MICROWAVE SINTERING OF ABRASION RESISTANT ALUMINA LINER TILES

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ABSTRACT

The need to move large volumes of highly abrasive materials, such as coal, grain, sand, and various waste products, places great demands on the piping systems through which they are conveyed. Excessive wear is a common problem, especially evident in the sections of pipe, which change the direction of flow of the abrasive material. . Due to their excellent wear characteristics, ceramic materials have become increasingly popular for use in lining pipe casing subjected to high abrasive wear conditions. Presently, tiles of high alumina are manufactured by our industry and applied in large pipes in thermal power plant, cement, fertilizer, coal washer etc. They are made by conventional process, which takes nearly 30 hrs for firing through a kiln. In addition, the kilns are oil fired, necessitating a re-look at an alternate process which is not oil dependent and less polluting and yielding products of better quality.

The potential advantages of using microwave processing of materials such as short process time, energy efficiency and improved properties have been long recognized. Only recently effort to use it in processing some special ceramics, tungsten carbide based (WC) composites, fabrication of transparent ceramics, and sintering of powdered metals have been successful. [1-4]

In this study, feasibility of microwave sintering of high alumina tiles [5] to realize the short process time and energy efficiency advantage of the process and improved properties of the tiles were established. The results also generated inputs required for scale-up of microwave processes for production such as the uniformity and reproducibility of material properties for given processing parameters.

Detailed investigations were carried out on tiles sizes varying up to 130 (L) x 50 (W) x 20 mm (t) using a 6 kW batch processing Microwave sintering oven . Initially 1" specimens were sintered in "sinterwave", a modified kitchen microwave (designed and fabricated by us) system to estimate the sintering temperature, densification and hardness. The 6 kW batch system form M/s COBER, USA was used for component sintering studies and test samples which are larger in size. The Microwave system was integrated with a Mikron 680 IR temperature measurement system of 0.68 μm spectral response and a Eurotherm PID controller 2404 to control the process parameters including ramp rate.

The critical components for microwave sintering include an insulation box and the susceptors. The insulation box consists of a small chamber fabricated from RATH 17/400 grade vacuum formed low density Fiber insulation board of 25 and 50 mm thickness. The casket were made with an ID of 170 mm x 150mm x 150 mm deep and a top cover with a 12 mm hole for IR temperature measurement. Si_3N_4 bonded SiC plates of size 150mm L x 100 mm wide and 10 mm thick were slipped on either side of the stack. The positioning of susceptors was significant in getting dimensionally stable and crack free sintered product. The caskets and the tiles are shown in Figure 1. The process was optimized obviating the need for any intermediate soaking for binder burnout in flat tiles. However for curved tiles intermediate soaking was required to avoid small cracks at the curvature. A maximum of 5.5 kW microwave field was used in the test conditions and the time to reach 1600 ^{0}C was varied from 60 – 90 minutes. During soaking, the turntable was stopped and the temperature variation could be limited to less than 1 degree.

Number of batches of test pieces were also repeatedly sintered under different conditions to estimate the optimum sintering parameters, uniformity of sintering and reproducibility of material properties such as hardness, water absorption, cold crushing strength (CCS), Modulus of Rupture (MOR) and relative abrasion resistance, which is measured by relative abrasion index (RAI) value to evaluate improvement in properties.

X ray diffraction was also carried out to evaluate the formation of alumina phase. In conventional processing, a total load of nearly 4 MT tiles of different sizes are stacked and sintered in an oil fired fast OTH furnace. The total process duration is 30 hrs. The specified properties for the product were taken as a reference to evaluate the properties obtained by Microwave sintering. Conventionally, the sintering temperature for the product is 1600 ^{0}C with a soaking period of 4 hrs. The raw materials used are of commercial grade materials with nearly 80 % alumina, clay and MnO.

The results of Microwave sintered tiles are very interesting considering that the basic raw materials are commercial grade materials and not as experimented usually in laboratories with high purity materials. The total mass of each batch was in 2 kg range.

