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SPECIFICATION OF MgO−CONCENTRATE OBTAINED FROM POST MORTEM MgO–C REFRACTORY MATERIALS

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The recycling process of post mortem MgO–C (MC) materials is based on carbon removal from the crushed material. The laboratory results of the annealing of the crushed post–mortem MC bricks in a static layer, in an air atmosphere in an electric furnace are presented. The quantity and quality of the fractions size was evaluated before and after annealing. The results show that the carbon inside the grains of size 6.3 - 15 mm is incompletely burnt out under the given conditions. The results from annealing of the fractions size below 6.3 mm confirmed that the carbon burning out decreases the strength and density of the agglomerates, which then partially disintegrate during annealing. A mechanical load is needed for the complete disintegration of the brittle agglomerates. In addition, the results of the MgO-fractions quality evaluation are discussed, which were obtained by annealing the grit from the post–mortem MC bricks from the pilot-plant experiment in a rotary kiln in the factory Hačava, from the company INTOCAST Slovakia a.s.. The obtained fraction size of MgO possesses a suitable chemical and phase composition. However, the strength of the MgO-grains core was lower. This confirms that the post–mortem MC materials may be recycled; however, it requires waste management and the related technical equipment for the recycling processes.

INTRODUCTION

Although most refractory materials from the lining of pig iron and steel production aggregates do not have a negative impact on the ecosystem of a landfill, the pressure on the reduction of landfill material and the economic recovery of post-mortem refractories are growing. The rise of basic raw material prices and land-filling of industrial waste lead to the development of specific recycling technologies allowing for the recovery of part of the refractory materials possessing suitable quality for other applications. The quality evaluation of the obtained recycled materials from worn linings is important for their correct reuse in the ceramic and metallurgical industry [1, 2]. The state of the post-mortem refractory linings determines the processing effectivity and the economy of the material recycling. The recovered materials may be used according to their quality as building materials (gravel) or as an additive to slags, also as secondary raw materials for the production of building ceramics, inorganic binding materials or some new non-shaped/refractories [3-10].

The present work is focused on the processing of dense periclase-carbon (MgO–C, MC) bricks from linings of high-temperature aggregates. The main target is to obtain a coarse fraction of a MgO−clinker. The periclase-carbon bricks are produced from fused magnesia or clinker, containing about 98 wt. % of MgO. To achieve a high density of the MC material, the mass is combined from several size fractions of MgO, mostly under 5 mm. The MgO particles are bound with carbon which is formed during the carbonisation of pitch with graphite. The content of carbon in the MC bricks is 6 - 10 wt. %. The bricks possess high density, high strength, and low slag wettability, therefore they are resistant to attacks of slag and molten metal up to a temperature of 1700°C [11-13].

Post mortem linings from MC-materials are suitable for recycling if they are not deeply disrupted by a corroding medium and are free of residual accretions. During the technological process, for example, about 1/3 to 1/2 of the volume of the lining MC bricks in a steel ladle are diminished due to the melt corrosion, however, the rest of the bricks are not impregnated deeper with the corrosive medium [14].

This work has verified the possibility to release coarse periclase particles from the agglomerates of crushed MC bricks after the decarbonisation process. The basic step of the processing is the destruction of the carbon bonds by burning it out in an oxidising atmosphere. The key processes for the MgO-concentrate recovery are: crushing, oxidising annealing, sieving, and the final classification of the fractions according to their quality.

EXPERIMENTAL

The effect of the carbon burn-out on the agglomerate strength decrease and the release of the MgO grains from agglomerates was studied in the laboratory.

The post-mortem MC 98/10 bricks were taken from the working lining of a steel ladle. The bricks surface was wrinkled but disrupted by corrosion only by a few millimetres (5 - 15 mm) in depth. The average chemical analysis of the material is listed in Table 1 together with the result of the analyses of the surface layer and the core of the bricks for comparison of the surface corrosion rate.

The post/mortem bricks were crushed to a grain size less than 15 mm (set 1) and less than 6.3 mm (set 2). The crushed material was sieved into more fractions. The fractions in batches of 100 g were gradually annealed in a thin layer (an area of 15 × 20 cm). The annealing of each fraction in air in an electric laboratory furnace at the temperature of 900°C was carried out for 30 min. The process of heating and annealing took about 120 min, then the material was cooled freely in the furnace.

