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A TEST METHOD FOR MEASURING THE FLOW IN REFRACTORY
MATERIALS AT HIGH TEMPERATURES

BY

CHRISTEN KNUDSEN

A

THESIS

submitted to the faculty of the
SCHOOL OF MINES AND METALLURGY OF THE UNIVERSITY OF MISSOURI
in partial fulfillment of the work required for the

Degree of

MASTER OF SCIENCE, CERAMIC ENGINEERING

Rolla, Missouri

1948

Approved by



Professor of Ceramic Engineering

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Introduction

One of the most important properties of refractory materials is the ability to resist stress which may either be directly applied or originate in the material.

The factors which govern the resistance to stress include the modulus of elasticity, the ultimate strength under different types of stress, and the "flow" characteristics which become of importance when the material is stressed at the temperature at which plasticity is developed.

Systematic determinations of the mechanical properties such as modulus of elasticity and ultimate or breaking stress is rather difficult or more or less impossible to carry out at the temperature at which the refractories are used. The reason for this is that refractory materials lose the property of almost perfect elasticity and tend to become plastic at temperatures which are far below the temperatures of fusion. Thus, in case of fireclay and fireclay-silica mixtures, this transition temperature lies within the range 800° - 1000°C.

Once the material has attained some degree of plasticity, no value for the breaking stress strictly comparable with that obtained with the elastic material can be derived, because the ultimate strength of the plastic material is dependent on the rate of loading - a condition which does not apply when the material is elastic.

Apart from the question of obtaining accurate values of the mechanical strength, the behavior of a plastic material under

stress is of considerable practical importance. In the temperature range where the material is elastic it will regain its original form when a stress on it is removed. At the higher temperatures at which it becomes plastic, the material may not show a constant deformation under constant stress, but "flow" or deformation which increases continuously with the time, may take place. If the stress is removed after acting, sometimes, the material will not regain its original form.

It seems evident that a systematic study of the flow characteristics of ceramic materials at high temperatures in general is of paramount importance. Knowledge of the relations between stress, temperature and rate of flow would enable us to predict the behavior of the different materials under service conditions. Such data could be used to determine the safe loading for furnace structures where a maximum allowable deformation during a specific working time and temperature is given.

The data would also find its application in controlling the burning process by preventing of warping caused by improper setting.

Review of Literature

In reviewing the literature on the flow properties of ceramic materials at high temperatures, it was seen that a very extensive work has been carried out by different authors in this field. F. H. Norton ⁽¹⁾ in his book on refractories lists forty-

(1) F. H. Norton, *Refractories*, Ch. 8, p. 437, McGraw-Hill Book Company, Inc., 1942.

six references under the chapter, "Lead-Bearing Capacity of Refractories".

The majority of the work carried out in this field constitutes suggestions of different types of test methods and listing of the results obtained with different refractory materials.

Some authors, however, go more deeply into the matter and try to explain what is going on during the flow period and also try to find some relation between the different rates of flow. Thus, A. L. Roberts and F. W. Cobb ⁽²⁾ ⁽³⁾ ⁽⁴⁾ have developed

(2) A. L. Roberts and F. W. Cobb, *The Behavior of Refractory Materials under Tension at Different Temperatures*, *Trans. Ceram. Soc.*, Vol. 32, pp. 22-24, 1933.

(3) A. L. Roberts and F. W. Cobb, *op. cit.*, Part II, *Trans. Ceram. Soc.*, Vol. 35, pp. 182-208, 1936.

(4) A. L. Roberts and F. W. Cobb, *op. cit.*, Part III, *Trans. Ceram. Soc.*, Vol. 37, pp. 296-311, 1938.

a very nice apparatus for determining the behavior of refractory

materials under torsion at different temperatures. By this test method they have been able very closely to determine the different temperatures at which refractory materials lose their property of almost perfect elasticity and tend to become plastic.

F. H. Norton (5) (6) divides the types of flow into differ-

(5) F. H. Norton, The Flow of Ceramic Bodies at Elevated Temperatures, J. Am. Cer. Soc., Vol. 19, 3, p. 129, 1936.

(6) F. H. Norton, A Critical Examination of the Load Test for Refractories, J. Am. Cer. Soc., Vol. 22, 10, p. 334, 1939.

ent groups which occur in different materials under different conditions. The flow types are summarized in Table I:

TABLE I - Different Types of Flow

<u>Body Structure</u>	<u>Type of Flow</u>	<u>Condition</u>
Crystals and Glass	$V = k_1 F^a$	Plastic
Crystals or Crystals and Glass	$V = 0$	Elastic
Crystals or Crystals and Glass	$V =$	Fractured
Glass	$V = kF$	Viscous

V = flow rate

F = force applied

k_1 = constant

a = varies from 3 to 5

The relation $V = k_1 F^a$ for plastic conditions at constant temperature has been experimentally obtained by loading ceramic materials up to 1000 hours.

For a constant rate of flow the relation is: $\dot{t} = k_2 \log F + b$ where \dot{t} is the temperature, k_2 and b are constants.

Horton indicates that if the constants in the above equations were determined for a number of materials and were found to hold over a sufficiently large field, a few experimental determinations of flow rates would permit the complete evaluation of any particular material.

Refractoriness - Under-Load Tests

Various standardized test methods have been developed in the different countries to determine the load bearing capacity of refractory materials at high temperatures.

In the United States, the A.S.T.M. has worked out a method (7)

(7) A.S.T.M. Designation C-16-41.

where a full sized brick is loaded with twenty-five lbs/in² and heated to a certain temperature, 1300, 1350, and 1450°C, respectively, depending upon the type of material to be tested. The desired temperature is reached after a given heating schedule, and held constant for 1½ hours. Then the furnace is cooled, and the shrinkage of the brick measured.

Objections to this method were put forward by A. F. Dale (8)

(8) A. F. Dale, The Relation Between Ordinary Refractoriness, Under-Load Refractoriness and Composition, Physical and Chemical, of Refractory Material, Trans. Ceram. Soc., Vol. 23, pp. 217, 1924.

and summarized by F. H. Clews and A. T. Green. (9) Dale indicated

(9) F. H. Clews and A. T. Green, Refractoriness Under-Load Tests, Com. Inst. Gas. Engr., No. 103, 1934.

that the use of a single specified loading might give misleading results, according to whether or not it exceeded the "yield value" of the material at the specified temperature. At the

same time he criticized the use of a single fixed temperature of testing because the material might resist deformation as (1) a rigid solid, (2) a plastic solid, or (3) a viscous liquid, and a slight increase in temperature might be sufficient to change the category in which the material could be placed.

F. H. Norton's (1) (5) main objections to the A.S.T.M. load test is that it does not provide suitable data for the designer of furnaces in which the brick is subjected to long, continued temperatures and loads. He therefore suggests a method by which the brick is tested long enough to measure a steady flow rate. He shows an automatically controlled electric furnace in which bricks can be tested over 500 to 1000 hours.

In Britain (10) and in Germany (11) the load test is con-

(10) A. F. Dale, Some Fallacies to be Avoided in the Standardization of Any Method of Testing the Load-Bearing Capacities of Refractories at High Temperatures; and a Suggested Method for Standardization, Trans. Ceram. Soc., Vol. 24, pp. 216, 1924-25.

(11) Deutsche Industrie Normen (D.I.N.) 1064.

ducted by cutting or drilling out of the brick a small test piece. The test piece is given a predetermined load and heated up in a furnace at a constant rate. The temperature is increased until the test piece has yielded 20% of its length. The shrinkage of the test piece magnified 10 times is automatically recorded during the experiment and plotted against the temperature.

F. H. Clewe and A. T. Green ⁽⁹⁾ have given a critical examination of the rising temperature load test. As a relatively rapid means of examining or comparing the behavior of refractory materials under load, the practical value of this test cannot be doubted. It must be admitted, however, that the test only provides limited information about the behavior of the material under certain standardized but arbitrary conditions. Furthermore, since the test piece is subjected to both stress and a rising temperature at the same time, the true effect of the stress on the material at any given temperature may not be indicated by the deformation curve obtained. In the test, the two variables, temperature and time, operate simultaneously, the results of which must be inconclusive.

Another objection to the test is that comparatively small variations in the experimental conditions, especially in the rate of the temperature rise, will effect serious errors in the final results.

D. Petit ⁽¹²⁾ has suggested a test method from which re-

(12) D. Petit, A New Method of Assessing the Stability of Refractory Structures, Trans. Cer. Soc., Vol. 38, pp. 313, 1939.

sults he, by some degree of accuracy, can determine the conditions under which a new installation may be operated. The method is the same as the common European rising temperature method with the exception that he runs a series of experiments and uses different pressures for each experiment.

As in most of the load tests, the time element is not considered in this case, a factor which is of most importance in an installation.

The plasticity of refractory materials at high temperatures has a special interest in the stopper and nozzle refractories used in steel ladles in the steel industry. L. G. Ekholm and L. D. Hower, Jr. ⁽¹³⁾ emphasize the fact that the finest quality

(13) L. G. Ekholm and L. D. Hower, Jr., *Pouring Techniques and Their Effects on Casting-Pit Performance*, A.I.M.E. Proceedings of the National Open Hearth Committee, Vol. 28, p. 120, 1945.

of steel can be reduced to scrap by improper pouring. Good pouring depends mainly on such things as a proper opening in the bottom of the ladle to prevent splashing of the stream, and a proper control over the rise in the ingot. These two factors are both governed by the properties of the nozzle and the stopper head refractories.

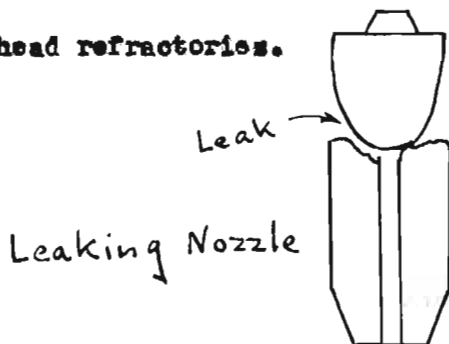


Fig 1.

During the pouring the stream of molten steel will give a very abrasive action on the nozzle and some of the ceramic material will be washed away, especially along the edge of the nozzle. When the pourer wants to shut off the stream, he will get a leak as shown in Figure 1. To prevent this leaking and to provide an effective shut off, the nozzle is often

made of a medium refractory material which will be plastic at the pouring temperature. This plasticity will allow the stopper head to be squeezed into the nozzle and give an effective shut off of the stream.

There are no standards or regulations given about how refractory or how plastic these nozzles ought to be at the pouring temperature of the steel. There is also not given a proper test by which this type of plasticity can be determined in a comparatively simple way.

Outline of Work

In the present work there was developed a simple test-method to determine the flow in ceramic materials at elevated temperatures. The test worked on the principle of constant temperature vs. rising pressure. The full sized test brick was heated up to a given temperature which was held constant. The pressure was applied on the brick at a constant rate, giving at last a maximum pressure of 100 lbs/in². The shrinkage of the brick was measured with short intervals and plotted against the pressure.

Different types of fireclay bricks, and steel ladle nozzles, were tested and the results compared with the properties of the tested materials.

A series of flow curves where the rate of flow was measured in each case for a constant temperature and pressure was taken as explained by F. H. Norton. (5) The results were compared with the results obtained by the increasing pressure test method. These experiments were carried out first to indicate the temperature at which the rate of flow would be most suitable for the increasing pressure test.

Procedure

Apparatus:

There has been designed and built for the experiment a gas fired furnace as shown in Fig. II and Fig. III. The circular furnace is built up of a 7" thick wall of refractory insulating brick. On the inside of the insulators is rammed up a 2" lining of alumina ramming mix to withstand the high temperatures. The furnace is placed about 35" above the floor on a rack made of beams and rests on a reinforced concrete slab. The heat is provided by a North American Fan Mixer, Type ERB-405 and the flame is blown in tangentially.

