New Kiln Furniture Solutions for Technical Ceramics

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Innovative Designs and Tailored Materials offer High Flexibility

Abstract

Kiln furniture concepts to fire technical ceramics such as high alumina products, electronic components and powders are discussed. The established standard systems such as box saggars and box setters and light weight rack systems offer a solid reliability on one side but offer also opportunities for innovation. Here the combination of design flexibility, material development and production skills offer new solutions with the target to achieve strongly improved service life and reliability and as well as higher quality consistency of the final product properties.

Introduction

The appropriate solution for a kiln furniture system in order to run a stable high temperature process for any product is always a matter of the two key criteria:

- material selection
- design [1].

Progress in material development, production capabilities and in design skills have lead to better kiln furniture system solutions. The end customer has eventually the benefit of having:

- kiln furniture systems of higher durability, i.e. longer service life and thus lower cost per firing cycle
- lower weight of the kiln furniture [2,3,4,5] i.e. lower energy consumptions.

Another considerably large market segment is the kiln furniture for technical ceramics and powders. The requirements for these applications are in fact broader and quite demanding. Several applications allow using standard kiln furniture based on SiC, mullite and cordierite, which indeed fulfill the requirements to a very good extend. On the other hand, there are several harsh application environments, where the limits of standard systems are quickly reached. These are very high application temperatures, sharp thermal gradients, high sensitivity to any kind of product contamination or chemically aggressive products. The challenge is to better cope with these sensitive or harsh environments by finding new system solutions.

Kiln Furniture for Technical Ceramics and Powders

Technical Ceramics is indeed a very comprehensive term with an enormously broad application field. It is not the intention to cover the full range but to focus on some of the main application fields with high volume throughput. Depending on the applications, a set of well established high temperature materials and kiln furniture system are applied. Tab. 1 and Tab. 2 illustrate these materials and systems and the selection criteria.

Product Applications and Standard Kiln Furniture

Oxide Ceramics – High Alumina

Most of the high Alumina products such as spark plugs, tap plates or

Application	Product	Criteria	KF material	Comments	
				Pros	Conts
Oxide Ceramics	spark plugs, grinding media, oxygen sensors, tap plates etc.	T_{max}: 1500 - 1700°C air atm.	high temp. Mullite, Alumina-Mullite	low creep with coarse refractory type microstructure	rel. thick walls required, higher weight
			RSiC	low weight, high durability	possible SiO_2 contamination, oxidation
			SSiC	low weight, very high durability	possible SiO_2 contamination. limited in size
	electrical insulators, catalyst carriers, DPF	$T_{max:}$ 1300-1450°C air atm.	NSiC, RSiC	low weight, very high durability	
			Mullite, Cordierite	high durability	higher weight
Electronic Components	MLCC, PTC, varistors, piezzo, LTCC, ferrites etc.	T_{max}.: 1100 -1400°C	high-purity Mullite, Alumina-Mullite, Al_2O_3 (with or w/o ZrO_2 coating)	good thermal shock resist., hi-temp. creep resist., good chem. resist. for T <1150°C	limited chemical inertness if porous, for T> 1150°C mostly ZrO_2 coating as inert layer required to avoid contact reactions
		chem. aggressive components, e.g. Ba, Sr, Fe, Pb, Zn, Mn, Co, Mo etc.	ZrO_2, Al_2O_3, MgO, Al_2O_3-MgO Spinel, ZnO	best in corrosive environm. for T> 1150°C, lowest contamination risk	low thermal shock resistance
		various atm. from N_2/H_2 to O_2-enriched synthetic air high sensitivity to contamination	sandwich Mullite – SiC	rel. durable, mainly for Ferrite	Only bulky design, high weight
Powder calcination	pigments, luminescent substances, raw material calcinates for electronic comp., battery components	T_{max}: 1200-1400°C	Al_2O_3, Mullite,	in most applications sufficiently stable and inert	rel. thick walls required, higher weight
		air atm.	Cordierite	for lower temp.-ranges best compromise in costs vs. durability	rel. thick walls required, higher weight
		chemically aggressive components	ZrO_2	best in corrosive environments for T> 1150°C, lowest contamination	poor thermal shock resistance
		sensitive to contaminations	SiSiC	very durable for pigments, luminescent substances	not applicable for electronic components and battery components due to free Si-diffusion

Tab. 1 Categories of Kiln Furniture Application in Technical Ceramics and Powder and Materials Selections

