High-End Al_2O_3 Ceramics: Easy to Get with New Ready-to-Press Material from Nabaltec

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There are two main reasons for using alumina in ceramic applications. First, there are technical reasons, when certain material characteristics are needed and second, there are commercial considerations, aiming to get these features at an affordable cost-value ratio. The most important reason is usually on technical side, as Al_2O_3 is an excellent choice when engineered parts are applied under rigid conditions. In this operation purpose the high number of material advantages of alumina is beneficial. Properties like excellent thermal creep resistance, high thermal conductivity and the remarkable electric insulation are the main reasons why alumina is used in a variety of E&E applications.

This area is growing quite a lot as products like high energy LEDs, computer chips and relays, become increasingly important. Last but not least, such parts are used to a greater extent in the ever increasing electric mobility market. But also attributes like the exceptional hardness and the high wear resistance in combination with a superior corrosion resistance make alumina the leading material when it comes to applications in the engineering industry [1]. In this connection, an often addressed challenge is the failure behaviour of ceramics based on statistical effects. Their breaking behaviour is described by a relatively broad cumulative failure distribution, which makes it unusual for the responsible engineers to calculate the necessary part dimensioning [2]. Therefore, as users have to be on the safe side they calculate the necessary part properties together with a big share of safety tolerance. As a result, the achievable breaking strength and hardness have to be continuously improved in order to meet the required material properties. This is not only done by adjusting forming steps and sintering procedures but also by tuning the raw materials itself under the premise not to increase the costs per part significantly.

Introduction

The key for its successful improvement lies in the microstructure of the ceramics. Commonly, disruption energy is not high enough to break alumina particles up directly, but cracks are growing alongside the grain boundaries. It is obvious that a defect-free single crystal of alumina has the maximum breaking strength and the highest hardness in contrast to polycrystalline ceramics which are finally a sum of grains in different sizes, varying grain boundaries, particle packing defects and chemical impurities spread between the grains.

All these influences reduce the maximal possible mechanical properties and are the starting point for the alumina producer like Nabaltec AG for improving the raw material leading to a number of specifically optimised GRANALOX®-products.

The origin of cracks in ceramic parts are in most cases defects already present on or near the surface whereas they act as an initiator when load is applied onto the ceramics. The fissure goes alongside the weakest points through the microstructure while defects like voids and crystal phases formed by chemical impurities reducing the necessary energy for breaking [3]. For crack propagation, the defect origin is not important, in contrast to the amount and volume of the voids within the microstructure. Furthermore, understanding the origin of the defects is exactly the approach by which the manufacturer and raw material supplier have to optimise the microstructure.

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Discussion

The possibilities at Nabaltec are focused on the raw material itself, as it has an enor-

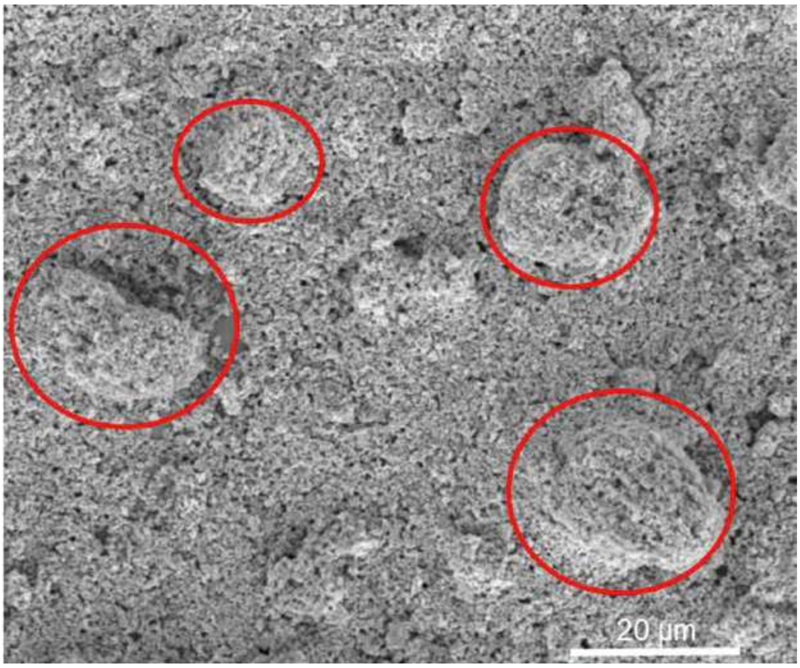

Fig. 1 **Partially milled alumina (agglomerates marked in red)**

mous influence on the physical ceramic properties depending on how the alumina is designed. Generally, there are two main options for improving the fracture strength, which are on the one hand optimising the particle distribution and on the other hand adjusting the feedstock formulation, e.g. when the material is used for dry pressing as forming process. The result is later tested by measuring bending strength on manufactured test specimens.

