

Study on failure mechanism of aluminosilicate refractories in the roofs of reheating furnaces

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Introduction

In reheating furnaces, conventional arch type roof has been replaced by suspended roof design. Various types of roof designs are in practice using different types of aluminosilicate refractories depending on furnace capacity and operational parameters.

In normal operating conditions, with low level of fluxing agent in furnace environments and with about 1 to 2% of free oxygen in atmosphere, the fireclay bricks gave a satisfactory service. The failure of these bricks are generally caused by spalling as a result of thermal fluctuations during operation. However when the furnaces were driven hard, particularly with liquid fuel, the fireclay refractories showed premature failure.

In authors' plant, various types of aluminosilicate refractories are used in different reheating furnaces. High heat duty, high alumina and imported aluminosilicate refractories (HHD) were used in the roof of reheating furnaces. In identical operating conditions their service performances indicated that

The service condition of reheating furnaces in authors' plant are discussed.

The different types of aluminosilicate and high alumina bricks have been used in the roofs of reheating furnaces and premature failure of high alumina bricks was observed. The physico-chemical properties of the bricks were determined and its relationships with service performance was studied.

It has been observed that the performance of the refractories largely depends on its thermal shock resistance and creep behaviours.

lives of indigenous high heat duty bricks were much inferior to imported bricks. Also contrary to the expectations, high heat duty bricks showed comparatively better life than high alumina bricks. The work was undertaken to find out the causes of the above behaviour during service of various types of bricks in the authors' plant. The findings of the above study is given in the present communication.

Operating conditions

The reheating furnaces in Tisco are continuous and multi-zone type with top and bottom firing arrangements. These furnaces have three zones viz. preheating zone, firing zone and soaking zone. The output varies from 45 to 60 tonnes per hour depending on the type of the charge. The two sets of burners each containing 6 numbers and only one set consisting of 5 burners are used. Burners are of pressurised type which help to maintain the furnace pressure at +0.02 to +0.03 w. g. and prevent air infiltration. A reducing atmosphere is always tried to maintain to reduce scale loss of the charged stock. But during discharge of the stock in some furnaces while the door opens, air is introduced and the furnace atmosphere is changed for a short period. The scale of the stocks gets accumulated and builds up the bottom of the furnace. After a certain period of the operation, the furnace is stopped for repairing the bottom. In Tisco this interval is around one month. The operating temperature of the furnace soaking zone is maintained in the range of 1250°C to 1300°C. During weekly shutdown period the fuel is completely shut off and the temperature comes down well below 200°C, often below 100°C. Also the furnace is heated up very fast during re-starting, reaching the operating temperature within 3 to 4 hours. It was observed that the bricks had some sort of vitrification at the exposed faces after some period of service, and cracks were also developed.

The nature of the cracks were suggestive due to both tensile and compressive stresses. The bricks failed due to the tearing of the cracked pieces.

Experimental procedure and results

Samples of reheating furnace roof brick were collected from different supplies. The types of bricks taken for investigation were high heat duty, high alumina and imported high heat duty quality. Samples of each brand were selected on the basis of similar porosities and their physical and chemical properties were evaluated. The high temperature strength and creep characteristics were determined on these samples as received, and also on the test pieces made after firing the sample bricks at 1300°C for 5 and 10 hours' duration. Spalling resistance was determined on as received samples by prism test at 1000°C and also by water quenching test as per GOST 1056 (57). Loss in strength was measured on the samples after they were exposed to a thermal shock treatment at various temperature levels. The details of reheating furnace roof bricks as received and fired at 1300°C for 5 and 10 hours are given in Table I.

Chemical analysis

The chemical analysis of different samples were done as per ISI test method and results are given in Table II.