The test brick is placed in the middle of the furnace between two 6" diameter silicon carbide cylinders. To prevent sticking a thin layer of pulverized flint is sprayed between the brick and the SiC cylinders in each experiment.

The pressure on the test piece is provided by a Hanna Air cylinder, Type LSM, with 12" stroke.

For other load tests the pressure in this case is applied from the bottom of the furnace. Supporting U-beams on the top and sides hold the ~~the~~ Test brick and SiC-cylinders in place.

Compared with the usual loading equipment where weights are used, the air cylinder shows a big advantage. The pressure can thus easily be regulated by means of a manometer and a control valve. For getting uniform pressure, the air cylinder is connected with an air tank which has a damping effect on the variations in the pressure of the air line.

Fig 2

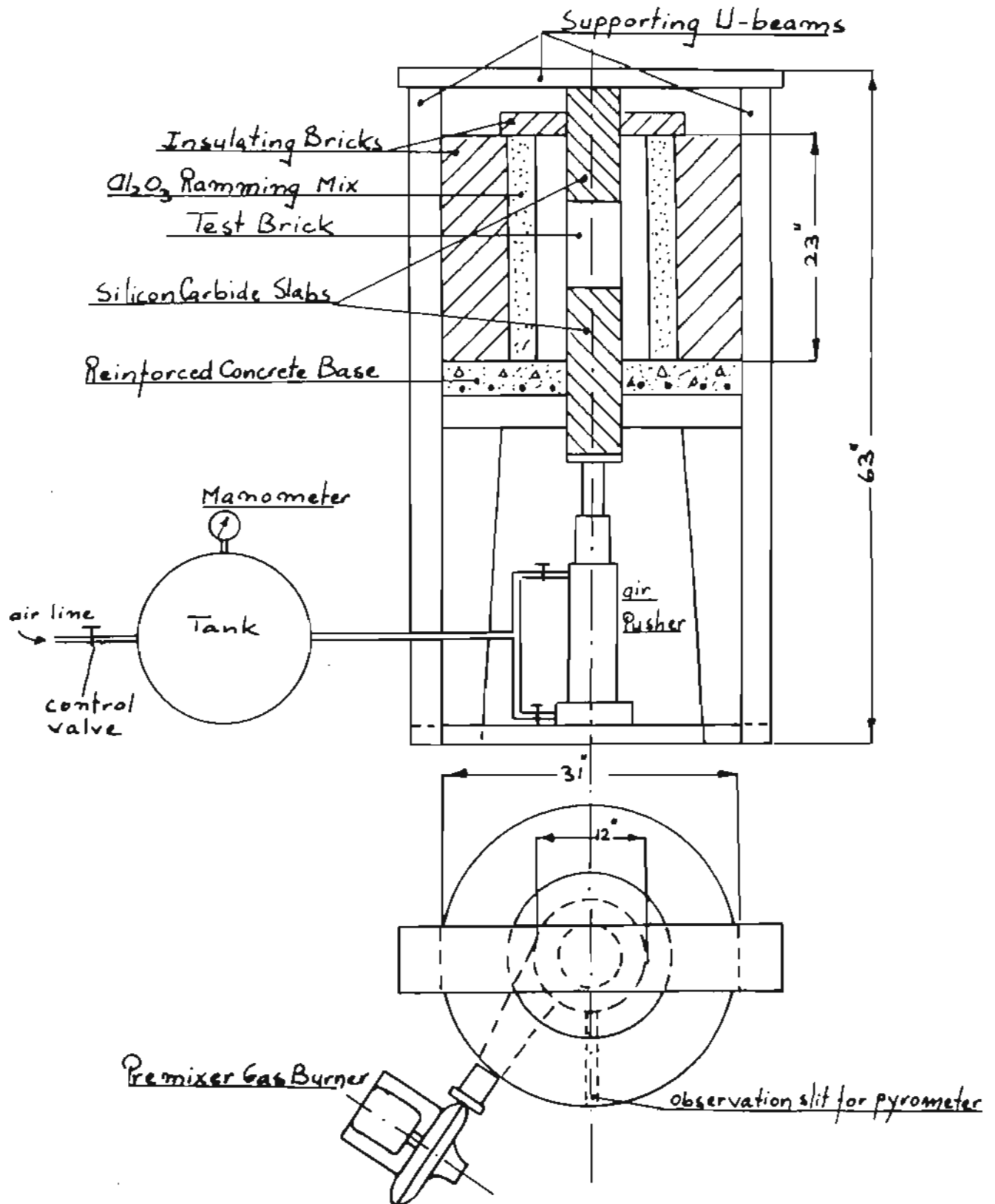
Load Test Equipment



Fig. 3 Load Test Equipment

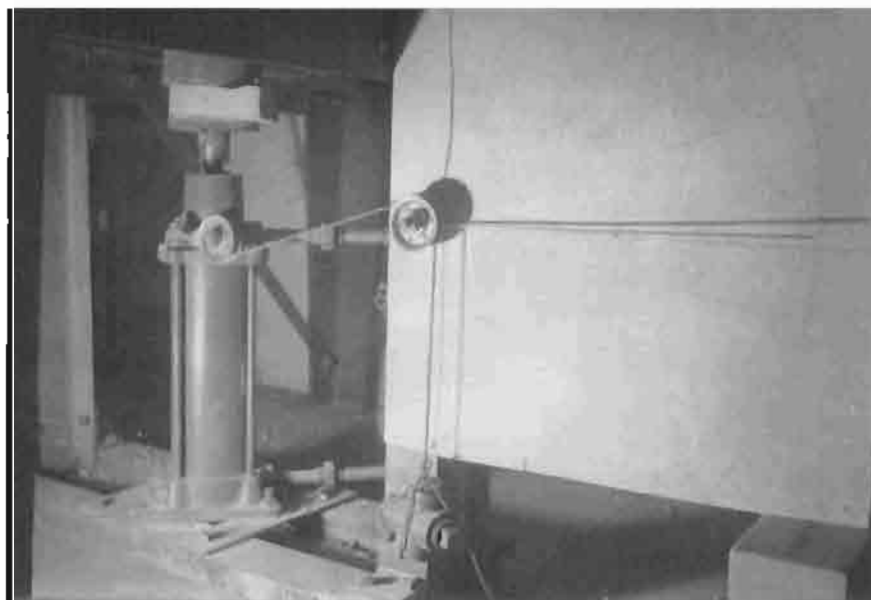


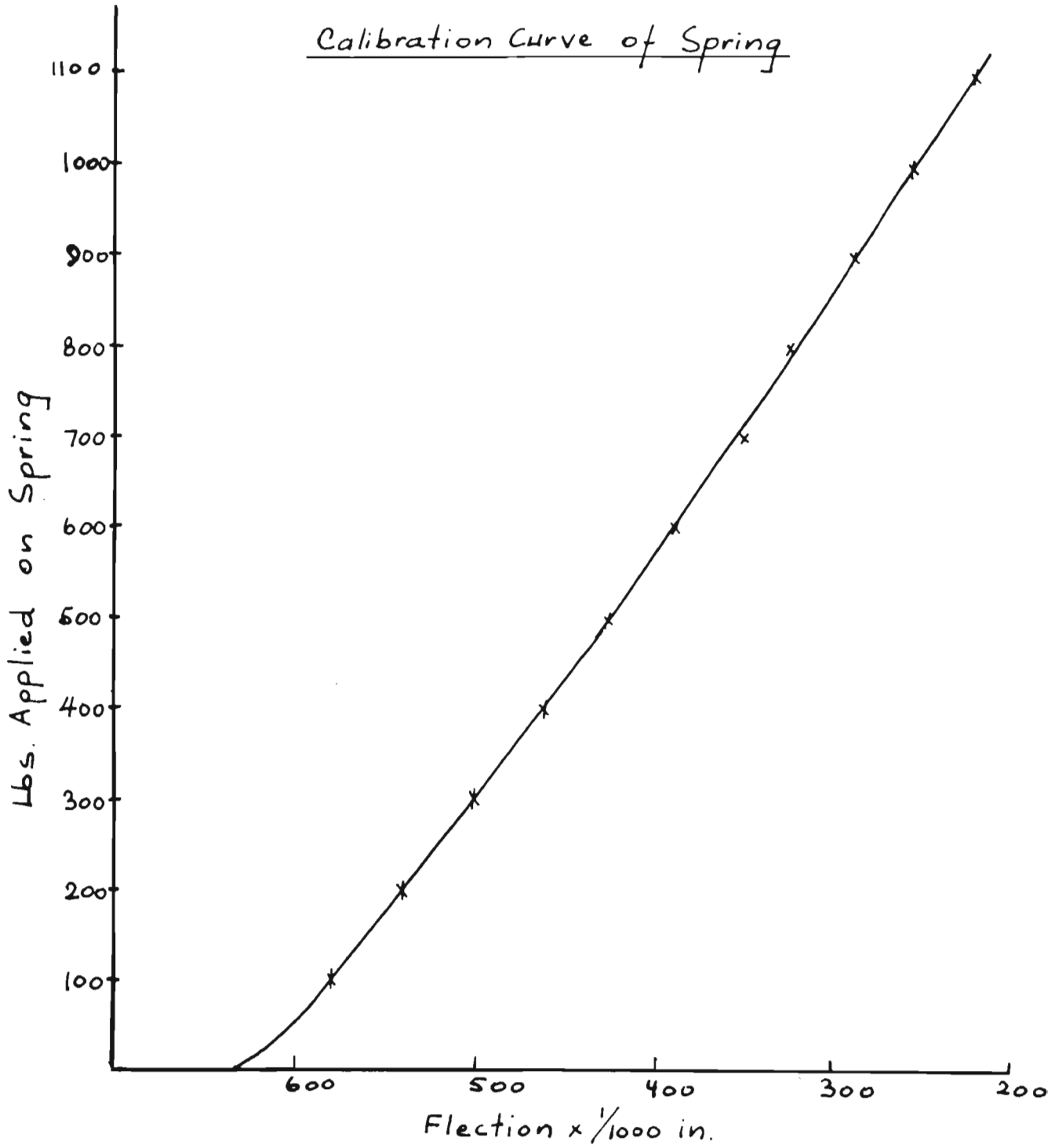
Fig. 5 Shrinkage Indicator

The air pusher and the manometer were calibrated by means of a spring. The deflection of the spring was first calibrated in a press where different known pressures were applied. The deflection was measured by means of a micrometer screw and plotted against the pressure as shown in Fig. IV.

The temperature was determined by means of an optical pyrometer.

The shrinkage of the test brick was taken by a simple arrangement consisting of a string which transfers the movement of the pusher via two pulleys (with ball bearings) over on an indicator as shown in Fig. V. The movement of the pusher was in this way magnified fifteen times.

Fig 4



Test Methods

Increasing Pressure Method:

For measuring the shrinkage under increasing pressure at constant temperature, the test brick was heated up to 1400°C according to the schedule shown in Fig. VI. The rate of heating selected was the most convenient for the control of the type of gas burner used.

When 1400°C was reached, this temperature was held as constant as possible. After ten minutes a pressure of five lbs/in² was applied. The pressure was then continuously increased at a rate of ten lbs/in² per three minutes up to a maximum of 100 lbs/in² increase in pressure beginning at twenty lbs/in².

For each brick the shrinkage was plotted against the pressure.

The reproducibility of the method is good as long as the temperature is kept constant within $\pm 10^\circ\text{C}$ during the test. The heating schedule up to 1400° must also be followed quite closely above 1000°C, due to glass formation in the brick. The bricks tested also must have the same porosity to give the same results.