The decarbonising effect on the disintegration and release of the MgO particles from the agglomerates during annealing, sieving and following the mechanical load was evaluated. Therefore, the following was observed: i) the weight loss of the annealed material and redistribution of the grains into fractions and the effect of sieving (15 min on sieves with a hole of: 1; 2.5; 3.1; 5; 6.3 and 15 mm; *laboratory equipment AS200 RETSCH GmBH*); ii) the disintegration of the aggregates due to the mechanical load (each sample fraction (100 g) in the thin layer was loaded by a tile (900 g) which rotated 3 times) and iii) the effect of the rinse of the sample fractions by water and drying. The phase purity of the fractions was measured by an X-ray diffraction analyser (*Rigaku MiniFlex 600)*.

The pilot/plant experiment was carried out in the rotary kiln in the factory Hačava, of the company INTOCAST, a.s. Slovakia, in cooperation with the company Messer Tatragas. The post-mortem MC bricks from the linings of an electric arc furnace, oxygen convertors and foundry ladles (from the company SSM Stražske, Slovakia) were crushed. A fraction of 1 - 12 mm size was used for the test only.

The basic parameters of the oxidising annealing in the rotary kiln are listed in Table 2. The kiln is heated with the burner operating on natural gas. The oxidising atmosphere was assumed by an oxygen input nozzle oriented on the material surface [15].

The fine fractions of size under 1 mm were separated from the burned-out material. The obtained MgO-concentrate with a grain size from 1 to 3.15 mm was evaluated and the results were compared with the pure fused magnesia and MgO-clinker which are used as the main raw material for the production of the new MC bricks. The results include qualitative and quantitative chemical and phase analyses, the loss on ignition, the moisture content (STN EN 1097-5, STN EN 15169), the granulomere analysis (STN EN 933-1), the determination of density (STN EN 1097–6), the strength of the grain in the fraction of 1 - 2 mm according to the Protodyakonov method [16, 17] and the macro – and microstructure (*light microscope/NEOPHOT 32, D).*

The measuring procedure of the grain strength in the fractions of 1-2 mm by a modified Protodyakonov method was performed as follows: a 100 g dose sample of 1 - 2 mm grain size was placed in a cylinder (Ø = 50 mm) using a dropping hammer (m = 6.35 kg) of a cam–ram piece of equipment. The hammer fell freely on the MgO sample 5-times from 115 mm in height. Consequently, a sieving analysis was undertaken. The difference between the weight of the 1 - 2 mm fraction before and after the test points to the strength difference of the individual grains.

Table 1. Chemical composition of the crushed post–mortem MC 98/10 bricks; loss on ignition /LOI.

Sample from worn MC 98/10	Chemical analysis (wt. %)										
	Al_2O_3	SiO_2	MgO	CaO	Fe_2O_3	Na_2O	K_2O	SO_3	MnO	Cr_2O_3	LOI
Average	4.4	1.5	79.7	4.5	1.6	< 0.05	< 0.05	0.00	< 0.25	0.13	9.2
Thin surface layer	5.6	1.4	62.6	19.7	1.6	0.15	< 0.05	0	< 0.25	0.9	8.2
Brick core	4.4	1.8	80.8	4.2	1.5	< 0.05	< 0.05	0	< 0.25	0.1	9.2

Table 2. Basic parameters of the annealing process in a rotary kiln of the MC materials.

Rotary kiln		MC-material	
Length × inside diameter	42 m × Ø 2.1 m	Size of the crushed MC material	1 - 12 mm
Revolutions	8 - 9 min^{-1}	Ratio MgO : C	90 % : 10 %
Initial temperature/head of kiln	429°C	Batch	1.46 $t \cdot h^{-1}$
Maximum temperature of material	1250°C	Product	0.96 $t \cdot h^{-1}$
Consumption of natural gas	314 $N \cdot m^3 \cdot h^{-1}$		
Consumption of oxygen in additional nozzle	109 $N \cdot m^3 \cdot h^{-1}$	Run time of experiment	123 H

RESULTS

Laboratory tests: The release of the MgO-particles from the agglomerates of the crushed MC brick

The granularity of the crushed MC material for the 1st set of tests is shown in Table 3 which includes the weight loss during annealing (900°C/30 min) of all the individually annealed fractions from 0-1 mm to 6.3 - 15 mm. Checking the decarbonisation level by loss on ignition confirmed that the residual carbon in the annealed fractions from 0 - 1 mm to 5 - 6.3 mm is less than 0.4 wt. %.

