Experiences with microporous calcium hexaluminate insulating materials in steel reheating furnaces at Hoesch Hohenlimburg and Thyssen Krupp Stahl AG Bochum

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Abstract

The steel reheating furnaces of the hot rolling mills at Hoesch Hohenlimburg GmbH and Thyssen Krupp Stahl AG Bochum have special requirements for the furnace roof lining. The variety of steel grades produced in these mills require numerous changes of the kiln temperature during use. Therefore, the furnace roof lining is made with lightweight refractories to accelerate the adjustment of kiln temperature between different steel grades to be reheated before rolling.

The fired insulating bricks used for the kiln roof show severe thermal shock damage, which occurs during the cleaning periods when the furnace is cooled down to ambient temperature. This requires either a gunning repair or the replacement of parts of the furnace roof. Therefore, new high temperature lightweight refractories based on microporous calcium hexaluminate have been developed and tested as lightweight gunning material and pre-cast shapes to replace the insulating bricks. Additionally, the new materials have been tested to replace refractory fibre linings for the stators of the walking beam furnace.

The properties of the new material and the practical experiences gained over a trial period of up to 3 years are discussed.

Introduction

The hot rolling mill at *Hoesch Hohenlimburg* (HHO), Hagen is running a walking beam furnace for reheating the steel slabs prior to rolling. It has an output of about 800,000 mt per year. HHO is producing special alloyed carbon steel, e.g. for automotive applications like axle carriers and other construction pieces. The slab pre-heating temperature ranges from 1350 °C down to 1100 °C for manganese containing steel grades. The reheating furnace has the following dimensions: length 29.9 m, width 12.2 m, and height 4.1 m. It has burners in the roof and in the side wall. The furnace is cooled down to ambient temperature twice a year for cleaning and maintenance purposes.

The *Thyssen Krupp Stahl (TKS) AG*, Bochum hot rolling mill is operating two walking beam furnaces and one pusher type furnace, each of them with a production capacity of about 250 mt per hour. TKS Bochum is producing about 230,000 mt/month low carbon quality steels, e.g. for automotive sheets, and about 110,000 mt/month stainless steels. The slab pre-heating temperature ranges from 1350 °C down to 1080 °C, e.g. for special alloyed steel. The kiln dimensions are about 35 m length, 10 m width, and 4 m height. The kilns have burners in the roof and in the side wall. For the weekly maintenance shift, the kilns are cooled down to about 1000 °C. After a period of about 9 months, each kiln is cooled down to ambient temperatures for cleaning and maintenance purposes.

HHO and TKS Bochum have a special requirement on the kiln refractory linings. Both hot rolling mills are producing various steel grades with different requirements of slab pre-heating temperature for hot rolling. Therefore, the kiln temperature needs to be changed frequently and the kilns need to react quickly to those changes. The HHO furnace and the two walking beam furnaces at TKS Bochum have lightweight refractory kiln roof linings, which provide a lower heat capacity compared to dense refractories (e.g. 2.5–2.8 g/cm^3), that are commonly used. Due to the lower heat capacity, the lightweight linings provide a faster adjustment of kiln temperature setting and also reduce energy losses, because of a lower heat transfer through the lining. These lightweight linings are the focus of this paper.

Lightweight furnace roof lining

In general, the following requirements apply for reheating kiln roof linings:

- The permanent linear change during use must be below +1.5% to avoid high stresses which can damage the kiln roof. This is of special interest for all monolithic or pre-cast shaped (dried at 400 °C) materials, as they are not fired prior to the installation. The sintering takes place during use.
- The refractories must withstand the service temperatures, and also local temperature peaks. In the burner area for example, the temperature can be up to 1500 °C. Therefore, the refractories should provide a safety range (e.g. 100 °C) to cover potential overrides of the usual application temperature in the kiln.
- The thermal shock resistance of the lining needs to be sufficient to withstand the cooling and heating cycles during the maintenance periods mentioned above.

In the 1990's, HHO and TKS Bochum developed lightweight refractory linings for their reheating kilns which are based on mullite-corundum insulating bricks of ASTM class 30. The complete roof lining is as follows (material data in **Table 1**):

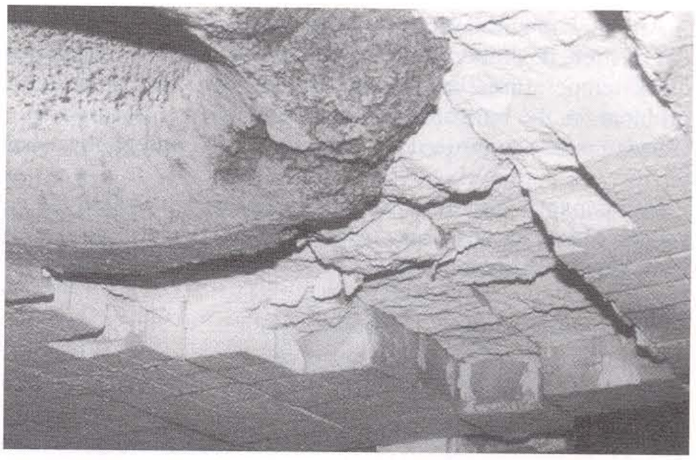

Fig. 1. Typical thermal shock damage of the lightweight brick refractory roof lining of a reheating furnace