Even with a soaking time of 60 minutes, the total sintering duration can be limited to nearly 90 minutes to get the required properties in test samples. The required product specification of the water absorption is <1 % , Thermal expansion , and hardness are realized even for samples sintered at 1500 – 1530 ^{0}C. However the cold crushing strength (CCS) & 3-point modulus of rupture (MOR) were consistently lower. Graphs 1-4 show the properties as a function of sintering temperature. Even prolonged soaking time did not improve these properties. The fractured surfaces indicated the absence of liquid phase sintering. The x ray analysis showed minor phases other than alumina peaks. However, x ray phases became refined from 1550 ^{0}C onwards. The fractured surfaces started showing liquid phase sintering. It was evident from MnO flux melting and migrating to the surface leaving a dark brown skin color and an ivory colored alumina core. In components sintered at 1500 – 1530 ^{0}C showed uniform light brown color on the fractured surface. This observation demonstrates that a minimum sintering temperature of 1550 ºC is essential for these components, which is nearly 30 degrees lower than conventional sintering temperature. Figures 1-4 show the properties as a function of sintering temperature. And it clearly indicates that there is an accelerated improvement in properties around 1500 C. The results indicate that the abrasion resistance is higher even for samples sintered at 1500 ^{0}C, in spite of slightly lower cold crushing strength and modulus of rupture (Table 1).

The reproducibility of the density, hardness, and microstructure was explored on component level. Higher soaking time was essential to realize all the properties in the component. The results of sintering studies have established that microwave sintered high alumina tiles have superior properties than as conventionally sintered component (Table 2 & 3). The cold crushing strength and modulus of rupture are consistently higher with very high abrasion resistance index. The process time has been significantly reduced to about 90 minutes in samples and to about 180 minutes in components as against 30 hrs in conventional processing. .The orientation of the sample is also important. Three / four tile stacking has not effected large variations in hardness between components due to masking effect.

Though it is not prudent to compare the processing cost per tile in a 6 kW microwave system and the conventionally tonnage OTH furnace, it appears that the former method is very advantageous. The results of the studies are convincing that if large batch systems of 1 - 2 cu. m loading capacity can be developed it will be commercially beneficial and more importantly we can realize products with improved properties using a green technology.

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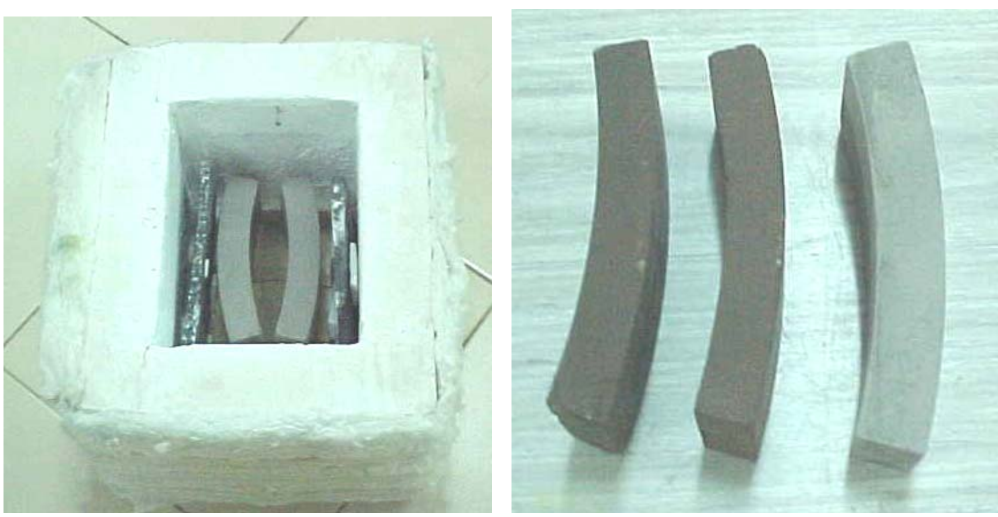

Figure 1 : a) Tiles loaded in a casket b) sintered and green tile.