Optimisation of the particle distribution is a very important step, as the original material are aggregates consisting of primary particles whose size can be controlled during the calcination process. During the following milling procedures, these aggregates are broken down to a certain degree whereas the targeted grain size distribution depends on the needs of the individual application.

In the case of engineering ceramics, the microstructure has to be preferably defect-free and homogeneous as these parts have to tolerate high shearing forces.

Therefore, the starting material has to be dispersed almost quantitatively to avoid agglomerates which disturb the packing during the forming process and result in defects within the microstructure after sintering.

The grinding process starts with basic calcined alumina with a mean grain size diameter of about 80 µm. These aggregates consist of about 0,4 µm primary particles which are separated during the further procedure. By using conventional milling technology there are plenty of agglomerates left in the material sizing up to 20 µm (Fig. 1). This causes defects in the following processes and leads to voids after the sintering process in the ceramic microstructure with the result of a low fracture strength of the ceramic part [4].

Nabaltec uses an advanced milling technology in order to disperse almost 100 % of agglomerates. The main difference between conventional and advanced milling is found in the maximal possible dispersing degree, where the energy might have been not high enough to destroy all agglomerates during the process. An indicator for that is the relatively high D_{90} value measured by laser diffraction which is at about 2,2 µm for a grade consisting of 0,4 µm primary particles (Fig. 2). By using the advanced milling approach the material can further be dispersed. The progress is checked by determining the D_{90} continuously which goes down gradually to a D_{90} of about 1,3 µm where the process is stopped.

These two different prepared alumina grades were used to check if there is a noticeable difference in the defect rate within the microstructure after sintering. Therefore, each grade was mixed with about 4 % of deionized water and test specimens were made by uniaxial compaction at 100 MPa. After sintering at 1600 °C with a dwell time of 2 h, the surfaces have been polished and thermally etched.

The microstructure checked by SEM-imaging shows that there are obviously differences of the defect rate between the two grades (Fig. 3). While there are many and big voids when the conventionally milled grade is used, the microstructure of the advanced milled grade is smooth with only few defects in the observed area. This is exactly the result needed for a raw material used for high-performance engineering ceramics.

Common forming methods for parts in this application are uniaxial or isostatic com-

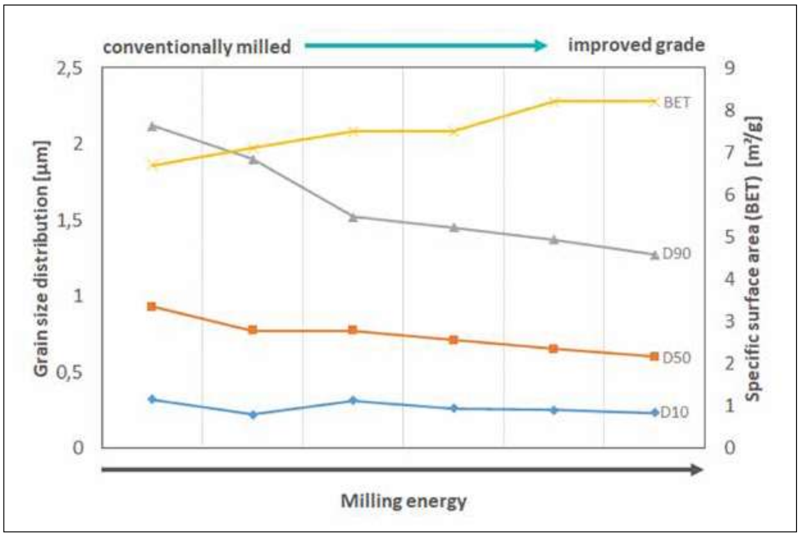

Fig. 2 **Dispersing progress of alumina; difference of conventionally and improved milled grades**