Fig. VII shows two sets of shrinkage curves for four bricks, two bricks of each kind. The maximum difference in the shrinkage at the same point are about .3%. Usually the results are more close for stiff mud bricks than for dry pressed.

Fig 6

Time Temperature Curve
for Load Test

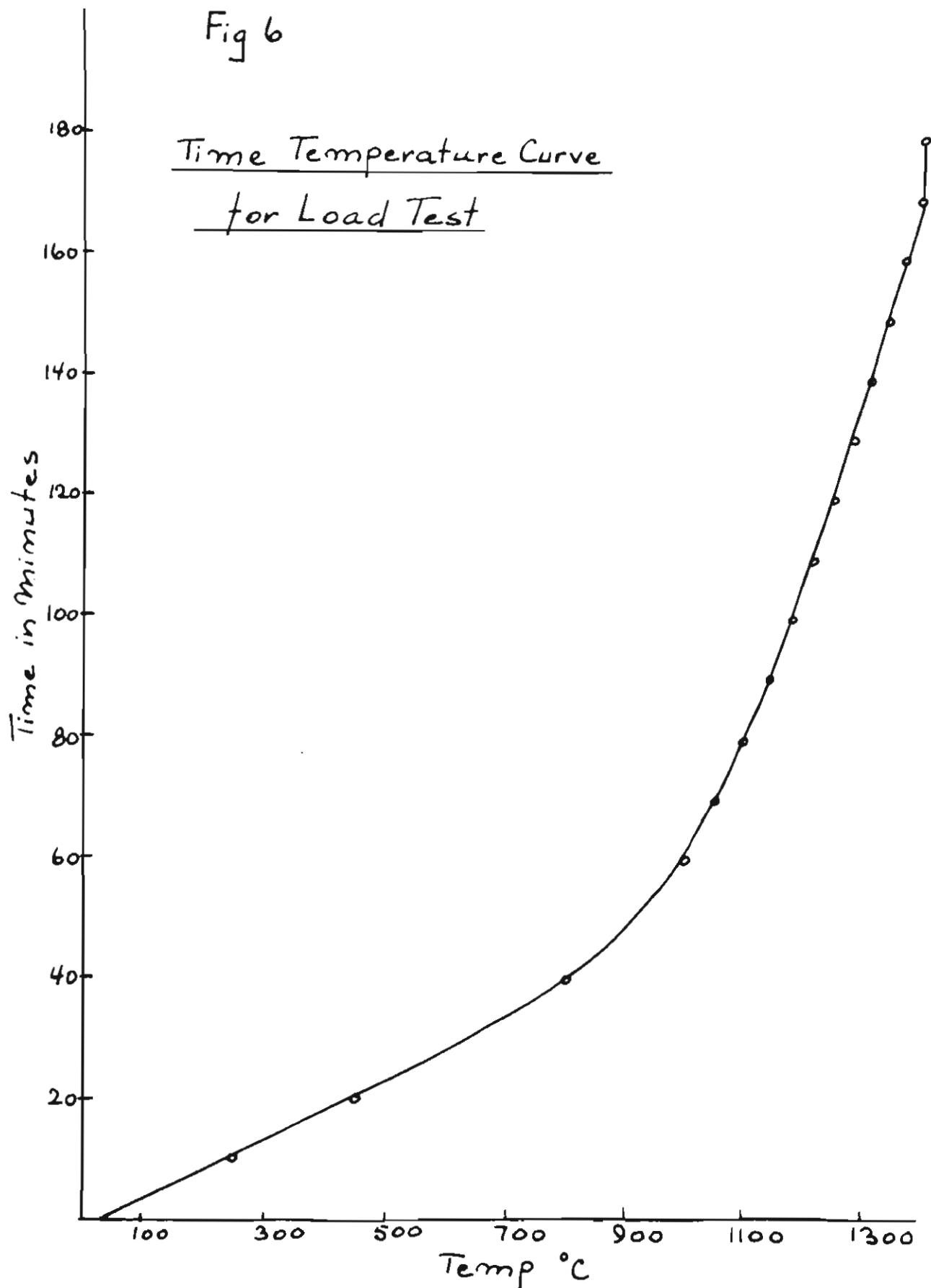
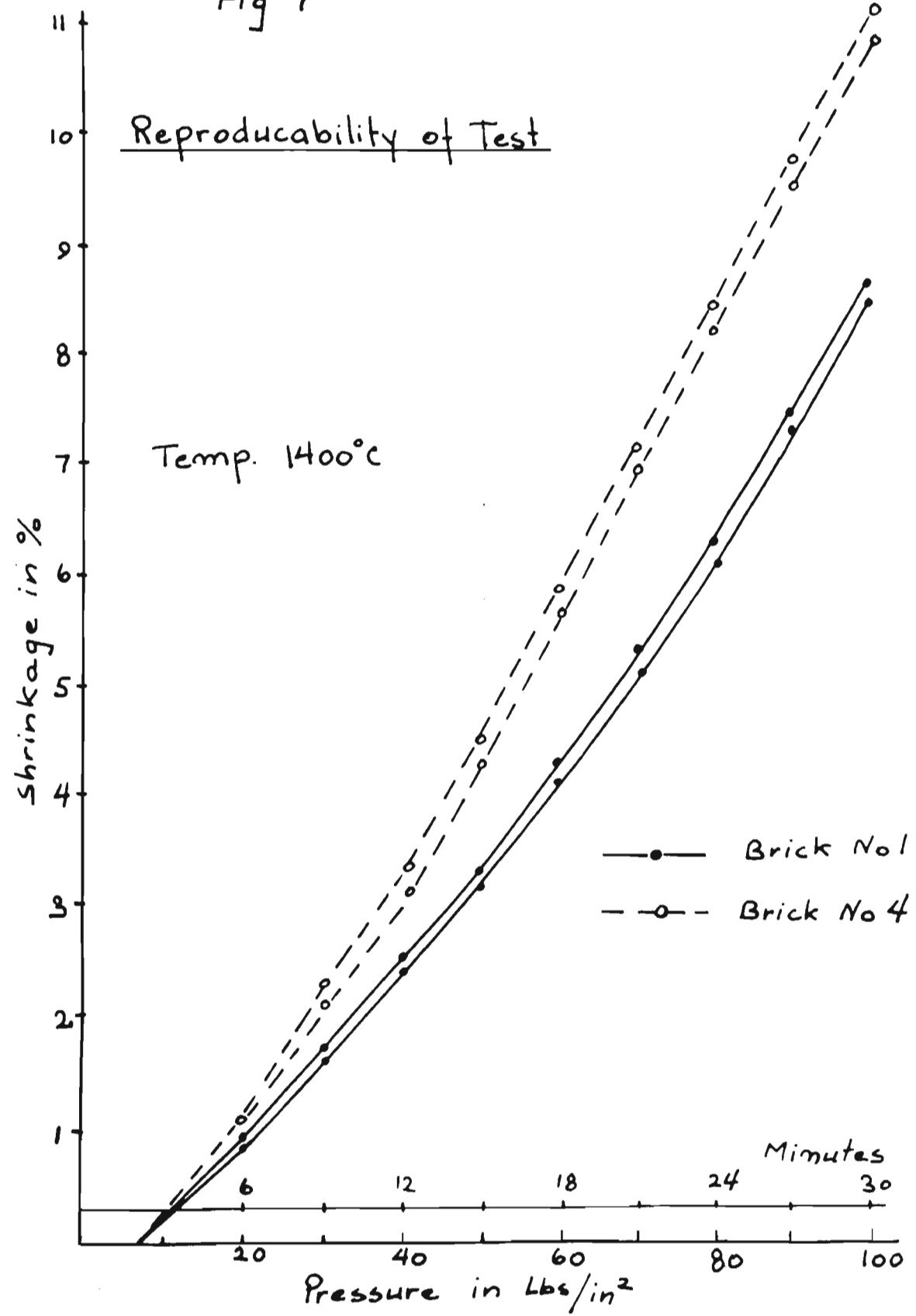


Fig 7



Constant Pressure and Temperature Method:

The brick was heated up at a fixed rate to 1300°C and 1400°C. For each temperature were applied three different pressures, 50, 60 and 100 lbs/in². For each of the total of nine experiments, the pressure and temperature were kept constant until the brick showed a constant rate of flow, usually requiring about 100 minutes. The shrinkage of the brick was recorded every ten minutes, except in one case where it was necessary to take the readings every five minutes because of the rapid flow. In this case the shrinkage of the testbrick was plotted against the time.

Materials Tested

As materials to be tested, stiff mud and dry pressed firebricks of Intermediate Heat Duty and High Heat Duty (14) quality

(14) A.S.T.M. Designation G27-41 for Dissification of Fireclay Refractories.

were selected. The bricks were manufactured by the three largest firebrick producers in Missouri. In addition to these bricks, two Super Duty bricks were tested, plus two steel ladle nozzles.

In the method for measuring constant flow rates, was used a Brand A Intermediate Heat Duty stiff mud brick.

The same experiment was also carried out at 1400°C with a Brand A Intermediate Heat Duty dry pressed brick. The physical data on the different types of bricks tested is given in Table II.

TABLE II

Physical Properties of Materials Tested

No	Brand	Quality	Type	PCF	% Porosity	% Shrink. under Load Test at 1350°C *
1	A	Interm. Heat Duty	Stiff Mud	32	11.	1.28
2	B	"	"	32	10.	2.15
3	C	"	"	31/32	15.	
4	A	"	Dry Pressed	32	20.5	2.27
5	B	"	"	31	17.8	4.45
6	C	"	"	31/32	21.7	
7	A	High Heat Duty	Stiff Mud	33	13.5	2.32
8	B	"	"	33	16.	2.70
9	C	"	"	32/32	13.6	
10	A	"	Dry Pressed	33	21.	1.47
11	B	"	"	33	19.	3.72
12	B	Super Duty	"	34	17.5	2.40
13	B	"	"	34	17.7	(at 1450°C) 4.90
14	S	Nozzle	Stiff Mud	16**	16.1	(at 1450°C)
15	CC	Nozzle	"	28	17.2	

* Figures given by producer

** The cones bloated during test, hard to set accurate P.C.F.

Results

The Constant Rate of Flow Test:

The results from the constant rate of flow experiments are given in Table III and the shrinkages are plotted against time in Fig. 8 and Fig. 9.

As it will be noticed, the rate of flow is always largest in the beginning, just after the load is applied. Then, after a while, the curves flatten out and become a straight line which means that the rate of flow in the brick is constant. The curve for the brick heated to 1300°C and loaded with 30 lbs/in² is not given as the rate of flow at this low pressure and temperature was almost zero.

From the linear part of the curves were taken the different flow rates (% shrinkage per hour). The flow rates at the different temperatures are plotted against the logarithm of the pressure giving straight line curves as shown in Fig. 10.

These curves are in good relation to the results obtained by F. H. Norton. (5) The curves are almost parallel except the one 1300°C. This is, however, as good as can be expected as long as the work is carried out with such a variable material as commercial firebricks. It might be mentioned that the flow curve for brick No. 4 does not necessarily have to be parallel with the flow curves for brick No. 1. Both bricks are of Intermediate Heat Duty quality, the only difference between them being that No. 1 is made by the stiff mud method and No.