X-ray diffraction analysis

The samples were crushed to 200

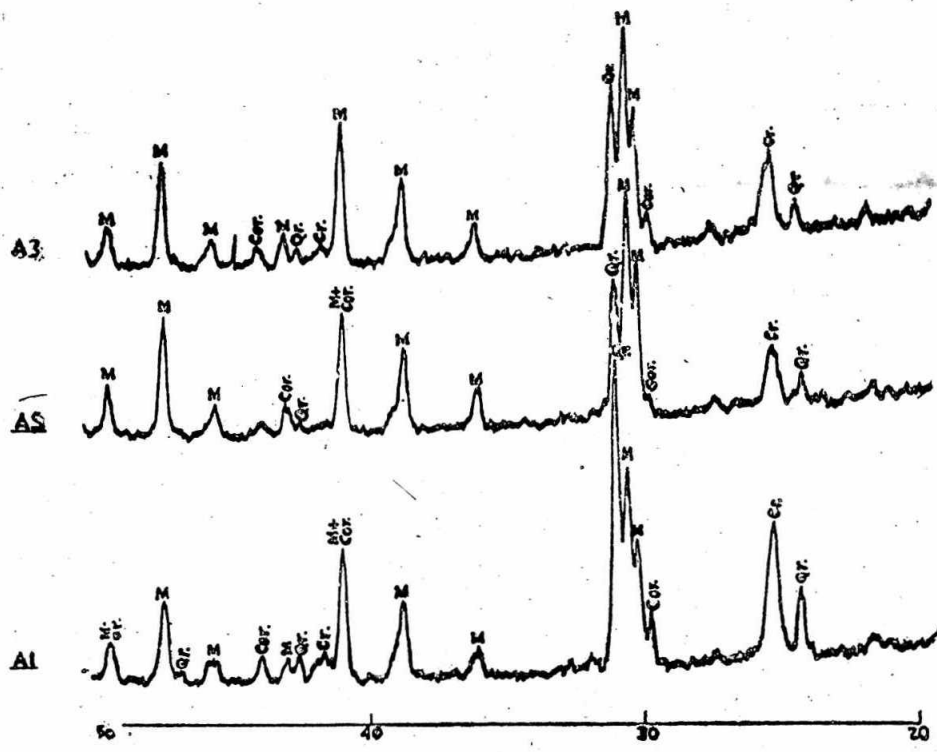


Fig. 1 X-ray diffractograms of samples A-1, A-2 and A-3.

