Study of different mortars used for soaking pit recuperators

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The efficiency of the recuperators in reheating furnaces depends upon their infiltration, thermal conductivity, thermal shock resistance etc. To make a gas tight joint in recuperator, suitable jointing material is essential. An attempt has been made to develop a suitable mortar for recuperator joints. The properties of different mortars are studied by their shrinkages, strength at different temperatures, air permeability, plasticity, workability and thermal behaviour. The characteristics of mortars are also studied with different bond contents and by varying the grain size.

The performance of different mortars in Tisco plant and the data showing the air pick-up during service are also mentioned.

Introduction

Effective utilisation of waste heat by the use of recuperators system has become more and more imperative due to the recent energy crisis. Although both the recuperator and regenerator are used to preheat air, the co-efficient of heat transfer is higher in the recuperators than that of regenerators (2.5—6.0 as compared to 1.5—3.5 Btu/ft^2/hr/°F.)[1]. A recuperator is a device whereby heat is continuously transferred from hot waste gases to combustion air and thus preheating it. In many modern soaking pits, batch or continuous type reheating furnaces, ceramic recuperators are used for achieving very high preheat temperature. The efficiency of the ceramic recuperative system depends upon the quality of the refractory tube and the mortars used for jointing material.

Mortar joints are inherently more porous than the bricks. Hence it is expected that a considerable quantity of air infiltration may take place through the numerous mortar joints in a recuperator system. Suitable quality of mortar obviously will help in minimising the air infiltration, thus increasing the efficiency of the recuperator.

In authors' plant, attention has been paid to minimise the air infiltration through the mortar joints and studies were taken up for developing a suitable mortar for recuperator joints. Mortars of different compositions, gradings and bond contents have been taken into consideration for studying the various properties viz. bonding strength, drying, firing changes, workability, plasticity, index, porosity and permeability at the ambient temperature and also at elevated temperature. The performance in actual service condition has also been followed up. The details of the above work have been given in the present communication.

Experimental

Different compositions of alumino-silicate mortars with various bond contents and varying grain sizes were made in the laboratory. The general chemical analysis and physical properties of the mortars are given in Table I. Briquettes of 175 mm x 25 mm x 25 mm. sizes and standard tensile briquettes were made with different compositions by hand ramming. These were dried and fired at 300°C, 600°C. and 1000°C. for one hour in an electric furnace in oxidising atmosphere. Room temperature properties were determined after drying at 110°C.

Tensile strength

The tensile strength of the briquettes are given in Table II and Fig. I.

Linear changes after firing

The drying and firing changes of the mortars are given in Table III.

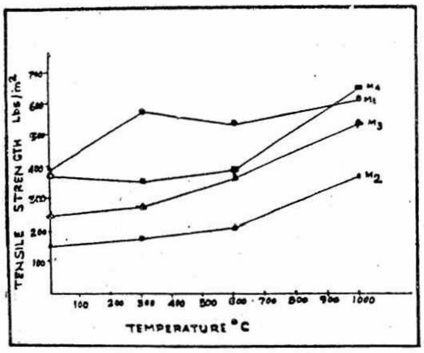

Fig. 1 Variation of tensile strength of different batches

Differential thermal analysis

The differential thermal analysis of different samples were done in the laboratory using conventional method and apparatus. The results are plotted in Fig. 2.

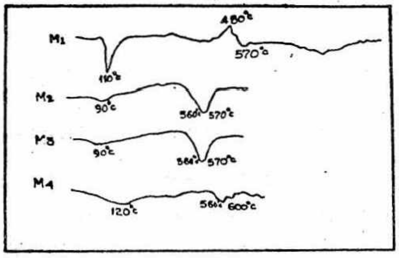

Fig. 2 D. T. A. curve of different composition

Thermo gravimetric analysis

Thermo gravimetric analysis were carried out on dried samples using standard apparatus and the heating rate was 10°C/min. From the cumulative weight loss curves, the total loss at a particular temperature range was noted and given in Table IV.

Apparent porosity

The apparent porosity of the mortars are given in Table V and illustrated in Fig. 3.

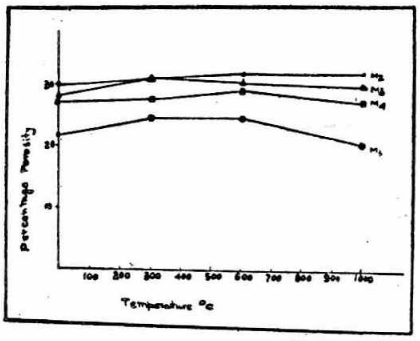

Fig. 3. Apparent porosity of different mortar compositions with increase of firing temperature

Permeability

The permeability of the samples dried at 110°C and fired at 1000°C/1 hr. were done as per B. S. 1902 using a standard apparatus. The results are given in Table VI.