50 mm	lightweight castable (on top)
13 mm	refractory ceramic fibre blanket (1260 °C fibres, 128 kg/m^3)
230 mm	insulating brick ASTM 30

The ASTM class 30 bricks provide the required low bulk density of 1.08 g/cm^3 and a high thermal stability of 1650 °C. The disadvantage is the low thermal shock resistance, which leads to spalling of the bricks during use **(Figure 1)**. This spalling occurs usually after 12–18 months and requires either a gunning repair of the damaged areas or a complete relining. The maximum lining life achievable with these bricks is about 24 months. The gunning repair is performed by high alumina gunning mixes (Table 1). These materials have a bulk density of 2 g/cm^3 or higher, and thereby increase the heat capacity of the roof lining, which is, of course, not desired. The lining life of such a gunning repair is a maximum of about 18 month.

Tab. 1. Typical data of refractories for lightweight reheating kiln roof lining

Material	Insulating high alumina brick ASTM class 30	Insulating plate	Conventional lightweight castable 1	Conventional lightweight castable 2	High alumina gun mix 1	High alumina gun mix 2
Mineralogical composition	Mullite/ Corundum	Calcium Silicate	Lightweight Chamotte	Vermiculite/ Silica	Andalusite	
Classification temperature resp. temperature limit of application [°C]	1650	1100	1100	1000	1600	1450
Chemical analysis [wt.-%]						
SiO_2	26	27-33	46	26	37	41
Al_2O_3	73	< 1	26	32	55	50
Fe_2O_3	< 0.5	< 0.6	8.4	10	0.6	1.3
CaO	< 0.1	27 - 33		23		5.0
MgO		2.5 – 6.5		8		
Bulk density [g/cm^3]	1.08	0.2 – 0.3				
Monolithics: at 110 °C			1.04	0.54		
at 800 °C				0.50		
at 1000 °C			1.0		2.3	2.08
at 1400 °C						
CCS [MPa]	2-3					
110 °C			3	1–2	52	15
800 °C			2–3	1	33	
1000 °C			1			
1200 °C					31	13
Thermal Conductivity [W/mK]						
at 400 °C		0.06	0.33	0.14	1.98	0.67/0.90
at 800 °C		0.14	0.35		1.80	
at 1000 °C	0.5					
at 1200 °C					1.97	

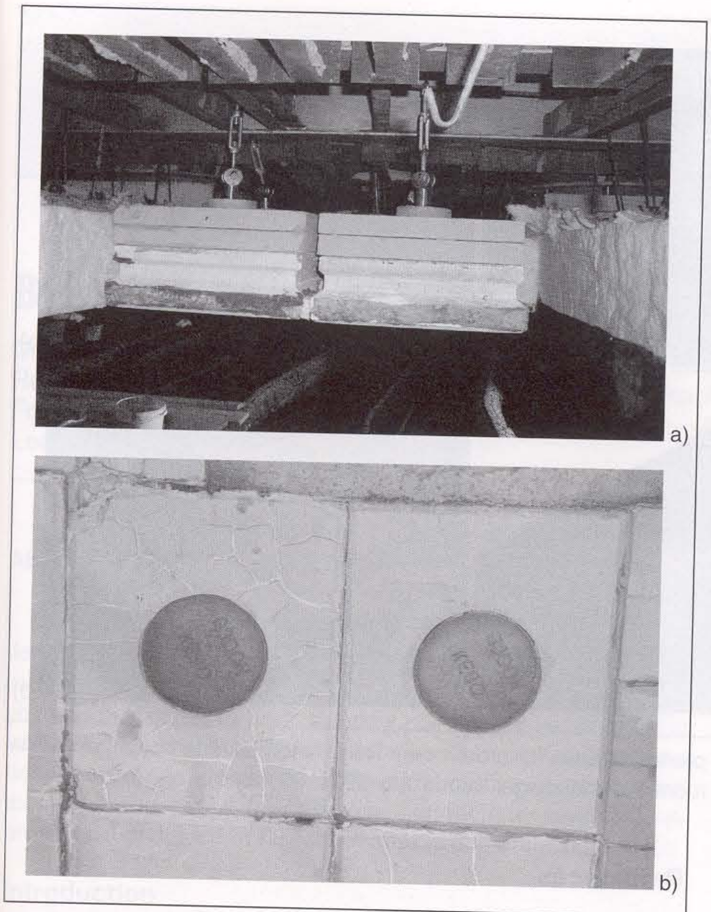

Fig. 6. Microporous pre-cast shapes for kiln roof, 400 × 350 × 160 mm with high alumina anchor brick and two 50 mm calcium silicate plates on top. a) during installation b) after 12 months: slight but different surface cracking (s.text), but no damage. Status July 2004: 19 months, ongoing

has been changed from vibrating to rodding, and the fines separation is avoided.