Tab. 2 Materials characteristics of applied kiln furniture materials to fire technical ceramics and powders

Product Type		Box saggars, -setters, pusher batts						Supports			Substrates			
		Aptamull HT	Aptakor 85	Aptazirox C4	S-Corit Q	Aptamull PB	SC90GB (SiSiC)	Aptamull E	Aptakor 99G	SC100RG (RSiC)	MgO-Al_2O_3	Mullite	Zr-Mullite	Aptazirox CF
application		hi-temp.	hi-temp.	powder calc., electr.	powder calc.	electr., hi-temp.	powder calc.	electr.	powder calc., electr.	electr.	electr.	electr.	electr.	electr.
max. appl. temp.														
ox. atm.	°C	1700	1600	1400	1360	1700	1350	1400	1550	1650	1400	1400	1400	1400
neutr. atm.	°C	1700	1600	1400	1360	1500	1350	1400	1550	2000	1400	1400	1400	1400
MoR	MPa	6	9	8	12	6	230	13	218	96	19	6	46	22
H_2O absorp.	%	8,5	7,0	7,0	12,0	6,3	0,0	7,5	0,00	5,3	5,7	11,3	12,3	7,0
Density	g/cm^3	2,5	2,7	4,0	2,0	2,9	3,0	2,8	3,9	2,7	2,9	2,5	2,3	4,1
Porosity	%	21,5	18,5	27,0	24,1	18,3	1,6	21,2	0,00	14,4	16,2	28,0	28,6	28,6
CTE $_{20-1000\,°C}$	$10^{-6}/K^{-1}$	5,3	5,2	9,0	3,2	5,5	4,3	5,6	8,1	4,8	11,1	5,6	4,9	
TSR		excellent	good	medium	excellent	excellent	excellent	good	medium	excellent	medium	good	medium	medium
Al_2O_3	%	80,7	83,1		45,8	90,3		90,9	99,7		19,4	76,8	52,1	
SiO_2	%	18,8	16,3		43,4	9,6		8,9	0,0		0,1	22,7	25,3	
Fe_2O_3	%	0,1	0,1			0,0		0,0	0,0		0,1	0,1	0,1	
MgO	%	0,1	0,1		8,4				0,0		79,9			
ZrO_2	%			95,0									22,5	95,0
CaO	%	0,1	0,1	5,0		0,1		0,1	0,0		0,1	0,0	0,0	5,0
TiO_2	%		0,1			0,0		0,0	0,0			0,0	0,1	
Na_2O	%	0,1	0,0			0,1		0,1			0,0	0,0	0,0	
K_2O	%	0,0	0,1											
SiC	%						85,0			>99				
Si	%						15,0							

grinding media have to run through firing temperatures between 1550 – 1700 °C. Batch kilns, pusher kilns, roller hearth kilns and tunnel kilns are applied, which basically depends on the required flexibility and volumes to be produced. In general the continuous kiln systems have smaller cross sections due to the very high temperatures in order to reduce any thermal gradients to the best extends. The products are in most cases placed in box saggar systems which run either as a monolayer through continuous kiln systems, such as roller hearth- kiln or pusher kilns, or the saggars are set up as a multi-layer system either by a direct stacking of the saggars onto each other or with a SiC-based support beam structure.

The two base materials that are applicable for these high temperatures in cyclic conditions are mullite, mullite –alumina and RSiC and SSiC.

Mullite HT

The design and the microstructure of the mullite based saggar systems is tailored to minimize any high temperature creep and to cope to the best extend with the thermal gradients during the heat cycles. So far the best established systems are based on high temperature Mullite materials which have typically a coarse grained mullite microstructure without any glassy phases. Mullite is here clearly superior to Alumina due to its lower high temperature creep rate. To further minimize any high temperature creep the box saggar design is relatively compact with wall thickness of typically between 14 – 18 mm. However, with this compact design there are automatically limits in the applicable sizes of the saggar systems, i.e. to large sizes would lead again to more creep and to higher thermal stresses in the box saggar itself under cyclic conditions which reduces the thermal shock resistance (Fig. 1, 2).