Study of crack formation

The recuperator tubes were laid with mortar M1, M2, M3 and M4 and these were air dried and also fired at 1000°C. Visual examination was made for assessing the nature of cracks formed during drying and firing. The cracks formed during drying of the joint laid with M2 mortar were found to be maximum whereas with M3 mortar hardly any crack was noticed. Cracks formed with M1 and M4 were of moderate nature. On firing at 1000°C, the same trend was noticed with increased severity. In case of M3 no crack was visible even after firing. Figs 4-A and 4-B show the nature of cracks formed during drying and firing respectively.

Plant performance

In authors' plant, soaking pits are having the refractory recuperation system. The recuperators are of cross flow design. The recuperator tubes are of 45—58% alumina quality with the shelf tiles of 45% alumina type. Each pit is connected to two recuperator chambers on either side. Each chamber is packed with approx. 980 tubes. All the four types of mortars were used in different pits for laying the recuperator tubes. Their performances were assessed by measuring the difference in the oxygen content in the flue gas before and after the recuperator chamber. The excess oxygen, if any, found in the flue gas was comput-

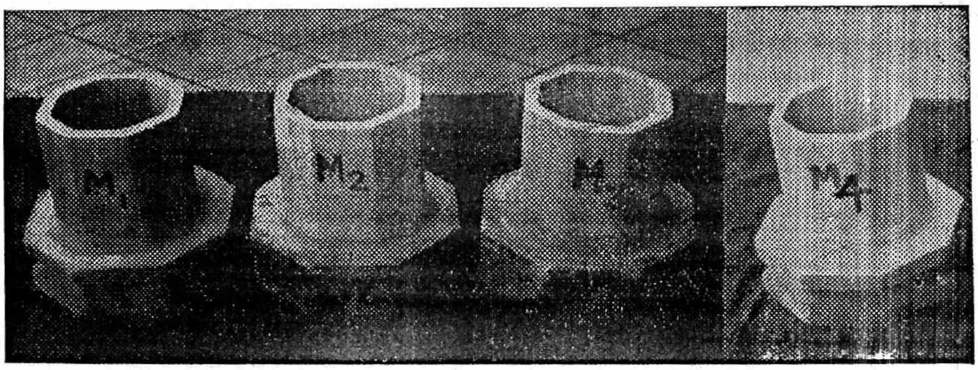

Fig. 4-A Nature of cracks formed after drying with different mortars

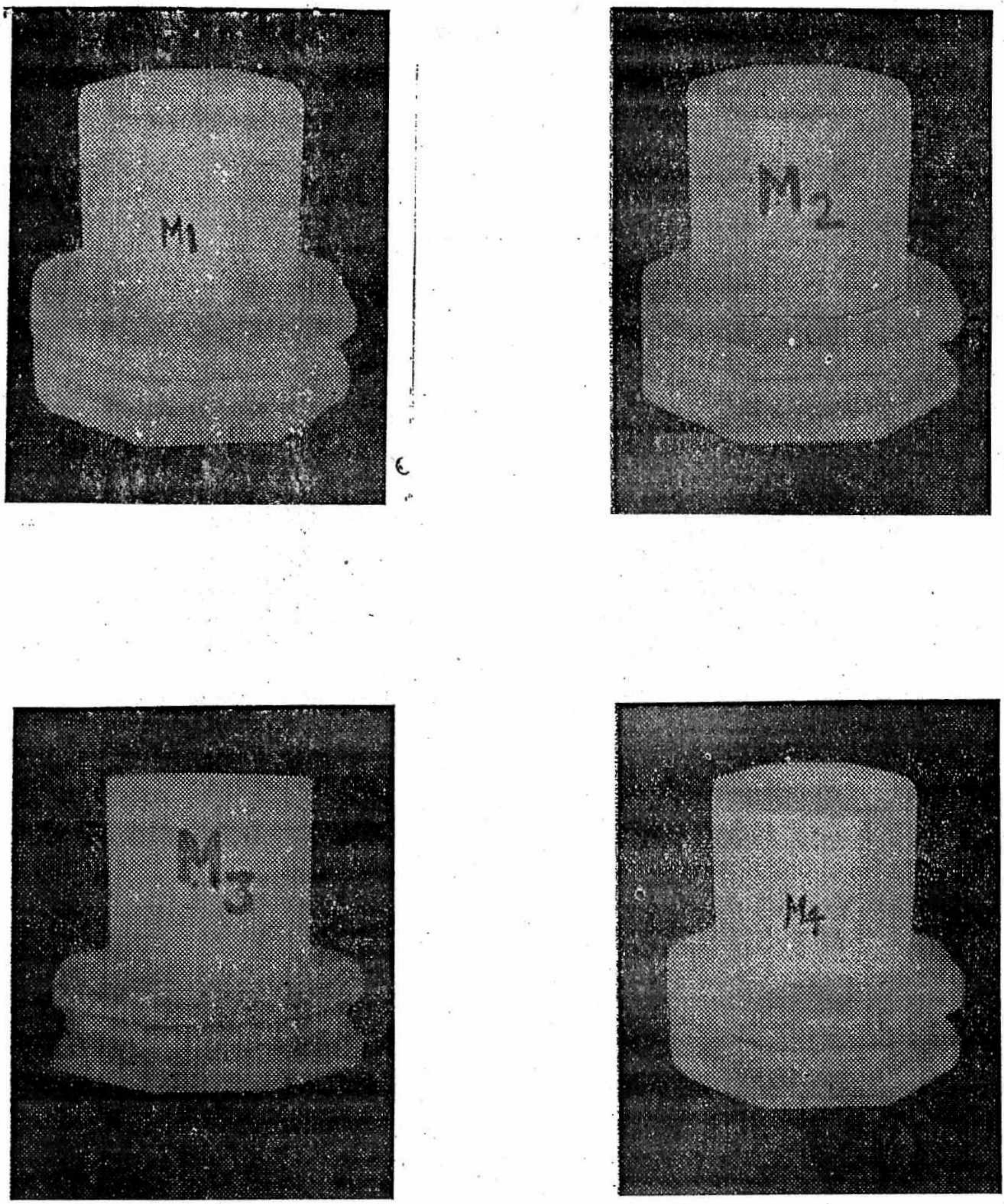

Fig. 4-B **Nature of cracks formed after firing to 1000 °C with different mortars**

ed for air infiltration. The air infiltration data thus obtained within a few months is given in Table VII.

Discussion

From Table I it can be seen that MI had lower alumina content than other mortars. MI and M2 were of fine grains, whereas M3 and M4 were comparatively coarser materials. The setting time of MI was much lower than those of other mortars, and its workability was better than others due to addition of air setting bond.

The refractoriness of MI was comparatively lower than M2, M3 and M4. This has also reflected in strength characteristic which showed increasing trend at a comparatively lower temperature.

The tensile briquettes fired at different temperatures indicated that the strength of MI with air setting bond was considerably increased at 300°C followed by a decrease in strength at 600°C. The strength increased at 1000°C. The increase in strength at initial stage was due to gel formation and partial evaporation of moisture upto 300°C. At higher temperature gel structure decomposed with consequent reduction in strength. This might be explained by the fact that over this temperature range, loss of honey-comb structure has occured with the formation of primary silica gel due to dehydration[4]. At 1000°C ceramic bond started developing with increase in strength.

