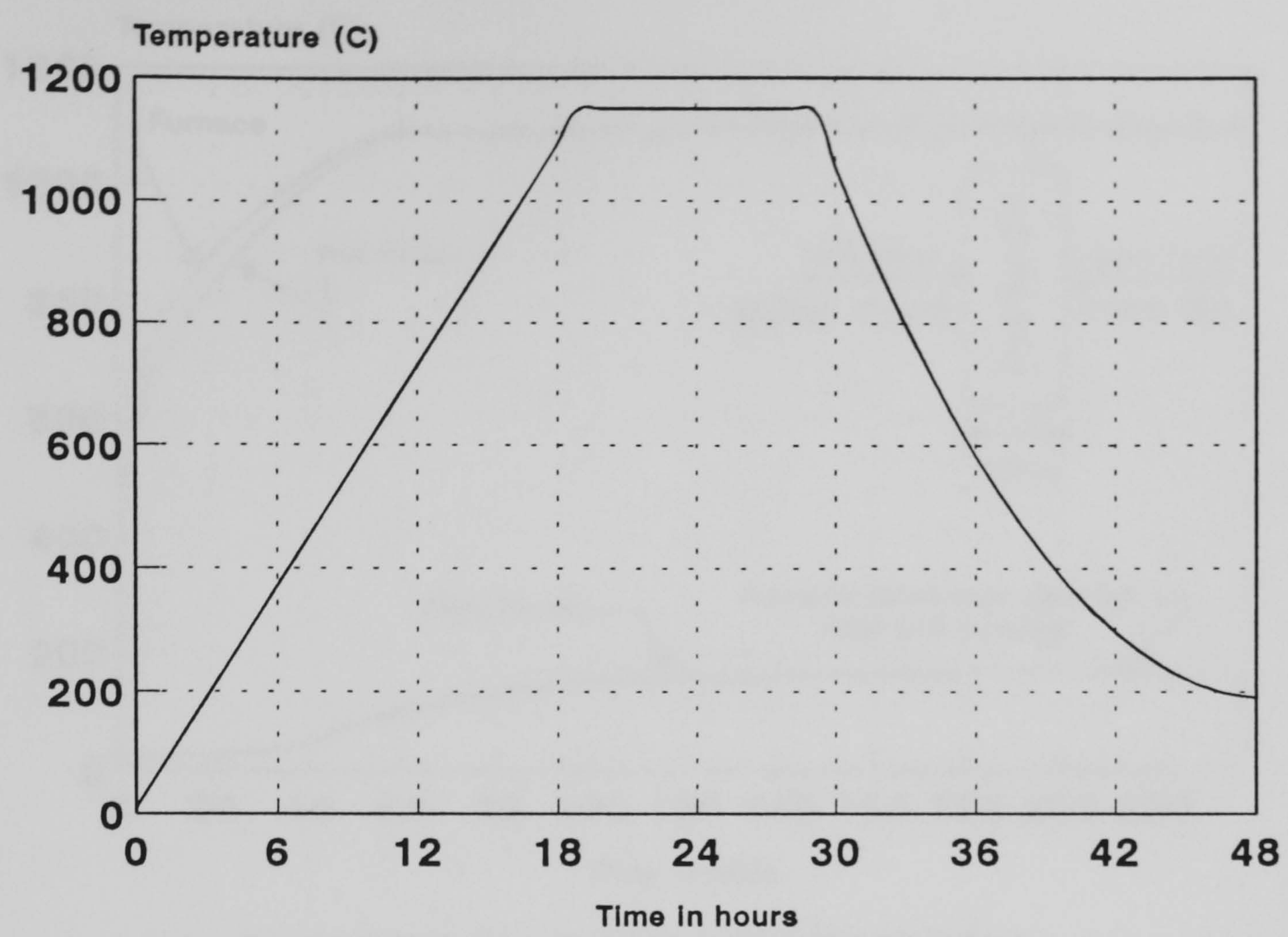
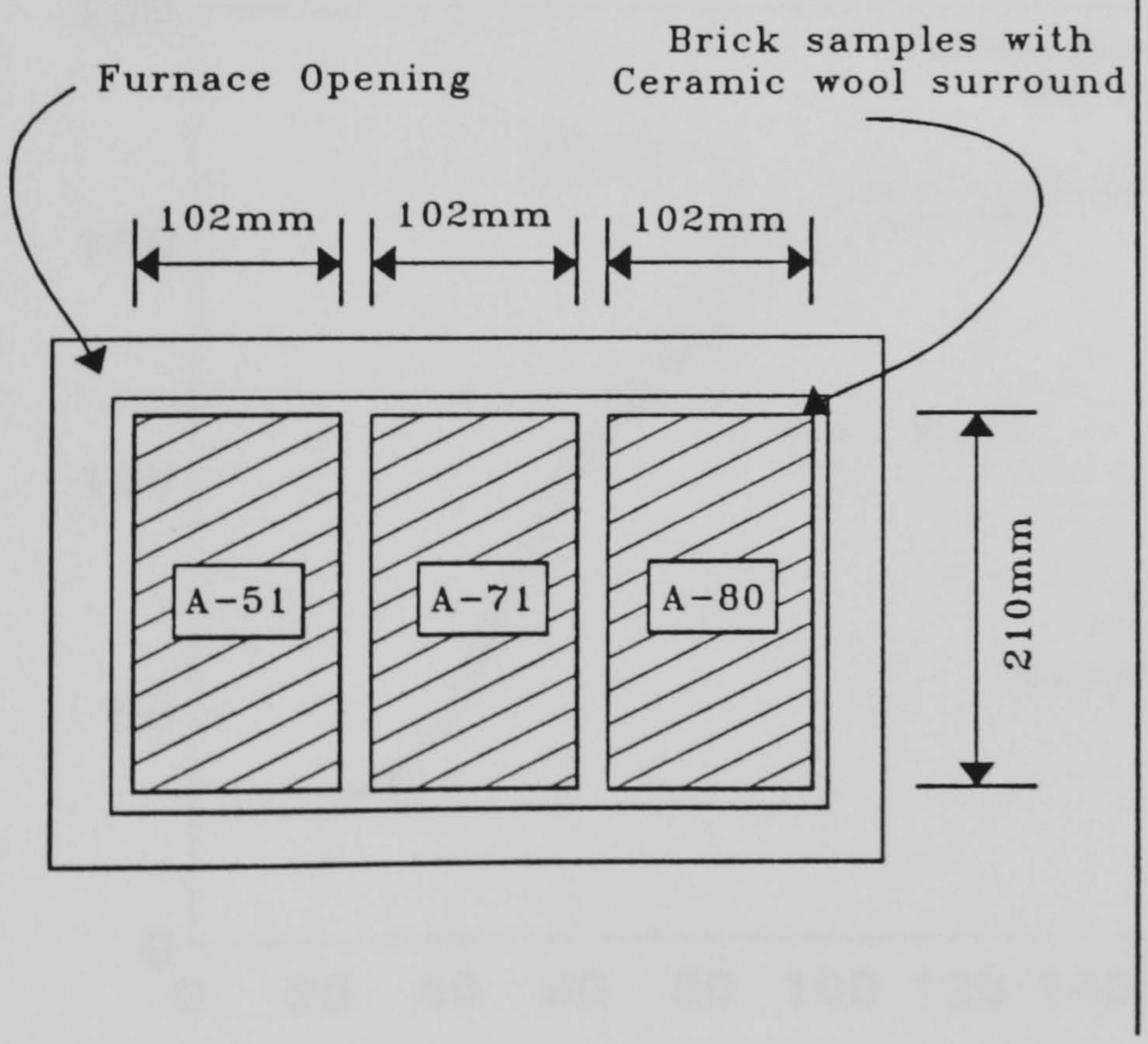


Figure A.4.1 - Carbolite Kiln Firing Cycle for New Furnace Lining Material



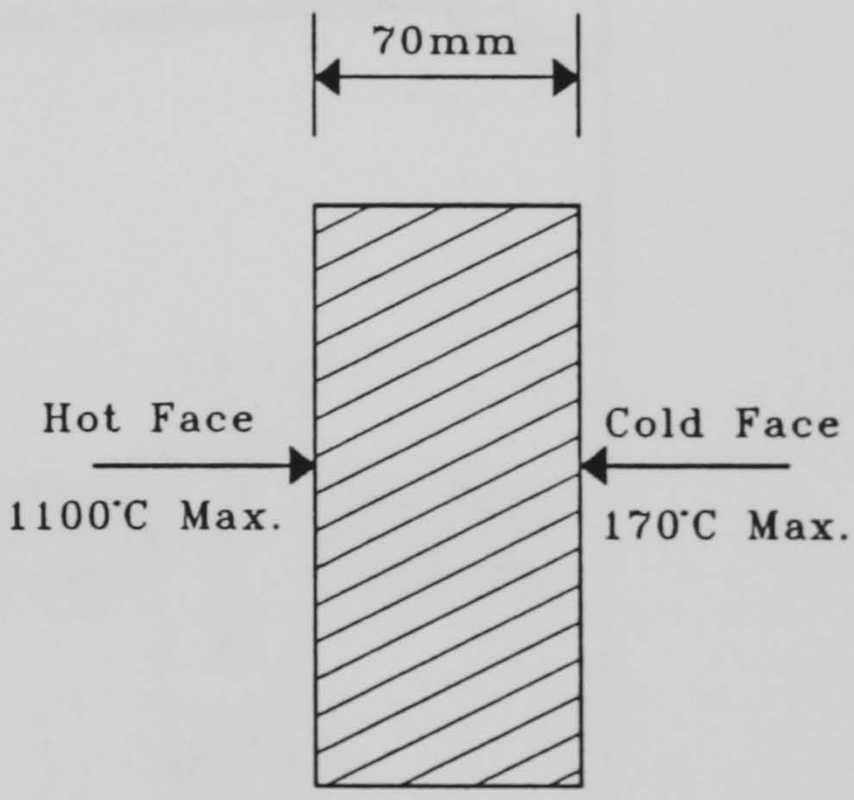


Figure A.4.2 General Arrangement of Preliminary Furnace Test

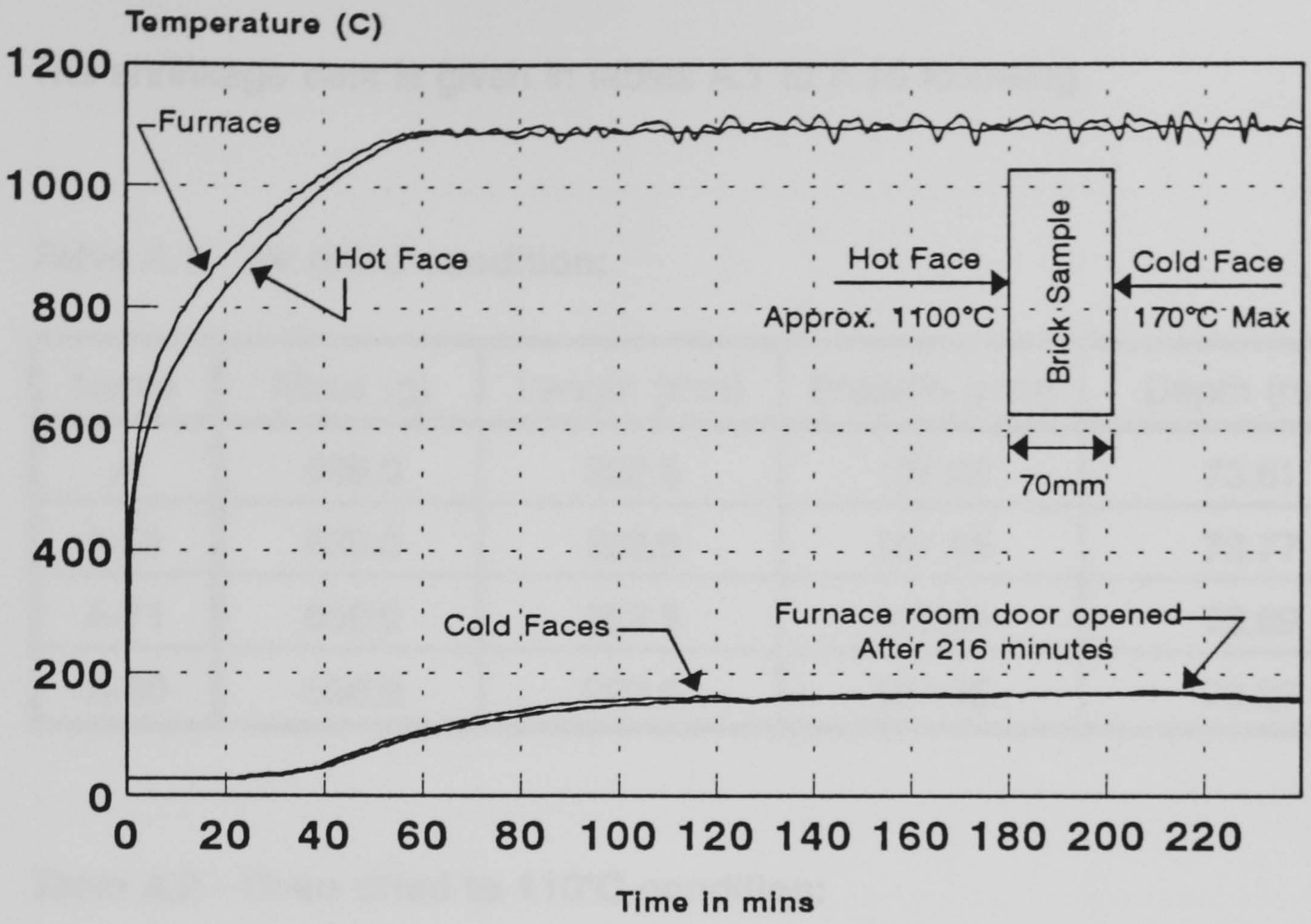


Figure A.4.3 - Insuline Brick Samples Fire Test
25-08-94 Salford University, Small Furnace.
Furnace, hot face and cold face temperatures.