Conventionally, the stators and cross beams of the walking beam furnace construction are lined with refractory ceramic fibres. The lining life of these fibres is up to 6 years, but less in areas close to burners (down to 1 year). Although the lining life in general is not an issue, fibres cause some health concerns, especially during break out of the used linings. Therefore the new microporous material has also been tested in these areas.

The stators have a diameter of 168 mm and have been lined with pre-cast shapes in a tongue and groove system without further fixing elements **(Figure 7)**. At the static stators after 24 months of use, some joints have formed due to broken tongues of the shapes. This damage has been repaired by the 43% Al_2O_3 mortar, and the installations are still in use (July 2004, 30 months). However, for the moving stators, the pieces fall off after a few weeks, probably due to the vibrations of the system. Here, an additional fixing system would be required for higher lining life.

Figure 8 shows microporous pre-cast shapes for the cross beam lining. No damage was observed after 17 months use, and the installations are ongoing (July 2004, 30 months). Some pieces had fallen off after 18 months, but for other reasons not related to failure of the refractory material. Summarizing the experiences, the trials have shown, that the microporous material can replace the fibre linings, and will provide a higher lining life especially in burner areas, due to a higher erosion resistance. However, for the moving stators of the walking beam furnace, a better mechanical fixing to the stator would be necessary to improve the lining life.

Temperature measurements have been made at the top of the kiln roof lining by thermocouples placed in two different kiln zones. They compare the insulating brick lining with the microporous pre-cast shape lining of the same dimensions. With the microporous material, the temperature on top of the roof lining is between 7.6 and 9% lower:

– 120 vs. 132 °C at the hot kiln zone (1300 °C inside kiln)
– 157 vs. 170 °C at the other kiln zone (1274 °C inside kiln).

For another kiln the larger pre-cast shapes have been compared with other, medium weight pre-cast shapes. Here, the overall insulation is higher with a temperature of about 80 °C at the top of the lining. The temperature difference is about 20 to 30 °C (or 25%) to the advantage of the microporous lining.

Calculations of the heat transfer have been made based on the thermal conductivity data given in Tables 1 and 2 acc. to the following equation:

Heat flux $\dot{q} = \lambda/d\,(T_1-T_2)$ W/m^2

λ = thermal conductivity, d=lining thickness, $T_{1,2}$=temperature range

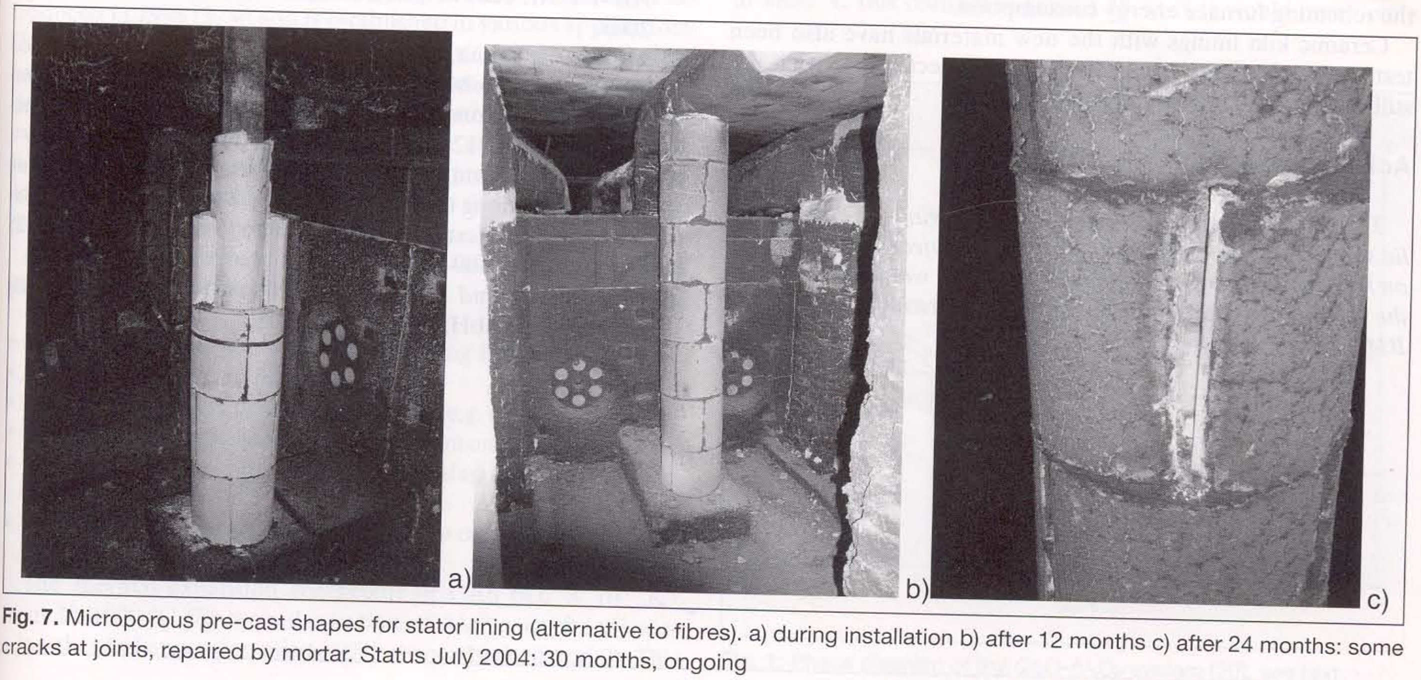

Fig. 7. Microporous pre-cast shapes for stator lining (alternative to fibres). a) during installation b) after 12 months c) after 24 months: some cracks at joints, repaired by mortar. Status July 2004: 30 months, ongoing