The results from the annealing indicate: The fine-grained fractions were enriched by carbon during crushing. The fractions of 0 - 1 mm to 5 - 6.3 mm are fully burnt-out. The lower weight loss by annealing of the 1 - 2.5 mm fraction by 7 wt. % is probably due to the higher amount of the primary MgO particles in the fraction. On the other hand, the low weight loss of the 6.3 - 15 mm fraction is caused by the incomplete decarbonisation of the large agglomerates in their core. This is confirmed by the agglomerates' high strength and black colour in their core.

Table 3. Fractions in the initial MC material after crushing on a grain size of less than 15 mm and weight loss by annealing (1st set of tests). Conditions of annealing: 900°C/30 min.

Fractions	Proportion of fraction (wt. %)	Magnetic ratio (wt. %)	Weight loss (wt. %)
6.3 - 15 mm	33.5	0.4	-4.9*
5 - 6.3 mm	12.1	0.6	-7.8
3.1 - 5 mm	17.5	0.4	-7.1
2.5 - 3.1 mm	8.2	0.7	-7
1 - 2.5 mm	14.3	0.85	-5.2
0 - 1 mm	14.4	0.6	-13.5

** incomplete burning out of the agglomerates in the fraction*

The effect of the annealing and sieving, followed by a short modest mechanical load and a final rinse by water and drying on the disintegration of the agglomerates of the initial fractions 3.1 - 5 mm and 5 - 6.3 mm are presented in Table 4.

The disintegration process of the annealed agglomerates pointed to: Decarbonisation, the destruction of the carbon bonds is insufficient for the complete disintegration of the brittle agglomerate during the consequential sieving. The brittle agglomerates require a mechanical load using an adequate force which ensures the release of the larger MgO-particles without breakage of the primary MgO-grains. Slight pressing between two moving plates contributes to their disintegration. As shown in Table 4, rinsing by water and drying also significantly assists the disintegration of the agglomerates, the dust particles are simultaneously removed from the surface MgO-grains. The fractions of the initial size of 5 - 6.3 mm and 3.1 - 5 mm crumbled up to 90 % and 70 %, respectively. Individual MgO grains remained on the sieve of the 5 mm and 3.1 mm mesh (Figure 1).

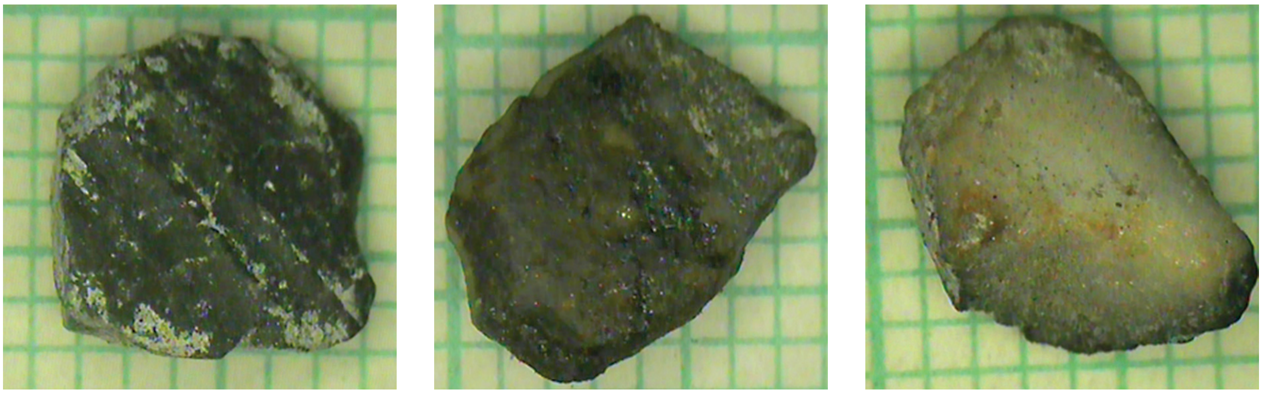
In these cases, the X-ray diffraction analysis confirms the presence of periclase and trace amounts of fero-periclase and spinele. In the fine fraction (-1 mm), the diffraction lines of graphite and brucite were also identified. The MgO dust particles reacted with water.