Table 1. Data on Microwave Sintered Test Samples.

SL. NO.	SINTERING CONDITION	DENSITY (g/cc)	ABSORPTION (%)	MOR (Kg/cm^2)	CCS (Kg/cm^2)	RELATIVE ABRASION INDEX
1	1450°C / 60 min	3.05 ± 0.5	4 -6 %	-----	------	-----
2	1500°C / 60 min	3.1 ± 0.5	2.47 ± 1.5	1370 ± 200	1860±250	5.0 ± 1.0
3	1550°C / 60 min	3.22 +/-0.01	1.47 ± 0.58	2158 ± 195	2070 ± 543	4.85 ± 1.3
4	1560°C / 30 min	3.31+/-0.04	0.41 + 0.42	1919 ± 268	2849 ± 1232	4.07 ± 1.79
5	1560°C / 60 min	3.38 +/- 0.02	0.08 + 0.3	2403 ± 43	3123 ± 1316	7.10 ± 1.55
6	1580°C / 30 min	3.34 +/- 0.04	0.19 + 0.29	2191 ± 239	3563 ± 1506	13.39 ± 3.05
7	1580°C / 60 min	3.39 +/- 0.05	0.16 + 0.43	2715 ± 746	4066 ± 1812	13.11 ± 8.215
Ref	1580°C/ 240 min, total time 30 hrs	3.2 -3.4	<1	>2200	> 3000	>4

Table 2 : Property Variation on Tile Component at Different Segment.
(Tile size 108 x 52/42 x 20 mm thick sintered at 1600 for 60 minutes)

SL. NO.	B.D (g/cc)	% A.P (%)	% W.A (%)
1.	3.348	1.106	0.330
2	3.378	0.661	0.196
3	3.360	0.752	0.224
4	3.373	0.771	0.228
5	3.343	2.136	0.635
6	3.368	0.736	0.218

Table 3. Effect of Sintering Condition on Bulk Density and Absorption on Tile Component .

Sl.No.	Sintering Condition	Dimension (mm)	B.D (g/cc)	% W.A (%)
	Rectangular flat tiles			
1	1580 C/60 min	127 X 51 X 19.5	3.47 ± 0.11	0.92 ± 0.92
2	1600 C/60 min	108 X 52/42 X 20	3.36 ± 0.018	0.30 ± 0.22
3	1600 C/90 min; int. soak at 1100	127 X 51 X 19.5	3.48 ± 0.075	0.95 ± 0.61
4	1600 C/90 min int. soak at1100	139 X 51 X 19.5	3.36 ± 0.08	0.58 ± 0.52
	Rect. Curve Tiles , int. soak to avoid cracks			
5	1580 C/90min	139 X 51 X 19.5	3.36 ± 0.05	0.88 ± 0.61
6	1580 C/90 min; int. soak at 1100C	139 X 51 X 19.5	3.48 ± 0.58	0.88± 0.44
7	1600 C/60 min, int. soak at 560	139 X 51 X 19.5	3.38 ± 0.02	1.23 ± 0.22
8	1600 C/90 min , int. soak at 560	139 X 51 X 19.5	3.43 ± 0.05	0.66 ± 0.37