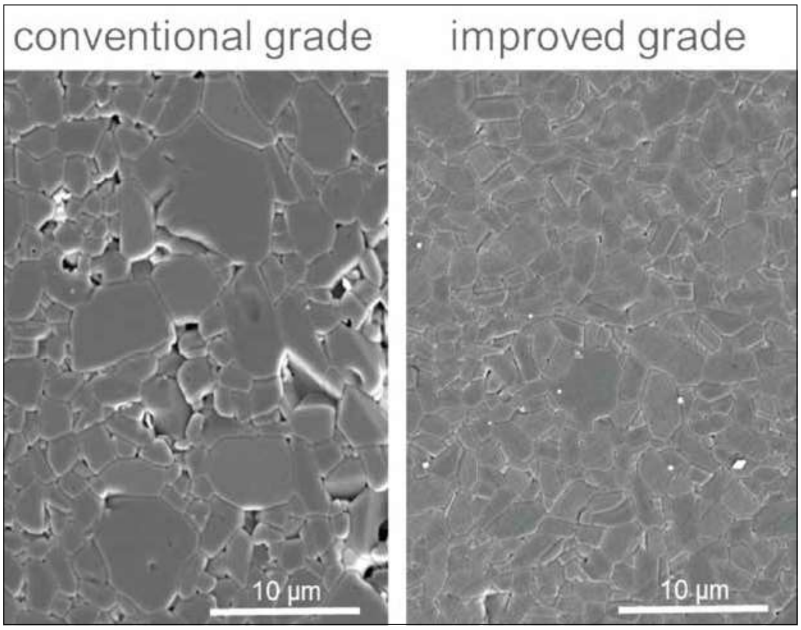

Fig. 3 **SEM-image of conventional alumina grade with defects (l.) and improved grade with almost defect-free microstructure (r.)**

paction where the alumina is conditioned by granulation. Therefore, the necessary organic feedstock has also to be improved and adjusted to the grain size distribution of the advanced milled alumina grade. Well-dispersed alumina without agglomerates and equal grain size distribution tends to make problems in the forming process. During compaction, the equal-sized particles hinder each other resulting in parts with low green density.

Thereby, pressed parts are produced which have low breaking strength and therefore it is difficult to operate them by green machining as they are too brittle. Nabaltec's production process makes it possible to tailor the organic formulation to provide grades which can be compacted under these difficult circumstances and at the same time offer high stability of the green body.

For this purpose, different types of organic modules have been defined which are used to generate granulates with particular material properties. First step of examining the behaviour of granules when getting compacted is testing them via the granule strength test [5]. Nabaltec established this test since several years and made it an essential tool in granulate development. The granule breakage can be observed during the test simultaneously by camera which gives an insight into the nature of the breaking behaviour itself. In addition, a prediction can be made how granulates will behave in the following pressing step in the bulk.

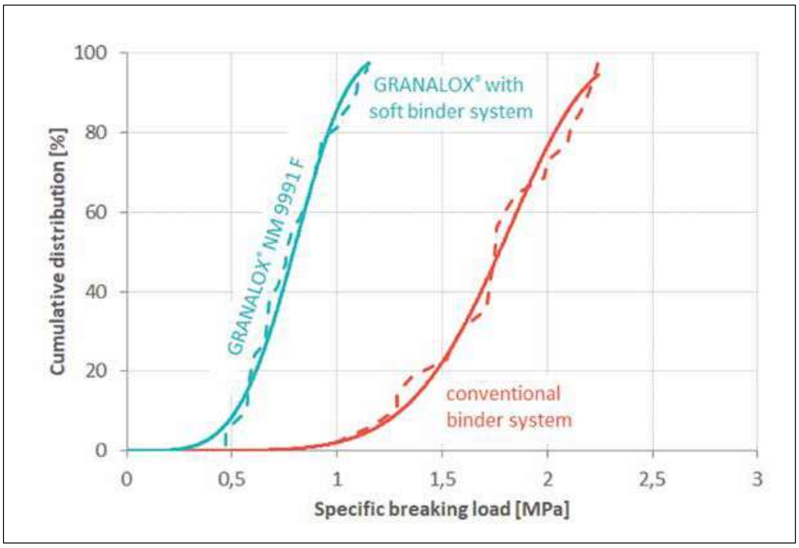

Fig. 4 **Comparison measurement of single granule breaking strength of two different binder types**

Based on this knowledge, the optimised alumina of the advanced milling has been formulated with a tailored organic composition in order to get a powder which ceramic fabricators can process easily to produce ceramic parts with high-end ceramic properties. The result of this optimisation can be characterised by the single granule breaking strength test, as the mean granule breakage was successfully decreased.