Table 4. The disintegration of the agglomerates (initial fractions: 3.1-5 mm; 5-6.3 mm) due to annealing and sieving (15 min), followed by a short modest mechanical load and also a rinse and drying.

Fraction	Contents (wt. %)					
	Initial fraction: 3.1 - 5 mm (100 %)			Initial fraction: 5 - 6.3 mm (100 %)		
	after annealing	+ after mech. load	+ after rinse and drying	after annealing	+ after mech. load	+ after rinsing and drying
5 - 6.3 mm	–	–	–	46	21	10
3.1 - 5 mm	61	48	30	33	19	15
2.5 - 3.1 mm	16	17	12	7	5	10
1 - 2.5 mm	12	17	25	7	20	24
0 - 1 mm	11	18	33	7	35	41

Table 5. The disintegration of the agglomerates of 0-6.3 mm; granularity: a) after crushing b) annealing and sieving, c) after tumbling in a mill, rinsing on sieves by water and drying.

Fraction	Content of fraction (wt. %)		
	Initial crushed material (a)	After annealing and sieving (b)	After annealing, then 2 h tumbling in a mill, rinsing and drying (c)
5 - 6.3 mm	18	3	2
3.1 - 5 mm	26	11	9
2.5 - 3.1 mm	12	10	7
1 - 2.5 mm	21	28	24 - 26
0 - 1 mm	23	48	56 - 58



a) b) c)

Figure 1. Residual grains in the 5 - 6.3 mm fraction after rinsing.

Wet sieving allows one to separate the 0.5 - 1 mm fraction from the powdery fraction, prevents dusting, but requires drying and forms a slurry with a higher concentration of $Mg(OH)_2$.

The goal of the 2nd set of tests was to determine which amount of the size fractions of the clinker can be obtained. The crushed MC material (0-6.3 mm) was annealed under the same condition as in the 1st set of tests (900 °C/30 min). The results of sieving (15 min) before and after the annealing and after 2 hours of tumbling, rinsing and drying are presented in Table 5.

The portion of the fine fraction increased markedly after the annealing and tumbling in slow-running mill. After the rinse and drying of the grains larger fractions than 1 mm, which represent the individual MgO particles, formed only about 40 wt. %.

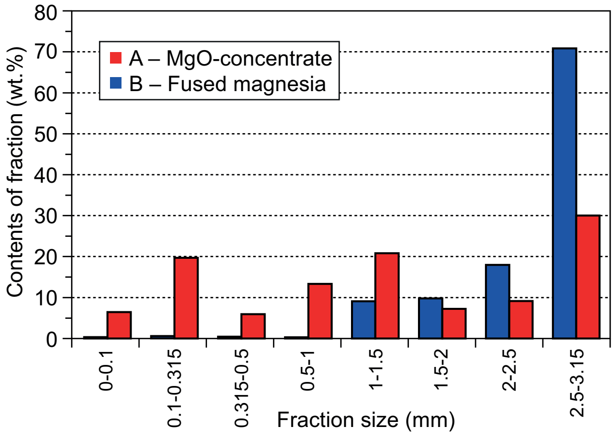


Figure 2. Comparison of particle size distribution of the MgO-concentrate (A) and the fused magnesia (B).

MgO concentrate obtained from the pilot plant experiment

The MgO concentrate of a fraction size less than 3.15 mm was obtained by processing the crushed post-mortem MC bricks (under 12 mm) in a rotary kiln in the Hačava factory (the company INTOCAST, a.s Slovakia). The granularities of the fused magnesia and the MgO-concentrate are presented in Figure 2.

The quality of the obtained MgO-grains of 1 - 2.5 mm fraction (sample A) was compared with the fused magnesia (sample B) and with the MgO-clinker (sample C), which are applied in the production of the new MC bricks.