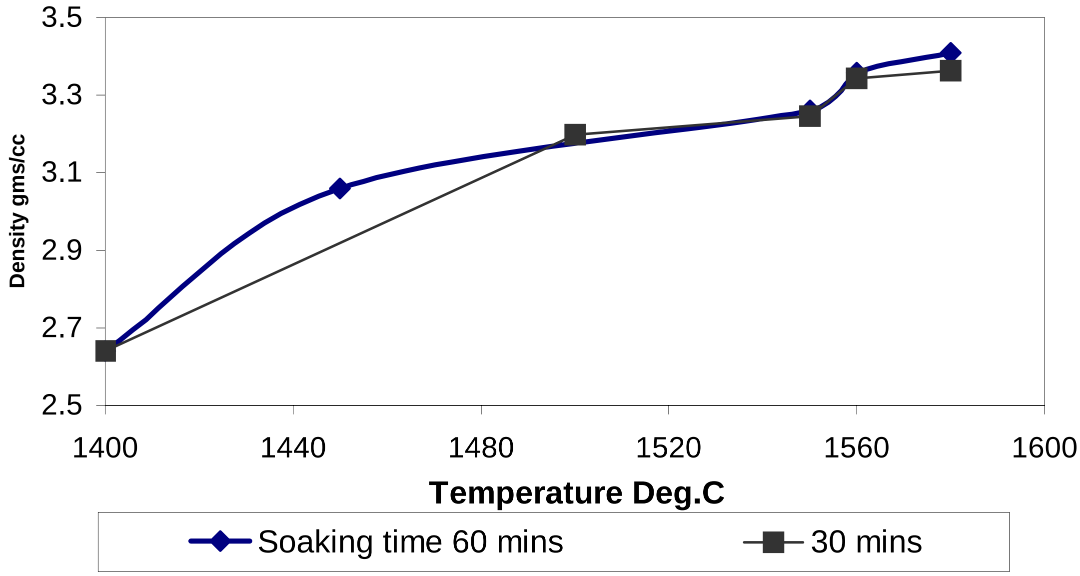

Figure 1. Bulk density of ceralin sample as a function of sintering temperature.

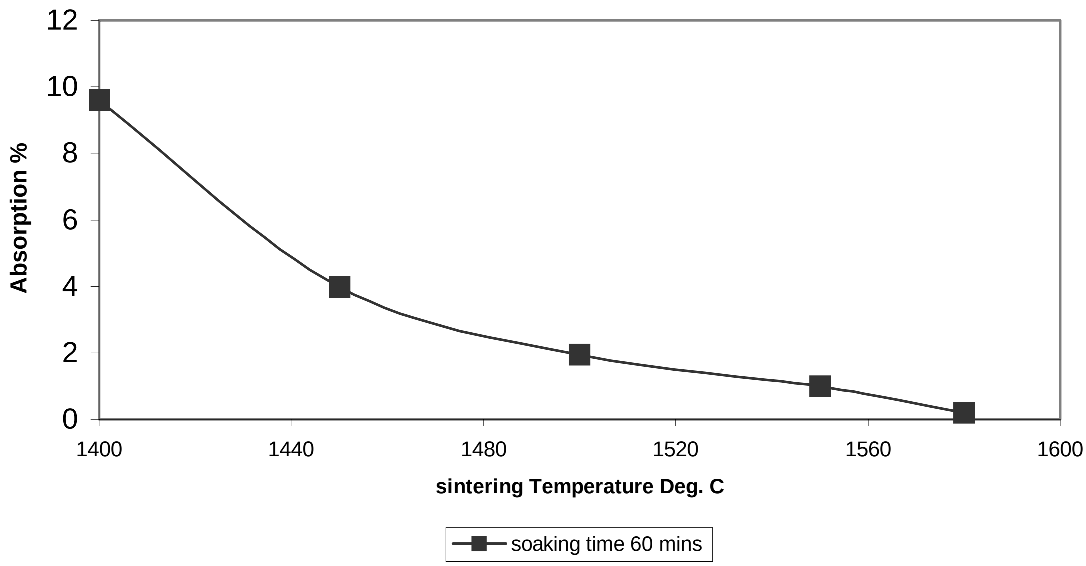

Figure 2. Water absorption of ceralin sample as a function of sintering temperature.