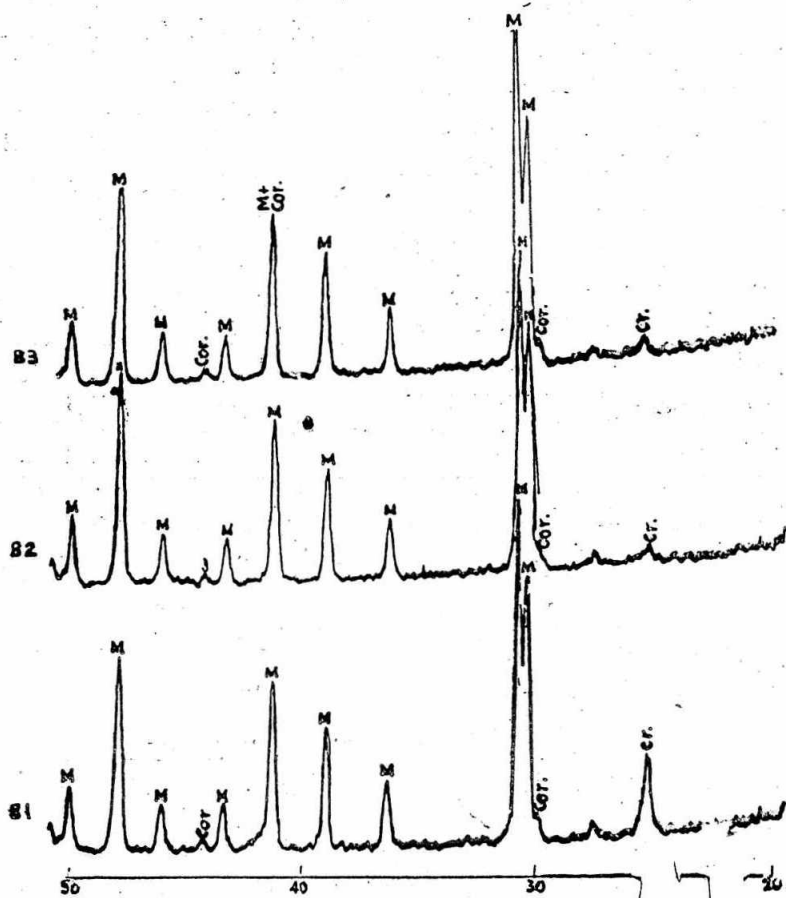


Fig. 2 X-ray diffractograms of samples B-1, B-2 and B-3.

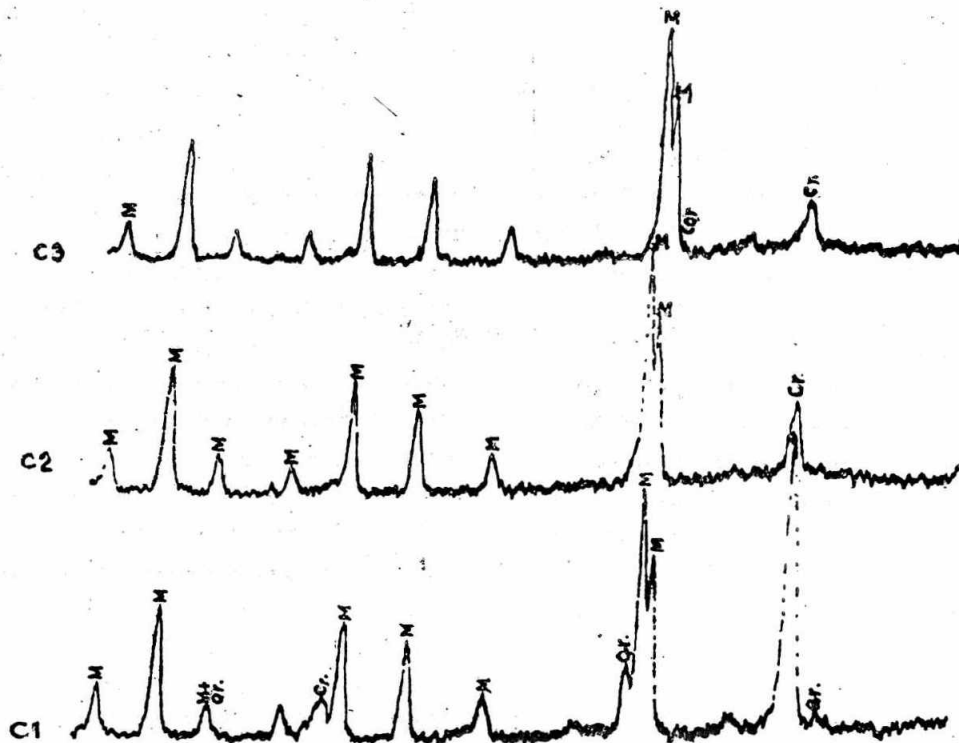


Fig. 3 X-ray diffractograms of samples, C-1, C-2 and C-3.

mesh fineness and subjected to X-ray study for identification of major and minor phases. A Phillips X-ray diffractometer was used for this purpose using Cok α radiation. Scanning speed of 1° per minute has been adopted for all the samples. The samples were screened between 20° and $50^\circ(2\theta)$ at 40 KVA and 20 MA settings. The results obtained are given in Table III and Figs. 1 to 3.