RSiC

Recrystallized SiC box saggar systems are another alternative, which are typically lighter then mullite systems with wall thicknesses of 8-10 mm. RSiC has the benefit to have zero high temperature creep and its thermal shock properties are indeed superior to Mullite based systems. With this the service life time of RSiC is higher,- this being in fact only limited by the oxidation of the RSiC microstructure. Today's RSiC qualities reach considerably long service lives. However, the RSiC oxidation does lead to SiO_2 formation which could lead to a contamination of the high alumina products. This is typically prevented intermediate inert layers between product and RSiC, e.g. alumina girt or -coating.

Roller hearth kilns run at these high temperatures with RSiC rollers, and it is here crucial to avoid any glassy phase formation between the RSiC roller surface and the box saggar- or roller batt material. This can be achieved by running RSiC rollers with RSiC saggars which are free of glass forming components, i.e. only RSiC surfaces touch each other during the transport of the saggars along the roller carpet at higher temperatures, - the interface between roller and saggar is "dry". A combination of RSiC rollers and e.g. oxide based saggars could lead to some glass formation between the interfaces. The friction at the interfaces leads to Al_2O_3-SiO_2-based dust, which forms easily a liquid phase particularly if some further contaminants are present. All this can then lead to hazardous sticking between saggars and rollers and disrupt the continuous kilning process.

Support structures in e.g. batch kilns or tunnel kilns are preferably designed using RSiC cross beam

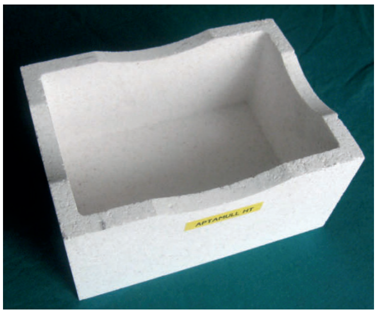

Fig. 1 (left) Standard box saggar in mullite-HT to fire spark plugs

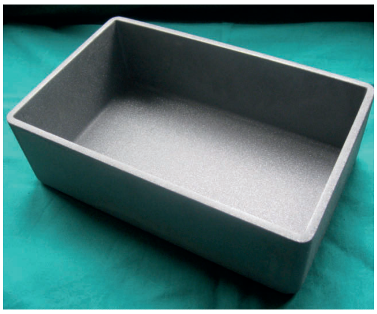

Fig. 2 (right) Standard box saggar in SC 100 RG (RSiC) to fire grinding media

structures. For pusher kilns the standard pusher batt material is similar to the saggars, i.e. a high purity, coarse grained Mullite which is tailored to have a high wear resistance to withstand the typical pusher kiln conditions.

Oxide Ceramics – Insulators, Catalysts and Filters

Electrical insulators, Cordierite catalyst carriers or filters are fired at relatively lower temperatures of approx. 1300 – 1450 °C. This is the typical temperature range for NSiC- or RSiC-kiln furniture for very light and flexible super-structures [1]. Fig. 3 and Fig. 4 illustrate a typical superstructure for electrical insulators in NSiC and an RSiC-Mullite assembly saggar for diesel particle filters respectively.

Electronic Components

The firing temperatures for electronic components range between 1100 – 1400 °C depending on the product compositions. The firing atmospheres vary from oxygen free N_2/H_2 gases to oxygen enriched synthetic air. Piezoceramic , MLCC, PTC and varistors are multilayer ceramics based on Titanate structures which contain relatively aggressive ingredients such as Ba, Sr, Pb, Fe, Mn, Zn, Mo, Co, Cu etc. Ferrites are dry pressed products with similar aggressive ingredients, i.e. Fe, Mn, Zn. Thus the key task is to cope with the contact reactions between product elements and kiln furniture materials. Interface diffusion between the electronic component and the kiln furniture material must be prevented or at least minimized to the best extend to assure a high consistency of the electrical performance of the products.
Today's standard solutions for e.g. MLCC are focused to be light, stable and inert to any inter-diffusion processes between the MLCC and kiln furniture material. The products run in most cases through pusher kiln systems with N_2/H_2 atmosphere, some of the high-end products are fired in smaller batch kilns. Two typical kiln furniture systems have been firmly established in this market segment:

Box setter Systems

These are stackable alumina- or mullite based box setters systems which are typically ZrO_2 coated (inner part only) or have ZrO_2- or ZrO_2 coated mullite substrates as inlays. The ZrO_2 prevents the contact reactions between MLCC-products and kiln furniture material .