TABLE III

Shrinkage of Bricks Loaded at Different Temperatures
and Pressures

Time in Min- utes	% shrink. brick No. four										
	at 1400°C		at 1300°C			at 1350°C			at 1400°C		
	30 psi	60 psi	60 psi	100 psi	30 psi	60 psi	100 psi	30 psi	60 psi	100 psi	
10	.9	1.4	.27	.49	.25	.76	1.12	.36	1.39	1.35 2.70 4.05	
20	1.9	3.2	.58	.85	.54	1.21	2.24	.76	3.00	4.75 5.25	
30	2.5	4.7	.90	1.25	.74	1.75	3.05	1.12	4.08	5.28 7.40	
40	3.	5.7	1.12	1.57	1.10	2.24	3.72	1.39	4.98	8.30 8.95	
50	3.2	6.5	1.26	1.80	1.44	2.74	4.45	1.62	5.70	9.44 9.95	
60	3.5	7.4	1.37	1.98	1.85	3.14	5.15	1.84	6.50	10.30	
70	3.8	8.2	1.48	2.15	2.10	3.64	5.96	2.06	6.90		
80	4.	9.1	1.57	2.34	2.20	4.08	6.71	2.24	7.51		
90	4.2	9.8	1.66	2.52	2.29	4.55	7.50	2.44	8.11		
100	4.4	10.6	1.73	2.70	2.42	4.98	8.20	2.66	8.75		
110					2.56						
120					2.69						
Flow Rate in % per hr.	1.61	4.45	.53	1.1	.8	2.8	4.4	1.25	4.	6.2	

Flow Curves for Brick No. 1

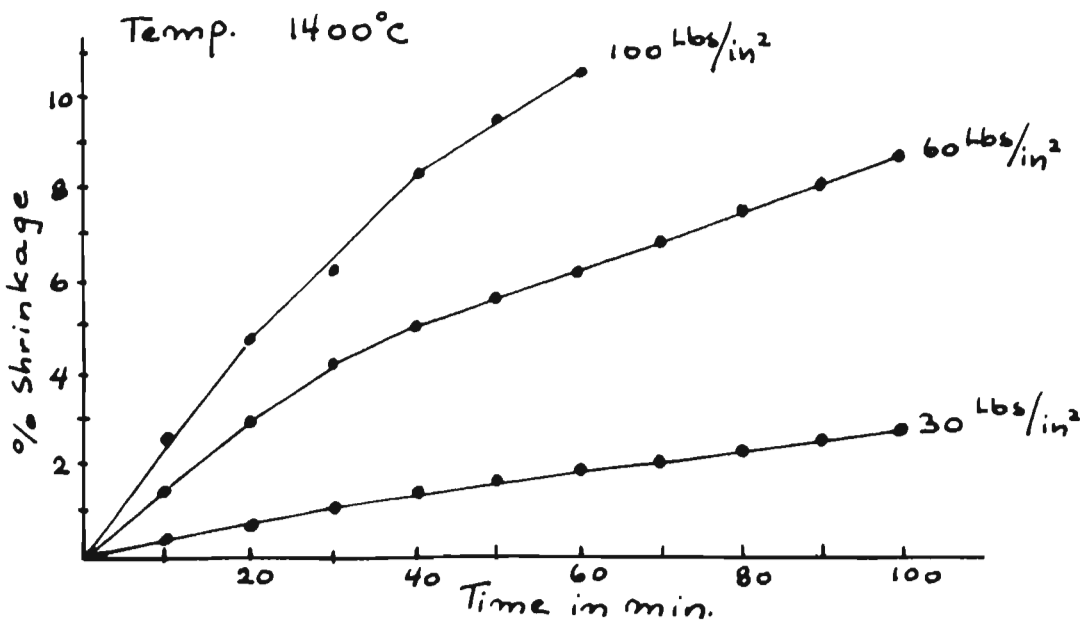
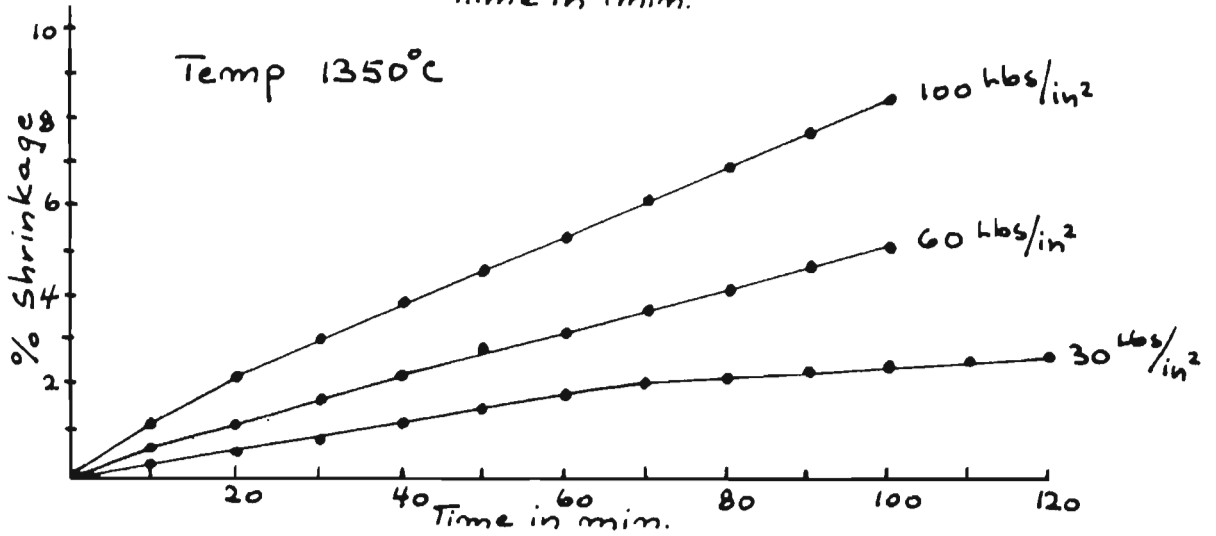
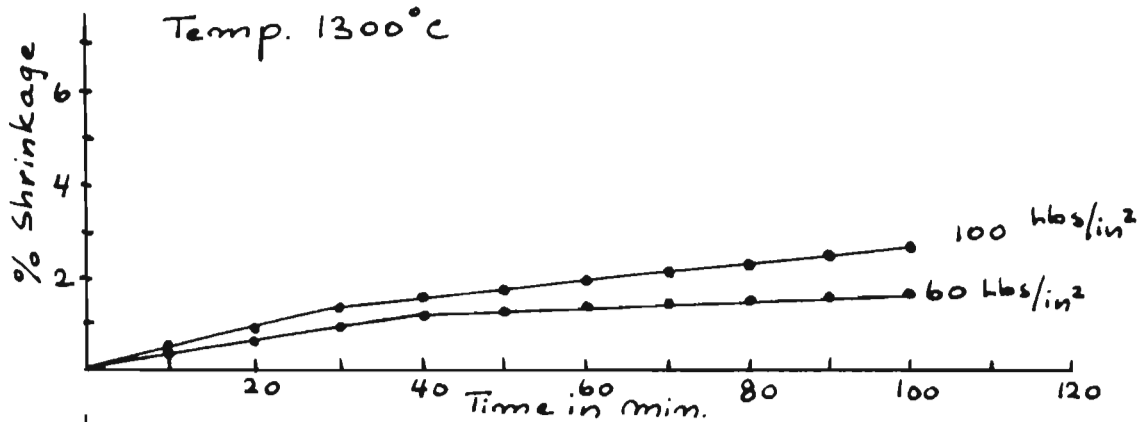


Fig 9

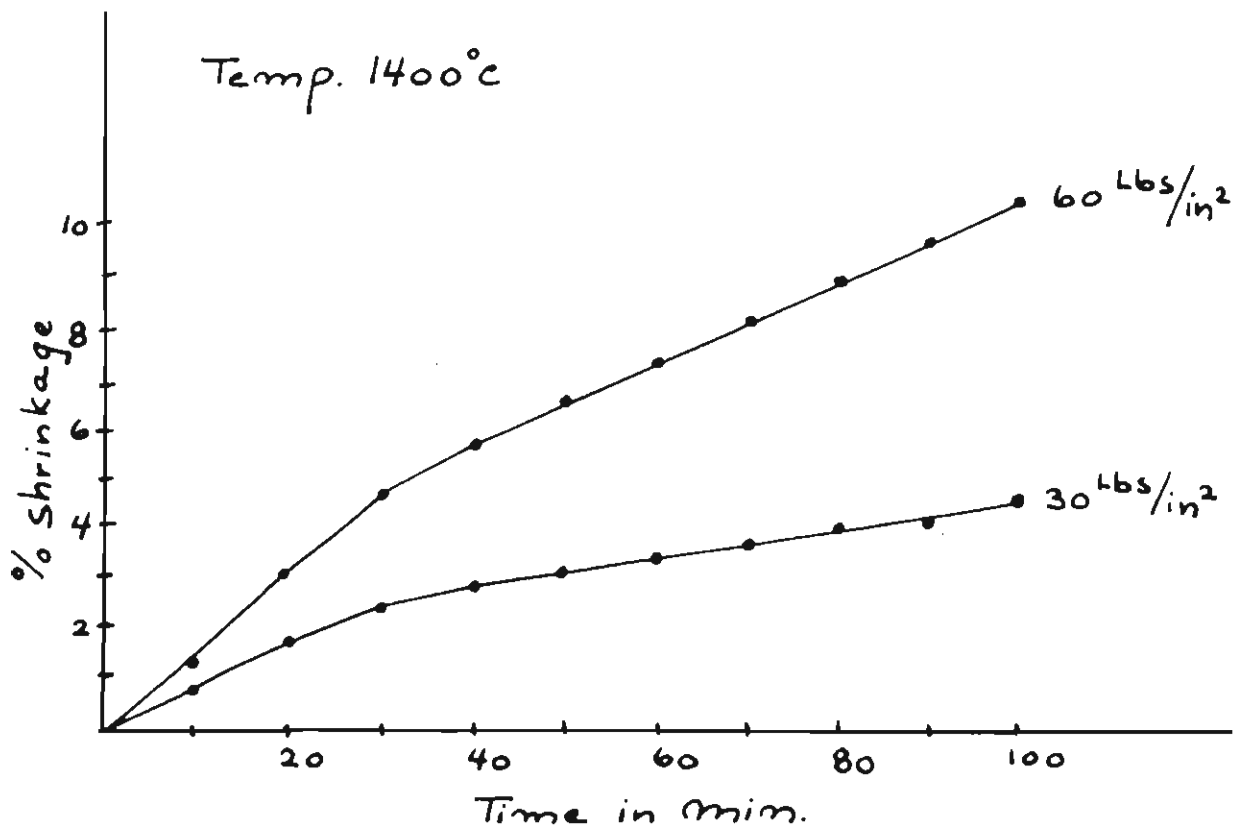
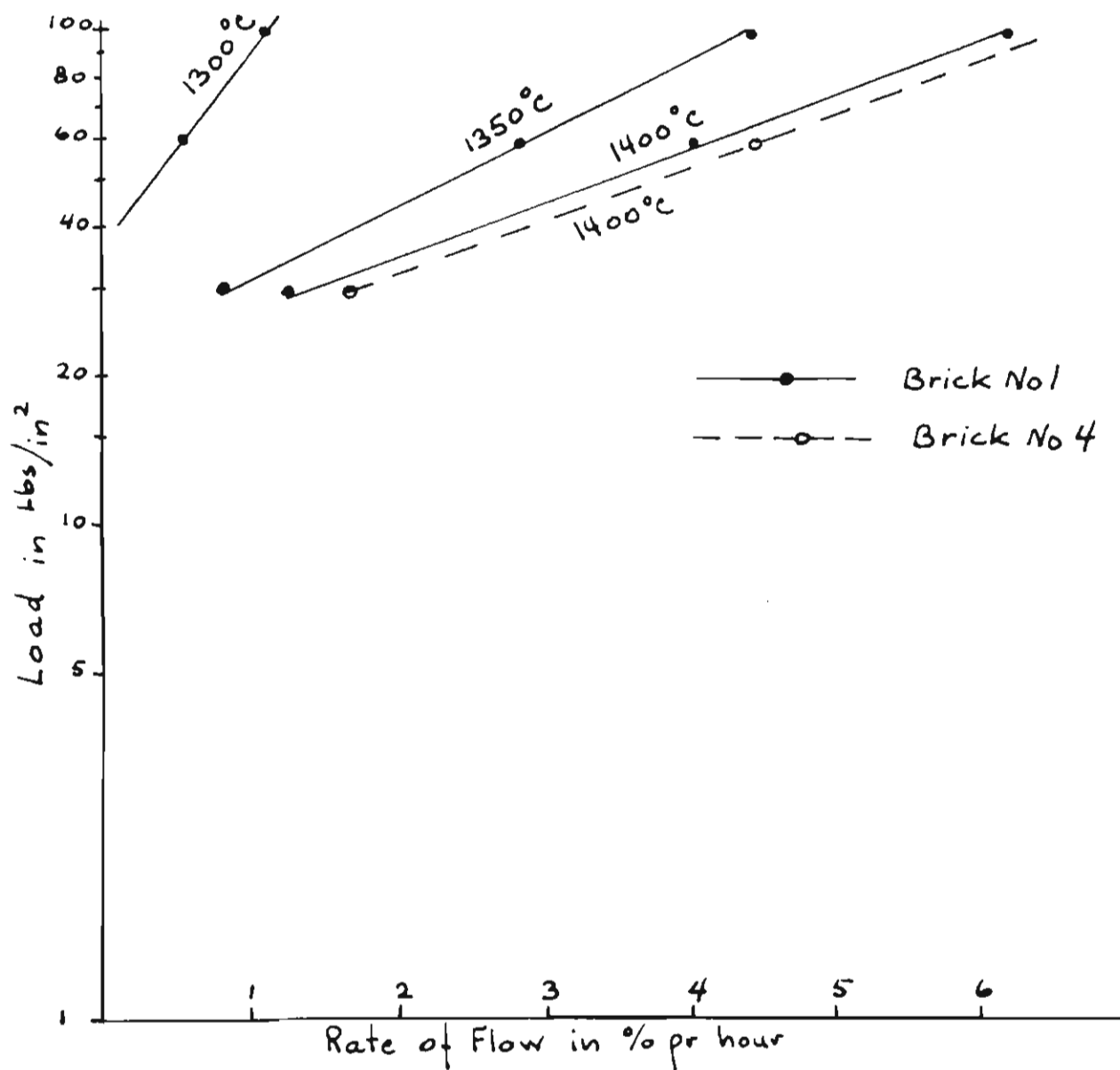
Flow Curves of Brick No. 4