Physical Properties

Apparent porosity, bulk density, permanent linear change on reheating, refractoriness under load, refractoriness and cold crushing strength were done as per ISI specifications. Spalling test of different samples were done as per ISI (prism spalling) and GOST

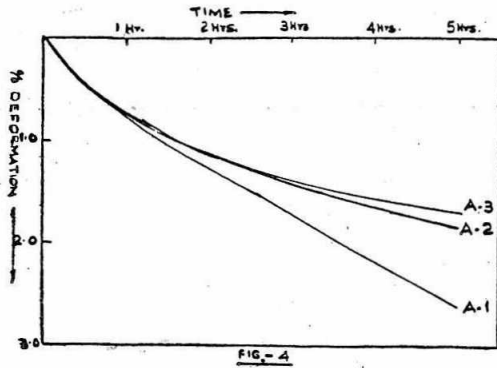
specification (1056—57) and results are given in Table IV.

Compressive creep

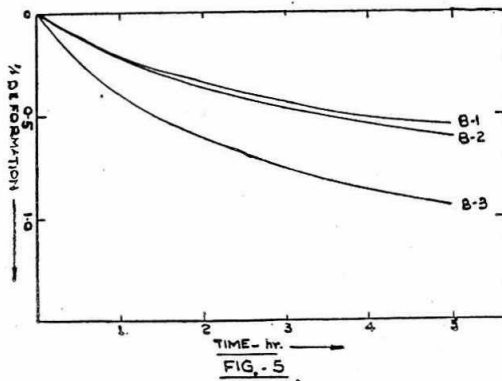
The compressive creep characteristics of different samples as received and heat treated were carried out in a standard apparatus. The diameter of test piece was 50 mm and a load of 2 kgs/sq. cm. was applied. The duration of testing was 5 hours. The rate of heating was maintained at $5^\circ\text{C}/\text{mt.}$ and after reaching the test temperature a soaking of 30 minutes was given before the load is applied. The deformation with time at the test temperature were plotted and given in Figs. 4 to 6.

Torsional creep

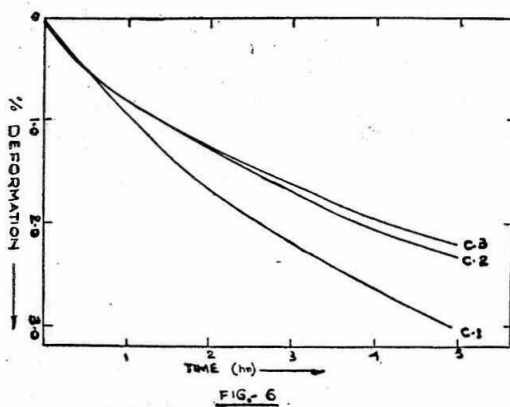
The tests were conducted in stan-



Percentage deformation under compressive stresses of A-1, A-2 and A-3



Percentage deformation under compressive stresses of B-1, B-2 and B-3.



Percentage deformation under compressive stresses of C-1, C-2 and C-3

standard equipment with automatic loading device The test specimens were 200 x

20 x 20 mms cut from the bricks. A torque of 4.57 kg. cm was applied per square centimeter of sample cross section. The tests were conducted at 1200°C which was raised at the rate of 6°C per minute and a soaking for 30 minutes were given before loading. The flow rate after the application of maximum torque was noted and given in Table V.

Hot modulus of rupture

The hot modulus of rupture of the samples were carried out in a standard equipment. The modulus of rupture determination was done at 1200°C or 1300°C. The heating schedule and load application was done as per ASTM. C-503-67 tests. The results are given in Table VI.

Cold modulus of rupture

The cold modulus of rupture was done on as received samples as well as on samples quenched from different temperatures. The specimens were soaked at 200°C, 400°C, 600°C, 800°C and 1000°C for 30 minutes and quenched in cold water. The drop in strength were measured and plotted in Fig. 7.

Permeability test

The percentage increase of permeability of different specimens quenched from different temperatures were determined. The results are plotted in Fig. 8.