In case of M2, M3 and M4 the strength was increasing gradually with the increasing of firing temperature. This was due to the formation of alumino phosphate. The lowering in strength of M4 at 300°C was due to higher moisture pick up caused by the presence of pyrophosphate in the specimen fired at a low temperature. M3 showed much improved strength as compared to M2 which was due to the presence of more P_2O_5 in the bonding media.

Crack formation during drying was maximum with M2 mortar which was due to the higher drying shrinkage. M3 had the minimum drying shrinkage, as such no crack was observed. The photographs of the recuperator joints revealed that micro-cracks developed during drying of the mortars MI, M2 and M4 had been extended further during firing at 1000°C. This indicated that the mortar M3 was less susceptible to spalling than the mortars MI, M2 and M4.

The differential thermal analysis of MI with air setting bond indicated that at 110°C, the endo-thermic peak was formed due to evolution of moisture present in the mix. The small exothermic flat peak at 480°C denoted that some oxidising material was present in the mix. The endothermic peak at 570°C indicated the presence of free quartz and decomposition of structural water of the clay. In case of M2, M3 and M4 with phosphate bonds, the first endothermic peaks were obtained at 90°C for M2 and M3 whereas the peak was observed at 120°C in case of M4. These were presumed to be due to the formation of mono-substiuted alumino phosphate[2]. The monoaluminium phosphate often transformed to aluminium pyrophosphate at lower temperature and then transformed to meta phosphate[3]. The endothermic peaks at 560°C to 600°C indicated that polymerisations were complete at that temperature range.

The thermo-gravimetric analyses showed a higher loss in case of M1 & M2 as compared to M3 and M4. This was due to the presence of more structural water in the former as because the mix contained more clay.

The apparent porosity of all the samples indicated an increasing trend upto 1000°C except in M1 where there was reduction in apparent porosity at 1000°C due to partial sintering of the mix. Decrease in apparent porosity was due to elimination of moisture and organic matter at initial stages. At 1000°C reduction in apparent porosity was not observed due to less vitrification. This was also compared with the linear changes of different mixes.