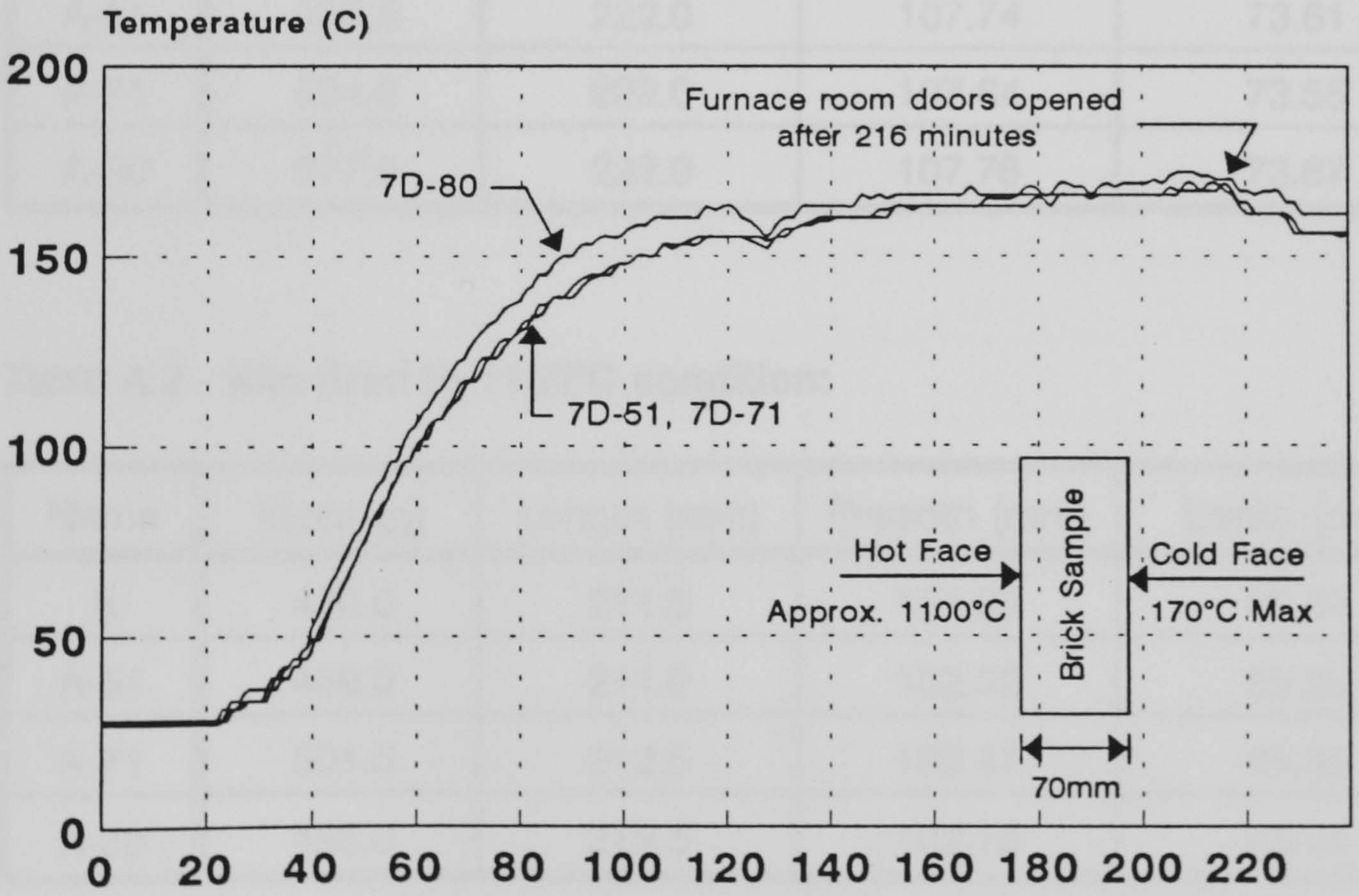


Figure A.3.5 - Insuline Brick Samples Fire Test
25-08-94 Salford University, Small Furnace.
Cold face temperatures only.

The shrinkage data is given in tables A.1 to A.10 following.

Table A.1 **- Air dried condition:**

Name	Mass (g)	Length (mm)	Breadth (mm)	Depth (mm)
A	596.0	222.5	107.99	73.61
A-51	630.0	222.5	107.98	73.77
A-71	656.0	222.5	107.84	73.69
A-80	598.0	222.5	107.98	73.93

Table A.2 **- Oven dried to 110°C condition:**

Name	Mass (g)	Length (mm)	Breadth (mm)	Depth (mm)
A	569.5	222.0	107.79	73.52
A-51	598.5	222.0	107.74	73.61
A-71	634.0	222.0	107.64	73.55
A-80	577.5	222.0	107.76	73.67

Table A.3 **- Kiln fired to 1150°C condition:**

Name	Mass (g)	Length (mm)	Breadth (mm)	Depth (mm)
A	470.0	211.5	101.88	69.40
A-51	489.0	211.0	102.02	69.56
A-71	501.5	212.5	102.47	69.86
A-80	496.0	212.5	102.18	69.44

Breadth and depth measurements made with digital Vernier rule.

Table A.4 **- Brick Densities:**

Condition	A	A-51	A-71	A-80
Cured/Air Dried	336.97	355.46	371.29	336.67
Oven Dried (110°C)	323.71	339.94	360.73	327.68
Air Conditioned	330.62	345.31	365.69	332.28
Fired (1150°C)	314.30	326.57	329.68	328.96

Densities given in Kg/m^3

Table A.5 **- Mix A Brick linear shrinkage (wrt original):**

Condition	Length(%)	Breadth(%)	Depth(%)
Cured/Air Dried	0	0	0
Oven Dried	0.2	0.19	0.12
Air Conditioned	0.2	0.16	0.18
Fired	4.94	5.66	5.72

Table A.6 **- Mix A-51 Brick linear shrinkage (wrt original):**

Condition	Length(%)	Breadth(%)	Depth(%)
Cured/Air Dried	0	0	0
Oven Dried	0.2	0.22	0.22
Air Conditioned	0.2	0.2	0.23
Fired	5.17	5.52	5.71

Table A.7 **- Mix A-71 Brick linear shrinkage (wrt original):**

Condition	Length(%)	Breadth(%)	Depth(%)
Cured/Air Dried	0	0	0
Oven Dried	0.2	0.19	0.19
Air Conditioned	0	0.15	0.18
Fired	4.49	4.98	5.20

Table A.8 - **Mix A-80 Brick linear shrinkage (wrt original):**

Condition	Length(%)	Breadth(%)	Depth(%)
Cured/Air Dried	0	0	0
Oven Dried	0.2	0.2	0.32
Air Conditioned	0.2	0.28	0.31
Fired	4.49	5.37	6.07

After the first firing cycle and subsequent cooling, the bricks were fired again to exactly the same cycle to determine if any further shrinkage or damage would be incurred. The inspection of the samples after the second firing showed that no extra damage had occurred and that the sample dimensions/shrinkages were as shown in the following tables.

Table A.9 - **Brick dimensions after second firing (including densities/shrinkages)**

Name	Mass (g)	Length (mm)	Breadth (mm)	Width (mm)	Density (kg/m^3)
A	470.0	210.0	101.73	69.28	317.6
A-51	487.0	210.0	101.83	69.43	328.0
A-71	500.0	212.5	102.39	69.76	329.4
A-80	495.0	211.5	102.01	69.38	331.1

Table A.10 - **Linear shrinkage after second firing (w.r.t original)**

(Shrinkage after first firing shown in brackets)

Name	Length(%)	Breadth(%)	Depth(%)
A	(4.94) 5.62	(5.66) 5.62	(5.72) 5.76
A-51	(5.17) 5.62	(5.52) 5.70	(5.71) 5.88
A-71	(4.49) 4.49	(4.98) 5.05	(5.20) 5.33
A-80	(4.49) 4.94	(5.37) 5.52	(6.07) 6.15

A.4.2 Further development work

The need to produce fired, crack-free samples was of paramount importance in the development work. For ease of volume retention it was decided to use Tilcon grade 2JS perlite, which has a lower bulk density than pack 3. The standard adopted mix was for an aggregate-cement ratio of 0.8 and a water-cement ratio of 1.35. Slabs of insuline were used for this section of the development work in order to ensure that all samples had the same processing conditions. In this way a slab could be made and have sections removed from it at the required curing duration, for instance 24 hours, 3 days, 7 days etc.

The slabs were manufactured with Secar 51 cement and 2JS. A 300 x 300 x 50mm slab had the following mix proportions (now referred to as mix 1):

Secar 51 - 833g, Perlite 2JS - 667g, Water - 1125g.

After the first cure period the samples were cut in half using a circular saw, and one half returned to its original curing conditions. The other half was cut again, one section being dried at 110°C, the other being placed in a roaster bag and heated at 110°C. The process of sealing in a roaster bag and heating to 110°C will be referred to as "re-heating" in the following text.

The code numbers listed in the results refer in sequence to the mix number, the curing temperature (i.e. RT = room temperature, 40 = 40°C cure temperature), the cure time in days, and the drying condition (R = dried in the bag). Some codes also carry the letter "W" meaning the sample was soaked in water prior to compression testing. The letter "P" means that the sample was tested after curing but without being dried. Edge samples where tested are marked with an asterisk, when making the slab if level grading was used the edge samples were weaker than the internal ones, if more mix was used in the edges they were stronger than the internal samples.

A.4.3 Compression test results for different curing methods.

Notes:

Samples 1-RT-1R correctly processed but air conditioned for 6 days after drying.

Samples 1-RT-3 were air dried after 3 days, then oven dried as normal.

Samples 1-RT-3R were left for 6 days before re-heating.

Samples 1-RT-7R were re-heated in a plastic bag which melted away causing damage to the surfaces of the tested material after being peeled off.

1-RT-1R	Dimensions	Density(kg/m^3)	σ_{cr}(N/mm^2)
A	46.2x49.9x51.1	364	0.556
B	46.2x48.9x51.1	371	0.589
C*	46.2x48.9x49.9	373	0.439

1-RT-3R	Dimensions	Density(kg/m^3)	σ_{cr}(N/mm^2)
A	47.1x50.2x50.0	355	0.410
B	47.1x49.9x50.3	367	0.604
C	47.1x49.9x50.6	395	0.466

1-RT-3	Dimensions	Density(kg/m^3)	σ_{cr}(N/mm^2)
A	46.5x50.3x50.0	368	0.496
B	46.5x50.6x49.8	371	0.599
C	47.5x50.6x50.4	406	0.580

1-RT-7	Dimensions	Density(kg/m^3)	σ_{cr}(N/mm^2)
A	47.7x50.2x50.9	361	0.740
B	47.8x50.1x51.2	380	0.879
C	47.1x50.3x51.3	383	0.765

1-RT-7W	Dimensions	Density(kg/m^3)	σ_{cr}(N/mm^2)
A	50.3x47.8x51.1	374(dry)	0.673
B	51.4x50.4x47.4	369(dry)	0.639
C	50.4x47.3x50.8	363(dry)	0.647

1-RT-7R	Dimensions	Density(kg/m^3)	σ_{cr}(N/mm^2)
A	46.1x50.1x50.6	401	0.755
B	49.4x50.5x51.3	352	0.653
C	49.6x50.0x50.6	377	0.661
D	49.3x50.3x51.1	390	0.599
E	49.6x51.1x51.2	374	0.696
F	46.5x49.9x50.3	386	0.693

1-RT-0R	Dimensions	Density(kg/m^3)	σ_{cr}(N/mm^2)
A	49.2x46.3x51.3	381	0.636
B	49.2x46.0x51.7	381	0.594
C	49.2x46.1x51.8	393	0.616

1-RT-0RW	Dimensions	Density(kg/m^3)	σ_{cr}(N/mm^2)
A	49.2x46.6x51.4	386	0.590
B	49.4x46.3x51.4	376	0.560
C	49.3x46.3x51.7	392	0.556

1-RT-1	Dimensions	Density(kg/m^3)	σ_{cr}(N/mm^2)
A	47.6X47.1x50.6	357	0.520
B	47.4x47.5x50.4	357	0.520
C	47.4x47.1x50.6	359	0.570

1-RT-1R	Dimensions	Density(kg/m^3)	σ_{cr}(N/mm^2)
A	46.2x47.4x50.6	381	0.450
B	47.4x47.2x51.2	385	0.560
C	47.4x47.4x51.0	384	0.550

1-RT-3	Dimensions	Density(kg/m^3)	σ_{cr}(N/mm^2)
A	47.2x47.0x50.4	358	0.530
B	47.4x47.1x50.9	362	0.570
C*	47.3x47.2x50.8	366	0.620

1-RT-3R	Dimensions	Density(kg/m^3)	σ_{cr}(N/mm^2)
A	46.5x47.4x50.7	367	0.580
B	47.6x47.4x50.9	378	0.640
C	46.4x47.5x50.7	363	0.490

1-40-0R	Dimensions	Density(kg/m^3)	σ_{cr}(N/mm^2)
A	49.4x49.1x50.0	351	0.450
B	49.2x49.2x50.1	367	0.460
C	49.2x49.1x50.7	364	0.450

1-40-1	Dimensions	Density(kg/m^3)	σ_{cr}(N/mm^2)
A	46.1x47.6x50.8	391	0.720
B	47.6x45.0x50.8	377	0.790
C	47.3x47.2x50.3	374	0.670

1-40-1R	Dimensions	Density(kg/m^3)	σ_{cr}(N/mm^2)
A	48.2x47.4x50.6	377	0.570
B	47.2x47.9x50.1	349	0.540
C	47.3x47.5x50.3	363	0.510

1-40-3	Dimensions	Density(kg/m^3)	σ_{cr}(N/mm^2)
A	47.2x47.3x50.8	363	0.570
B	47.3x47.3x50.8	370	0.600
C*	47.1x46.6x51.2	382	0.770

1-40-3R	Dimensions	Density(kg/m^3)	σ_{cr}(N/mm^2)
A	46.7x47.4x50.4	368	0.610
B	46.9x47.4x50.7	373	0.730
C	47.6x47.4x50.5	369	0.660

1-40-7	Dimensions	Density(kg/m^3)	σ_{cr}(N/mm^2)
A*	47.7x47.7x51.0	413	0.980
B	47.7x45.9x50.7	374	0.830
C	47.9x45.9x50.5	361	0.700

1-40-7R	Dimensions	Density(kg/m^3)	σ_{cr}(N/mm^2)
A	47.1x47.1x50.8	365	0.760
B	47.2x47.4x50.6	367	0.800
C*	47.2x46.8x50.5	390	0.810

1-40-7P	Dimensions	Density(kg/m^3)	σ_{cr}(N/mm^2)
A	47.3x47.6x50.9	510(wet)	0.550
B	47.5x47.4x50.8	516(wet)	0.630
C	47.3x47.3x50.5	491(wet)	0.530

As expected it can be seen from the results that dried samples are stronger than damp or saturated specimens. Heating to 110°C in a roaster bag appeared to have a detrimental effect on compressive strength in all cases. This could possibly be due to accelerated conversion of the high alumina cements. Also for both curing temperatures the compressive strengths are not consistent. The one day cure strengths are higher than the three day, and the seven day strength higher than the one day.

A.4.4 Investigation into the Effect of Density and Curing time on Strength of Samples.

In the following tables the samples were made brick shaped (220x110x70mm) and tested without cutting to get a more representative set of results for expected strength.

In *Table A.11*, all bricks were made to mix 1 ratios (aggregate-cement ratio = 0.8, water-cement ratio = 1.35), with varying quantities of wet-mix used for the target densities stated. In all cases the cement used was Secar 51, aggregate was perlite 2JS. Samples were cured for 24 hours, bagged at room temperature.

Table A.11 - **Compressive Strength Variation with Density for 2JS, Mix 1 Bricks.**

Target Density (kg/m^3)	Actual Density (kg/m^3)	Comp. Strength (N/mm^2)
400	437	1.07
400	409	0.82
400	419	0.91
375	399	0.75
375	392	0.76
375	406	0.81
350	371	0.57
350	376	0.69
350	374	0.61
325	351	0.41
325	350	0.41

The compression test results are shown in **Figure A.4.5** on a following page.

Table A.12 following shows the fast gain of strength which can be promoted by mixing the constituents in warm conditions (ie warmed cement and aggregates, hot water, and warm cure - bagged - at 40°C) The results of this experiment would tend to indicate that a very fast turn around of the samples may be possible as the brick units need have very little compressive strength in their own right. The samples were dried in a preheated oven at 110°C immediately after the quoted drying time was finished.