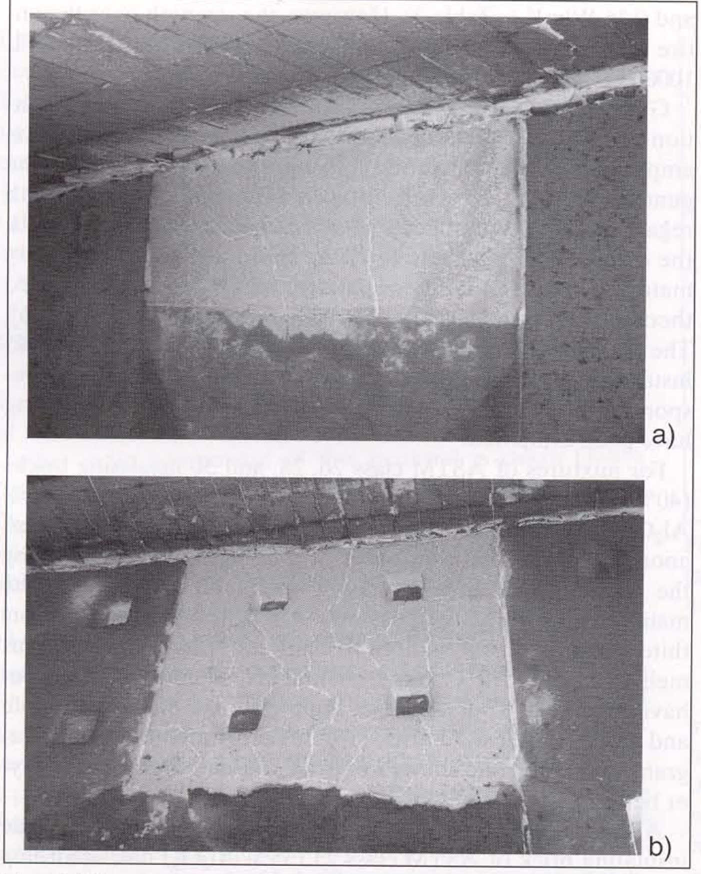

Fig. 4. Microporous gunning lining of kiln sidewall, 220 mm thick, a) with metallic anchors, after 18 months b) with ceramic anchors, after 18 months. Status July 2004: 23 months, ongoing

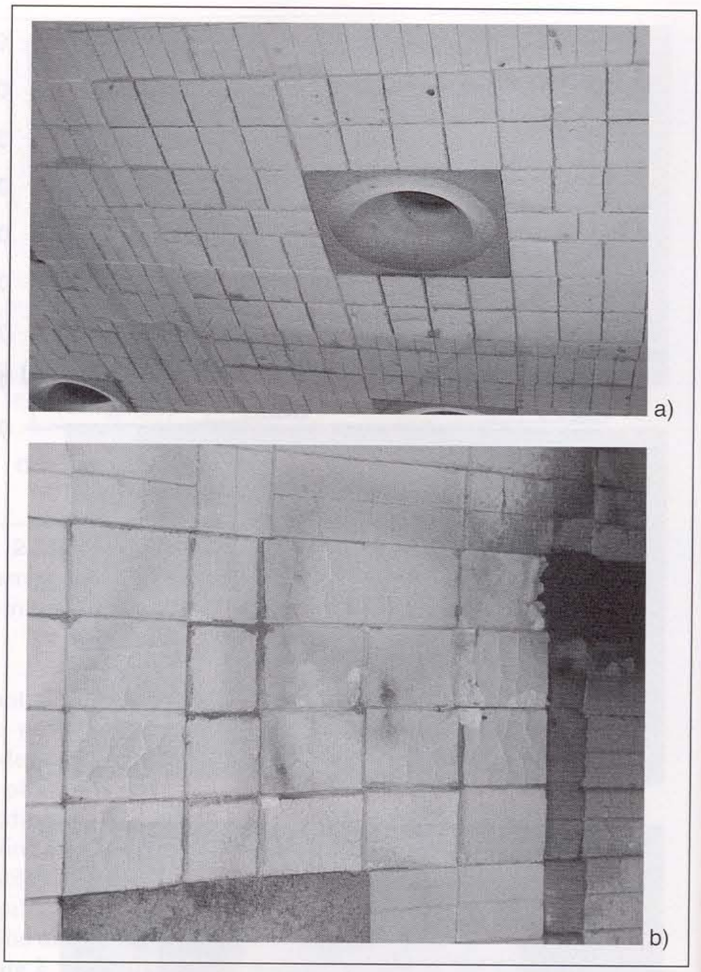

Fig. 5. Microporous pre-cast shapes for kiln roof lining, 230 × 154 × 230 mm a) after 6 months, no visible damage, samples taken for lab tests (s. text) b) another area after 18 months, Status July 2004: 23 months, ongoing

orous test field is due for relining in October 2004 (after then 25 months use).