Permeability of M1 dried at 110°C and fired at 1000°C increased due to the formation of micro cracks in the specimen during cooling. The specimen M2, M3 and M4 indicated better results due to less vitrification with the formation of aluminium phosphate in the matrix. Though the porosity of M2. M3 and M4 were high as compared to M1, the permeability of M3 was minimum after firing at 1000°C. This might be due to more P_2O_5 content which has helped in reducing the channel porosity by formation of more alumino phosphates.

The service performance showed minimum air pick-up with M3 mortar. This might be due to lower drying and firing shrinkages and also for low permeability.

Conclusion

1) Air infiltration can be reduced by proper quality of mortar.

2) Optimum grain size is required for minimising the crack formation and to control the drying shrinkage.

3) Low drying and firing shrinkage of the mortar is of paramount importance.

4) Air permeability of the mortar should be minimum so as to control the air infiltration.

5) Too much sintering as depicted by the strength characteristic is not considered favourable which may increase the glass content with consequent loss in spalling resistance.

Acknowledgement

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References

1, DAVID, A. VAN CLEAV "Heat recovery Equipment puts waste to work". Iron Age Vol. 220 No. 3, 1977, pp 30.

2. S. K. MITRA etai, "Phosphate bonded monolithics". Proc. of seminar on monolithic refractories, April 1971, pp. 91--96.

3. G. N. DUDEROV, "Use of alumino-phosphate bonds in the manufacture of unfired refractories" Refractories (Translation of Ogneupory) 1964 (9—10), pp. 485—91.

4. H. M. GLASS, "Soluble silicate as bonding media". Discussion, Trans. British Ceram. Soc. Vol. 48, 1949, pp. 161—176.

TABLE No. 1 Chemical and Physical properties of the mortars.

Comp.	Bond used	Chem. analysis, % SiO_2	Al_2O_3	P_2O_5	Screen analysis, % −0.8 to +0.2mm	−0.2 ot +0.10mm	−0.10 mm	P. C. E.	Workability index	Plasticity index Atterberg	Final Setting Time Hr. Min.
M1	Air setting	51.60	37.25	—	7.4	32.8	59.9	1590°C	Excellent	5.8	2 30
*M2	Soluble phosphate	47.80	39.23	0.75	12.3	18.7	69 0	Orton 31—32	Good	9.11	5 05
*M3	Soluble phosphate	42.60	43.08	1.61	5.5	49.1	45.5	Orton > 32	Good	7.9	7 15
*M4	Phosphoric acid	46.68	41.93	1.02	4.8	44.6	50.5	Orton > 32	Good	6.5	More than 8 hrs.

* With varying bond contents.

TABLE—II Tensile strength of different mortar compositions

Mortar marked	As such (dried at 110°C) psi	300°C/1 hr. psi	600°C/1 hr. psi	1000°C/1 hr. psi
M1	384.3	579.0	539.6	611.0
M2	147.3	193.0	203.3	363.0
M3	247.0	273.0	381.3	540.0
M4	373.3	354.0	388.6	650.0

TABLE—III Drying and firing changes of different mortar compositions

Mortar marked	Drying shrinkage, %	300°C/1 hr. %	600°C/1 hr. %	1000°C/1 hr. %
M1	—2.86	—0.329	—0.414	—0.601
M2	—4.13	—0.156	—0.146	—0.627
M3	—1.47	—0.052	—0.155	—0.240
M4	—1.55	—0.153	—0.085	—0.102

TABLE—IV Thermo gravimetric analysis of various mortars

Mortar marked	Temperature range, °C	Total weight loss (%)
M1	260 — 900	4.06
M2	280 — 940	4.82
M3	440 — 900	3.20
M4	320 — 970	3.86

TABLE—V Apparent porosity of various mortars

Mortar marked	As such dried at 110°C/ %	Fired at 300°C/ 1 hr. %	Fired at 600°C/1 hr. %	Fired at 1000°C/1 hr. %
M1	21.7	24.9	24.6	20.3
M2	28.0	31.2	32.0	32.4
M3	29.8	31.2	30.7	31.6
M4	27.5	28.0	29.4	29.5

TABLE—VI Permeability air/cm^2/cm/sec/cm/head of water

Mortar marked	M1	M2	M3	M4
As such (Dried at 110°C)	0.0490	0.0425	0.0462	0.0213
Fired at 1000°C/1 hr.	0.06525	0.0446	0.0321	0.0482

TABLE—VII Average percentage of air pickup accross the recuperator during service for different mortars

Mortar marked	%
M1	19 — 25
M2	20 — 30
M3	12 — 18
M4	19 — 25