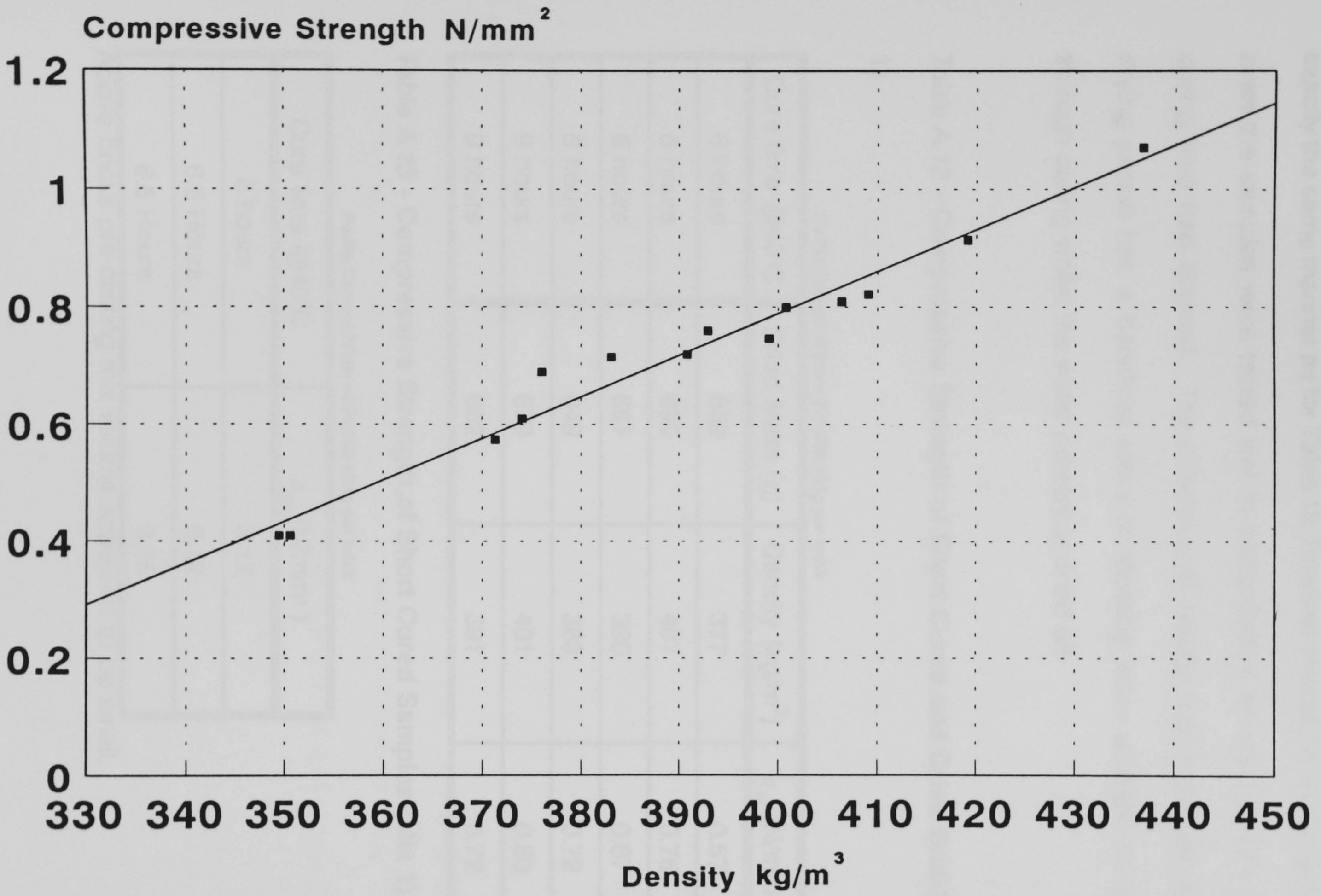


Figure A.4.5 - Brick Compressive Strength .v. Density
Perlite Grade 2JS
Mix 1 - Densities 330-450 kg/m³

Table A.13 following gives the results for the brick samples manufactured in exactly the same manner as for Table 12, however instead of being dried in the oven the samples were tested wet in compression as soon as the quoted curing time had elapsed. The difference in results would indicate that the drying period has a beneficial effect of allowing more strength to develop through curing whilst the water content is dried off.

***Table A.12* - Compressive Strength of Short Cured and Dried BrickS (Mix 1)**

Perlite:Cement:Water=239:298:403g per brick

Cure time @40°C	Dried Mass (g)	Density (kg/m^3)	σ_{cr} (N/mm^2)
6 hours	639	377	0.57
6 hours	690	407	0.76
6 hours	661	390	0.67
9 hours	649	383	0.72
9 hours	679	401	0.80
9 hours	662	391	0.72

***Table A.13* - Compressive Strength of Short Cured Samples (Mix 1).**

Perlite:Cement:Water=239:298:403g per brick

Cure time @40°C	σ_{cr} (N/mm^2)
6 hours	0.12
6.5 Hours	0.12
6.5 Hours	0.16

Above bricks pre-casting mix volume appeared to be small.

Due to the wet mix volume being particularly low for the low end densities with perlite 2JS it was decided to use a grade with a lower bulk density, this grade

was 2JL. *Table A.14* following shows the compressive strengths for bricks made with perlite 2JL, and 3JS (a much higher bulk density) which were made to investigate the effect on Perlite grade and density to higher extremes. The figure after the perlite grade in the sample name is the predicted density of the dried sample.

Samples 3JS-600 cured at room temperature had very poor edges due to the mix being over wet when moulding, hence faces being pulled away when the mould was released.

Samples 3JS-600 and 3JS-700 cured at 40°C had a reduced water content (due to the reduction of perlite surface area with using a coarser aggregate). The aggregate-cement ratio was unchanged, however the water-cement ratio was reduced to 0.89 from 1.35 (i.e. was reduced by one third).

After curing for the time stated in *table A.14* all samples were oven dried at 110°C for approximately 5 hours prior to testing - some samples had not reached a stable water content (i.e. fully oven dried) however the rate of water loss was slow for the hour prior to structural testing.

Table A.14 **- Compressive Strengths and Densities for 2JL and 3JS Bricks**

Sample	Approx. Cure Time (Hours)	Cure Temp (°C)	Density (kg/m^3)	σ_{cr} (N/mm^2)
2JL-300	48	20	339	0.21
2JL-300	48	20	343	0.21
2JL-300	48	20	347	0.22
3JS-600	48	20	659	1.16
3JS-600	48	20	687	1.17
3JS-600	24	40	609	1.31
3JS-600	24	40	613	1.48
3JS-700	24	40	712	2.43
3JS-700	24	40	701	1.80

As can be seen from *table A.14* a very high compressive strength may be obtained at still relatively low density. These brick densities have not been pursued further due to the obvious increase in cost of raw materials, and the likelihood that the distinct increase in density would lead to a much poorer material with regards to thermal efficiency.

Due to the seeming success in manufacturing bricks at the low end of the density range with perlite 2JL it was decided to manufacture a series of samples in the density range of 275-350kg/m^3. These bricks were manufactured once again to mix 1 proportions (aggregate:cement = 0.8, water:cement = 1.35) and were all mixed at elevated temperature using hot water (notionally 50°C). All samples were cured for 24 hours at 40°C in polythene bags to maintain a high humidity atmosphere, followed by immediate

drying at 110°C in a pre-heated oven. After the drying period all samples were inspected thoroughly and all samples appeared to be free from cracks. The compression test results for these bricks are shown in *table A.15*.

A further set of 2JL samples were mixed and cured at room temperature as a comparative sample. The samples were given a longer curing period of 48 hours, once again in polythene bags to maintain a high humidity atmosphere. The samples were tested in the wet state (ie no drying to promote the hydration of the cement and formation of the C_3AH_6/AH_6 structure). The compression test results for these samples are given in *table A.16*.

Table A.15 **- Compression test results for 2JL bricks (275-350kg/m^3)**

Sample	Approx. Cure Time (Hours)	Cure Temp (°C)	Density (kg/m^3)	σ_{cr} (N/mm^2)
2JL-275	24	40	284	0.37
2JL-275	24	40	270	0.31
2JL-275	24	40	275	0.34
2JL-300	24	40	297	0.36
2JL-300	24	40	289	0.32
2JL-300	24	40	308	0.40
2JL-325	24	40	334	0.52
2JL-325	24	40	320	0.46
2JL-325	24	40	310	0.44
2JL-350	24	40	362	0.71
2JL-350	24	40	343	0.60
2JL-350	24	40	332	0.55

Table A.16 - **Compression Test Results for 2JL Room Temperature Cured Bricks**

Sample	Approx. Cure Time (Hours)	Cure Temp (°C)	Density (kg/m^3)	σ_{cr} (N/mm^2)
2JL-300	48	20	339	0.21
2JL-300	48	20	343	0.21
2JL-300	48	20	347	0.22

The compression test results for the 2JL bricks cured at 40°C are shown on the following page in graphical form. **Figure A.4.6** shows the straight line trend of the compression test results.

A.4.5 Investigation of the Factors Effecting Drying and Firing Shrinkage and Cracking.

The problem of samples cracking during drying and firing was a distinct hindrance to the research development. If samples were to be evaluated fully it was necessary to investigate the effects of density, mix proportions, manufacturing methods and firing times on cracking of the samples, and to be able to manufacture crack free samples in good condition (i.e. a viable material from a mass production point of view).

The following sections describe samples made in order to evaluate the correct method required for production. In many cases, if a material was cracked after the drying cycle it was not fired as the effect would only be to open the fault

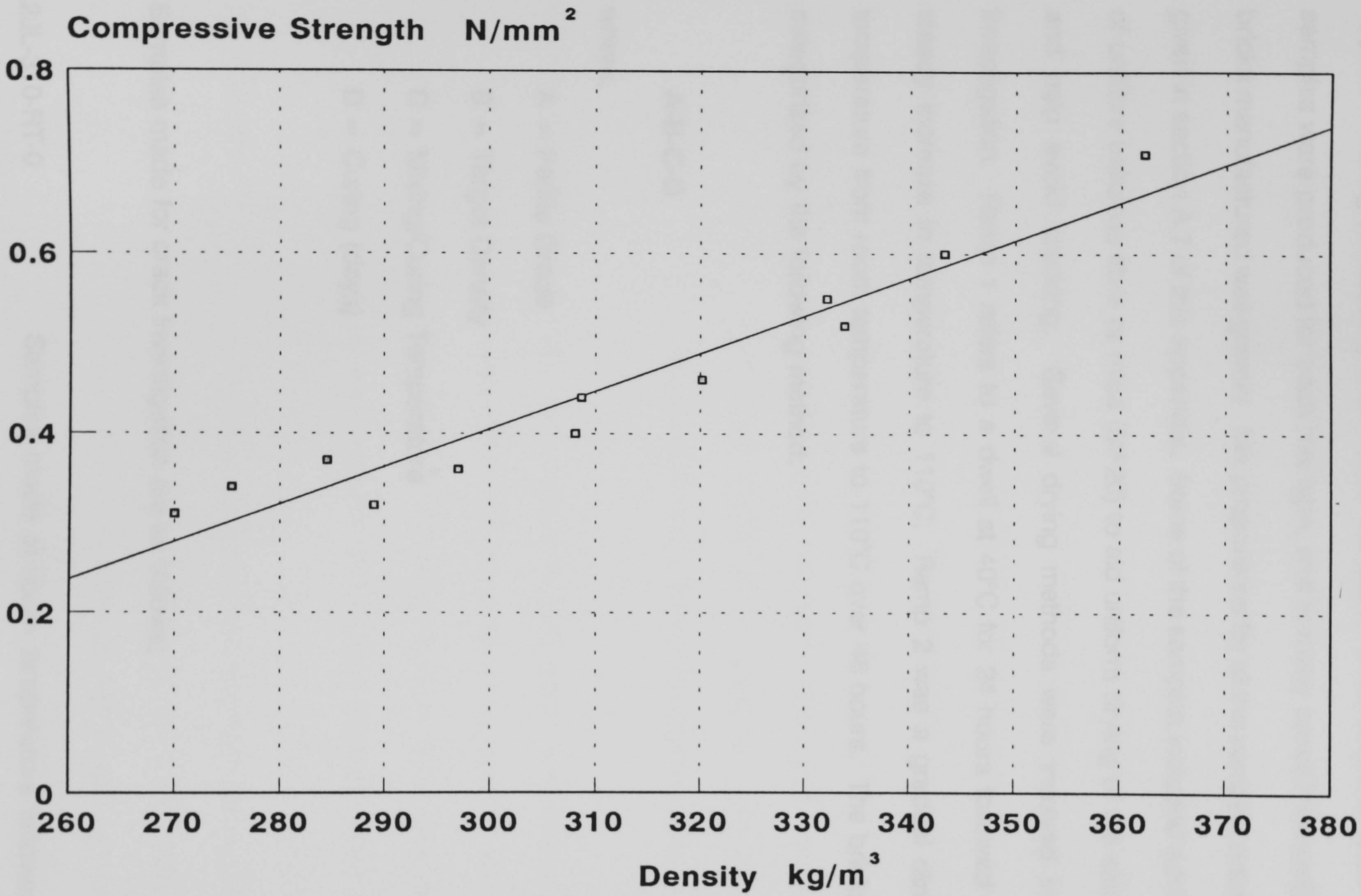


Figure A.4.6 - Brick Compressive Strength .v. Density
Perlite Grade 2JL
Mix 1 - Densities 270-370 kg/m³

further. An attempt has been made to describe the flaws (if any) which were apparent during processing of the brick samples. In all cases at least three samples were produced for each mix type, and in many cases the number of bricks manufactured was greater. Mix proportions for all the samples made are given in section A.7 of this appendix. Some of the samples included additions of unifibre cellulose fibre or mica (MF20) to aid uniform drying of the samples and help avoid cracking. Several drying methods were involved in this investigation. Ramp 1 refers to a dwell at 40°C for 24 hours followed by a steady increase in temperature to 110°C. Ramp 2 was a gradual climb in temperature from room temperature to 110°C over 48 hours. The bricks are categorized by the following method:

A-B-C-D

where,

A = Perlite Grade

B = Target Density

C = Mixing/Curing Temperature

D = Curing (days)

Samples made for crack investigation are as follows;

2JL-310-RT-0	Samples made at room temperature followed by drying immediately in a fan assisted oven at 110°C.

3 no. bricks appeared undamaged during this drying method. When the method was repeated with another 6 no. bricks all samples appeared to have cracked during drying.

2JL-310-RT-0(AIR)	Samples were made at room temperature and allowed to air dry at RT for 24 hours before drying in the oven at 110°C. All samples appeared to be uncracked after the drying cycle.
2JL-310-40-0	Samples made with warm water and immediately dried at 110°C after moulding. All samples appeared to crack during the drying cycle.
2JL-310-40-0(AIR)	Samples made with warm water and allowed to air dry at RT for 24 hours before being dried at 110°C. All samples had very slight cracking after drying, however edges were noticed to be extremely poor
2JL-275-RT-1	Samples made at room temperature followed by 24 hours bagged at room temperature. Samples were then dried immediately at 110°C and all samples appeared to have a slight degree of cracking/damage after drying.

2JL-310-RT-1	Curing/drying process same as 2JL-275-RT-1 above. Again, after drying all samples appeared to have some slight degree of damage.
2JL-350-RT-1	Curing/drying process same as 2JL-275-RT-1 above. All samples appeared to have cracked during the drying cycle.
2JL-310-RT-1(C2%)	Samples contained an addition of 2% weight of dry components of cellulose fibre which was dispersed in water prior to mixing. The curing/drying process was the same as 2JL-275-RT-1 above. All samples appeared to be damaged or cracked after the drying cycle.
2JL-310-RT-1(M2%)	Samples contained an addition of 2% weight of dry components of Mica (MF-20) which was dispersed by mixing dry prior to addition of water. The curing/drying process was the same as 2JL-275-RT-1 above. All samples appeared to be undamaged after the drying cycle.
2JL-310-RT-1(M4%)	Samples contained an addition of 4% weight of dry components of Mica (MF-20) which was dispersed

by mixing dry prior to addition of water. The curing/drying process was the same as 2JL-275-RT-1 above. All samples appeared to be slightly cracked after the drying cycle.