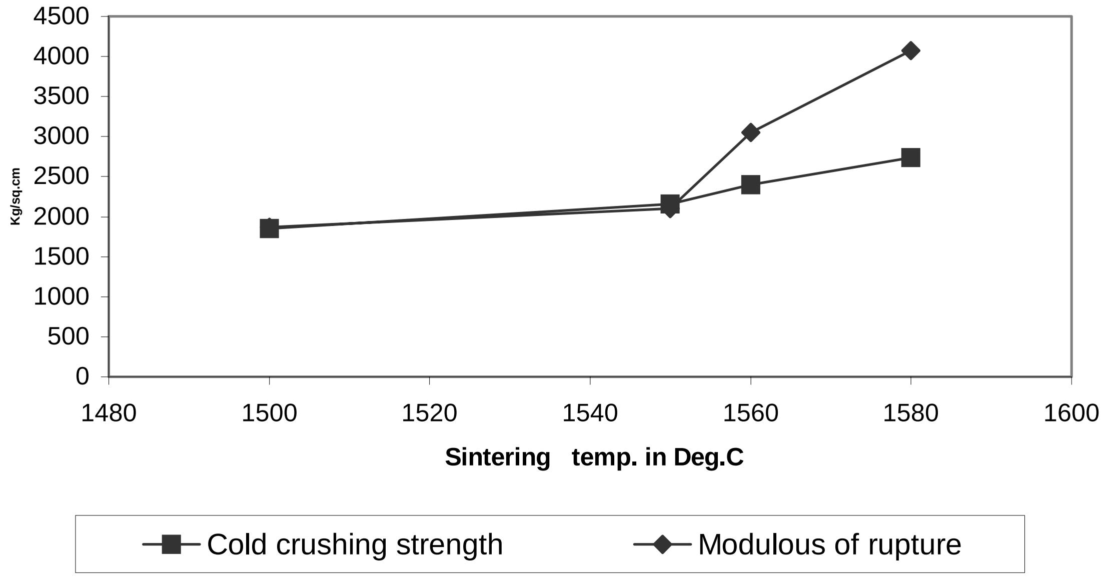

Figure 3. CCS & MOR of ceralin sample as a function of sintering temperature.

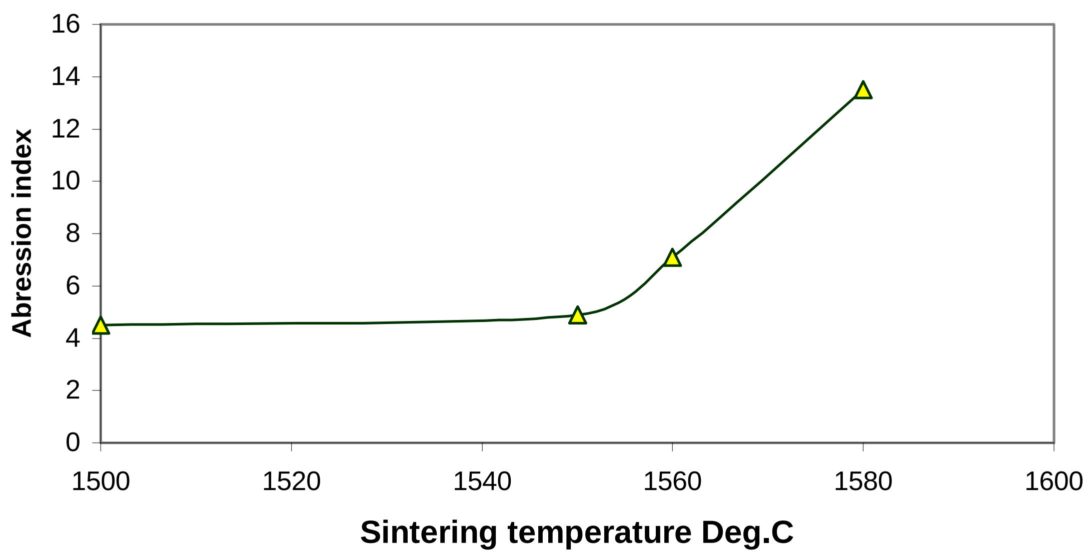

Figure 4. Abrasion index of ceralin sample as a function of sintering temperature.