Fig 10

Rate of Flow vs. Load

4 by the dry press. The parallel curves might indicate that the bricks are made approximately of the same raw materials.

From the relation, $V = K_1 F_1^a$, "a" is found to be around 1.8. This is lower than found by Norton, who sets "a" to be between three and five. In his experiments with firebricks, however, he used higher temperatures and lower pressures than in the present work.

The Increasing Pressure Test:

During the experiments it was found that there are mainly three different properties which govern the shrinkage of the brick under the present test method. These are; refractoriness, porosity, and texture of the brick. As these properties vary considerably in the different bricks, it is sometimes difficult to explain and correlate the results. Results in Table IV.

Effect of Refractoriness on the Shrinkage:

The refractoriness of a brick is, to a certain extent, given by the amount of glass formed in the brick which again will govern the flow rate of the material.

Fig. 11 and Fig. 12 show the effect of the refractoriness on the shrinkage of two stiff mud bricks of different refractoriness. As it will be noticed, the difference in one case in refractoriness does not seem to effect the shrinkage too much.

Fig. 13 and Fig. 14 show the effect of different refractoriness on dry pressed bricks. Here the difference in shrinkage is more marked and the difference between the cone 31-33 Intermediate

TABLE NO. IV

% Shrinkage vs. Pressure

Brick No.	% Shrinkage at								
	lbs/in ² 20	lbs/in ² 30	lbs/in ² 40	lbs/in ² 50	lbs/in ² 60	lbs/in ² 70	lbs/in ² 80	lbs/in ² 90	lbs/in ² 100
1	0.9	1.6	2.4	3.2	4.2	5.2	6.1	7.4	8.6
2	0.4	0.8	1.4	2.1	2.7	3.6	4.5	5.3	6.3
3	1.1	1.9	2.8	3.7	4.7	5.7	6.4	7.9	9.4
4	1.	2.	3.1	4.5	5.7	7.1	8.3	9.8	11.
5	1.1	2.2	3.5	4.8	6.7	8.6	11.		
6	1.2	3.	5.	6.8	8.5	10.5			
7	0.9	1.6	2.3	3.1	4.	4.9	5.8	6.7	7.4
8	1.5	2.5	3.8	4.2	5.1	5.9	6.7	7.6	8.6
9	1.	1.7	2.6	3.7	4.8	5.9	7.	8.2	9.3
10	1.1	2.1	3.4	4.8	6.1	7.4	8.8	10.1	11.6
11	1.7	2.8	3.9	5.1	6.4	7.5	8.7	9.8	10.7
12	1.	1.8	2.6	3.5	4.5	5.4	6.1	6.9	7.7
13	1.2	2.3	3.2	4.	4.8	5.5	6.2	6.8	7.4
14	4.6	10.8							
15	1.1	2.5	3.5	5.3	7.4				

Tested at 1400°C

Tested at 1250°C

Fig. 11

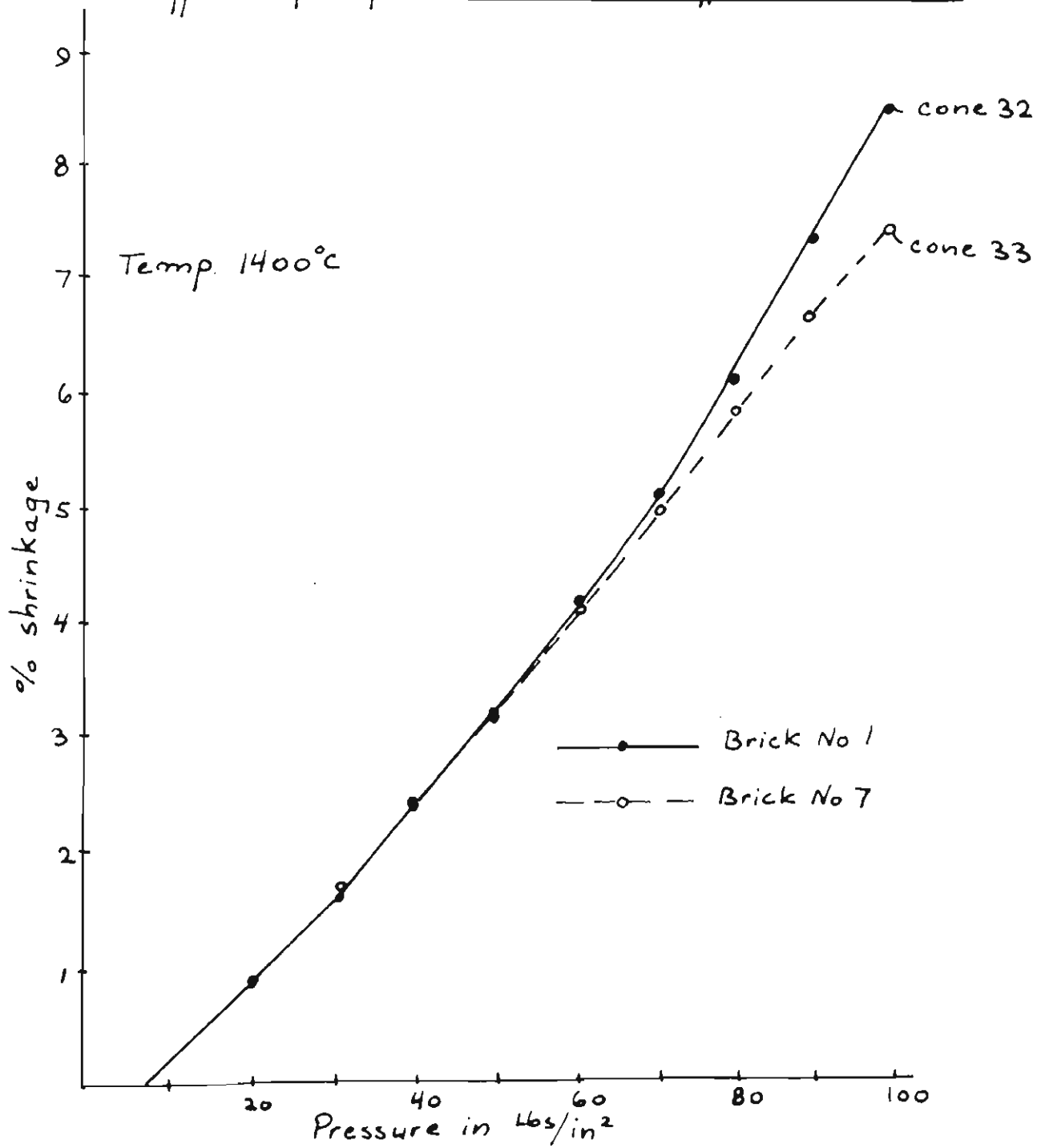
Effect of Refractoriness on Stiff Mud Bricks

Fig 13

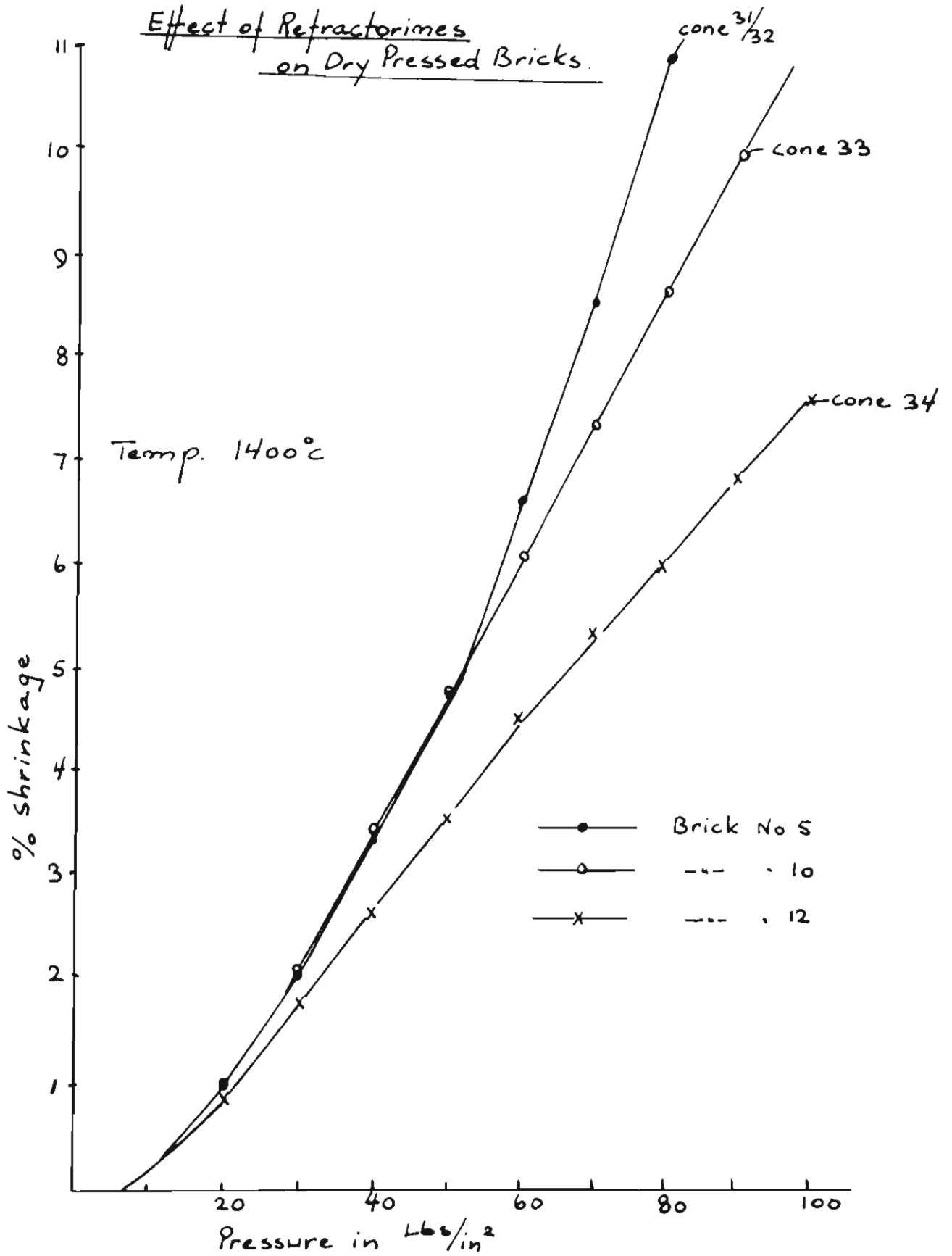




Fig. 12 Tested Stiff Mud Bricks



Fig 14 Tested Dry Pressed Bricks

Heat Duty brick and the cone 34 Super Duty brick is considerable.

