Kiln Furniture for Oxide Ceramics: Technical Properties for Increasing Demands

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Abstract

SiC-based kiln furniture has a maximum service temperature of 1600 °C. Beyond that point, mullite-corundum materials are required. Here it is important that high hot bending strength resp. thermal fatigue in conjunction with adequate thermal shock resistance be achieved through selectively designed microstructures and appropriate raw materials.

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Introduction

The development of kiln furniture for the production of oxide ceramics is probably more widespread than can be concluded from the number of publications in this field. There are relatively few papers which report on the effects of raw materials, granulometry, and bonding systems on corundum kiln furniture. This may be due to the fact that, during the last few years, many oxide ceramics producers have developed their own kiln furniture programs in response to their special demands. Refractory companies which produce kiln furniture should try to cover the whole firing range of oxide ceramics including the most widely varying conditions with respect to setting character, firing temperature, heating rate, and composition of the products to be fired. During the last few years, the qualitative demands on oxide ceramic components have grown noticeably which also implies the necessity of improving the servicing potential of kiln furniture. Strong competition in this area necessitates cost savings in all areas of kiln furniture production and application. The producers of kiln furniture will have to look for products with improved application properties and the most cost effective prices possible. Another important aim is the improvement of the ratio of mass of the product to be fired to the mass of the kiln furniture. Maximizing load capability and thermal shock resistance aim at perfecting the kiln furniture and minimizing the energy-consumption in these ceramic firing processes. For this reason, the thermal and mechanical properties of kiln furniture materials have to be improved during the course of ongoing development projects.

Depending on the Al_2O_3-contents of oxide ceramic materials, firing temperatures range from 1600...1800 °C. To fire these materials kiln furniture made of corundum or mullite with an appropriate bonding phase are employed. Some of the most important parameters are the choice of raw materials, the granulometric set-up, and the composition of the bonding phase. Great care also has to be taken during batch preparation, pressing, and production firing.

Demands for Kiln Furniture Products

There are certain application-oriented demands which have to be fulfilled by kiln furniture products:

Flatness: Especially for the production of substrates, there are very low tolerances with respect to the warpage of kiln furniture batts. Typical tolerance data will have a maximum value of about 0,2% of the batt-length which means a warpage value of 0,4 up to a maximum of 0,5 mm for substrates fired to standard postcard size.

Product strength: Usually batts are used for storage and transport outside the furnace. To allow for uniform handling, room temperature bending strength of about 10 N/mm^2 is necessary. Brittle edges and corners that release refractory grains from the batts mean stains and defects on the articles to be fired.

Hot strength: The service life of the kiln furniture is mainly limited by its hot strength, creep resistance, and static fatigue behaviour. Repeated mesurements which regulate the straightness of the batts and turn them after each firing have proven successful in prolonging the service life.

Contact reactions between kiln furniture and oxide ceramics: For the sake of high product standards, there must be no contact reactions between the kiln furniture and the supported oxide ceramic goods to be fired.

Thermal shock resistance: Due to the relatively low thermal conductivity, the relatively high thermal expansion, the thickness of the batts and the relatively dense setting of batts in the kiln car superstructure, major problems may arise if temperature gradients stress the kiln furniture and produce thermal shock crackings. There are ways of altering thermal shock resistance, for instance, by changing the granulometric composition, although this method has certain drawbacks in that it affects the surface smoothness of the batts' setting area.

Kiln furniture-geometry and configuration: The geometric dimensions of the various types of kiln furniture must be well-adapted to the products to be fired. The absolute dimensions of the batts, e.g. should be kept at a minimum while keeping in mind the typical material and processing parameters mentioned earlier.

This paper compares four different high-alumina refractories having different bonding systems and granulometry.

In addition to the standard testing criteria, particular attention was paid to the use of a nondestructive method for the measurement of thermal shock resistance. The resonance frequency of the various samples was determined. Furthermore, the thermal fatigue behaviour of the materials was included in the testing.

The effectiveness of parameter variation is also demonstrated by some photographs of the ceramic microstructure under the light- and scanning electron microscope (Figs. 1 – 4).