2JL-310-RT-1(RAMP1)	Samples were made at RT, followed by 24 hours bagged at room temperature. The sample were then dried at 40°C for 24 hours followed by a ramped climb from 40-110°C over 24 hours. All samples appeared to be undamaged after drying.
2JL-310-RT-0(RAMP2)	Samples were made at RT followed by immediate drying, unbagged, on a ramped temperature climb from RT to 110°C over 48 hours. All samples were cracked on the underside edges where the drying path had been obstructed by the moulding board/plastic sheet.
2JL-310-RT-0(RAMP1)	Samples were made at RT followed by immediate drying, unbagged, on a ramped temperature climb from RT to 110°C over 48 hours. All samples were cracked on the underside edges where the drying path had been obstructed by the moulding board/plastic sheet.

2JL-310-RT-1(RAMP1)	12 samples were made exactly as the 2JL-310-RT-1(RAMP1) samples previously. Bricks were measured carefully prior to drying cycle to get shrinkage data. Samples were dried to ramp 1 as described previously. After the drying cycle had been completed, inspection of the bricks showed severe cracking both longitudinal and across the bricks.
2JL-310-RT-5(RAMP1)	Samples made at room temperature as described previously, bagged and cured at room temperature for 5 days (to give a fuller curing period), removed from bags and dried to ramp 1. After drying, all samples were cracked on all faces.
2JL-310-RT-1(RAMP1)(P)	It was decided that the variation of particle size distribution may make a difference to the cracking of samples. To this effect 10, 20 and 30% of grade 2JL perlite was replaced by a corresponding mass of grade 2JS perlite in different samples. These samples were manufactured at room temperature and cured (bagged) at room temperature for 24 hours prior to drying to ramp 1. All samples were

cracked after the drying cycle.

2JL-310-RT-1(V) Samples were similar to those described for the grade 2JS perlite substitution above, however in this case fine grade vermiculite was used instead. Samples were cure for 24 hours (bagged) at room temperature followed by drying in a pre-heated oven at 110°C. Some samples showed faint drying cracks, but others appeared to be undamaged.

2JL-310-RT-1(V10)(M) Samples were manufactured with a 10% vermiculite substitution and also an addition of 4% Mica (MF20). The samples were mixed and cured (24hrs) at room temperature. The bricks were then transferred to a hot oven at 110°C and dried for 24 hours. All samples appeared to be undamaged after drying.

Throughout the investigation into different mix proportions and manufacturing method described above several bricks were chosen (when undamaged) and fired either to the cycle described in section A.4.1 or a faster cycle in a small cylindrical kiln. The faster cycle consisted of a ramp to 1150°C of 230°C/hr followed by a 5 hour dwell and natural cooling. **Figure A.4.7** on the following page shows a comparison of the two cycles. In all cases up to this point the

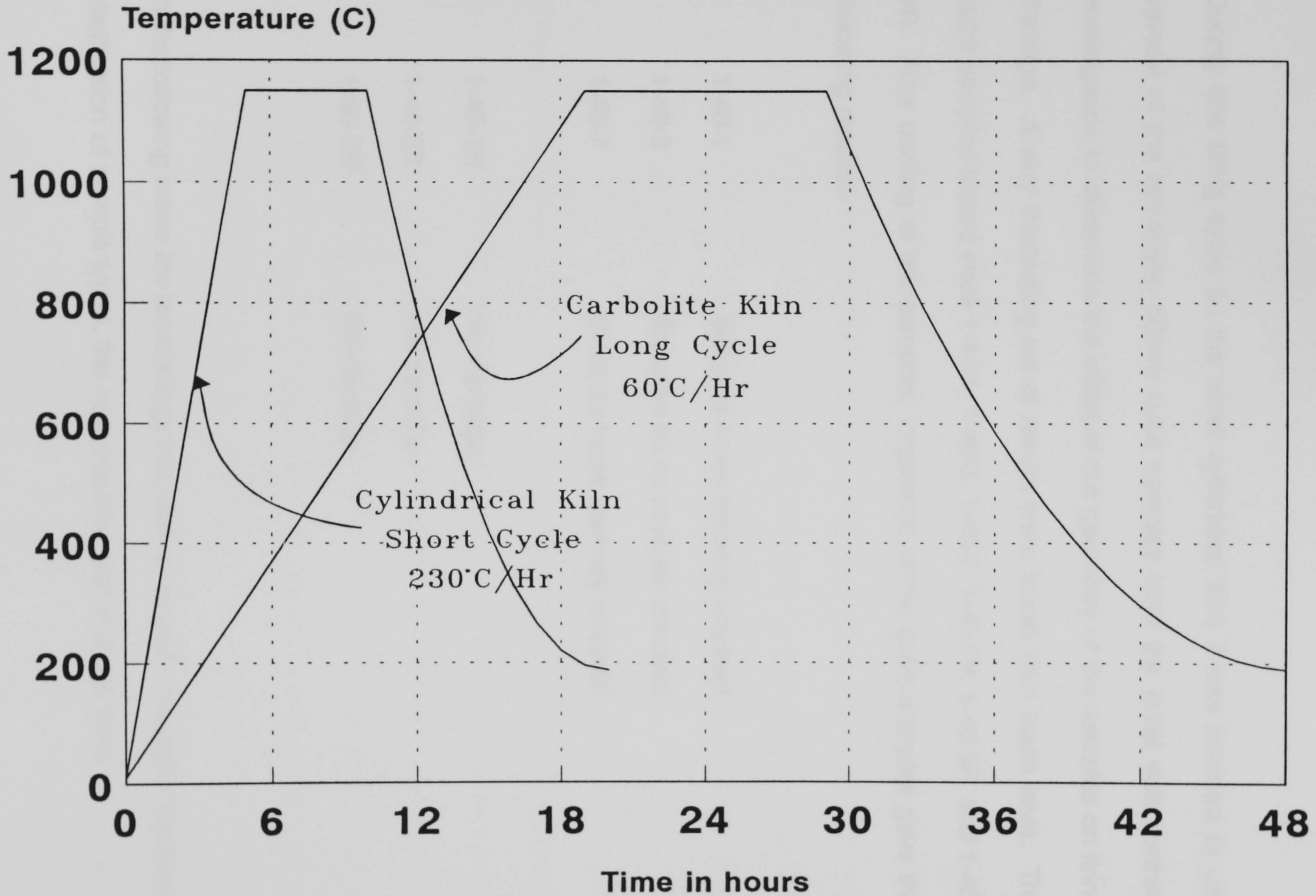


Figure A.4.7 - Comparison of the two firing cycles for New Furnace Lining Material

bricks were severely cracked after the firing cycle and the mix and manufacturing method was modified further.

During one firing cycle (in the small cylindrical kiln) it was decided to use several of the remaining 50mm cube samples from the initial slab method investigation to determine the effect of the geometry of the samples on firing damage. A very interesting set of results were found from these tests. The cube samples tested were 1-40-1, 1-40-3, 1-40-7, 1-40-1R, 1-40-3R, and 1-40-7R. After cooling of the samples, inspection of the cube samples gave the following results:-

1-40-1	Samples faces severely cracked
1-40-3	Samples faces severely cracked
1-40-7	Samples faces severely cracked
1-40-1R	No damage
1-40-3R	No damage
1-40-7R	No damage

In the following mixes the terminology has been changed once again for easier clarification of sample types, the new classification is of the form:

Perlite grade - Curing temperature - Days curing - #Mix number

2JL-RT-1-#2 Cracking problems may have been due to insufficient supply of cement to wet the perlite surfaces. It was decided to make a mix at the envisaged extremes: Aggregate:cement ratio = 0.5, water:cement = 1.35. Samples were made and cured at room temperature. After curing samples were dried in a pre-heated oven at 110°C. All samples were cracked after drying.

2JL-RT-1-#3 Samples were made the same as 2JL-RT-1-#2 above, however the aggregate:cement ratio used was increased to 0.65. After curing the samples were placed to dry in a pre-heated oven at 110°C for 24 hours. After the drying period approximately half of the 12 samples made were carrying minor cracks, other appeared undamaged.

2JL-RT-1-#4 Samples made were exactly the same as #3 above, however the bricks were left in the mould, covered with paper towelling and had an extra 200g water poured onto the sample. The samples were then covered with a plastic sheet and left to cure for 24 hours. Samples were dried as for #3 above, and after drying, no signs of cracking could be seen.

2JL-RT-1-#3(R) 9 no. samples were made as mix #3 above and cured for 1 day at room temperature. The samples were then placed in roaster bags and placed in the oven at 110°C

(i.e. re-heated). The samples were dry after 48 hours re-heating after which they were removed from the oven and removed from the bags. 4 no. of the samples were measured and placed in the large kiln for the firing cycle, 4no. samples were fired in the small kiln. After firing it was found that the samples were undamaged by the firing cycles.

The eight reheated 2JL-RT-1-#3 were fired to a temperature in excess of their service temperature, 4 in the small cylindrical kiln and 4 in the larger kiln. Of these samples no cracking appeared to have taken place during the firing cycle. The small kiln was programmed for a very rapid rise in temperature (230°C/Hr) where as the larger kiln was programmed for a slower rise in temperature (60°C). The samples in the larger kiln were carefully measured before and after firing to get shrinkage and density change information. The dimensions were as shown in the tables below.

Table A.17 **- Pre-Fired dimensions of #3-R bricks**

Brick No.	Length (mm)	Width (mm)	Depth (mm)	Density(kg/m^3)
1	221.0	111.3	72.3	329.8
2	221.0	111.2	72.4	335.3
3	221.0	111.4	72.2	338.4
4	221.0	111.2	72.6	336.0

Table A.18 - **Post-Fired dimensions of #3-R bricks**

Brick No.	Length (mm)	Width (mm)	Depth (mm)	Density(kg/m^3)
1	211.0	103.50	68.16	323.8
2	211.0	103.92	68.14	328.6
3	211.0	103.26	68.68	330.8
4	211.0	103.50	68.22	334.3

Table A.19 - **Shrinkages and density changes of #3-R bricks during firing cycle**

Brick No.	Length (%)	Width (%)	Depth (%)	Density (%)
1	4.52	7.00	5.73	1.82
2	4.52	6.55	5.88	2.00
3	4.52	7.31	4.88	2.24
4	4.52	6.92	6.03	0.51

Although the samples were uncracked after the firing cycle the fired samples were extremely friable to the touch (ie could be eroded easily, and may not perform well in a high velocity flame situation) and had poor edges and corners. This however could be worked upon, and it seemed that the re-heat method of drying was essential for the manufacture of uncracked samples.

Further samples were made using the re-heat method of drying in order to manufacture a full thickness (210mm) test panel in order to be able to compare the performance of the new lining material with an equivalent thickness of ceramic wool. In addition to the #3-R samples made the following samples were manufactured:

2JS-RT-1-#5R Perlite grade 2JS was adopted for this sample due to a

temporary shortage of grade 2JL. Aggregate-cement ratio = 0.8, water-cement ratio = 1.35. Samples were mixed and cured for 24 hours at room temperature (bagged) then re-heated in a pre-heated oven for 48 hours at 110°C. Samples were fired in the small kiln to the short firing cycle. No visible damage had occurred after the firing cycle.

#5-R bricks were fire tested to the hydrocarbon curve on the small furnace to a similar test arrangement as shown in **figure A.4.2** previously. The test results are shown in **figures A.4.8 and A.4.9** which indicate a steady state cold face temperature of approximately 190°C.

2JL-350-RT-3R — Aggregate-cement ratio of 0.8, Water-cement ratio of 1.35, target density 350kg/m^3. Mixed and cured (3 days) at room temperature, re-heated for 24 hours at 110°C. No sign of damage after the drying cycle upon inspection. Samples fired to the fast cycle in the small kiln and showed faint cracks and distortion after firing.

2JL-RT-1-#6R — Aggregate-cement ratio of 0.73, water-cement ratio of 1.35. Samples were mixed and cured for 24 hours at room temperature followed by re-heat drying at 110°C for 24

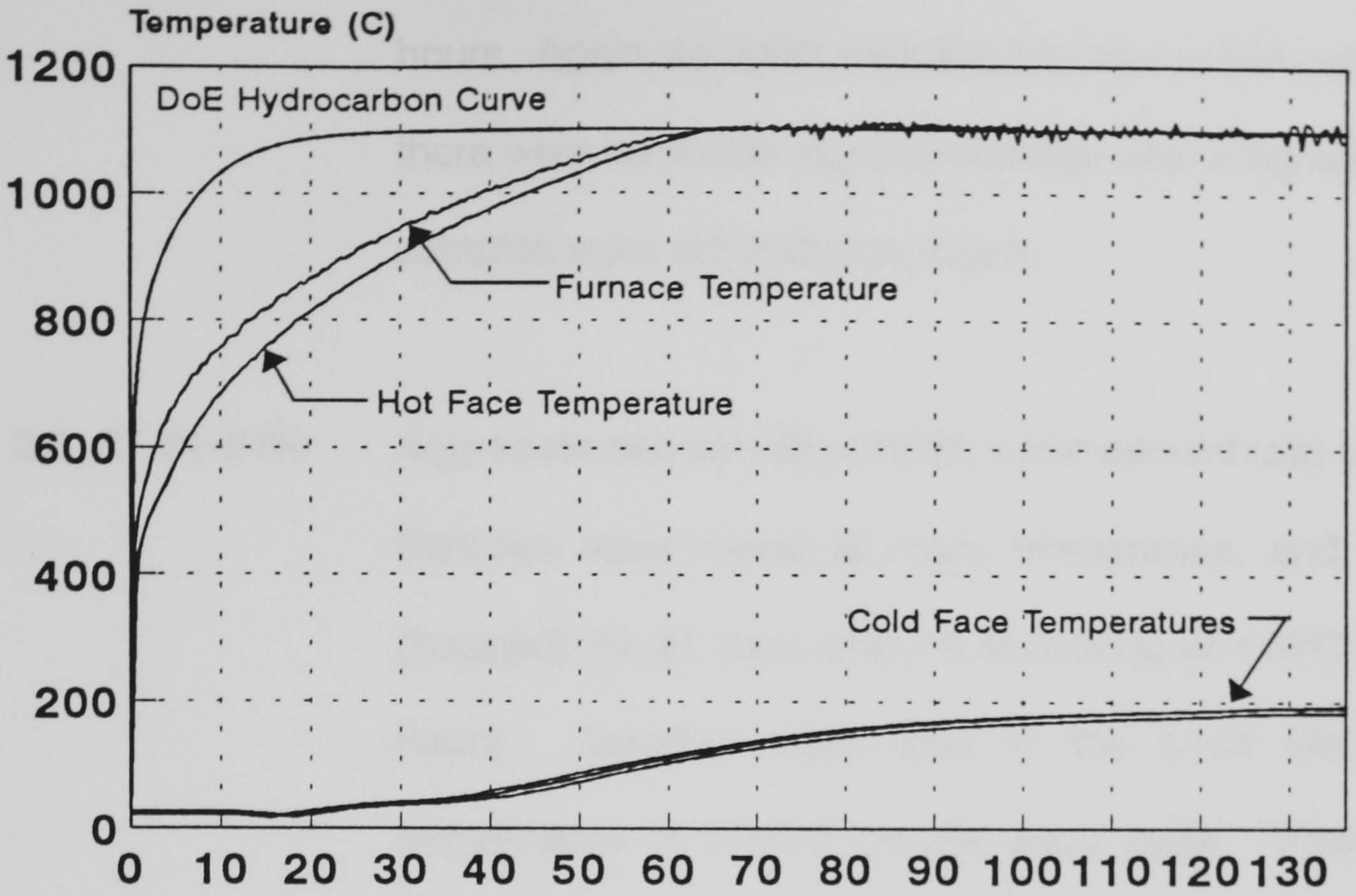


Figure A.4.8 Fire Testing of Samples #5-R
Small Furnace - Salford University 13-01-95
Sample Thickness ≈ 70mm