Effect of Porosity on Shrinkage:

In Fig. 15, the curves for bricks of different shrinkages are plotted together for comparison. It will be noted that the porosity of the material has a very definite effect on the shrinkage curves. This can be readily explained, as a brick of high porosity will have less load bearing capacity than one of the same material with a lower porosity, as there is less material in the brick to carry the load in the latter case.

As long as the difference in refractoriness is not larger than one or two cones it seems that the porosity is the major factor which governs the rate of flow. Of interest is to notice that a dense cone 32 Intermediate Heat Duty, Stiff Mud brick with a porosity of 10% shows less shrinkage than a cone 34 Super Duty brick with a porosity of 17.5%.

Fig. 16 and 17 show a comparison between different brands of Intermediate Heat Duty and High Heat Duty bricks.

As the graphs show, there is considerable variation between the different brands of brick even for the same type. This is especially the case for the Intermediate Heat Duty quality. The variations in this class are so large that they cannot be explained by the variations in porosity alone. This is especially the case for the stiff mud bricks. Brick No. 1 has a porosity of 11%, while No. 3 has 10%. The variation in shrinkage is, however, considerable.

Fig. 15

Effect of Porosity on Shrinkage

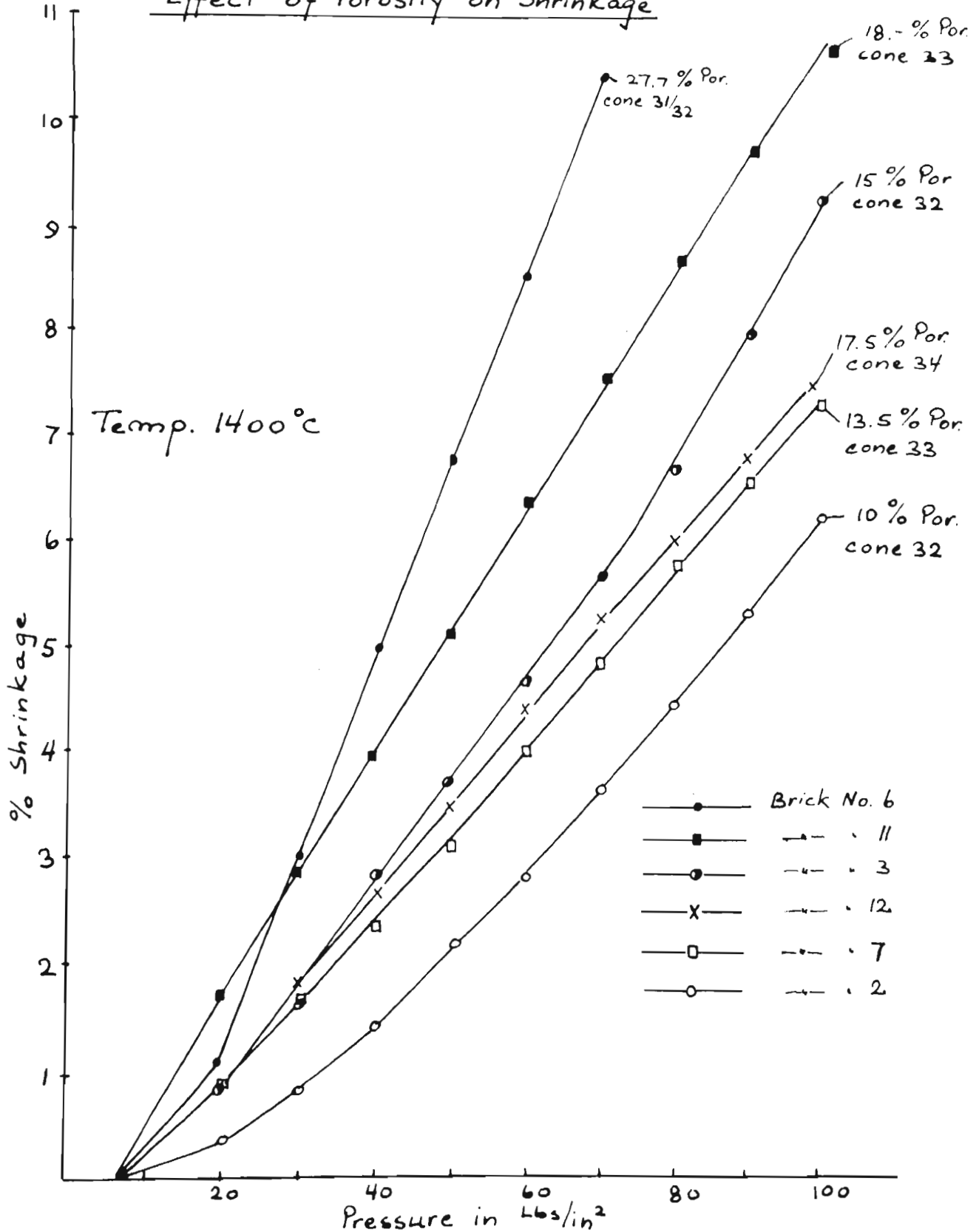


Fig. 16

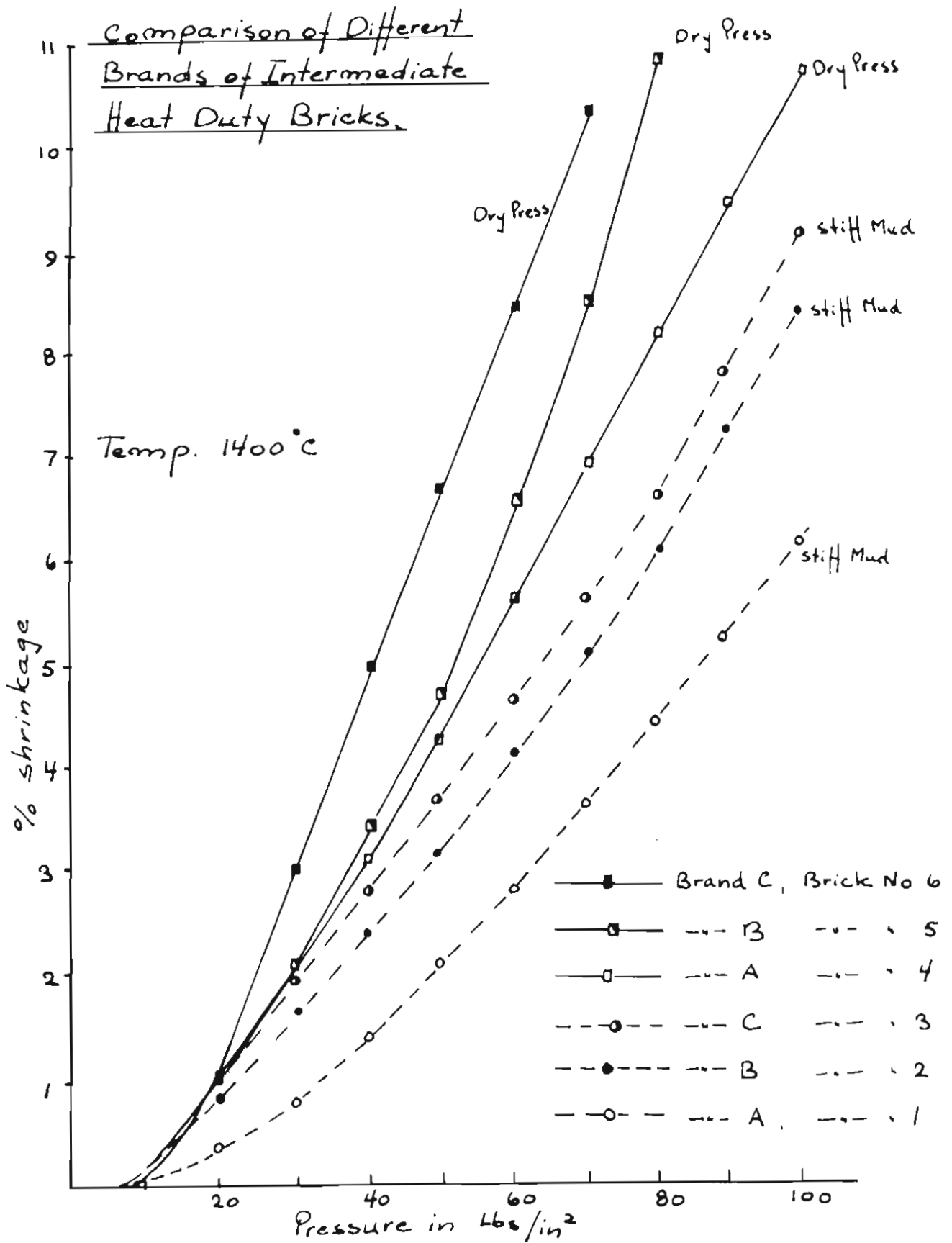


Fig. 17

Comparison of Different Brands of High Heat Duty Bricks

Temp. 1400°C

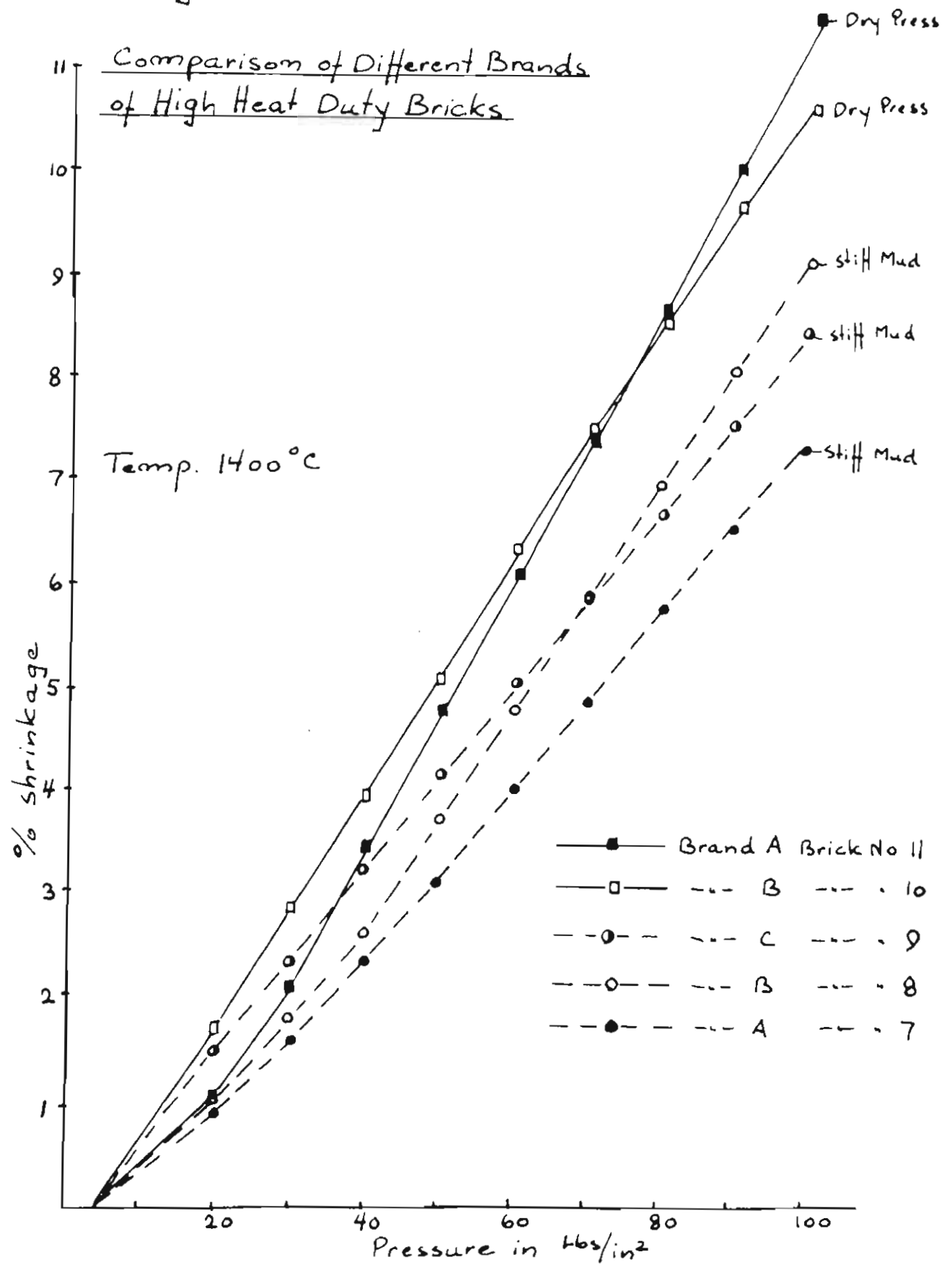




Fig. 18 Lamination Cracks in Stiff Mud Bricks

