Development of creep resistant Magnesite checker brick for glass tank furnace regenerator

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Abstract

In this paper, different grades of Magnesite has been characterized and found that, lowest creep at 1500°C is given by the material with very high purity fused magnesia.

Introduction

A Glass tank furnace regenerator consists of a regenerator chamber in which a chimney blocks or checker work of refractory bricks has been stacked with different qualities of Magnesite in different zones. In one cycle the checker is heated up by flue gases, subsequently in the following stage (20-30 minutes) the heat is transferred to combustion air. These furnaces are provided with 2 or more (an even number) re-generators. In principle the optimum half-cycle time depends on the pull of the melting tank (thermal load). During the burner reversal, lasting about 30 - 60 seconds, there are no flames within the furnace. Regenerators utilize checker brick to improve efficiency by taking advantage of the excellent heat exchange properties inherent in ceramic materials. As the furnace exhausts through the checker packing, the bricks are preheated by the waste gases, providing a source of energy to preheat the combustion air when the cycle is reversed. Regenerator efficiency can be affected by a variety of factors, from pack design to regenerator size.

In Magnesite based refractories, the creep is affected by the presence of impurities like SiO_2, CaO, Al_2O_3, Fe_2O_3, B_2O. These impurities are present at the grain boundaries of the periclase. The grain boundaries of the material are the most reactive sites. At high temperature these impurities become liquidus and facilitates grain to grain sliding. The creep of Magnesite bricks also depends on the CaO/SiO_2 ratio, which determines the low melting phases formed. Another issue governing this creep property is the viscous flow and migration of dislocation. The influence of pores is more sensitive with material having large crystal sizes. So, the creep of a material is based on combination of the above factors involved that work together.

According to Simonov et al, the deformation due to creep is due to occurrence of several factors like grain boundary slippage, viscous deformation and climbing of dislocations. The influence of porosity is more sensitive in materials with large crystal size and basic refractories with the lowest silicate content having $CaO/SiO_2=2$ is the most deformation resistant.

According to Banerjee et al, creep characteristics of Magnesite refractories showed no deformation at 1450°C when compared to forsterite brick which shows poor strength even at low temp of 1200°C. The Magnesite proved to superior than chrome-mag and mag-chrome bricks. The strength of chrome-mag and mag-chrome bricks showed decrease in strength at 1350°C.

In this paper, study has been done on the properties of Magnesite brick with a special attention to its creep property. Magnesite used for manufacturing of bricks are taken from different sources and the comparative properties of the brick has been discussed. We have also added different additives to achieve the most desired property, that is creep at 1500°C less than 0.2%.

Experiment:

As per the Andreasen equation different quantity of these materials with optimum grading was taken to prepare different compositions with different q values to achieve the highest packing density. Twenty one different batches of bricks were manufactured with different q values from 0.32 to 0.52 and compared their apparent porosity.

Commercially available different types of high purity magnesia grains with different size fractions as per Table 1 were used as major raw materials. The gradation was decided based on the best value obtained from the Andreasen equation. Four different batches of bricks were manufactured and compared their physical, chemical and thermo-mechanical properties along creep at 1500°C.

Component	T 1	T 2	T 3	T 4
97% Sintered Magnesia	√			
99% Sea Water Magnesia		√		
97% Fused Magnesia	√		√	
99% Fused Magnesia		√		√

Table 1: Different formulation with various Magnesite

The set of trial samples consisting of five different recipes were shaped into bricks by using industrial hydraulic press with a specific pressure of 1.8 Ton/cm^2 and samples were dried at 110°C for 24 hrs. After drying, the samples were fired at 1680°C with predetermined heating schedule and soaking time in high temperature tunnel kiln. The fired samples undergo testing as per the industrial testing practices. Apparent porosity (AP), bulk density (BD), cold crushing strength (CCS) and Creep at 1500°C. Micro structure analysis were done by using Optical Microscopy (Leitz orthoplan pol optical microscopy with image analyzer). Creep was tested using creep instrument Netzsch 422, Germany, microscopy was done in Carl Zeiss Scope A1 model no.- 10002158. Each value of the tested samples was average of three parallel samples.

Results and discussion:

Table 2 shows the chemical properties of different types of Magnesia used. It shows that higher the percentage of Magnesia in the raw material, lesser is the impurities.