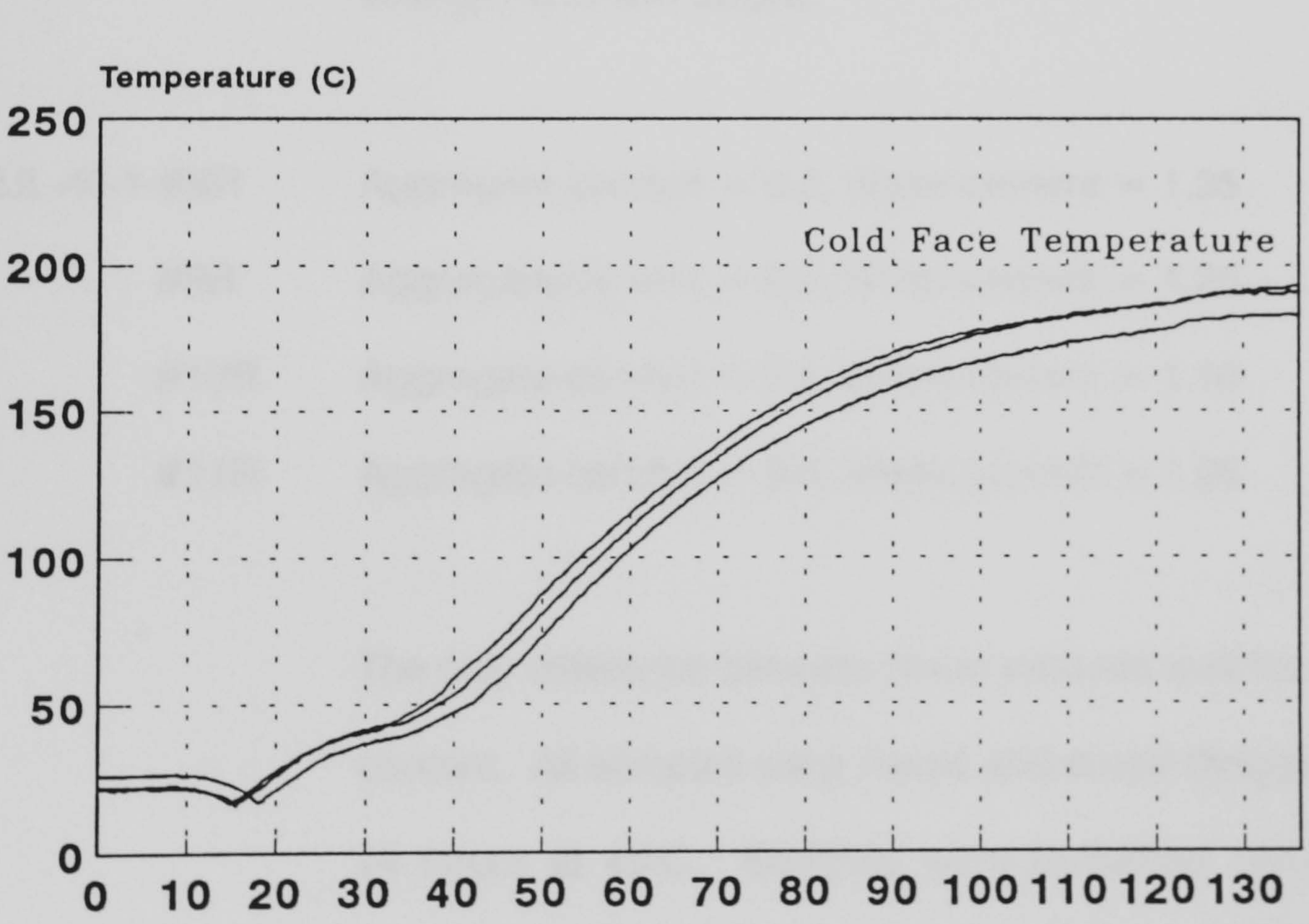


Figure A.4.9 Fire Testing of Samples #5-R
Small Furnace - Salford University 13-01-95
Sample Thickness ≈ 70mm

hours. Again, samples were fired to the fast kiln cycle and there were no visible signs of damage after firing although samples were still friable to touch.

2JL-RT-21-#7R Aggregate-cement ratio of 0.57, water-cement ratio of 1.07. Samples were mixed at room temperature, and cured (bagged) for 21 days prior to re-heating at 110°C for 24 hours. Samples were fired in the small kiln to a temperature of 1150°C on the short cycle. Post firing inspection found minor cracks where samples had been in contact with each other, otherwise samples had good strength and firm edges.

2JL-40-1-#8R Aggregate-cement = 0.8, Water-cement = 1.35

#9R Aggregate-cement = 0.8, Water-cement = 1.26

#10R Aggregate-cement = 0.8, Water-cement = 1.16

#11R Aggregate-cement = 0.8, Water-cement = 1.06

The only difference between these samples was the water content. All samples were mixed and cured (bagged) for 24 hours at 40°C. Samples were re-heated (when still warm from curing cabinet) in pre-heated oven at 110°C. Bricks were fired in the small kiln to the short firing cycle, post firing inspection gave the following results:

#8R	Samples had short, medium width (approx 0.5mm) cracks in the edges of the bricks, however otherwise the bricks were in good condition, firm corners and apparent good strength
#9/10/11R	Samples were uncracked after the firing cycle. Edges were firm and faces were in good condition. Bricks had apparent good strength. Some of the bricks had distorted during firing due to the stacking method used. Of all samples, #11R seemed to be in slightly better condition than the other samples.
2JL-#3R(300S)	Two fired samples of 2JL-RT-1-#3R were soaked in a mixture of 300ml of colloidal silica and 1100ml of additional water between them. The samples were then dried at 110°C for 24 hours and re-fired on the short firing sequence. Post firing inspection showed the samples still to have poor edges, but no cracking of the samples had occurred, and there appeared to have been an increase in strength of the samples (faces).

A.4.6 Maximum Temperature - Short Term Exposure.

As the bricks manufactured with secar-51 cement were fired at 1150°C it was obvious that they would survive this temperature during service. However the pyrometric cone equivalent suggested that the operating temperature my be far in excess of this (1440°C).

Several representative bricks of #11R were rapidly fired to 1300°C and held at that temperature for 5 hours. Complete melt down of the bricks occurred, and the bricks were hardly recognisable when inspected after cooling. A glassy surface to the melted surface was evident also. Figure A.4.10 following shows a photograph of the post-heated samples.

The test was repeated at 1200°C and the bricks survived satisfactarily with no noticeable damage. It should be noted that the samples were fully immersed within the furnace, and this is a much more severe condition than just one face exposed which applies to lining situations. The discrepancy between the pyrometric softening point of the cement, and the melting temperature of the tested samples was explained by the combination of the cement with perlite which acted as a flux to promote a lower melting point.

Three Secar-71 bricks (aggregate/cement ratio = 0.8, density = 370kg/m^3) were made following the normal procedure, however following the drying stage they were fired directly to 1300°C. The firing cycle was RT to 1150°C over five

hours, followed by 1150-1300°C in three hours with zero dwell at 1300°C. This was then followed by natural cooling to room temperature.

Following cooling of the samples inspection showed the typical length to be approximately 206mm, however there were some cracks in the bricks, in particular in one brick which had been arranged as a beam between two others. This length indicated similar shrinkage as would be expected for the Secar-51 samples fired to 1150°C. No development work was carried out on the Secar-71 bricks as a substantial amount of work would be required to find the necessary mix proportions and curing-drying-firing cycle to prevent cracking of the bricks. However, this brief investigation does show that similar compositions to those investigated fully could satisfy much more demanding temperature resistance requirements.

Figure A.4.10 - Condition of #11-R samples after heating to 1300°C

A.5 Full thickness panel testing.

For the purpose of evaluating the new furnace lining material at its proposed full thickness it was decided that a test panel should be manufactured which could be tested for extended periods. The long runs of the tests were to ensure that the cold face temperatures of the samples had stabilised, and hence steady state conditions achieved.

The test panel arrangement can be seen in **figure A.5.1** on the following page, and consisted of a Vermiculux surround which was lined with ceramic wool and contained the brick samples tightly packed in the centre. The test was performed on a programmable electric kiln as it was decided that a test running unsupervised on the gas fired furnace would not be safe. The manufacture of the test panel was such that it would fit into the aperture of the kiln and the thickness of the panel which extended into the kiln was similar to the wall thickness of the kiln. Hence the test panel formed an integral part of the kiln wall, and the ceramic wool surround provided an effective seal around the edges of the test panel. The ceramic wool and Vermiculux in the non-exposed area of the sample provided a good insulation against loss of heat from the panel edges.

The test panel was manufactured using a "dry fit" method (i.e. the was no mortar or sealant used in the joints between the bricks). Many of the bricks had slight distortions after the firing cycle due to the method of stacking within

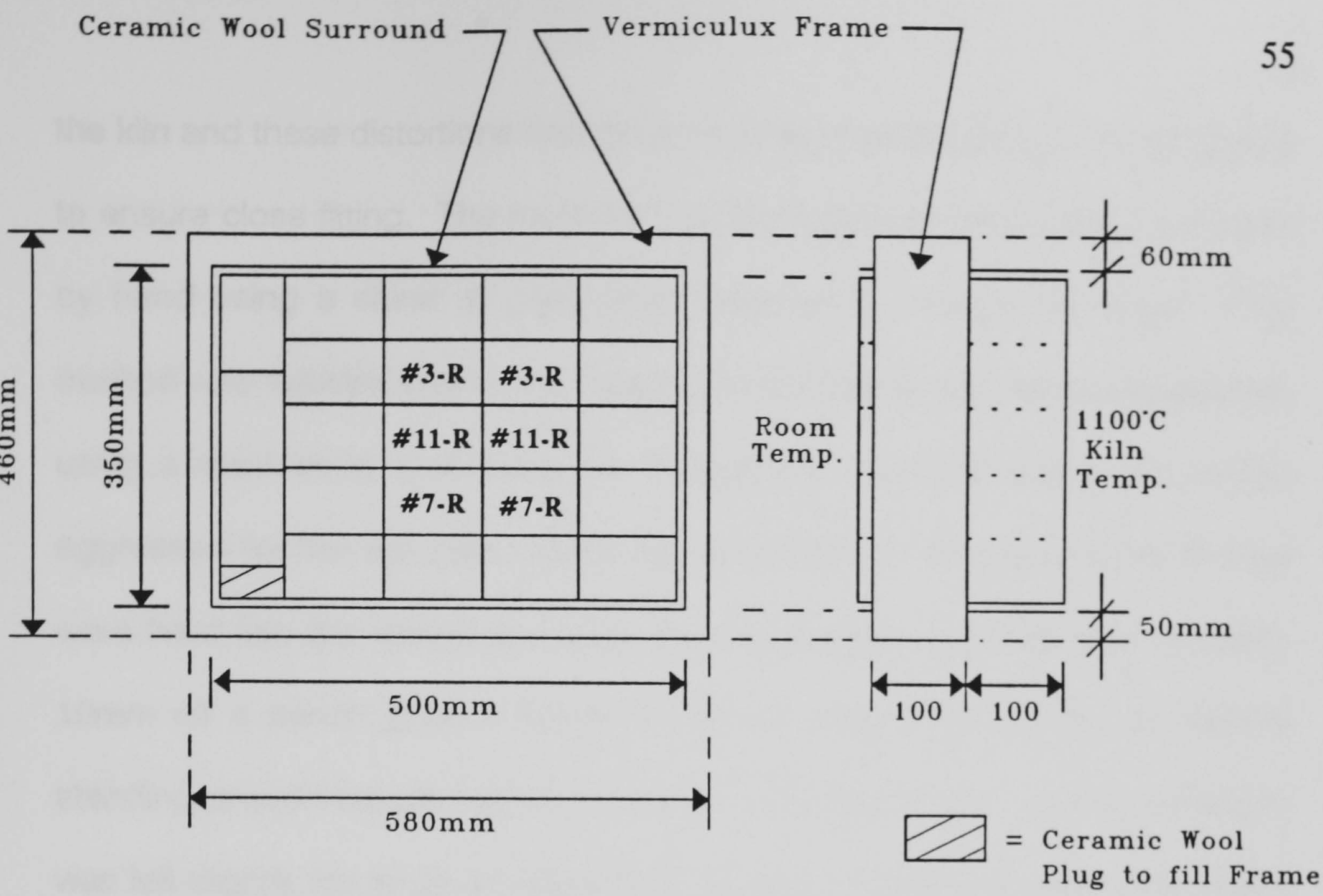


Figure A.5.1 General arrangement of Test Piece

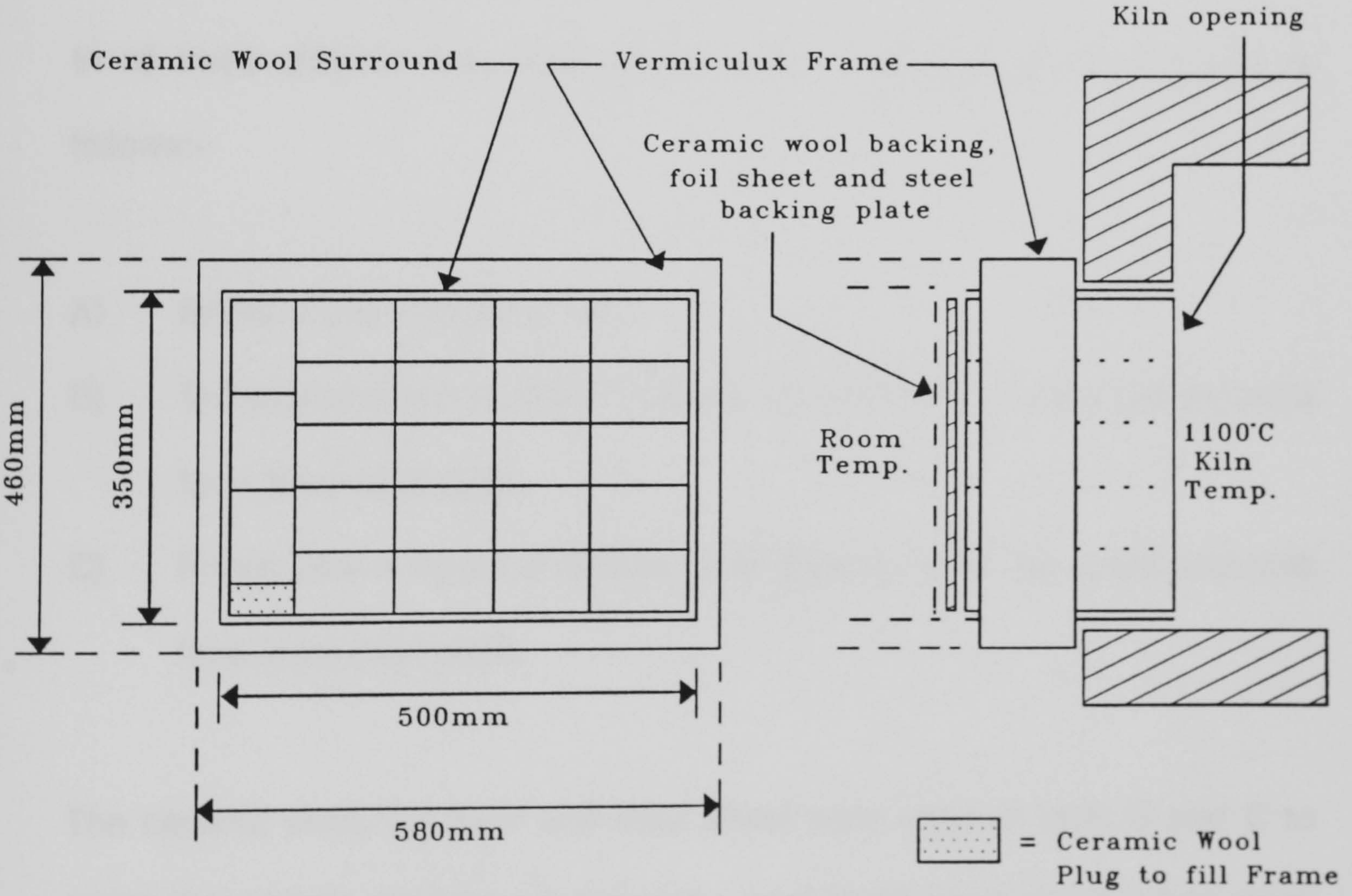


Figure A.5.2 Test Arrangement for Ceramic Wool Backing

the kiln and these distortions had to be removed before using in the test panel to ensure close fitting. The machining of the individual bricks was performed by hand using a sheet of glass paper stapled to a large flat board. This method was suitable due to the nature of the bricks and it was envisaged that using a mechanical machining (i.e. cutting with a circular saw) may be too aggressive for the samples. Once the samples had been sanded to fit they were fitted into the Vermiculux surround which was supported approximately 10mm off a bench (thus if frame is 100mm deep, there would be 100mm standing proud from the top of the frame). The final brick in a row or column was left slightly too large and fitted by forcing the sample between the others already mounted, in this way a very tight fit could be managed.