Rack systems

Racks are ultra-light, stackable supports in alumina or mullite, e.g. "Spaghetti" racks [8], onto which thin ZrO_2 coated Mullite substrates are placed. The service live time of these box setter- and rack systems are is mainly limited by the gradual mechanical weakening of the materials during the thermal cycling. The oxide material systems tend to some high temperature creep, which in fact limits the automatic loading and unloading for these high volume products. Furthermore the durability of the ZrO_2 coating is crucial and could also limit the service life. The chemically more resistant materials based monolithic ZrO_2, MgO or Al_2O_3-MgO Spinel have just a limited application due to the relatively lower thermal shock resistance. Their applications are limited to smaller product geometries and have so far no high volume application.

Powder Calcination

Luminescent substances, pigments, raw material calcination for electronic- and battery components etc. run through high temperature treatments to transform the initial raw material ingredients to the required final phase composition, i.e. titanates, ferrites etc. In case of relatively short soak times smaller rotary kilns are the most economical solution. For longer soak time, lower volumes or higher flexibility, the calcination process is preferably done in classical box saggar systems which are either running through continuous pusher- or tunnel kilns or stacked onto each other in batch kilns. It is a fairly simple process, however, any contamination with the calcined powder product and the box saggar material ought to be avoided or at least minimized to the best extends. Considering the powder calcination for electronic components, the same issues occur as with the firing of the MLCC or piezo ceramics themselves, i.e. contact reactions with Ba, Pb, Zn, Sr, Fe, Mn etc. and the kiln furniture material. Due to the fact that the calcination temperatures are usually lower then the firing temperature of the final product, the contact reactions are less severe and can in most cases be tolerated. Thus box saggar systems based on Cordierite and Mullite are very well establish as a standard. For the calcination of luminescent substances, dense saggar systems such as dense Alumina or SiSiC are frequently applied.

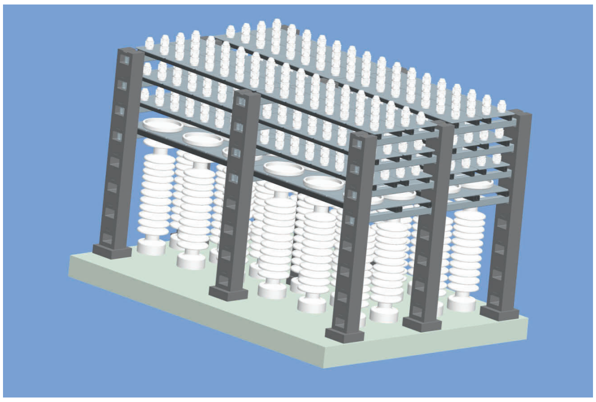

Fig. 3 Isolator firing kiln car superstructure with SiC posts and connectors in AptaSiC N (NSiC), aptasinit beams and –batts (NSiC)

New Solutions with Flexible Design- and Material Combinations

All of the above described kiln furniture system are well established. The pros and conts of the basic solutions are listed in Tab. 1. Each of these systems has certain limits. The target of the presented innovation approach is to overcome the current hurdles that limit the service life and performance of box saggars and – setters, spaghetti racks etc. Thus a new, very flexible design solution is necessary which allows an intelligent combination of the most suitable materials for kiln furniture systems to eventually improve their service life and to eliminate basic deficits.

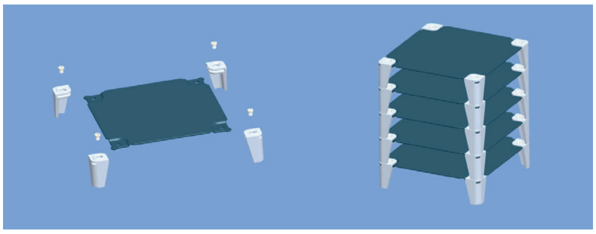

Fig. 4 SC1OORG–mullite E assembly as a stackable setter system for DPF firing (RSiC and mullite, patented design [6])

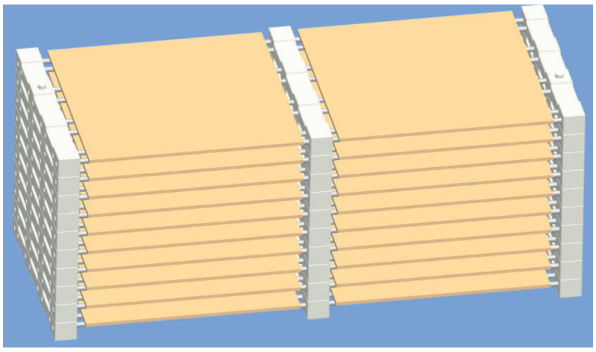

Fig. 5 Mullite based "spaghetti rack" system with alumina or mullite rods glued into mullite support segments. ZrO_2 coated mullite substrates are placed on the rod to hold the MLCC [8]