The chemical and phase composition of the recycled MgO is well in accordance with the fused magnesia

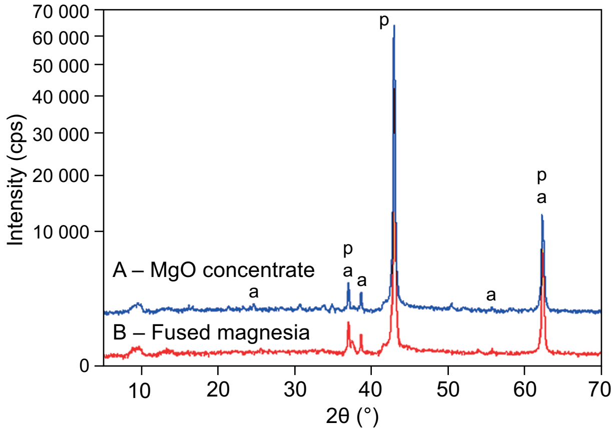
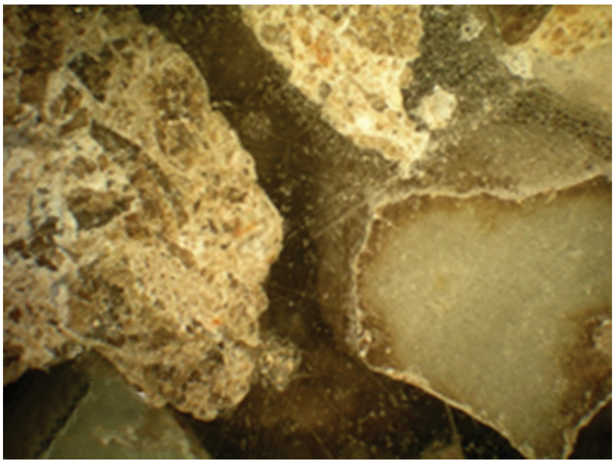


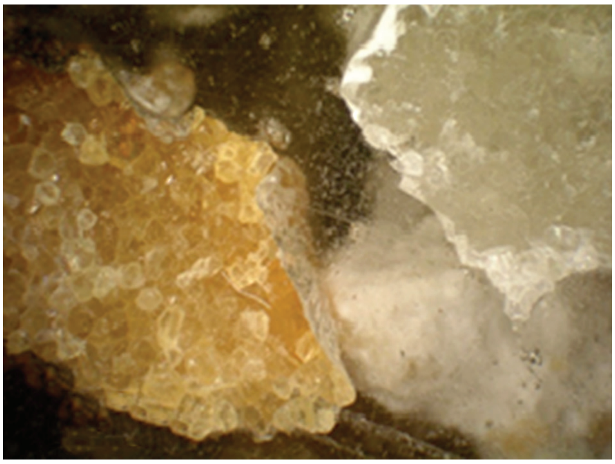
Figure 3. The diffraction pattern of the recycled MgO-concentrate (A) and the fused magnesia (B); fraction: 1.5 - 2 mm; p – periclase, a – non-stoichiometric TiO_x.

Table 6. Chemical compositions, loss on ignition (LOI) and moisture (M) of the recycled MgO (A) and fused magnesia (B).

Sample	Contents (wt. %)									
	MgO	Al_2O_3	SiO_2	CaO	Fe_2O_3	MnO	TiO_2	Na_2O	K_2O	LOI	M
A	93.07	0.26	2.54	2.79	1.04	0.24	0.03	0.01	0.01	1.72	0.85
B	93.75	0.46	1.13	2.43	1.80	0.26	0.03	0.01	0.01	1.02	0.55



a) recycled MgO



b) fused magnesia

Figure 4. Grains of the recycled MgO (A) and the fused magnesia (B); in a polarised light; magnified 25×.

Table 7. Bulk density of the recycled MgO, the fused MgO and the MgO-clinker and a comparison of the particle strength of the fraction 1 - 2 mm.

Sample	Bulk density of the particle in the fraction: 1.5 - 2 mm ($g \cdot cm^{-3}$)	Rest on sieve of 1 mm mesh after strength particle test (wt. %)
A – MgO-concentrate	3.53	38.6
B – fused magnesia	3.54	92.2
C – MgO-clinker	3.09	50.1

(Table 6, Figure 3). Both of them consist of periclase and the x-ray diffraction analysis confirmed a trace of a non-stoichiometric TiO_2.

The bulk density of the 1.5 - 2 mm fraction of the MgO/concentrate and the fused magnesia and the grain strength which was determined by the modified Protodyakonov method [16] are compared in Table 7. The grain strength of the recycled MgO is slightly lower than that of the new MgO-clinker grains, but very low in comparison to the fused magnesia.