In all three different test arrangements were investigated, those being as follows:-

A) Bricks alone (210mm thick)

B) Bricks plus 25mm nominal thickness of ceramic wool, foil sheet and cold face 3mm steel plate.

C) Bricks plus 4 layers of 3.5mm thick ceramic wool, foil sheet and cold face 3mm steel plate.

The ceramic wool, foil layer and steel sheet were used in tests B and C to mimic the system used for a full ceramic wool lining used at present in many kilns. The ceramic wool used in test B was excess material taken from the

3mx3m ceramic wool lined test furnace under construction at the University and as such would be directly comparable to the ceramic wool systems, and their quoted figures for cold face steady state temperatures. The ceramic wool used in test C was a medium density blanket of approximately 190kg/m^3, and the foil sheet was the one which was removed from the ceramic lining used in test B. Cross section test arrangements for tests B and C (including kiln opening) can be seen in **figure A.5.2**.

The main samples being tested were located in the centre of the test panel and each had a thermocouple attached to monitor cold face temperature. Other thermocouples were located inside the kiln (mounted through the test panel) and in open air slightly above the test panel to measure ambient temperature. The samples to which the thermocouples were mounted are shown on **figure A.5.1**, and were the best bricks in terms of low density (#3-R), finished condition (#7-R) and (#11-R). Temperature measurement on the cold face of samples was via K type copper disc thermocouples as prescribed in BS476 with 30mm square by 1.5mm thick insulating cover pieces to restrict cold face thermal losses. Ambient temperature and kiln temperature were read with inconnel sheathed 1.5mm diameter K type thermocouples.

The three brick samples under test, and their dimensions etc were as follows:-

#3-R

Perlite	Cement	Water
695g	1069g	1439g

Aggregate:Cement Ratio = 0.65, Water:Cement ratio = 1.35

Average fired density of bricks = 328kg/m^3

Average heat path (hot-cold face) = 211mm

#11-R

Perlite	Cement	Water
829g	1035g	1100g

Aggregate:Cement Ratio = 0.80, Water:Cement ratio = 1.06

Average fired density of bricks = 367kg/m^3

Average heat path (hot-cold face) = 213mm

#7-R

Perlite	Cement	Water
800g	1400g	1500g

Aggregate:Cement Ratio = 0.57, Water:Cement ratio = 1.07

Average fired density of bricks = 380kg/m^3

Average heat path (hot-cold face) = 215mm

Figures A.5.3 and A.5.4 on the following page show the first two tests of the furnace lining material at full thickness with no backing. Problems were encountered where the furnace program crashed overnight, however from inspecting data files it can be seen that the cold face temperatures had reached steady state conditions.

Figures A.5.5 and A.5.6 show the test results for the test panel with backings of 25mm ceramic wool and 14mm ceramic wool respectively. In both cases it

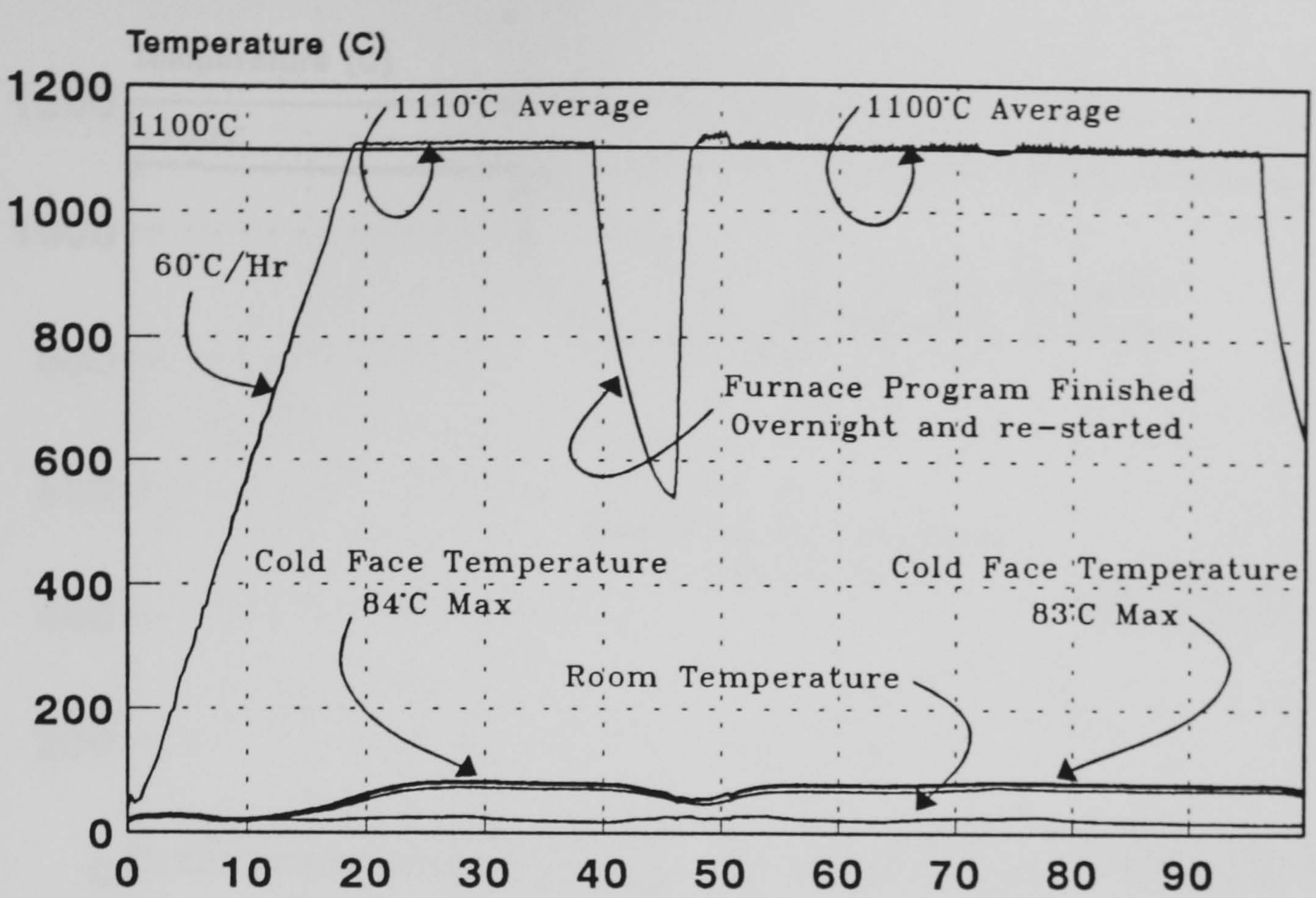


Figure A.5.3 - Kiln Fire Test of New Furnace Lining Material
Samples 210mm thick 24-27th January 1995
Mixes #3-R, #7-R and #11-R, No backing

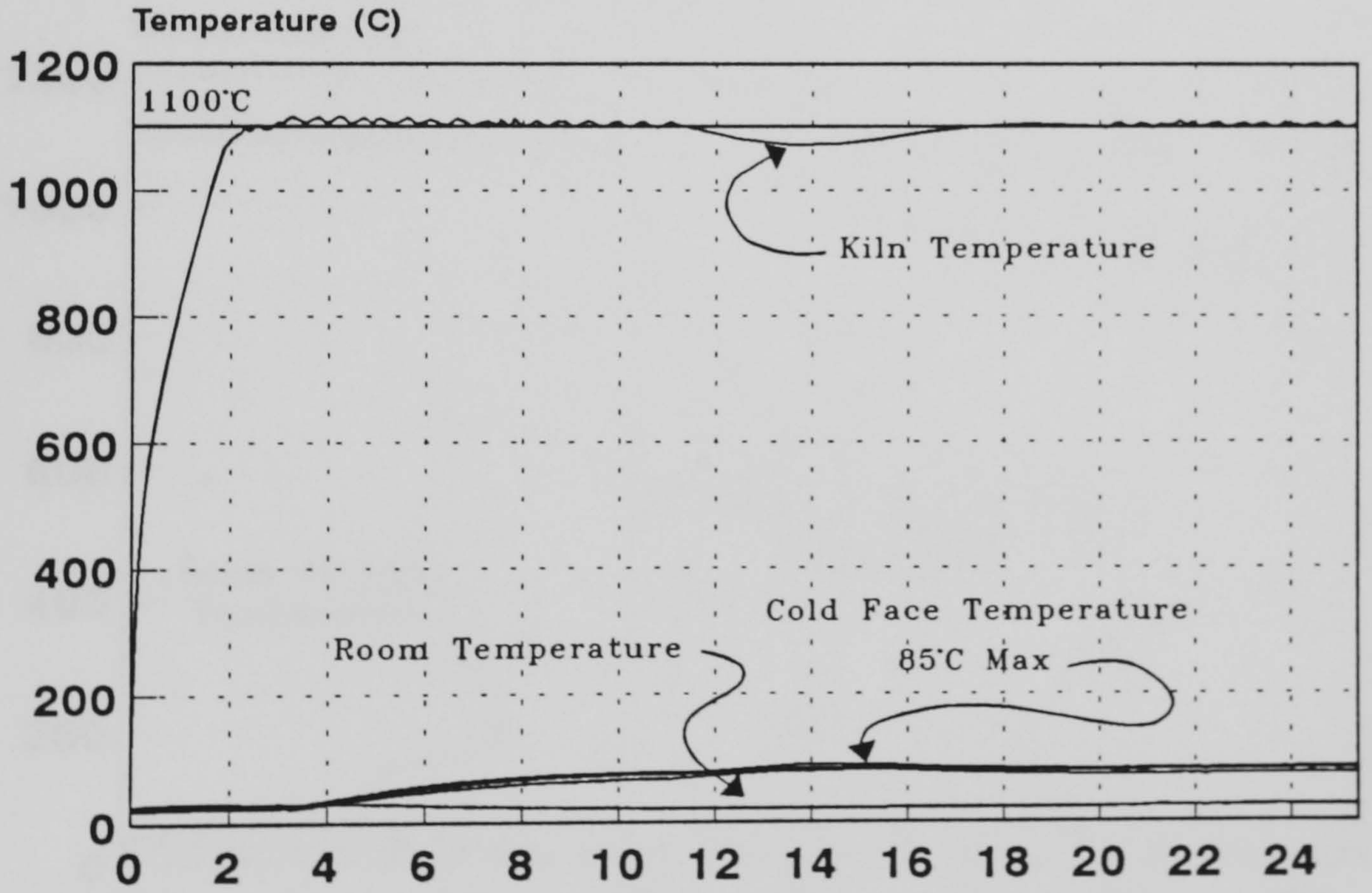


Figure A.5.4 - Second Test of Brick Samples. Carbolite Kiln.
No cold face insulation, 30/01/95-31/01/95
Mixes #3-R, #7-R and #11-R

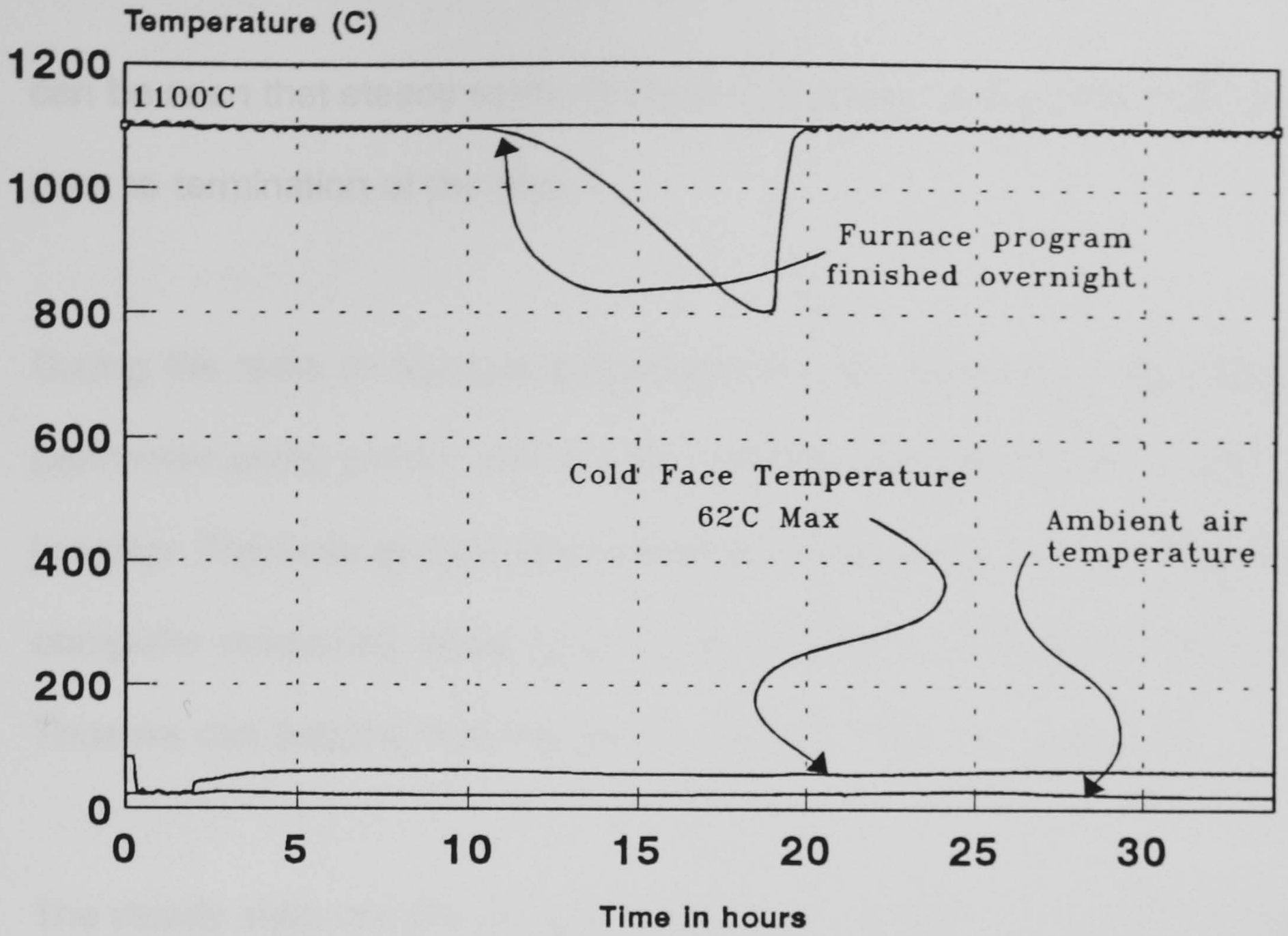


Figure A.5.5 - Test of New furnace Lining Material.
Backing of 25mm ceramic wool + foil layer + steel.
31/01/95-01/02/95.