By examining the bricks after the test, however, some of these, especially No. 1 and No. 3, showed lamination cracks, Fig. 18. These cracks never occurred in brick No. 2. This explains the difference in shrinkage as a brick with a texture which would develop lamination cracks during loading, definitely would be weaker.

It might be noticed that the difference between the brands is larger in the Intermediate Heat Duty group than in the High Heat Duty group. The reason for this is probably that the producers have a closer control with the raw materials, and with the manufacturing for the higher quality groups of bricks.

It is somewhat difficult to compare the results obtained, with the A.S.T.M. load test data given by the manufacturers for the different qualities of brick, as these data mostly are 6-7 years old.

Comparison of Super Duty Bricks:

From Table II giving the physical properties of the materials tested, it will be noticed that for the Super Duty bricks, No. 12 and No. 13, the percent shrinkage after the A.S.T.M. load test at 1450°C were 2.40 and 4.90 respectively.

These two bricks were loaded with 25 lbs/in² and heated up to 1538°C. This temperature was held for about 50 hours and the shrinkage measured regularly. The results after 30 hours are shown in Fig. 19. It will be noticed that brick No. 13 between 8 and 13 hours shows a much larger shrinkage than brick

Fig 19

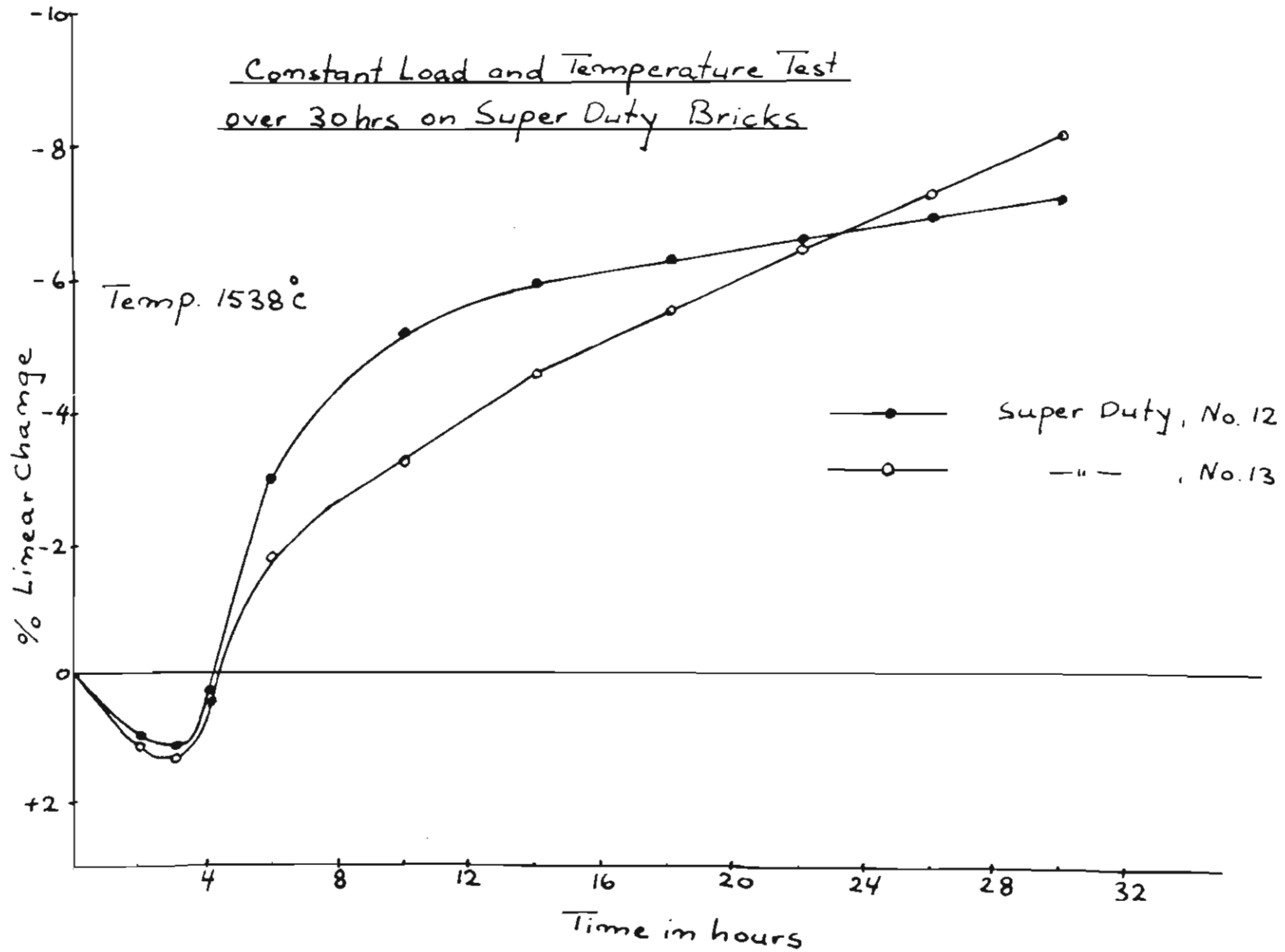
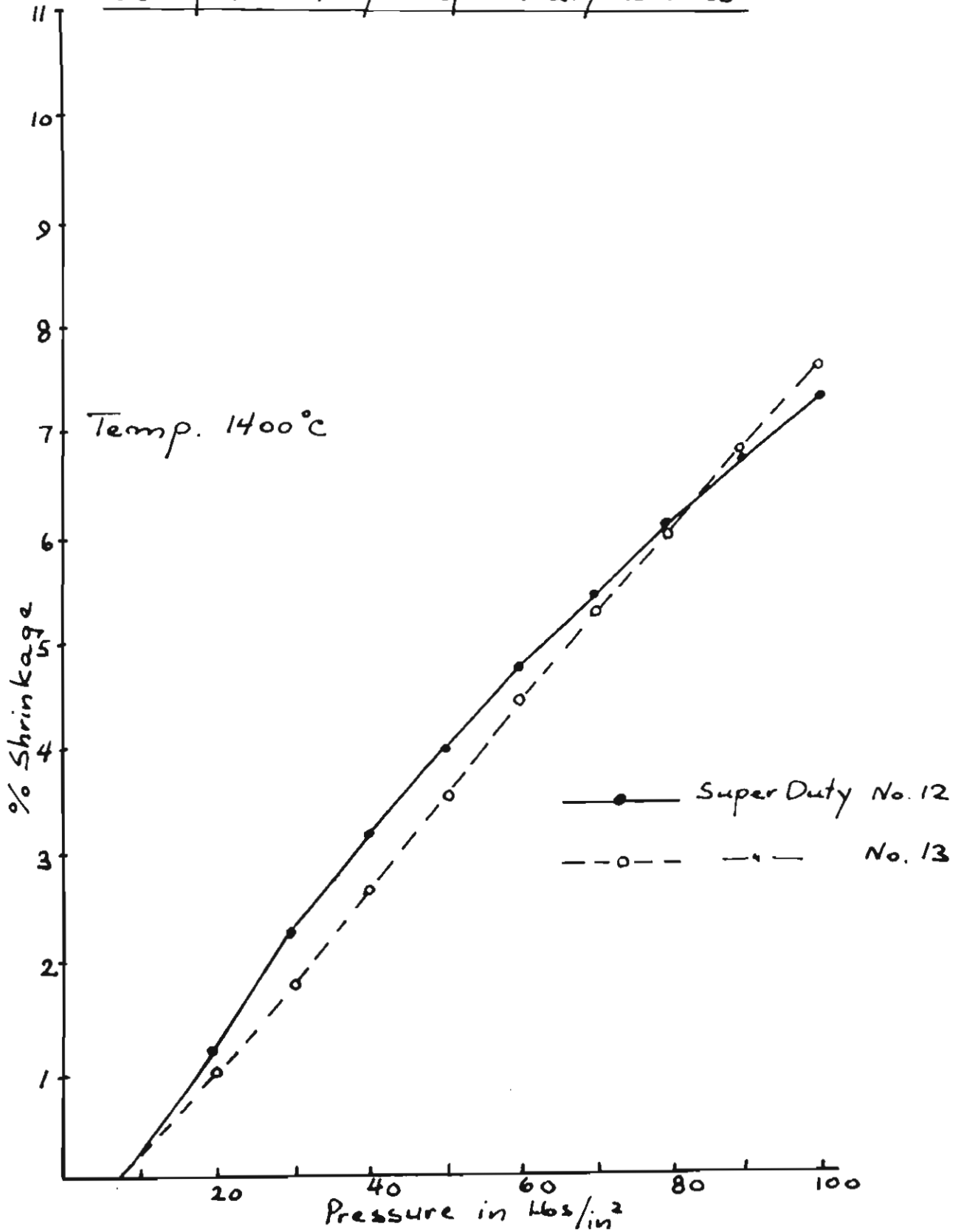


Fig 20

Comparison of Super Duty Bricks

No. 12. This is quite in correspondance with the results from the A.S.T.M. test. On further heating, however, the rate of flow decreases. After 25 hours, the shrinkage of brick No. 12 is larger than the one of No. 13 and the difference keeps on increasing on further heating. In this case the results from the A.S.T.M. load test will give a wrong picture of the load bearing capacity of the two bricks.