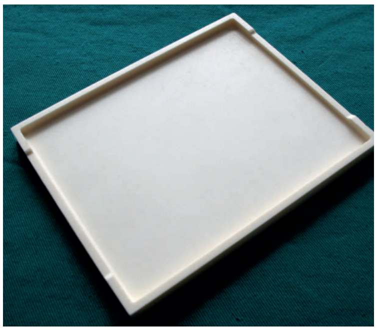

Fig. 6
Stackable box setter system in mullite E to fire MLCC. ZrO_2 coating will be applied onto the setter to prevent contact reactions

Design Innovation for Oxide Ceramics

Box saggars as such have limits in weight, size and durability. However, the establish kiln systems at the customer are set up to run theses geometries. Thus the task is to re-design the box saggar system and keep its basic function rather then to redesign the total firing system.
Box saggars for high temperature firing have their limits in sizes, thermal shock resistance and in the case of RSiC saggars a potential contamination of the oxide ceramic products with SiO_2 can occur. The actual reason for the build-up of high thermal stresses in box saggars is directly linked to their geometry and size. In the case of mullite based saggars relatively thick walls are applied to minimize the high temperature creep to the best extends. RSiC is the best material as far as thermal shock resistance and high temperature creep stability is concerned whereas Mullite is the best material to avoid any contamination with the oxide ceramic product. Thus the simple logic is to combine these two materials by using an assembly design [6]. The materials are applied right there where their properties are required. Furthermore the assembly solution as such reduces considerably the total built-up of thermal stresses during the high temperature cycles since individual kiln furniture segments are relatively small.
Fig. 8, 9 illustrate three principal approaches, which all have in common that the actual box saggar is assembled by an RSiC base plate to prevent any high temperature creep at the bottom of the saggar. To avoid contact reaction between the high alumina products and the RSiC, a second mullite-HT cover plate is positioned on top of the RSiC plate. The side elements are typically in mullite-HT with the options to have them as a single ring holding the base plates or the have them assembled by side-and corner elements which are connected by a ceramics screw connectors. These saggar systems can of course be stackable and, if required, have additional inlays to hold e.g. the spark plugs.

Conclusion

Assembly of mullite and RSiC:
- apply materials only where key properties required, avoid SiO_2 contamination of alumina products
- reduce thermal stresses; replacement of individual parts rather then complete saggar

→ increase service life time
→ reduce costs per cycle

Design Innovation for Electronic Components

MLCC Racks

The limitation of the service life time of the ceramic "spaghetti" racks is mainly due to their gradual deformation and –cracking over the service time. ZrO_2 coated substrate placed onto these spaghetti racks do also suffer from gradual cracking and deformation which again limits their durability and automatic handling.
An alternative approach [6,9] has been successfully tested on a production scale in several MLCC-production lines. The concept is to apply an RSiC "butterfly rack system" which is stackable with mullite-E connector elements (Fig.). These 3 mm RSiC-butterfly racks are coated with a synthetic mullite layer which perfectly interlocks to the open porous RSiC-microstructure. Thus any contact reactions between the RSiC butterfly rack and component are safely prevented. Standard substrates based on ZrO_2-coated Mullite are place on the racks to hold the MLCCs.
The following benefits arise from this new system:
1. RSiC has zero high temperature creep and a superior thermal shock resistance which leads to an outstanding stability of the systems and thus very high service life in the N_2/H_2 atmosphere.
2. The higher thermal conductivity of the RSiC butterfly racks leads to an excellent, very precise heat transfer to the MLCCs which eventually results in a better consistency of their electric properties
3. The stacking system allows a safe manual or automatic handling of the complete stack (typically 15-17 layers)
4. The flexibility of the stacking system allows various distances between each rack layer by simply exchanging the Mullite-connector elements. The packing

Fig. 7
Box saggar for powder calcination:
left: Aptazirox C4 saggar (ZrO_2);
right: S-Corit Q saggar (Cordierite) for e.g. electronic component calcinates and battery components respectively