Discussion

The chemical analysis of A-1, B-1 and

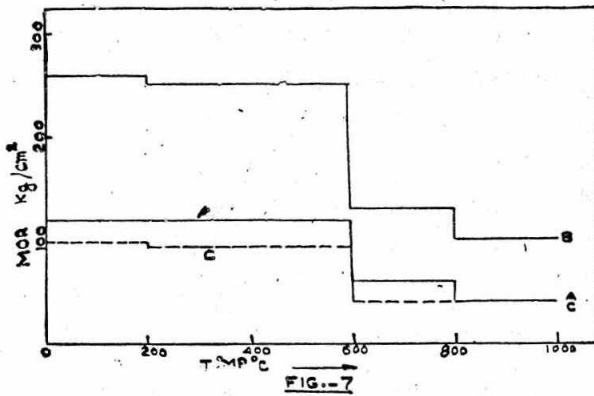


FIG.-7
Cold modulus of rupture of different water quenched specimens.

C-1 indicate that the alkali content of B-1 is slightly higher than A-1 and C-1. Other impurities i.e. iron oxide, TiO_2 are high in A-1 and C-1 but CaO and MgO are high in B-1.

$SiO_2-Al_2O_3-K_2O$ -system¹ showed that with increase of K_2O , the amount of liquid formation increases at eutectic temperature of $985^\circ C$. It is also observed that with the increase in CaO and MgO content the liquid formation in aluminosilicate refractory, increases and eutectic temperature reduces to a larger extent.

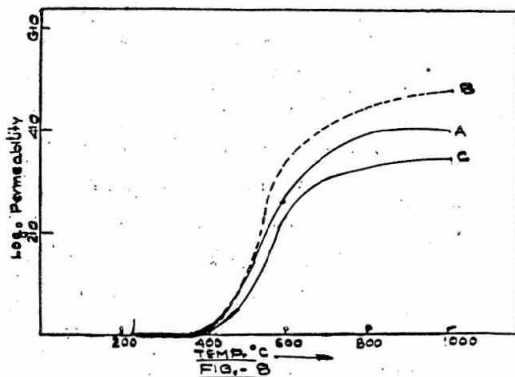


FIG.-8
Percentage increase in permeability of different water quenched specimens.

The x-ray analysis of different specimens indicated that cristoballite and quartz content of A-1 and C-1 is higher than B-1 with lower mullite content. Heat treated specimens indicated gradual decrease of cristoballite in A-2 and A-3 and C-2 and C-3 which is due to formation of more interlocking mullite structure. In B-2 and B-3 cristoballite peaks are also reduced but to a lesser extent with no appreciable increase in mullite peak suggests

that the formation of more liquid in B-2 and B-3 during heat treatment.

The physical properties of different specimens indicated that on heat treatment at $1300^\circ C$ for 5 and 10 hours the sample Nos. A-2, A-3 and C-2, C-3 showed expansion, whereas B-2 and B-3 showed shrinkage. The shrinkage in high alumina quality (B-2 and B-3) on reheating is suggestive of presence of low melting compounds in the matrix. This is also corroborated by the chemical and x-ray analysis. The prism spalling test indicated similar spalling resistance characteristics of all the samples but water dip spalling test done as per GOST specification showed that B-1 is inferior as compared to A-1 and C-1. This may be due to high liquid content in the matrix of B-1

The compressive creep rate of A-1, B-1 and C-1 as shown in Figs. 4 to 6 indicated that B-1 has got better creep resistance as compared to A-1 and C-1. Also A-1 is superior to C-1. The creep resistance of the heat treated sample

showed that while there was an improvement in the creep resistance properties of C-2, C-3 and A-2, A-3 samples the values of B-2 and B-3 deteriorated. During the heat treatment the crystalline phases are constantly re-arranged and increase the crystal-crystal bonding, but presence of low melting liquid in the matrix of B-1 the creep rate of B-2 and B-3 after heat treatment indicated increasing trend.