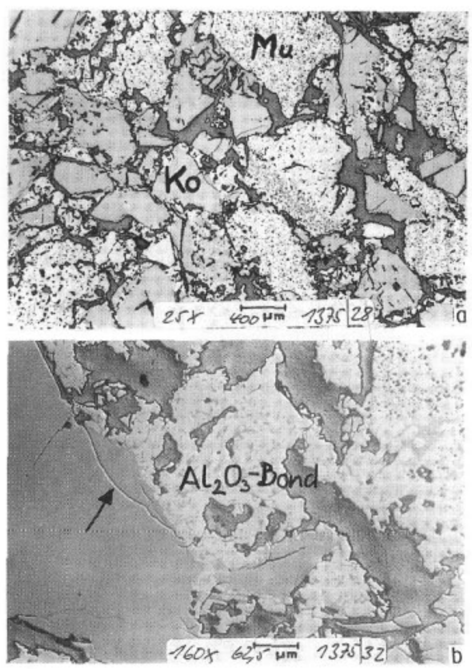

Fig. 1 a/b Structure of material 1; coarse grains of mullite with a fine grained Al_2O_3-bond

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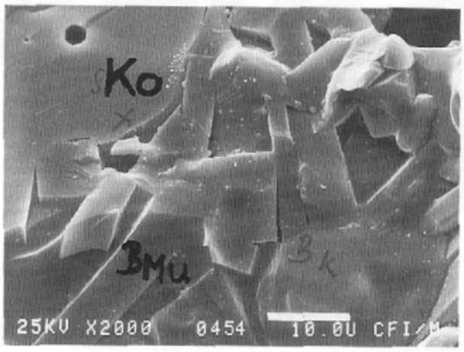

Fig. 2 Very compact crystals of secondary mullite (BMu) as bonding agent

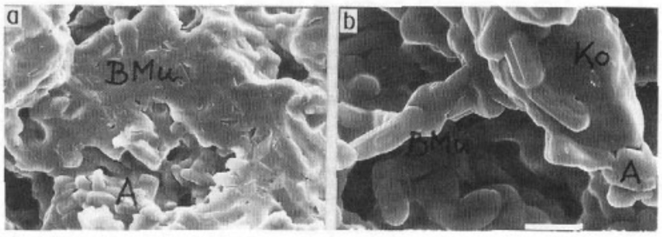

Fig. 3 a/b Fracture surface of material 3: mullite-bonding crystals (BMu), corundum (Ko), Al_2O_3 (A)

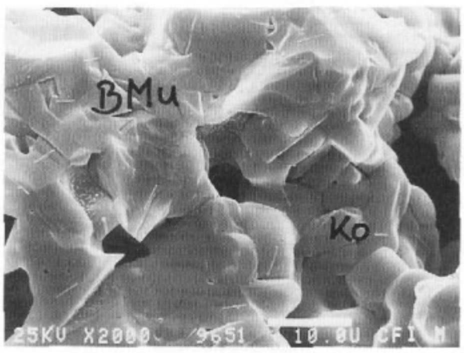

Fig. 4 Very densely packed mullite crystals with a relatively compact instead of acicular configuration

Results

Table 1 shows the thermo-mechanic properties of the four different alumina refractories. Type 1 has a pure Al_2O_3-bonding. The SiO_2-content of the bonding phase is less than 0,5 %. Figs. 1a and 1b show the structure of Type 1: Coarse grains of mullite (Mu) with a fine-grained alumina bond (Al_2O_3-Bond). Numerous fine cracks and partly inadequate contact between bond and grains are the reasons for rather low strength data.

The material exhibits a relatively high thermal shock resistance due in part to its rather low modulus of elasticity. This can be attained by the addition of rather coarse corundum grains together with a relatively high percentage of low reactive mullite. The refractoriness under load of Type 1-material is – as are the other three types – found above 1700 °C. The demand for high strength, improved flatness, improved surface-smoothness as well as improved thermal shock resistance gave rise to Type 2 test material.

As can be seen from the test results, Type 2 has a mullite bonding with some excess Al_2O_3 to avoid free, unreacted SiO_2-residues. Very compact crystals of secondary mullite (BMu) as the bonding phase provide high strength for this refractory (Fig. 2). By changing the grain size composition, a noticeable improvement in the surface character of the batts can be attained. The data on higher strength and lower thermal expansion hint at an improved quality level. This can be proved by checking two properties: static fatigue and thermal shock resistance.

Measuring hot bending strength and refractoriness under load will give some information on the short term behaviour of the materials at elevated temperature. Measurements of static fatigue will allow for some long range prognoses. During this test, bending bars were loaded with 0,1 N/mm^2 at a temperature of 1700 °C. The elapsed time before the break was measured (Figs. 5, 6).