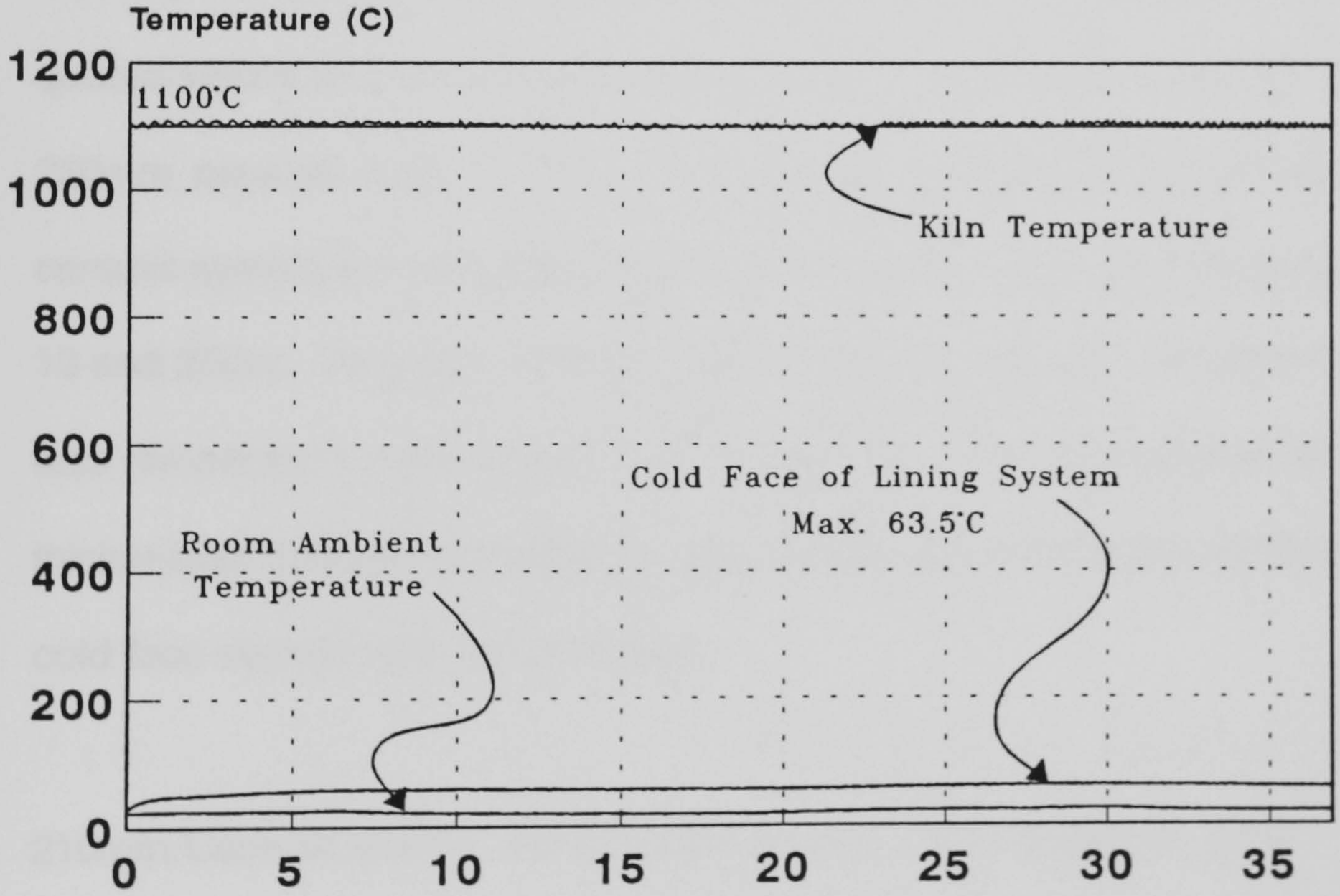


Figure A.5.6 - Test of New furnace Lining Material
4 Layers of 3.5mm thk ceramic wool cold face, plus foil.
02/02/95-04/02/95

can be seen that steady state conditions had been achieved on the cold face prior to termination of the test.

During the tests an independent thermocouple temperature verification was performed using a bare wire K type thermocouple with a well insulated cold junction. The independent measurement corresponded extremely well with the computer measured value (only 0.1°C difference between the two values). Thus we can assume that the results are both valid and accurate.

The steady state conditions on the cold face were 62°C for the 25mm ceramic wool backing and 63.5°C for the 14mm ceramic wool backing. These values are very favourable when compared to a full ceramic wool system where the quoted values from a manufacturer for 200mm ceramic wool is 81°C, and for 250mm ceramic wool of 71°C. The thickness of the backing lining of the ceramic system is not quoted but it is known that the type used varies between 13 and 25mm. Assuming that the 13mm backing was used, and that the cold face temperature measurements are reasonably linear between the two lining thicknesses quoted it is possible to draw a reasonable comparison between the cold face steady state temperatures:

210mm Ceramic wool + 13mm Backing wool + Foil Sheet = 79°C

210mm New Lining + 14mm Backing wool + Foil Sheet = 63.5°C

Thus it can be seen that an improvement of 15.5°C may be possible. It should be remembered that the test performed on the new furnace lining was in an electric heated kiln, and not in a gas fired one, which could possibly alter the results (this needs to be investigated further). However assuming these values to be correct the corresponding heat losses (W/m^2) for the two systems may be interpolated to give the following values:

Ceramic Wool 210mm system = $655W/m^2$

New Lining System 210mm = $450W/m^2$

On a following page a predictive calculation is given for the possible savings in terms of thermal insulation which may be obtained if the new lining material were to be used in the clay brick manufacturing industry. The new material however may have uses reaching much further than this, and other industries to which it may be applied could include the metal industry (smelting and section forming), the whole of the pottery industry and even down to insulating panels for industrial and domestic appliances such as cookers.

It can be seen from before that a possible saving of $200W/m^2$ could be achieved when using the new lining material as opposed to ceramic wool. If a typical brick tunnel kiln is 100m long, with the hottest zone being 30m long, the area of the hottest zone may be $30x(2+5+2)=270m^2$.

If a saving of 200W/m² can be achieved, the possible energy saving per year is area x time x saving:

Total Energy Saving ≈ 270 x (24 x 365) x 200 ÷ 1000 ≈ 473,040 kW hours/year.

Assuming the remaining 70m of the tunnel kiln may achieve a similar saving as for the hottest zone the full potential saving per kiln is 946,080 kW hours/year. This is approximately 32,000 therms. Gas firing is normal, and at the current cost of 25p/therm a saving of £8,000 per kiln per year may be possible.

The easiest way to assume a yearly potential saving for clay brick firing kilns is to assume an equivalent number of kilns. The normal annual brick production is in the order of 3×10^9 bricks and the annual output per tunnel kiln is typically 25×10^6 bricks. This gives 120 equivalent kilns which would result in a potential saving of almost £1m per year if all furnaces were to be re-lined with the new material.

Following the extremely encouraging results given in the full thickness test it was decided that two further tests should be performed, the first on mix #11-R which had a fired density of approximately 370kg/m^3 with aggregate-cement ratio of 0.8, water-cement ratio of 1.06, and the second additional test panel would consist of a new mix #12-R with a fired density of approximately 420kg/m^3, aggregate-cement ratio of 0.65, water-cement ratio of 1.07.

The following graph shown as **figure A.5.7** gives the full test data for the full thickness test panel using #11-R bricks alone with a 14mm ceramic wool foil backed lining and steel backing sheet. The constructed panel was of the same form as used for the multiple sample panel, and after testing for approximately two and a half days a cold face steady state temperature of 59°C was achieved. This cold face temperature corresponded to an ambient room temperature of 24°C. It was noticed however that room temperature had only a very slight effect on the panel cold face temperature. Using the steady state cold face data it is possible to calculate the yearly potential energy saving for clay brick firing kilns as £1.18m pa.

Figure A.5.8 shows the full test data for the full thickness test panel using #12-R bricks only. These denser bricks felt to be much stronger after the firing cycle, and once again a full thickness panel was constructed and tested mounted to the front of an electric kiln. The panel was again lined with a 14mm ceramic wool blanket, foil layer and steel backing sheet. The steady state temperature on the cold face of the panel was observed to be 59°C which would again correspond to a potential yearly energy saving of £1.18m pa for clay brick firing kilns when calculated as shown previously. This cold face temperature was again verified using the independent thermocouple arrangement with a heavily insulated cold junction.

Figures A.5.9 and A.5.10 show the post test condition of the full thickness test pieces for the assorted samples and for the #11-R bricks respectively.

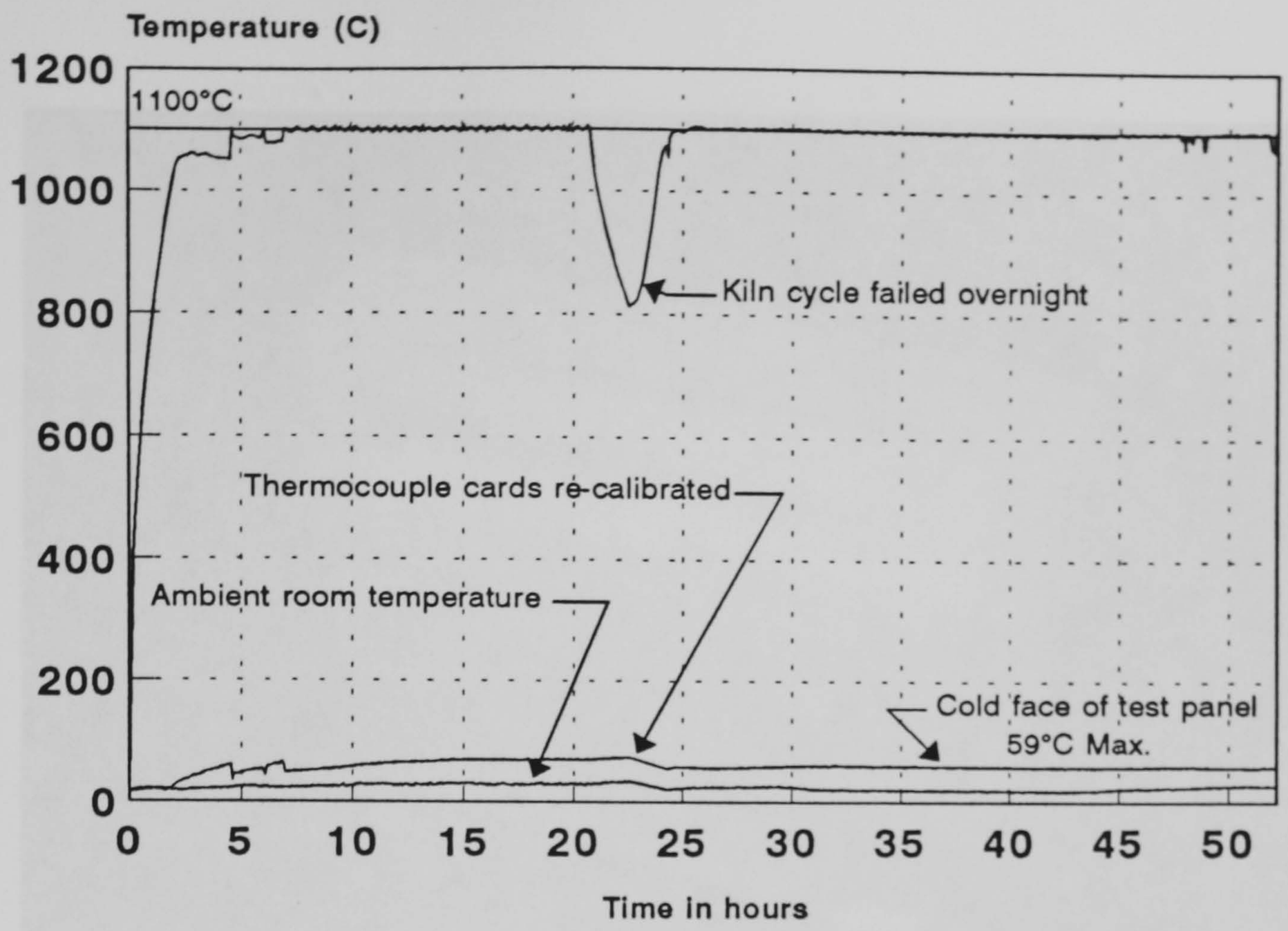


Figure A.5.7 - Full thickness test of #11-R bricks
New furnace lining material development
27-02-95 to 01-03-95

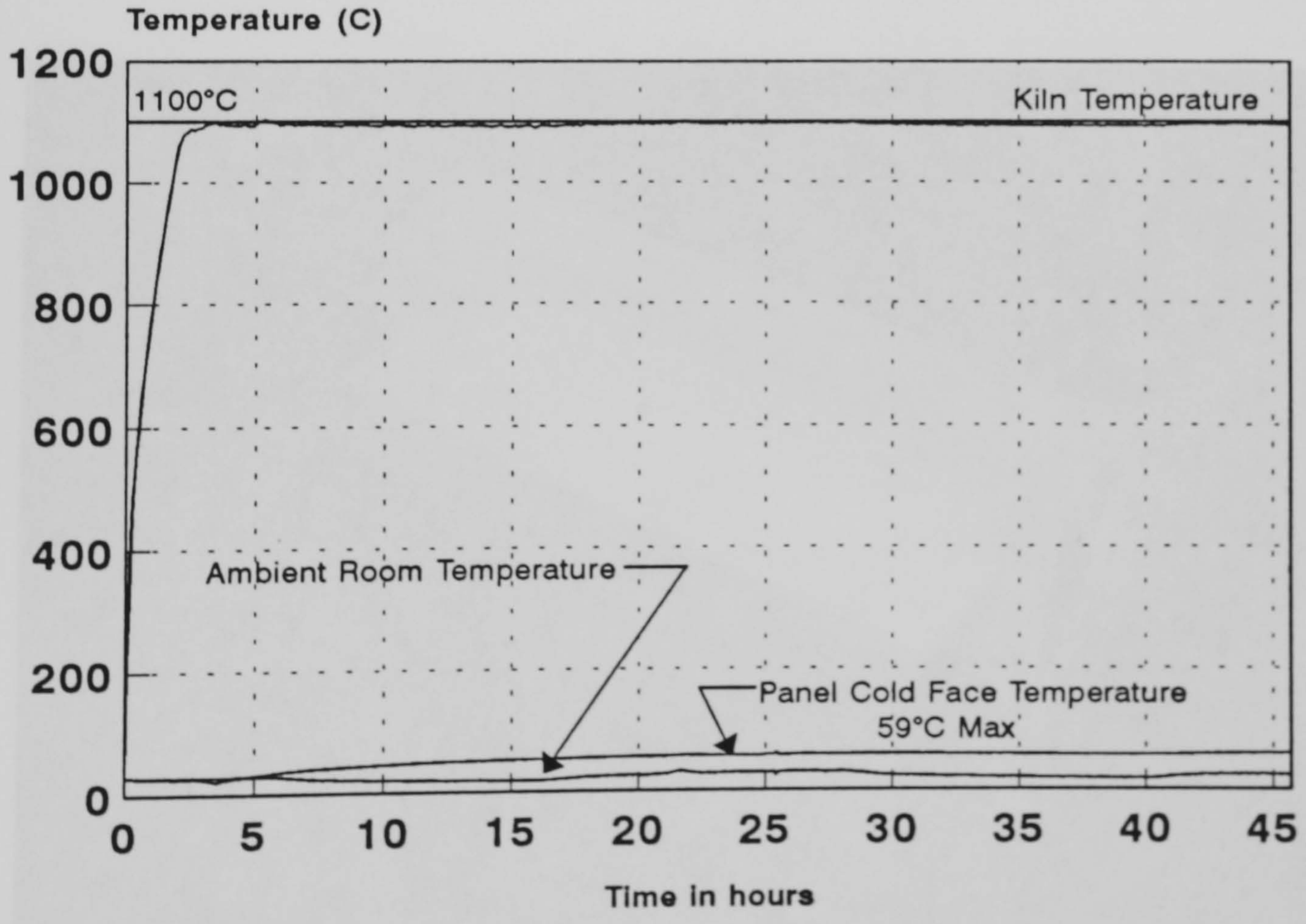


Figure A.5.8 - Full thickness test of #12-R bricks
New furnace lining material development
07-03-95 to 09-03-95

Figure A.5.9 Post test condition of assorted Insuline samples

Figure A.5.10 Post test condition of Insuline #11-R samples

A.5.1 Thermal Conductivity of Insuline

Following the encouraging results from the ad-hoc testing of Insuline bricks it was decided to send representative samples for high temperature thermal conductivity testing. This work was performed at Ceram Research, British Ceramic Research Limited, to BS 1902: 5.5: 1991 and is a NAMAS approved testing facility.