Based on the positive experiences with the first microporous gunning applications, trials with pre-cast shapes to replace the insulating fire brick have been started. The shapes of dimension 230 × 154 × 230 mm (size of two combined lightweight bricks) have been cast under vibration with an integrated metallic anchor sheet to fix the shape to the roof construction. They were tempered at 400 °C before installation. A chemically bonded, 43% Al_2O_3 mortar was used as joint fill between the pre-cast shapes. During the installation, the lower edge strength of the shapes compared to the lightweight bricks required some care during handling. Therefore, the edge strength was improved for following trials by slight modifications of the material. On top of the shapes, a refractory ceramic fibre blanket (13 mm) and 50 mm layer lightweight castable (0.5 g/cm^3) have been installed, as is used for the lightweight brick construction.

Figure 5a shows a trial area with microporous pre-cast shapes of about 3 m^2, surrounding a burner block in the kiln roof, after six months of use. The slight cracks formed on the surface are not considered to be critical, and the slight edge damage from the installation has not further deteriorated. In this area some shapes were purposely broken out after six months, to investigate the material in the lab. Some of the shapes showed a cracking at a distance of above 70 mm from the hot face. The cold crushing strength of the used shapes (INTOVAL VL 1000 HT/1) is between 3–4 MPa for both the cold and the hot end. So the strength has not decreased during use.

Another test area with the same kind of pre-cast shapes is shown in **Figure 5b**. The microporous shapes do not show spalling after 18 months of use, and this installation is ongoing (July 2004, 23 months). Summarizing the experience with this kind of pre-cast shapes, the lining life is definitely higher compared to the lightweight bricks ASTM class 30, which show extensive spalling after 18 months use. At least a part of the microporous shapes also shows some cracking at a depth of 70 mm. However, in the first 24 to 30 months a spalling damage did not occur. As the field trials are ongoing, a final comment of the lining life cannot be given here.

Parallel to the pre-cast shapes of 230 × 154 × 230 mm size, larger pre-cast shapes were also tested. They have a different system for fixing at the steel construction of the kiln. These are shown in **Figure 6a**. Their size is 400 × 350 × 160 mm and they are fixed to the kiln roof by a fired high alumina conical anchor brick. The joint between the pre-cast shapes is filled with a castable. Above the pre-cast shapes, two 50 mm thick calcium silicate insulating plates have been placed. The whole construction is covered by a lightweight castable of 50 mm thickness to avoid hot air convection out of the kiln.

Figure 6b shows two of the shapes after 12 months use. No damage has been observed up to 19 months (HHO) and 22 months (TKS Bochum) and the installations are still ongoing (status July 2004). As can be seen in Figure 6b, some shapes show a more intense cracking of the surface compared to others. Overall, these surface cracks are more intense for the larger compared to the smaller shapes. They result from fines separation at the bottom of the pre-cast shape due to the vibration during casting. The fines layer contains a higher content of binder, and therefore shows a higher sintering shrinkage compared to the body material. Based on this observation the casting process