Table 1 Thermochemical properties of 4 different high alumina refractories

Refractory No.	1	2	3	4
open porosity [Vol %]	22	22	20	20
bulk density [g/cm^3]	2,75	2,75	2,80	2,75
bending strength at 20 °C [N/mm^2]	9	14	14	15
bending strength at 1600 °C [N/mm^2]	4	14	13	17
bending strength at 1700 °C [N/mm^2]	2	6	8	10
RUL T_{05}[°C]	> 1700	> 1700	> 1700	> 1700
thermal fatigue at 1700 °C [h]	Br. 1650°C*	0,2	1,4	1,4
thermal expansion [$K^{-1} \times 10^{-6}$] from 20...1100°C	6,9	6,6	6,5	6,4
Young's modulus at 20 °C [N/mm^2 x 10^4]	1,5	4,4	2,9	3,7
mineral composition [%]				
corundum	58	54	49	42
mullite	38	41	46	56

* Br. = rupture at

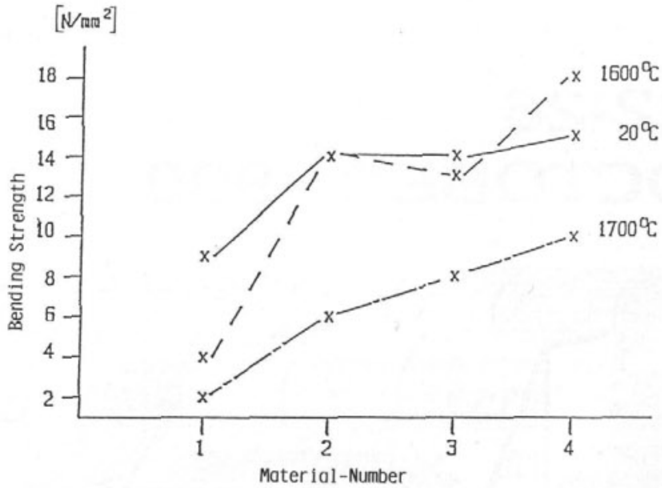

Fig. 5 Bending strength at various temperatures

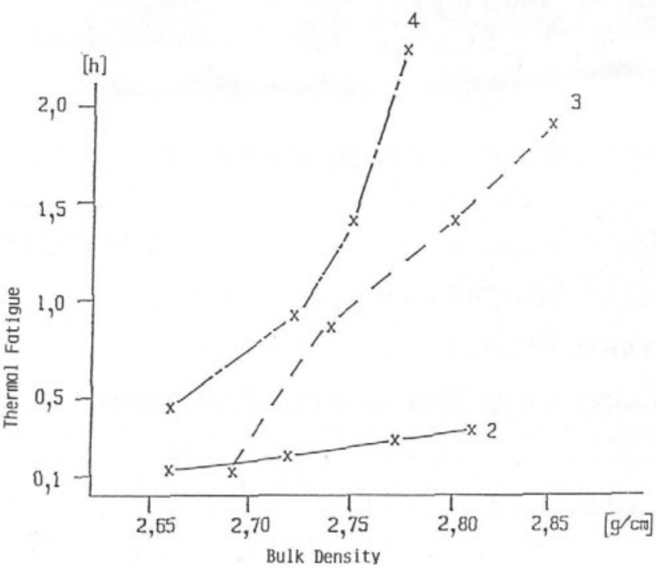

Fig. 6 Thermal fatigue vs. bulk density

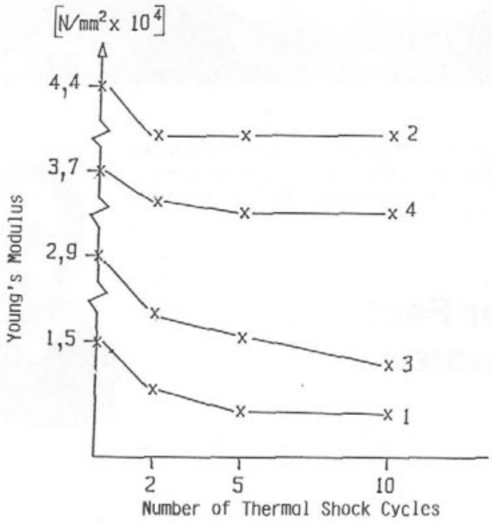

Fig. 7 Youngs' Modulus vs. thermal shock cycling

To determine the thermal shock resistance of the four materials, the Youngs' modulus of each was measured by means of resonance frequency in the original state as well as after 2 to 10 thermal cycles during which samples were heated to 950 °C and then cooled to room temperature in calm air. As can be seen in Table 2 and Fig. 7, the decrease in the Youngs' modulus of Type 2 is much less than Type 1. This is an indication of the fact that no deterioration of the ceramic structure takes place due to thermal cycling with Type 2. Parallel test measurements of room temperature bending strength after thermal shock confirmed the same tendency. In contrast, either the Youngs' modulus or the strength of alumina-bonded Type 1 decrease with an increasing number of thermal shock cycles.