RAW MATERIAL	MgO (%)	SiO_2 (%)	CaO (%)	Al_2O_3 (%)	Fe_2O_3 (%)
97% Fused Magnesia	96.14	0.88	0.85	0.70	0.56
97% Sintered Magnesia	97.32	0.34	1.26	0.18	0.64
99% Sea Water Magnesia	98.28	0.18	0.86	0.18	0.32
99% Fused Magnesia	98.88	0.1	0.58	0.16	0.2

Table 2: Chemical analysis of raw materials used

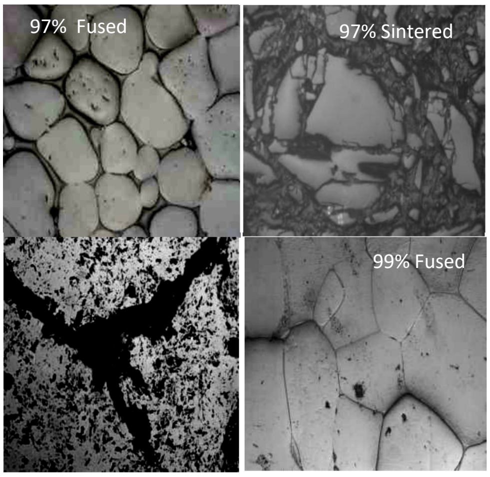

Figure 1: Microstructure of different Magnesites

From the micro-structure of the raw materials, it can be clearly stated that in 99% Fused Magnesia has much better grain structure and larger grain size than the other Magnesites. More over, as the grain boundary is much higher in 99% Fused Magnesia, the chance for presence of low melting impurities in the grain boundary is lesser. The average grain size in 99% Fused Magnesia is around 430µm whereas for 97% Fused Magnesia it is 390µm, 97% Sintered Magnesia it is 55µm and for 99% Sea Water Magnesia the average gain size is 65 µm.

Processing of particulate systems is determined by particle packing, hence particle size distribution and particle morphology. Packing theories are widely applied in refractory engineering. Andreasen defined the particle size distribution modulus, q, given by Eq. (1), as a measure of the contribution of the various ingredient size classes that compose the mixture to the overall particle size distribution.

$$\mathrm{CPFT}(\%) = 100\left(\frac{D^q - D_S^q}{D_L^q - D_S^q}\right) \quad (1)$$

Where,
CPFT = Cumulative Percentage Finer Than
q = Distribution co-efficient
D_S = Minimum Particle Size
D_L = Larger Particle Diameter
D = Average Particle Diameter

Figure 2 shows the different Apparent porosity values for all the batches of 0.32 to 0.52. From the plot, it is clear that at q value o.37, the best packing has been achieved as the Apparent Porosity is lowest in q=0.32.

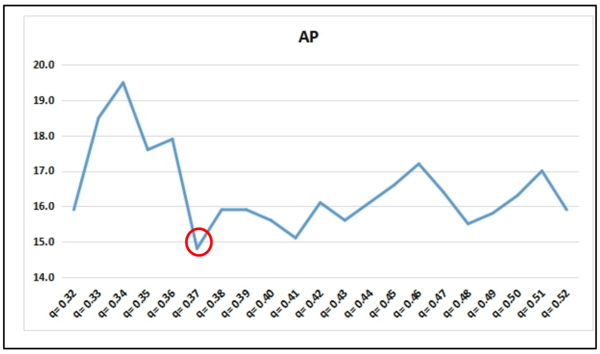

Figure 2: The variation of AP with respect to different q values

Figure 3 shows the physical properties of different batches of the Magnesite brick. T-4 showed low porosity, high bulk density as well as high CCS. Optimum quantities of different size fractions are selected based on the Andreasen's equation to achieve highest packing density and subsequently low porosity. As, impurity is very low and matrix is dense due to proper selection of coarse to fine ratio, the brick T-4 shows better physical properties.

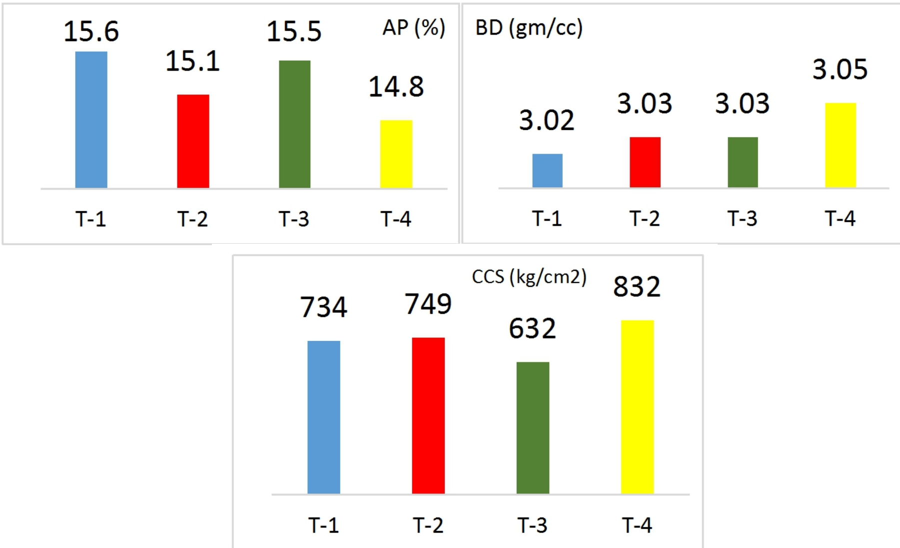

Figure 3: Physical Properties

Creep at 1500°C (Figure 4a, 4b, 4c, 4d) shows that Z_{5-25} for T-4 is minimum. The value is 0.20%. As the impurities are less, at high temperature firing, the impurities will not form high amount of low melting phases which causes grain dislocation. So, material with 99% Fused Magnesia shows lower creep value at 1500°C. Moreover, we have seen in Figure 1, that the grain boundary for 99% Fused Magnesia is maximum among the Magnesites and the pores are less, thus it gives better creep resistant property that is very important for Glass Regenerators.