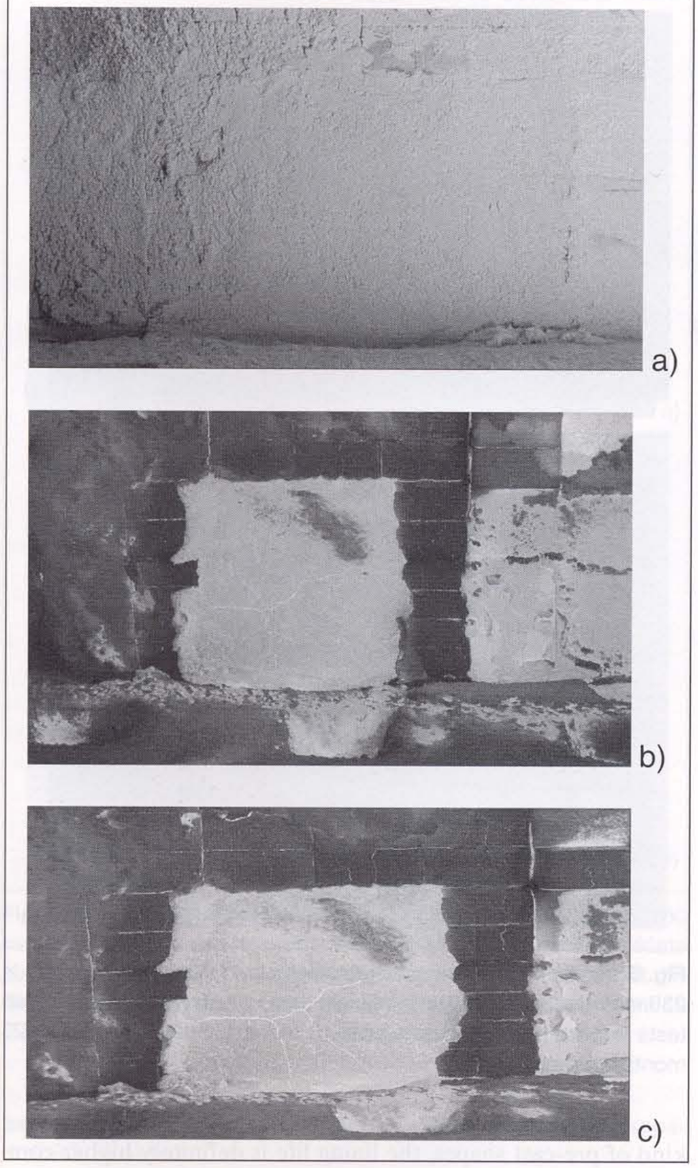

Fig. 3. Microporous gunning repair of kiln roof applied on thermal shock damaged lightweight bricks, about 70 mm thick. a) new installation b) after 12 months: surrounding lightweight bricks dark, microporous gun repair bright with just some slight cracks at the surface c) after 16 months: no change, status July 2004: 30 month, ongoing

during the installations and caused minor damage, which has not been considered critical for the applications discussed here. During the project slight modifications were made to improve the edge strength of the pre-cast shapes.

The thermal shock resistance by air quenching is > 30 cycles (950 °C → room temperature) and confirms the high thermal shock resistance of the microporous structure of SLA-92. The thermal conductivity of INTOVAL VL 1000 HT is between 0.37 and 0.46 W/mK, which is lower when compared to the ASTM class 30 brick. This is due to the microporous structure which restricts heat transportation by radiation.

As INTOVAL VL 1000 HT contains some SiO_2 from bonding additives, its temperature limit for application is 1350 °C. Above this temperature a partial melting occurs which can be expected from the phase diagram CaO-Al_2O_3-SiO_2 [6] and leads to shrinkage of the refractories. Therefore, the silica free versions INTOVAL VL 1000 HT/1 CAST and GUN have been developed to achieve a refractoriness of 1500 °C. These improved materials have an even lower thermal conductivity between 0.28 and 0.36 W/mK (s.Table 2). However, the strength over the entire temperature range is somewhat lower than INTOVAL VL 1000 HT, but the edge strength did not decrease.

Gunning of lightweight mixes allows an easy repair of insulation linings, especially when only parts of it are damaged, for example due to thermal shock spalling as described above. The gunning versions can also be troweled for smaller repairs. With regard to gunning applications on damaged insulating bricks, the contact reactions between CA_6 based insulating refractory materials and Al_2O_3-SiO_2 insulating bricks have been evaluated theoretically by the ternary phase diagram CaO-Al_2O_3-SiO_2 [6]. The composition of a 1:1-mixture of SLA-92 based castable and insulating brick has been drawn in the diagram and the corresponding phase triangle and temperature at the onset of melting have been evaluated.

For mixtures of ASTM class 26, 28, and 30 insulating bricks (40%, 60%, 73% Al_2O_3) with the SLA-92 refractory (89% Al_2O_3, 10% CaO) the stable phase composition is corundum-anorthite-mullite with an onset of melting at 1512 °C. During the reaction with such insulating bricks, hibonite (CA_6), the main phase of the SLA-92 castable, will be transformed to anorthite and mullite, but without formation of phase compositions melting below 1512 °C. Experimental investigation of creep behaviour at 1400 °C of a contact sample from a SLA-92 castable and an ASTM class 30 brick (JM 30) confirmed the phase diagram conclusion and showed only a 0.5–1 mm thin reaction layer between the different insulating materials.

Also, combination of the SLA-92 castable with a lower grade insulating brick of ASTM class 23 (35% Al_2O_3) has, according to the phase diagram, a stable phase composition of mullite – anorthite – cristobalite with an onset of melting at 1345 °C. Considering the higher amount of impurities (i.e. alkalis) contained in this type of insulating bricks, a much more severe contact reaction could be expected at temperatures above 1300 °C, but this is already above the classification temperature of 1260 °C for a class 23 brick.

Industrial application

The industrial applications trials with the newly developed materials started in April 2001 and are still ongoing. The gunning mix has been tested first and was applied on thermal spalling damaged insulating bricks ASTM class 30 with a thickness of about 70 mm. The first gunning field trial in the less demanding soaking zone of the furnace lasted for 27 months, before the whole area was completely relined to avoid an unplanned failure and unscheduled later kiln repair in the future.

Figure 3 shows another gunning repair in the kiln roof after installation and after 16 months of use. Apart from some slight cracks at the surface, which are not considered as critical, the microporous material shows no change after 16 months and is still in use (July 2004, 30 months). Afterwards, tests with gunning installations in the side wall have been performed **(Figure 4)**. The wall thickness is 220 mm (about 800 mt material per trial area) and applications with metallic and ceramics anchors were successful, and the installations are still ongoing (July 2004, 23 months).