A conventional 99,7 %-alumina granulate (Fig. 4, red line) shows in production a moderate compaction behaviour resulting in a sintered density of almost 98 % of the theoretical density. This grade was tested in the single granule strength test showing a mean breakage of about 1,7 MPa.

By modifying type and amount of the organic ingredients, the pressing behaviour can be optimised. The mean breaking load for the optimised granulate is reduced to a lower favoured pressure of about only 0,8 MPa (Fig. 4, green line). Furthermore, all granules break almost at the same load, which leads to a homogeneous compaction with low defect rate. In addition, breaking load is high enough to make sure that the granules do not break while shipping them to the customers.

The final step is manufacturing of test specimens at a production press and a subsequent sintering test in the company's lab kilns. In this context, the improved grade in comparison to a common 99,7 %-alumina granulate is also tested. When the conventional 99,7 %-Al_2O_3 material is used, typically a theoretical density of 97,7 % is reached. By using the company's improved GRANALOX® NM 9991 F, the sintered density can be increased up to 99,2 % of theoretical density which is remarkable for using an alumina coming from the Bayer process.

Such high densities are commonly only achievable by applying high purity alumina or by using expensive methods like hot isostatic pressing. Therefore, this is quite impressive particularly as reached by applying conventional pressing and sintering techniques (uniaxial compaction at 100 MPa, 1600 °C/2 h).

What also was observed while testing the new granulate under production conditions was the uncomplicated behaviour while pressing as almost no material adhesion was noticed on moulds and punches in uniaxial compaction. For that reason, this grade is the right choice when parts with high throughput have to be manufactured. The optimisation of both, particle size distribution as well as tailored feedstock formulation, can also be seen in the microstructure determined by SEM-imaging. Parts made of GRANALOX® NM 9991 F show only small and few voids in combination with an equal particle size.

This is the result of the optimal grain size distribution, where sintering starts with alumina particles which are all the same size. In the following sintering process, also the grain growth is very uniform and gives only few abnormal grain growth avoiding microstructure defects.

To get a better understanding of the ceramic properties of GRANALOX® NM 9991 F, specimens were checked by the three-point bending strength test. The results showed an enormous increase of the mean bending strength of up to 60 % (Fig. 5).

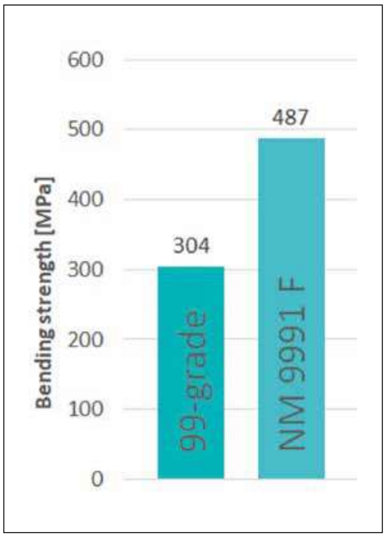

Fig. 5 **Comparison of 3-point-bending strength between conventional 99,7 %-Al_2O_3-grade and the new GRANALOX® NM 9991 F**

When a conventional 99,7 %-Al_2O_3 granulate is used, the testing bars break at already 304 MPa. This may come from structural defects which are present in the microstructure and be also seen in the comparatively low sintered density of the ceramic. When GRANALOX® NM 9991 F is used, a mean breaking strength of 487 MPa was determined demonstrating the success of alumina particle and organic recipe optimisation.

Summary

Nabaltec has been successful in developing the ceramic body GRANALOX® NM 9991 F where both the alumina particle distribution and the corresponding organic feedstock composition is optimised. This results in ceramics which can be used especially for applications in engineering ceramics where high breaking strength is needed. The adjusted granulate properties allow the customers to use the material for uniaxial compaction as well as isostatic forming and allowing different shaping methods afterwards. The organic composition is especially tailored to reduce the sticking behaviour in manufacturing to enable a high part throughput at the customer.

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