The same bricks were tested by the increasing load method and the results are shown in Fig. 20. By comparing Fig. 19 and Fig. 20, it will be seen that the curves obtained are of the same type. This is a fact which proves that the discussed test method may be more reliable than the present A.S.T.M. method. If the time of the testing methods is taken into consideration, the increasing pressure method needed three and one-half hours to give approximately the same results as obtained after 24 hours with the constant load and temperature method.

Comparison of Results Obtained from Constant Load Tests and Increasing Load Tests:

By comparing the results obtained in Fig. 8, 9, and 10 and Fig. 7, it will be seen that there is a big difference in the results obtained. Without doing any calculations, it is noticed in Fig. 7, that even though the pressure is increased all the time, the rate of flow is almost constant, especially for brick No. 8 and 11. This is strictly opposite the results obtained in Fig. 10 where the rate of flow was found to be proportional to the 1.8 power of the pressure.

This discrepancy can only be explained by the fact that during the increasing load test the flow in the brick never will come to an equilibrium. During the whole test, the rate of flow will be in the steep part region of the curves in Fig. 8 and 9 before these flatten off and become straight lines. The time required for the test will, however, tend to flatten out the shrinkage curve while the increasing pressure will tend to make it steeper. These two factors will work in each direction and partly eliminate each other giving an almost straight line curve.

By taking from Fig. 7 the shrinkage at 65 lbs/in² and subtracting the shrinkage at 55 lbs/in² and by knowing that this shrinkage took place over a period of three minutes, the approximate rate of flow for the brick at 60 lbs/in² can be calculated.

The same calculations are made with both bricks at 60 lbs/in² and 30 lbs/in². The results are compared with those obtained from Fig. 10 in Table V

TABLE V

Comparison of Flow Rates Obtained from Different Test Methods

	Brick No. 1		Brick No. 4	
	60 lbs/in ²	30 lbs/in ²	60 lbs/in ²	30 lbs/in ²
Flow rate from const. Load Test	4. %/hr.	1.23%/hr.	4.03%/hr.	1.61%/hr.
Flow rate from increas. Load Test	20. %/hr.	12. %/hr.	24. %/hr.	18. %/hr.

As seen from Table V, the flow-rates obtained by the increasing pressure method are from 5 to 10 times greater than from the constant temperature pressure method.

The fact that an increasing load will cause such a great increase in the flow rates in the brick has quite some interest in the construction of a refractory brick work. The bricks will not only have to take the load from overlaying brick, which is constant, but will also have to stand the load and stress due to expansion or contraction in the wall. These last types of stress will vary all the time and will also give considerable variations in the flow rates in the brick. It would, therefore, be incorrect

to estimate the shrinkage of a refractory wall just on a simple constant load test even though the results from the test together with practical experience would be of great value.

Testing of Open-Hearth Ladle Nozzles:

Two different brands of nozzles were tested by the increasing load method. The conical parts of the nozzles were cut off before they were tested, so that the test piece became a 9" long cylinder.

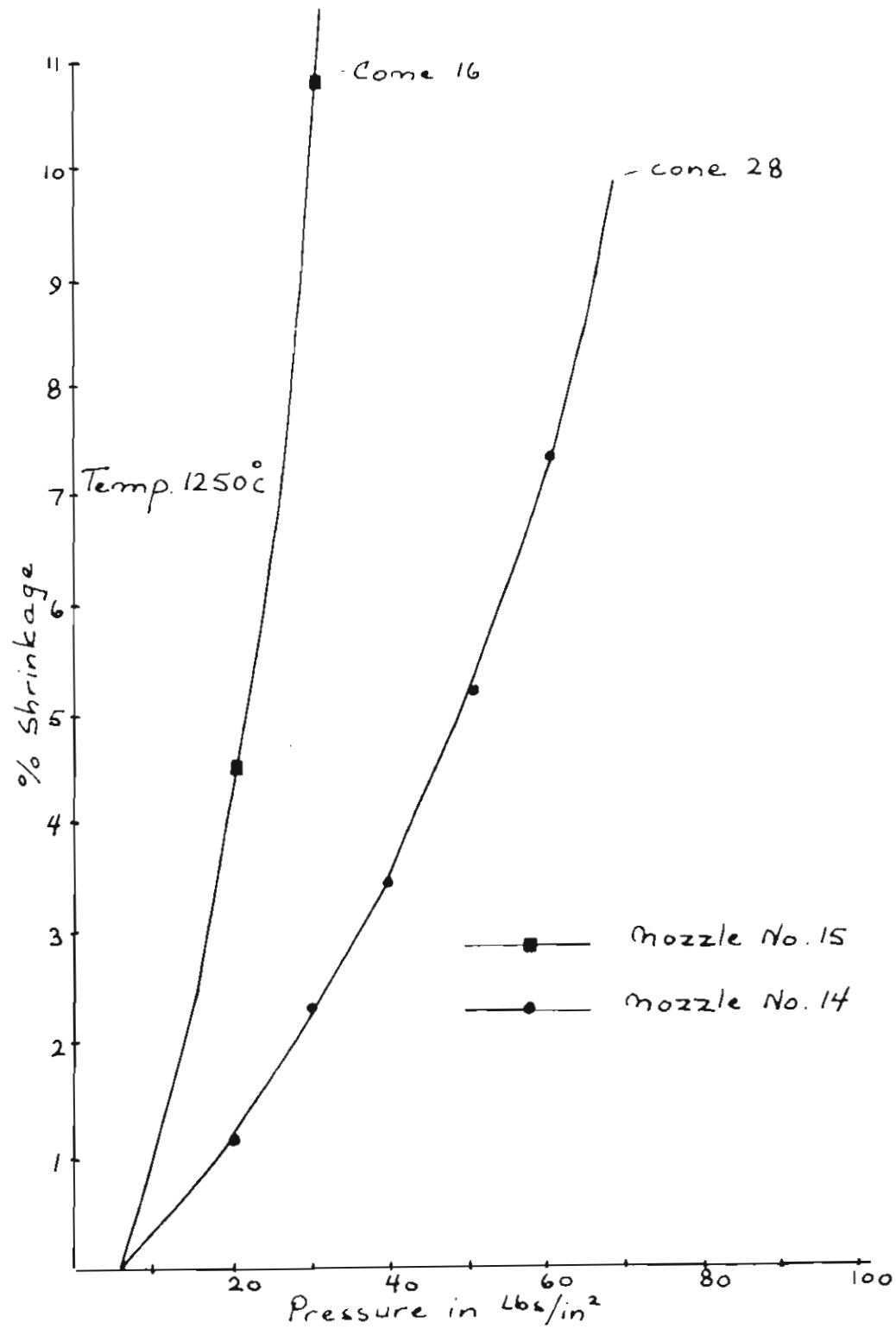
Because of the low refractoriness of the nozzles, the test was carried out at 1250°C. The results are given in Fig. 21 and 22.

As it will be noticed, the cone 16 nozzle has a very high rate of flow already at 1250°; it also shows considerable expansion on bloating. The cone 28 nozzle, however, has a moderate shrinkage, but shows some bad cracks. According to information from open-hearth operators, the pouring results with the cone 16 nozzle are better than for the cone 28 nozzle. This could be expected after the present test, because the cracking of the cone 28 nozzle definitely will cause bad leaking during the pouring process.

The soft cone 16 nozzle will, however, give a good shut off and the expansion will seal all kinds of cracks. Another factor which is discussed by R. B. Snow (15) is the erosion

(15) R. B. Snow, Mechanism of Erosion of Nozzles in Open-Hearth Ladles, presented at the Convention of Am. Cer. Soc., 1948.

Fig 21

Comparison of Nozzles.

of the steel. While the erosion of ordinary steel on a cone 19 type nozzle is 24.5%, it is only 16.0% on a cone 28 nozzle. This is a factor which is against the low cone nozzles. The erosion for different types of steel varies, however, very much so that the results always must be combined with practical experience.

The increasing pressure method will, however, give results which are very valuable for the development of the right type of nozzles.



Fig 22 Tested Nozzles

Conclusions

- (1) The increasing load test gives at the same temperature and load a flow rate in the tested brick which is five to ten times higher than measured in tests with constant loads.
- (2) During the test, the flow rate of the brick is almost constant, even though the load is continuously increased. This is also radically different from what is encountered in the constant load tests.
- (3) Experiments with different types of firebricks show that the most important property of the brick which governs the flow rate in the brick during the test is the porosity. The flow rate also depends upon the refractoriness, but the porosity is more important, as long as the refractoriness does not vary more than two cones.
- (4) Experiments with different brands but same quality of bricks show that the texture and possible laminations in the bricks have important effects on the rate of flow. In some cases, lamination cracks were brought out during the test.
- (5) Two Super Duty bricks which needed thirty hours at 1536°C in an ordinary constant load test to show the real relationship between their load bearing capacities were tested by the increasing load method. The results from this method indicated a similar relationship after three hours testing.
- (6) Two different qualities of nozzles for open hearth ladles were tested and the method proved itself very useful for controlling the plasticity of this kind of material at high

temperatures. The test also brought out bad cracks in one type of nozzle.

The test seems to give more valuable information about these properties in the nozzles than other methods used heretofore.

(7) The results obtained from the test method can only be used as empirical values in connection with practical experience, but are very valuable as such. In some cases, the results seem to be more reliable than those obtained by the A.S.T.M. constant load test.

(8) The test method discussed is simple and the whole test can be carried out in three hours.

Bibliography

1. A.S.T.M., Designation C-16-41
2. A.S.T.M., Designation C-27-41 for Dissification of Fireclay Refractories
3. Claws, F. H. and Green, A. T., Refractoriness Under Load Tests, Com. Inst. Gas Engr., No. 103, 1934
4. Dale, A. F., the Relation Between Ordinary Refractoriness and Composition, Physical and Chemical, of Refractory Material, Trans. Ceram. Soc., Vol. 23, p. 217, 1924
5. Dale, A. F., Some Fallacies to be Avoided in the Standardization of Any Method of Testing the Load-Bearing Capacities of Refractories at High Temperatures, and a Suggested Method for Standardization, Trans. Ceram. Soc., Vol. 24, p. 216, 1924-25
6. Deutsche Industrie Normen (D.I.N.) 1064
7. Ekholm, L. G. and L. D. Hower, Jr., Pouring Techniques and Their Effects on Casting-Pit Performance, A.I.M.E., Proceedings of the National Open Hearth Committee, Vol. 28, p. 120, 1945
8. Nadai, A., Plasticity; a Mechanics of the Plastic State of Matter, McGraw-Hill Book Company, Inc., 1931
9. Norton, F. H., A Critical Examination of the Load Test for Refractories, J. Am. Cer. Soc., Vol. 22, p. 334, 1939
10. Norton, F. H., The Flow of Ceramic Bodies at Elevated Temperatures, J. Am. Cer. Soc., Vol. 19, p. 129, 1936
11. Norton, F. H., Refractories, Ch. 8, p. 437, McGraw-Hill Book Company, Inc., 1942
12. Petit, D., A New Method of Assessing the Stability of Refractory Structures, Trans. Cer. Soc., Vol. 38, p. 313, 1939
13. Roberts, A. L. and Cobb, F. W., The Behavior of Refractory Materials Under Torsion at Different Temperatures, Trans. Ceram. Soc., Vol. 32, p. 22, 1933

Bibliography (continued)

14. Roberts, A. L. and Cobb, F. W., op. cit., Part II, Trans. Ceram. Soc., Vol. 35, p. 182, 1936
15. Roberts, A. L. and Cobb, F. W., op. cit., Part II, Trans. Ceram. Soc., Vol. 37, p. 296, 1938
16. Snow, R. E., Mechanism of Erosion of Nozzles in Open Hearth Ladles, presented at the Convention of Amer. Cer. Soc., 1948

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