Other gunning installations have been performed at the lower part of the access wall and on the upper sidewall at the exit of the pusher type furnace. Here, the thermal shock behaviour of the refractory lining is of special interest, because of temperature changes between 1300 °C and ambient temperature every time the kiln is opened for the slab charging. At the kiln entrance, the lining has been in use for 22 months (July 2004, ongoing). The whole area at the kiln exit including the microp-

Although the insulating brick lightweight roof lining provides the desired flexibility of the reheating furnaces in respect of quick temperature changes, the spalling behaviour is a major problem for the hot rolling mills and has been focus of several German public sponsored R&D projects [1–3] and publications [4, 5].

In this paper, an alternative to the lightweight bricks is discussed. This is based upon a newly developed microporous calcium hexaluminate material.

Microporous material SLA-92

The name SLA-92 of the newly developed insulating raw material stands for **S**uper **L**ightweight **A**ggregate of **92**% Al_2O_3. The other main component of this material is calcia (7–8% CaO), and the level of total impurities is very low. SiO_2 and Fe_2O_3 contents each are maximum 0.1%. The base mineral composition is calcium hexaluminate ($CaO \cdot 6Al_2O_3$) or CA_6 · CA_6 exhibits the best thermal properties among minerals in the calcium aluminate system, having a melting point above 1830 °C. The bulk density of SLA-92 is around 0.75 g/cm^3.

Figure 2 shows the microstructure and pore size distribution of SLA-92. Small CA_6 platelets are arranged like a house of cards with micropores between the crystals. This gives a homogeneous structure with a high internal porosity (typically 75%). The pore size distribution shows a narrow range of pore sizes between 1 and 5 µm with an average pore size of 3–4 µm. This structure accounts for the two key properties of SLA-92 described below, which make SLA-92 superior to conventional insulating raw materials.

The microporosity hampers heat transport by radiation, which is the main transport mechanism at high temperatures, resulting in the *low thermal conductivity* of SLA-92 especially at temperatures exceeding 1000 °C. In general, insulating materials are susceptible to spalling caused by thermal shock, because a temperature change creates a steep thermal gradient that causes high thermal stresses. But thermal spalling occurs only if a crack developed by thermal stress propagates through the material. The microporous house of cards structure of SLA-92 hampers crack propagation and contributes to the *high thermal shock resistance*. In addition to the low weight, is an essential property for the new reheating furnace lining.

Microporous refractory castables and gunning mixes

Intoval VL 1000 HT, a newly developed lightweight insulating material, is based on the microporous raw material SLA-92. The material is hydraulically bonded using high alumina cement. A castable and a gunning version have been developed. The gunning version has a lower top size of 3 mm compared to the castable's top size of 6 mm.

The gunning version is applied with a dry gunning machine with water addition made at the nozzle. The rebound is about 15%, which is considered acceptable for an insulating gunning mix using such a machine. The rebound can be reduced with alternative wet gunning technology, where a pre-mixed slurry is used. However, this technology has not been used for the trials described below.

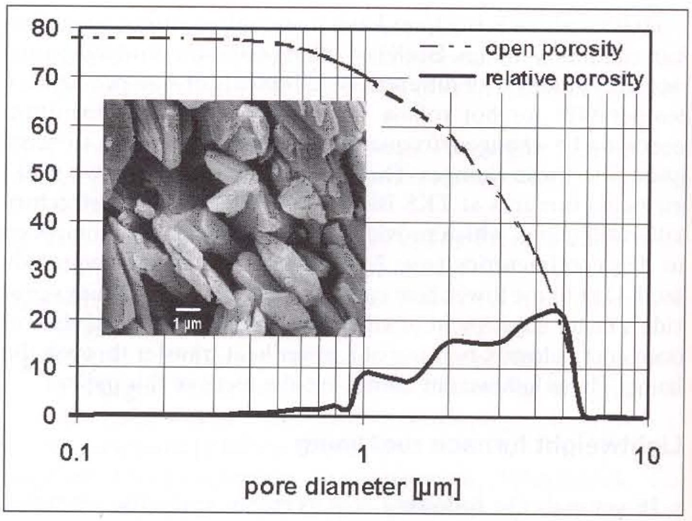

Fig. 2. SLA-92 microstructure of calcium hexaluminate platelets (scanning electron microscope) and micropore size distribution (Hg-intrusion method)

Initially, Intoval VL 1000 HT has been cast under vibration into pre-cast shapes using 55–60 wt.-% mixing water. After hardening, the shapes were tempered at about 400 °C to remove the physically and chemically bonded water from the hydraulic bonding. Later, and based on learnings described below, the casting process was done without vibration but by rodding to enable de-airing. This is the common technique for insulating castables.