The compressive creep characteristic of firebrick indicates that the plastic flow of firebrick^{3, 4, 5, 6, 7} primarily depended on the flow of glassy matrix at higher temperature. The crystalline phases in the matrix reduces the quantity of glass resulting in an increase of viscosity of the system. In B-2 and B-3 though more crystalline bonds were expected, yet the higher creep rate of these samples are presumed to be due to the low fusible nature of the liquid.

The torsional creep rate of the samples indicated also the same trend as in case with compressive creep. However since the torsional creep is more dependent on the nature of liquid formed, slight variation in the characteristic was observed. Specimens of A-2, A-3 and C-2, C-3 showed increasing trend in the creep resistance whereas the creep rate of B-2 and B-3 increased to a larger extent. This confirms the earlier observations made during the compressive creep testing. The hot modulus of rupture of the specimens indicates that the heat treated specimens C-2, C-3 and A-2, A-3 showed increased strength as compared

to C-1 and A-1 which confirms the more intercrystalline bonding of the heat treated specimens. In B-2 and B-3, the hot strength has gradually decreased due to more liquid formation, in the matrix during heat treatment.

The quantitative assessment of thermal shock resistance is not possible with normal testing procedures. The water dip and prism spalling tests provide only a comparison of thermal shock resistance of different samples. No significant differences were observed while the samples were tested as per the above procedures. It is proved that thermal shock damage will be optimum in the temperature range below initial liquid formation^{8, 9, 10} where refractory behave as a brittle material. D. P. Hassel Man¹¹ observed that increasing the size of Griffith's flaw by increasing grain size and introducing micro cracks in the brick, the thermal shock failure can be restricted. In C-1 the evenly distributed porosity and larger grain size showed better thermal spalling resistance as compared to other two samples.

The bending strength of thermally shocked specimens indicated that the drop of strength is a function of temperature difference (Fig. 8). It is observed that optimum temperature difference is required to initiate a fracture which is indicated by a sharp loss in strength. Thereafter no change in the strength characteristic upto certain temperature range is observed which indicated that no crack propagation took place. At 600°C the drop of

strength is observed due to crack initiation¹² and there is a change in the extent of cracking as compared to original sample. This is corroborated by the increase in permeability at that point. At around 800°C again a drop in strength was observed presumably due to the crack propagation. David and Tapin¹² also observed that drop strength is the function of temperature difference of refractory.

The samples A & C having initial low strength, showed lower drop in strength after shock treatment as compared to B-I, having higher initial strength. This is obviously due to the less initiation of crack in the samples compared to B-I. Similar observations have been reported by others¹³.

Conclusion

During the service, poor performance of the indigenous high alumina bricks used in reheating furnace roof have been investigated in the laboratory. Also tests were conducted to find out the difference in characteristics of high heat duty bricks obtained from indigenous sources as well as from imported origin.

From the results of various laboratory experiments, the following conclusion can be drawn.

- a) High alumina bricks having more impurities in the matrix on heat treatment developed low melting liquids. On the other hand high heat duty bricks both from indigenous and imported origin,

showed improved intercrystalline bonding in the matrix.

- b) The high alumina bricks showed higher shrinkage due to the presence of low melting liquids.
- c) The hot strength of high alumina bricks decreased on heat treatment whereas the high heat duty bricks showed improvement.
- d) The spalling characteristics when measured by loss in strength of thermally shocked test pieces showed that the drop in strength of high alumina bricks were more as compared to high heat duty bricks.
- e) The better performance of imported high heat duty bricks was due to better spalling characteristics as compared to indigenous high heat duty bricks.

Acknowledgement

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TABLE—I Characteristic of bricks fired at 1300°C for 5 and 10 hours

Manufacturer	Type of bricks	As such	Fired at 1300°C 5 hrs	Fired at 1300°C 5 hrs	Macro
A	H. H. D.	A-1	A-2	A-3	Mixed grain
B	High alumina	B-1	B-2	B-3	Fine grain
C	Imported (H. H. D.)	C-1	C-2	C-3	Coarse grain.