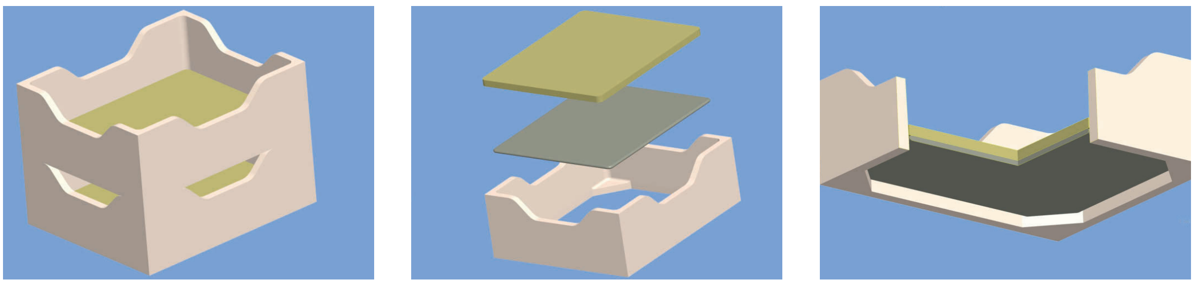

Fig. 8 a-c High temperature box saggar assembly with SC1OORG- (RSiC) base plate and square side element and cover plate in mullite-HT

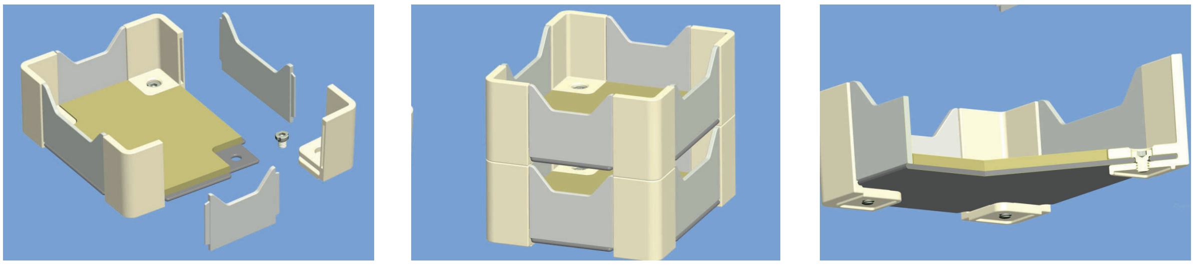

Fig. 9a-c High temperature box saggar assembly with SC1OORG (RSiC) base plate, cove plate in mullite HT and side elements and connector elements in mullite HT and mullite E (patented design [6])

density of the stack system can be easily optimized for the individual product- or process requirements.

Conclusion

Mullite and coated RSiC assembly:

- best service stability due to zero creep, no contact reactions
- improved stability of electrical parameters
- safe manual- and automatic handling
- high flexibility for optimizing packing densities

→ lower cost per cycle
→ higher process stability
→ improved product quality consistency

MLCC Box Setters

As similar assembly approach using Mullite coated RSiC box setter base elements, Mullite connectors and ZrO_2 substrates is illustrated in Fig. 11. The mullite coated RSiC base setter has a reduced wall thickness of just 3mm which results in comparison to tradition box setters (Fig. 6) in approx. 30 % less weight and less height. Therefore the number of stacked box setters can be increased giving a higher production capacity. The selection of substrate material placed into the box setter can then eventually determine the best thermal heat transfer rate for the MLCCs to better control the electrical parameters of the MLCC.

Conclusion

Coated RSiC base setter assembly:

- lower weight and height lead to increased production capacity
- heat transfer rate variable to optimize product needs
- improved service life expectations due to excellent thermal shock and –creep stability
- flexible box setter height with exchangeable connector elements

→ increased production capacity, lower specific production costs
→ safe handling
→ improved product quality consistency

Design Innovation for Powder Calcination

It is typically the powder calcination line that massively determines the final product qualities. The best solutions in this crucial process are needed to maximize the consistency of the powder qualities. Potential contaminations from interface reactions between the powder the kiln furniture must be minimized. Thus saggar materials with the lowest chemical affinity to the powder should be applied. MgO-Al_2O_3 Spinel, ZrO_2 or Al_2O_3 and SiC offer these properties; the selection of the best material combination depends eventually on the powder. A monolithic box saggar in MgO-Al_2O_3 Spinel, ZrO_2 or Al_2O_3 would be limited in size due to their low thermal shock resistance. The ceramic assembly solution does offer the opportunity that only ZrO_2 elements touch the powder as illustrated in the Figure . The box saggar consists of ZrO_2 side- and bottom segments that are assembled by mullite-E corner- and connector elements and a SiC base plate. The total thermal stresses are significantly reduced in all assembly elements, which is particularly important for the ZrO_2 parts. The mullite- and RSiC elements assure a solid durability in the thermal cycles. It is obvious that the inner ceramic elements are flexible, i.e. Al_2O_3, MgO, Spinel and other material can be applied as well.