The data of Intoval VL 1000 HT CAST and GUN are given in **Table 2**. The bulk density, which depends on the pre-firing temperature, is between 1.18 and 1.04 g/cm^3. The cold crushing strength is between 5 and 12 MPa, and over the entire pre-firing temperature range, it exceeds the strength (1–3 MPa) of typical insulating fired bricks of ASTM class 23–30. However, the edge strength of some pre-cast shapes after drying at 400 °C has been lower than the fired insulating bricks. This required some care

Tab. 2. Typcial data of microporous lightweight refractory castables and gunning mixes

	Intoval VL 1000 HT		Intoval VL 1000 HT/1	
	CAST	GUN	CAST	GUN
Temperature limit of application [°C]	1350	1350	1500	1500
Chemical analysis [wt.-%]				
Al_2O_3	86	84	89	89
CaO	10.5	10	10	10
SiO_2	3	4	< 0.2	< 0.2
Fe_2O_3	< 1	< 1.5	< 0.5	< 0.5
Bulk density [g/cm^3]				
110 °C	1.17	1.18	1.11	1.28
1000 °C	1.05	1.10	1.10	1.10
1400 °C	1.04	1.18	1.12	1.20
CCS [MPa] 110 °C	7	5	5	6
1000 °C	8	8	3	2
1400 °C	10	12	5	5
PLC [% 110 °C	– 0.1	– 0.1	– 0.1	– 0.1
1000 °C	– 0.4	– 0.4	– 0.4	– 0.3
1400 °C	– 1.2	– 1.5	– 0.5	– 0.8
1600 °C	n.d.	n.d.	– 2.4	n.d.
Thermal Conductivity* [W/mK]				
at 400 °C	0.37	0.37	0.30	0.30
at 1000 °C	0.36	0.37	0.28	0.29
at 1200 °C	0.46	0.46	0.36	0.36

* measured by hot wire method (DIN EN 993-15)

For the microporous material INTOVAL VL 1000 HT/1, the heat flux is about 30% lower compared to the insulating brick ASTM class 30 (1056 W/mK vs. 1602 W/mK).

Both, the practical measurements and the calculation indicate a potential for energy savings with the newly developed microporous refractories.

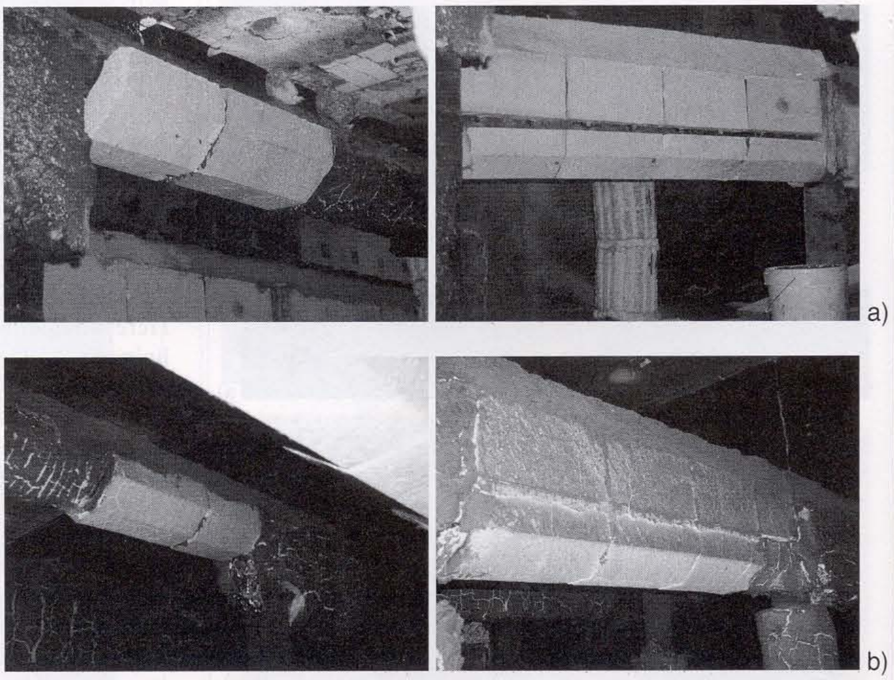

Fig. 8. Microporous precast shapes for cross beam lining (alternative to fibres). a) new installation b) after 17 months: no damage. Status July 2004: 30 months, ongoing

Conclusions and Outlook

The new microporous calcium hexaluminate material SLA-92 and the gunning and casting refractories INTOVAL VL 1000 HT developed with this material provide an interesting combination of properties such as low weight (1–1.2 g/cm^3), high refractoriness (up to 1500 °C), low thermal conductivity (between 0.28 and 0.46 W/mK), and high thermal shock resistance. The new material has been successfully tested over a 3 year period as gunning and pre-cast shaped installations, in steel reheating furnaces, where most of the trial installations are still ongoing.

The new microporous refractories offer a better alternative to higher density gunning mixes, fired insulating bricks, and refractory ceramic fibres. Gunning repairs with the new materials have a much lower bulk density (1.12 g/cm^3) and provide a much higher lining life compared to the standard materials (high alumina, about 2 g/cm^3). The pre-cast shapes outperform the insulating bricks ASTM class 30 with regard to thermal shock resistance and insulating behaviour. Trial installations with a lining life of 19–23 months are still ongoing. Refractory ceramic fibres have been successfully replaced by microporous pre-cast shapes for stator and cross beam linings. For moving stators an improved mechanical fixing would be required.

Temperature measurements and heat flux calculations indicate a potential for energy savings with the new microporous materials. Expanded trials over larger areas of the kiln roof lining would be necessary to prove this by practical figures from the reheating furnace energy consumption.

Ceramic kiln linings with the new materials have also been tested with another partner during this project. These trials are still ongoing and may be reported later.

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