The structure of the recycled MgO grains (A) and the new fused magnesia (B) observed by an optical microscope is shown in the Figures 4a, and 4b. The surface of both samples is covered by powdery particles, but the surface layer of the recycled MgO particles is chemically attacked and more sintered.

DISCUSSION

Laboratory annealing (900°C) tests of dense MC crushed material confirmed that the carbon bond elimination during oxidising annealing leads to brittle agglomerates and their partial disintegration. The annealed material contains less than 0.4 wt. % of residual carbon. As the carbon burns out, the carbon bond vanishes and the particles consistency significantly reduces. However, a total release of the individual MgO particles from the aggregates does not occur. As Ikonnikov mentions in his work [18], excluding the carbon bond does not guarantee the disintegration of the agglomerates as the present impurities can act as a binder of grains of the periclase, too. The multicomponent admixtures contribute to the strength of the decarbonised agglomerates.

Next to the temperature and oxidising atmosphere, the dimensions of the agglomerates are highly important for their complete decarbonisation, as well as their tumbling which supports their decomposition. The decarbonised layer forms a barrier opposing the transportation of CO_2/CO to and from the reacting zone, which moves to the core of the dense particle. Therefore, the initial maximal dimension of the agglomerates and the conditions of the decarbonising process must be selected so that fast heating and the mechanical load supports the agglomerate disruption, but does not disintegrate the integrity of the primary periclase particles.

The coarse fraction of the purified MgO can be obtained by an intensive sieving process. The fraction of the fine particles (0-1 mm) creates approximately 50 wt. %, which was confirmed by the laboratory and pilot-plant test, too (Table 5 and Figure 2).

The pilot-plant test carried out in the Hačava factory, at the company INTOCAST, a.s. Slovakia confirmed that the selection of the annealing conditions of the crushed post mortem MC bricks in a rotary kiln (Table 2, oxidising atmosphere) were adequate for the decarbonisation of the MC material of 1 - 12 mm granularity. Tumbling of the batch stimulates the disintegration of decarbonised agglomerates and continually provides a new surface for the oxidation. After decarbonising, intensive sieving is needed to obtain a coarse fraction of a similar chemical and phase quality and a bulk density for the new MgO raw materials. The strength of the MgO-grain of 1 - 2 mm size was not adequate to that of the fused magnesia and clinker (Table 7). The processes of crushing (the method of disintegration, the dimension of the initial fraction), fast heating/cooling at the input/output of material to the kiln can significantly influence the grain strength [19]. The differences in the weight ratio of the coarse fractions between the recycled MgO and the granularity of the new mass can point to the degree of the MgO-particles disturbance during the recycling processes. Because the granularity and strength are important parameters for utilising the MgO-clinker, they must be checked continually.

CONCLUSION

Laboratory and pilot-plant tests showed that the total decarbonising of the agglomerates requires an agglomerate of accurate size to assume the annealing condition, i.e., an oxidising atmosphere, a decarbonising temperature (1100 - 1200°C) and the intensive tumbling of the materials during the process of the carbon burning out in the kiln. The post–mortem MC lining material should be crushed into the size 2 times bigger than the maximum primary MgO-grain size in the materials. The problem is to achieve a complete disintegration of the decarbonised agglomerates by the intensive sieving of the fractions. The material can be slightly mechanically loaded before and during the sieving, applying the force which does not break the primary periclase particles, but only contributes to the destruction of the agglomerates.

The chemical and phase purity of the obtained coarse fraction are comparable with the new MgO raw materials used for the production of the refractory materials. However, the strength of the primary MgO grains is questionable. It is hard to tell whether the decrease in strength is caused by the quality of the MgO-particles, which deteriorated during the usage of the bricks in the high temperature aggregates or due to the damage during the crushing process or the temperature shock in the course of the decarbonisation.

Despite the mentioned problems, the obtained coarse fraction of MgO from the post–mortem MC bricks in the recycling pilot-plant process provides material which due to its quality is acceptable for new refractories production. The basic requirement for the application is that only the individual MgO particles can be present in the fractions. The partial replacement of this material for the clinker in the less resistant refractory material is possible.

The fine fractions can be incorporated into synthetic slags and powder in steel making.

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