Conclusion

- Assembly design minimizes any cross contamination risk
- Assembly design minimizes thermal stresses

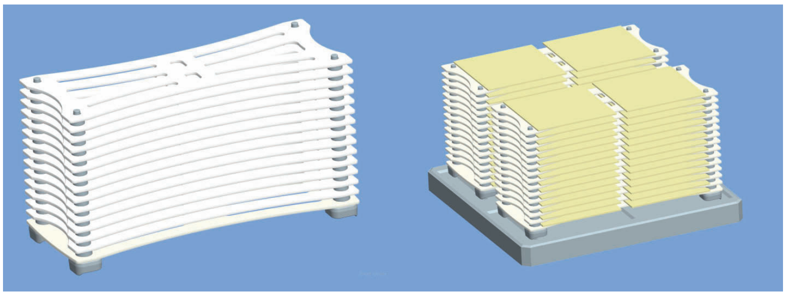

Fig.10 a, b Stackable butterfly rack system for MLCC firing; left: SC100RG- “butterfly racks” (3 mm, RSiC) with synthetic mullite coating assembled to a stack (patented design [6,9]); right: double stack with ZrO_2 coated mullite substrates, all placed on a mullite PB pusher batts

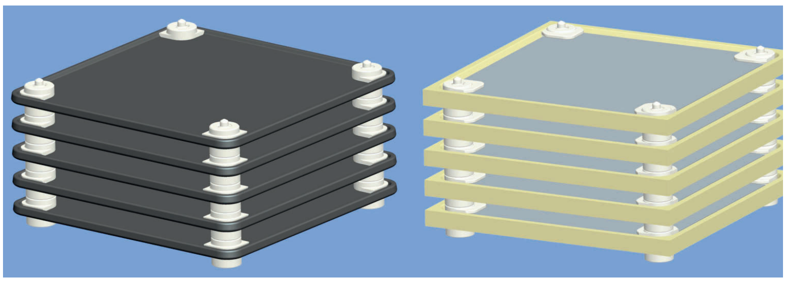

Fig. 11 a, b Box setter assembly system consisting of synthetic mullite coated SC100RG base plates (RSiC, 3 mm), mullite E- connector elements and ZrO_2 substrate inlays (fixed by connectors). Low product height allows additional layer in the stack and increases production capacity (patented design [6])

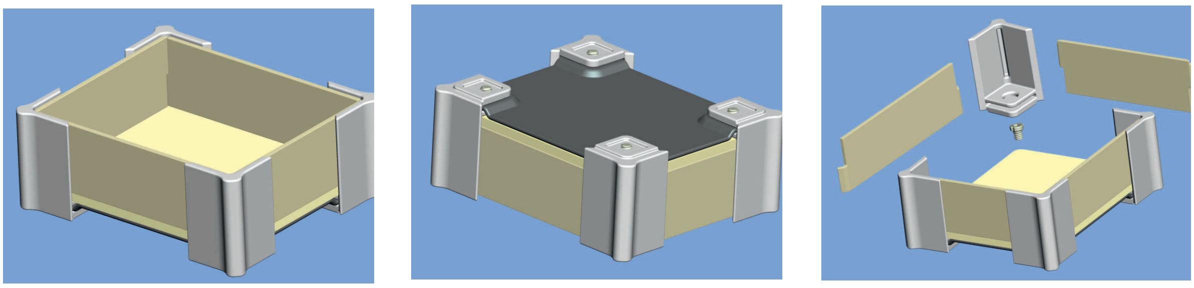

Fig. 12 a-c Box saggar assembly for powder calcination without cross contaminations: SC100RG base plate (RSiC), mullite-E corner- and connector elements, aptazirox C4 cover plate and - side elements (ZrO_2), patented design [6]

→ increased service life, lower cost per cycle
→ improved powder purity, higher product quality consistency

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