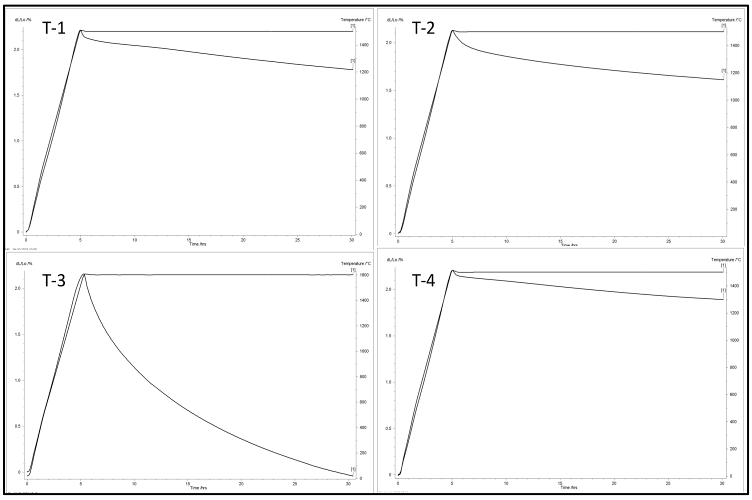

Figure 4: Creep Curves for T-1 to T-4

Recipe	Creep Z_{5-25}(%)
T-1	0.46
T-2	0.26
T-3	0.25
T-4	0.20

Table 3: Z_{5-25} values for T-1 to T-4

Micro-photograph (Figure 5) shows that there is compact matrix with high degree of bonding between grains of periclase with very little impurity phases in the grain boundary for T-4. In T-1 and T-3, the amount of pores as well as the liquid phases in the grain boundary is clearly visible. From Table 1 and 2, it can be seen that the impurity percentage in 97% Fused Magnesia and 97% Sintered Magnesia is much higher than that of 99% Fused Magnesia and Nedmag. But in Nedmag, the grain boundary is smaller than that of 99% Fused Magnesia. Nedmag grains are more prone to dislocation than 99% Fused Magnesia because the grain size is higher in 99% Fused Magnesia than that of Nedmag.

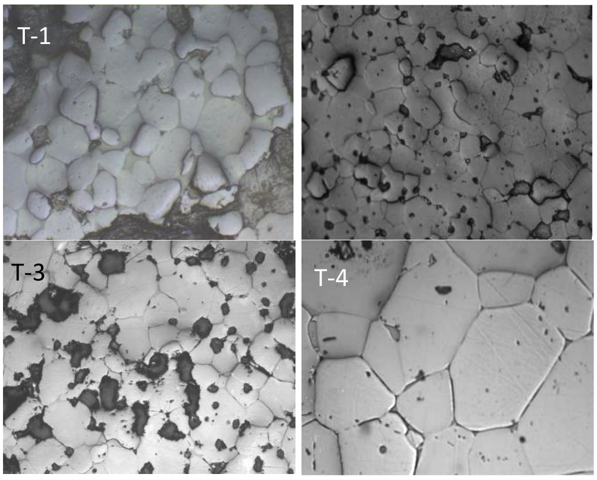

Figure 5: Micro-photograph of T-1 to T-4

Conclusion:

Creep at 1500°C and microstructure studies were conducted on all the above trials with grades of Magnesia to develop a Magnesite brick with creep value (Z_{5-25}) less than equals to 0.20%.
The following conclusions may be drawn from the study .
1) With formulation T-4, as the impurity percentage is less and the grain size is more, grain dislocation could not happen easily, thus providing Z5-25 value as 0.20%.
2) In T-1, the 97% Sintered Magnesia and 97% Fused Magnesia has more impurities and less grain size, thus resulting in low creep.
3) In T-2, the creep is better than T-1 as 99% Sea Water Magnesia and 99% Fused Magnesia has less impurities and high the grain size than 97% Sintered Magnesia and 97% Fused Magnesia. But, the grain size of 99% Sea Water Magnesia is lesser than 99% Fused Magnesia, so T-4 shows better creep.
4) In T-3, the creep is better than T-1, but poorer than T-2 and T-4 as grain size of 97% Fused Magnesia is less than that of 99% Fused Magnesia. Along with that, impurity percentage in 97% Fused Magnesia is much higher than that of 99% Fused Magnesia or 99% Sea Water Magnesia.