Table A.20 below gives comparisons of cold face temperatures between ceramic fibre anchored modules at 100mm thick with 20mm backing layer of ceramic wool plus foil sheet, and those results obtained in the thermal conductivity test of insuline (notionally 75mm thickness).

Ceramic Wool Anchored Modules @100mm thick + 20mm + foil		Insuline Mix #12R @75mm thick	
Hot Face °C	Cold Face °C	Hot Face °C	Cold Face °C
800	80	805	85
900	92	904	95
1000	104	1002	108

Table A.20 Comparison of Insuline #12R and Ceramic Wool Anchored Modules

Table A.21 overleaf gives the thermal conductivity test results obtained for samples of Mix #12-R tested at notionally 800, 900, 1000, and 1100°C and comparisons with trade figures for the thermal conductivity of ceramic fibre products at differing densities.

Sample	Density kg/m^3	Temperature °C	Thermal Conductivity W/mK
Insuline Mix #12R	431	805	0.172
		904	0.180
		1002	0.195
		1102	0.205
Ceramic Wool	96	800	0.22
		1000	0.30
		1200	0.41
	128	800	0.17
		1000	0.23
		1200	0.36
	160	800	0.16
		1000	0.21
		1200	0.32
Ceramic Fibre Board	290-310	800	0.15
		1000	0.21-0.24
		1200	0.36

Table A.21 High Temperature Thermal Conductivities of Insuline and Ceramic Wool

Figure A.5.11 gives a graphical representation of thermal conductivity as given in table A.21 above.

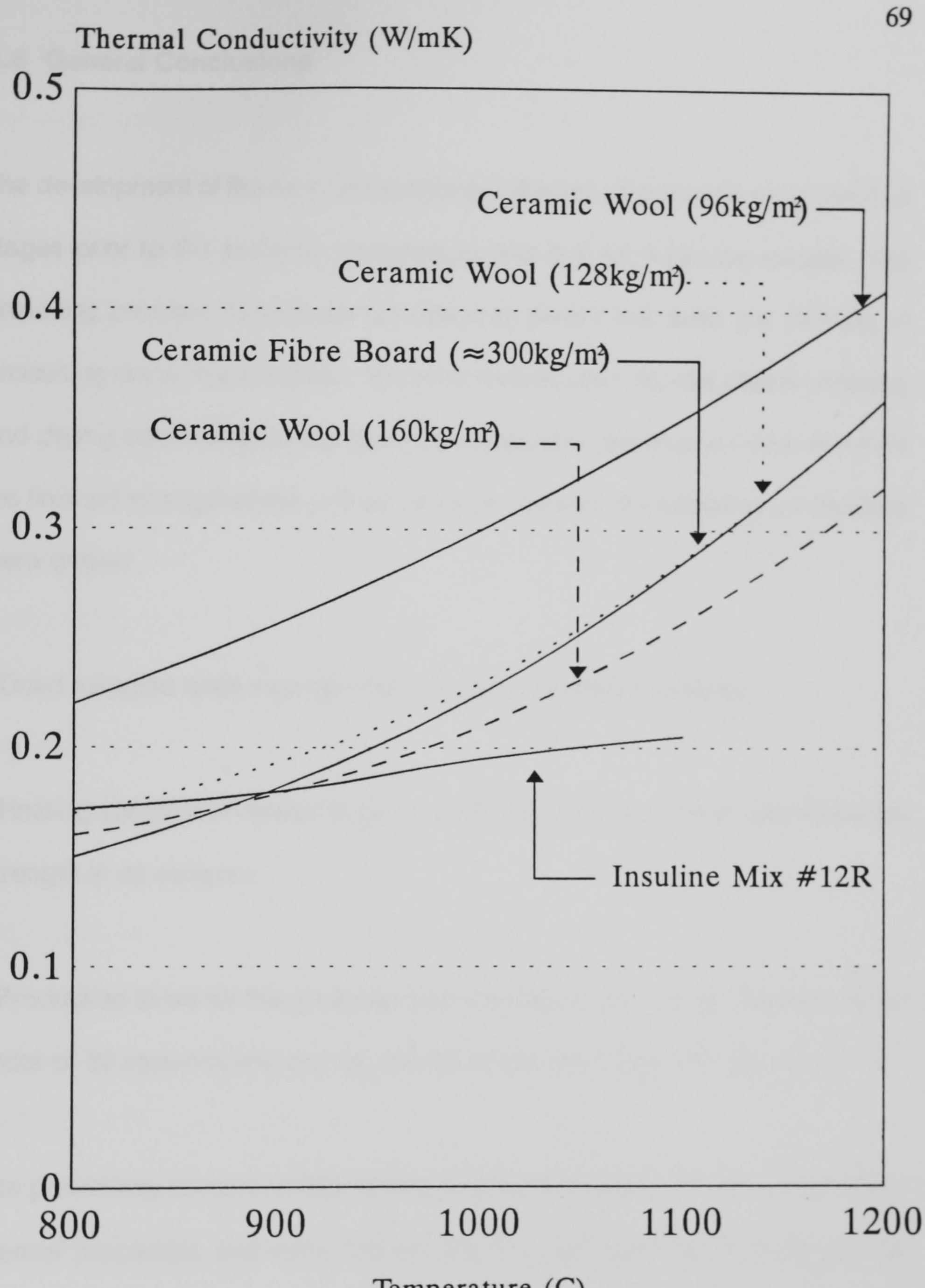


Figure A.5.11 Graphical Comparison of Insuline Mix #12R with Ceramic Fibre Products of Various Densities

A.6 General Conclusions

The development of the new furnace lining material has passed through several stages prior to the ability to manufacture and test full thickness panels. The prevailing problem throughout the research period has been the difficulty in producing crack free samples. The initial investigation into the effects of curing and drying conditions did not give fully conclusive evidence on their effect on the finished strength of the unfired product, however the following conclusions were drawn:

- Dried samples were stronger than damp or re-wetted samples.

- Heating samples in roaster bags to 110°C to dry had a detrimental effect on strength in all samples.

- Production times for the samples could be very short: mixing time was of the order of 30 seconds and curing periods could be of less than 24 hours.

The preliminary furnace testing of the samples showed them to have excellent thermal properties, and numerical analysis showed that if the material were to be developed fully it may be possible for the new furnace lining material to compete with ceramic wool in terms of high temperature performance. It would also have the inherent benefit of being non-fibrous and hence free from the danger of air-borne fibre inhalation and related disease.

The standard method of drying was observed to encourage crack and fault formation within the brick samples. Inspection with a crack microscope showed that these cracks tended to propagate via perlite-perlite boundaries within the sample, however using long careful hand mixing techniques did not solve this problem. The re-heat method of drying was observed to promote a weaker structure which is well known within the ceramics industry to provide a higher degree of thermal shock resistance.

Additions of different grades of perlite, fine grade exfoliated vermiculite, cellulose fibre or mica did not prevent the formation of cracks within the samples during normal drying. The additions were used in an attempt to reduce the shrinkage of the samples during the drying cycle by providing an aid to water loss from the sample. The additions were observed to reduce the severity of the cracks formed both in magnitude and occurrence, but cracks were still present both after the drying cycle and also after the firing cycle. The use of cellulose fibre was also observed to retard the set and cure of the cement and due to difficulty in dispersal of the fibres during mixing large localised shrinkage of samples was observed during the firing cycle.

Increasing the water-cement ratio (whilst maintaining aggregate-cement ratio and density) to ensure full wetting of the cement particles has been shown to have little if no effect on the prevention of crack formation during the drying cycle. Change of density was also found to have no significant effect in crack prevention, either by increasing the volume of mix used per brick, or by

increasing the water and cement content of the sample.

The use of programmed drying cycles (i.e. using slow rates of climb in temperature with or without plateaus during the cycle) was investigated. The effect of the ramped drying cycles was if anything to aggravate the drying cracking problem of the samples.

Bricks of very low density (and assumedly the best thermal performance) appeared to be extremely friable to the touch and had very poor edges after firing. These samples may not be suitable for an eroding or aggressive environment when used alone, however there is the possibility that they may well be ideal in a situation where a high density refractory facing layer is used.

Addition of colloidal silica prior to the first firing cycle has been shown to promote large shrinkage and severe cracking during firing. When the sample is pre-fired and then given a flat wash of diluted colloidal silica no cracking was promoted during the subsequent drying and firing cycle. The addition appeared to increase the overall compressive strength of the samples, but did not effect the friability and edge conditions significantly. Addition of colloidal silica to higher density samples has not been investigated, and may well prove beneficial in terms of sample integrity and strength however, it is more than likely to reduce the thermal resistance of the material, and may well reduce the thermal shock performance of the samples as well.

An increased cement content in fired samples does not appear to be as beneficial as general increase in density. It is thought that the cold condition of the fired higher density samples is mainly effected by the 'glazing' effect of the perlite after its melt temperature has been achieved. The best samples in terms of post-fired condition to date have been #11-R and #12-R with notional fired densities of 370kg/m^3 and 420kg/m^3 respectively.

Full thickness panel tests have been performed on a) assorted samples, b) #11-R samples alone and c) #12-R samples alone. The panels were mounted to the front of an electric kiln which was programmed to run at 1100°C for long periods. The initial test performed of assorted samples showed very good thermal performance with a steady state cold face temperature of 85°C max. This test panel was then modified to incorporate a cold face lining of 25mm ceramic wool, foil and steel backing sheet. The effect of this backing was to reduce the steady state cold face temperature to 62°C. When a similar test panel arrangement was constructed, this time using 14mm (4x3.5mm layers) ceramic wool, the cold face temperature measured at steady state was 63.5°C. This cold face temperature could indicate very large monetary savings in long firing duration kilns when compared to ceramic wool systems of a similar thickness.

Both full thickness test panels using #11-R and #12-R with the 14mm ceramic backing system showed a steady state cold face temperature of 59°C. This cold face temperature was verified using independent measuring equipment.

This would indicate that the density of the sample does not have a great effect on the insulation properties of the samples at these high temperatures. It was also noticed that the cold face temperature

was not particularly sensitive to fluctuations in room temperature.

Post firing testing of cut samples from a brick showed that the fired strength of #12-R was of the order of 0.27N/mm^2. This is very weak compared to the unfired strength, however, when considered fully, it would indicate that if the fired samples were to be stacked, a height of 65m may be reached before the bottom bricks would begin to fail in compression.

Preliminary investigations have shown that the new furnace lining material is capable of withstanding temperatures in excess of 1200°C when Secar 51 cement is used, and temperatures of 1300°C+ may be possible when using a higher grade cement (Secar 71). When the standard samples were heated to 1300°C they melted down, and when cooled they had formed a very strong ceramic. This ceramic had a glossy appearance, and this would tend to indicate that the cement does pass into a glassy phase upon reaching its melt temperature.

A.7 Mix proportions for samples.

Mix A) Perlite 729g, Cement 594g, Water 954g, 885g wet mix per brick for 220x110x70mm samples.

Mix 1) Perlite 667g, Cement 833g, Water 1125g for a 300x300x50mm slab sample.

Mix 1 proportion brick samples were made with perlite 2JS at varying density by changing the mass of wet mix used per brick. Wet mix per brick at differing densities were as follows:-

325kg/m^3 - 868g per brick.

350kg/m^3 - 935g

375kg/m^3 - 1000g

400kg/m^3 - 1068g

Perlite 3JS bricks were made at higher density by reducing the water content of the mix:

Perlite 1193g, Cement 1492g, Water 1333g and for following densities:

600kg/m^3 - 1250g wet mix per brick

700kg/m^3 - 1458g

Perlite 2JL was used to investigate the lower density end of the range and mix proportions were as follows:

Perlite 783g, Cement 979g, Water 1321g and for following densities:

275kg/m^3 - 688g wet mix per brick

300kg/m^3 - 750g

310kg/m^3 - 775g

325kg/m^3 - 813g

350kg/m^3 - 875g

Cellulose fibre and Mica additions were calculated as a percentage of dry mass of the aggregate and cement. The increase in wet mix used per brick was as follows:

310kg/m^3 density, 4% addition - 20g extra wet mix, 2% - 10g extra.

#2) Perlite 695g, Cement 1390g, Water 1872g, 1123g wet mix per 220x110x70mm brick sample.

#3) Perlite 695g, Cement 1069g, Water 1439g, 910g wet mix per 220x110x70mm brick sample.

#4) As #3, but left in mould with extra water added after pressing.

#5) Perlite 2JS 753g, Cement 941g, Water 1270g, 1200g wet mix per 220x110x70mm brick sample.

#6) Perlite 2JL 783g, Cement 1069g, Water 1439g, 940g wet mix per 220x110x70mm brick sample.

#7) Perlite 2JL 800g, Cement 1400g, Water 1500g, 1050g wet mix per 220x110x70mm brick sample.

#8) Perlite 2JL 829g, Cement 1035g, Water 1400g, 988g wet mix per 220x110x70mm brick sample.

#9) Perlite 2JL 829g, Cement 1035g, Water 1300g, 966g wet mix per 220x110x70mm brick sample.

#10) Perlite 2JL 829g, Cement 1035g, Water 1200g, 933g wet mix per 220x110x70mm brick sample.

#11) Perlite 2JL 829g, Cement 1035g, Water 1100g, 900g wet mix per 220x110x70mm brick sample.

#12) Perlite 2JL 884g, Cement 1360g, Water 1455g, 1150g wet mix per 220x110x70mm brick sample.

A.8 List of Main References

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