TABLE—II Chemical analysis of different bricks

Type of bricks	SiO ₂ %	T. Fe as FeO	Al ₂ O ₃ %	TiO ₂ %	CaO %	MgO %	K ₂ O & Na ₂ O %	Loss %
A-1	52.88	1.05	43.67	1.42	Tr.	Tr.	0.50	—
B-1	35.0	1.0	62.60	0.55	0.30	0.29	0.60	—
C-1	52.00	2.70	42.20	1.58	0.80	—	0.50	0.20

TABLE—III X-ray analysis of different heat treated bricks

Type of bricks	Quality	Major phases	Minor phases	Remarks
A-1	HHD (as such)	Mullite	Cristoballite, quartz, corundum	More cristoballite
A-2	HHD (Fired)	„	„	Low cristoballite
A-3	HHD (Fired)	„	„	Low cristoballite
B-1	High alumina (as such)	„	Cristoballite, corundum	—
B-2	High (Fired)	„	„	Cristoballite, and mullite peaks have changed very much
B-3	High (Fired)	„	„	—
C-1	Imported (as such)	„	Cristoballite & quartz	More cristoballite
C-2	HHD (Fired)	„	Cristoballite	Low cristoballite
C-3	HHD (Fired)	„	Cristoballite	Low cristoballite

TABLE-IV Physical properties of different bricks

Sample	Description	Apparent porosity (%)	Bulk Density gm/cc	Appr. Density	P. L. C. at (%)	R. U. L. Ta°C	P. C. E. (Orton)	C. C. S. kg/cm ²	Prism Spalling test	Thermal Spalling Water dip- ped test GOST 1056-57
A	H. H. D.	24.8	2.17	2.89	+ 1.3 at	1430	31-32	240	+ 30	+ 15
					1450°C/2 hrs.					
					+ 0.62 at					
B	High alumina	25.0	2.23	3.03	1300°C/5 hrs.	1510	Greater than 32½	380	+ 30	+ 10
					+ 0.51 at					
					1300°C/10 hrs.					
C	Imported	18.5	2.08	2.55	- 0.8 at	1420	30-31	210	+ 30	+ 15
					1600°C/2 hrs.					
					- 0.02 at					
					1300°C/5 hrs.					
					+ 1.2 at					
					1450°C/2 hrs.					
					+ 1.16 at					
					1300°C/5 hrs.					
					+ 1.51 at					
					1300°C/10 hrs.					

TABLE—V Torsional creep characteristic of different specimens

Sample	Description	Torsional creep rate mR/hr. at 1200°C
A-1	HHD (as such)	32.20
A-2	HHD (1300°C/5 hrs)	16.50
A-3	HHD (1300°C/10 hrs.)	12.31
B-1	High alumina	Failed after 45' applying torque
B-2	High alumina (1300°C/ 5 hrs)	Failed after 40' applying torque
B-3	High alumina (1300°C/10 hrs.)	Failed after applying torque
C-1	Imported (as such)	Failed after 20' applying torque
C-2	Imported (1300°C/5 hrs.)	Failed after 40' applying torque
C-3	Imported (1300°C/10 hrs.)	40.90

TABLE—VI Hot modulus of rupture of different specimens

Samples	Description	MOR at 1300°C (kg/cm ²)
A-1	HHD (as such)	88
A-2	HHD (1300°C/5 hrs.)	94
A-3	HHD (1300°C/10 hrs.)	98
B-1	High alumina (as such)	88
B-2	High alumina (1300°C/5 hrs)	62
B-3	High alumina (1300°C/10 hrs)	47
C-1	Imported (as such)	70
C-2	Imported (1300°C/5 hrs)	77
C-3	Imported (1300°C/10 hrs)	85