In Type 3, the mullite bonding was newly designed by means of another type of raw material. By changing the granulometry, the bulk density of the material could be increased. Figs. 3a and 3b show the fractured surface of Type 3. The mullite bonding crystals (BMu) form sinternecks to corundum grains (Ko) with some excess alumina (A).

Due to the increased secondary-mullite content, the strength data are rather high in spite of the relatively coarse grain composition. One of the drawbacks of this type of material is its vulnerability to thermal shock effects. The surface character of the batts will not meet the demands of a high-standard application.

Type 4 is a further development of Type 3. By reducing the maximum grain size, the surface quality should be improved. The amount and the character of the bonding was also improved thus exhibiting higher strength data in spite of a lower bulk density. Detailed observations of the bond area of Type 4 indicate very densely packed mullite crystals with a relatively compact instead of acicular configuration (Fig. 4).

This again resulted in a rather low Youngs' modulus indicating a reduced vulnerability to thermal shock. The results in this respect are very similar to Type 2. Type 4 also performed exceptionally well in the static fatigue and thermal shock tests which indicate a rather high service potential for Type 4.

Table 2 Variation of Young's Modulus after thermal shock cycling

Material	Young's Modulus [$N/mm^2 \times 10^4$]	Decrease [%]		
		2x*	5x	10x
1	1,5	11	16	17
2	4,4	3	3	3
3	2,9	7	10	13
4	3,7	3	4	4

* cooled from 950 °C to RT in calm air

Conclusion

Tests of four different high alumina materials having different types of bonding demonstrate the advantages of pure mullite bonding in comparison to alumina bonding systems. In the latter high thermal stress may occur in the ceramic microstructure thus deteriorating important application-oriented properties like high temperature strength, creep resistance or thermal shock resistance. The results of the laboratory tests can also be confirmed in praxis. As raw materials, fused mullite and sintered corundum have proved to be attractive for this kind of refractory material. For a good mullite bond phase, the firing temperature of the kiln furniture material should exceed 1700 °C. The importance of constant production control can also be inferred from the dependence of static fatigue on bulk density.

In general it may be stated that only raw materials of high purity should be used. Contaminations will increase the amount of glass phase and the viscosity of the glass phase will lower the strength data of the materials at high temperatures. The amount and character of the bond phase are very important with respect to static fatigue and creep behaviour of the various refractories and their practical service potential. It has been found that increasing the amount of the bond phase will improve short term strength data, yet negatively influence the long time strength. In other words, the goal of development projects should be to strike a compromise between sufficiently low amounts of bonding phase, and short-term high temperature strength data as well as the necessary creep and static fatigue properties.

Finally, using a practical test at the factory customers will decide whether a refractory material will meet specific criteria in oxide ceramic firing. There will never be an end to the developments. Many small steps towards maximizing special properties will always remain necessary. Close cooperation between users and producers of kiln furniture will be of great importance. □

Kurzfassung – Résumé – Resumen

Brennhilfsmittel für Oxidkeramik: Technische Eigenschaften für wachsende Anforderungen

Brennhilfsmittel auf SiC-Basis weisen eine maximale Betriebstemperatur von 1600 °C auf. Jenseits dieser Grenze sind Mullit-Korund-Werkstoffe erforderlich. Bei diesen ist Voraussetzung, daß hohe Heißbiegefestigkeit bzw. thermische Beständigkeit in Verbindung mit entsprechender TWB erzielt werden, was durch selektiv entwickelten Gefügeaufbau und geeignete Rohstoffe möglich ist.

Matériaux d'enfournement pour céramiques oxydes: caractéristiques techniques pour des exigences accrues

Les matériaux d'enfournement à base SiC ont une température maximale de service de 1600 °C. Au delà, on a besoin de produits demullite-coridon. Il est important d'obtenir des microstructures et de choisir des matières premières qui mènent à une haute résistance mécanique en flexion et à une bonne tenue à la fatigue et aux chocs thermiques.

Material refractario para cerámicas a base de óxidos: propiedades técnicas para requerimientos más estrictos

El material refractario para el equipamiento de hornos a base de SiC tiene una temperatura máxima de servicio de 1600 °C. A temperaturas más altas, se requiere material de mullita-alúmina. En este caso es importante obtener una alta resistencia a la flexión en caliente y una adecuada resistencia al choque térmico a través de microestructuras diseñadas en forma selectiva y materias primas adecuadas. □