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Advances in Characterisation of Machined Green Compacts

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Abstract

The behaviour of a ceramic component is determined by the material, by the shaping and other manufacturing operations and by the service conditions. In this paper the development of characterisation methods for the quality control and quality assurance of green compacts before final densification is discussed. Two aspects are considered. The first is the numerical simulation of uniaxial compaction. The aim is to determine the surface regions with high gradients, which should be removed by green machining. The second aspect is the evaluation and optimisation of testing methods for green compacts machined by different methods and under different conditions. Due to the fact that a shaping of green compacts results in high volumes of removed powders, the recovering of these powders is discussed. © 1996 Elsevier Science Limited.

Das Verhalten keramischer Bauteile wird im wesentlichen durch das Material, die Formgebung und andere Herstellparameter sowie die Einsatzbedingungen bestimmt. In diesem Artikel wird die Entwicklung von Charakterisierungsmethoden zur Qualitätskontrolle und Qualitätssicherung vor der Verdichtung durch Sinterverfahren beschrieben. Der erste Schritt ist die Möglichkeit der numerischen Simulation uniachsialer Grünformgebungsverfahren. Das Ziel der Simulation ist die Bestimmung der oberflächennahen Regionen mit hohen Eigenschaftsgradienten, die durch eine Bearbeitung im Grünzustand beseitigt werden sollen. Der zweite Schritt ist dann die Bewertung und Optimierung von Prüfverfahren für Grünkörper, die durch unterschiedliche Verfahren und Bedingungen bearbeitet worden sind. Da die Bearbeitung von Grünkörpern ein nicht unerhebliches Volumen an Abtragsprodukten ergibt, wird die Möglichkeit zur einfachen Wiederverwendung dieser Abspanprodukte diskutiert.

1 Introduction

The complex shape of ceramic components requires an expensive machining if the shaping is performed in the sintered state. For example the relative cost for final machining of silicon nitride valves is 44%, whereas the relative cost for shaping is 6%. 23% of cost is the powder.[1] Due to economic demands a shifting of the machining to the green state is favourable, as is cost reduction by recovering of removed powders.

Machining in the green state is not only favourable to achieve a complex shape in the early stages of ceramic processing but also to reduce shape distortions after sintering which are a result of the density gradient after uniaxial pressing. With the help of a numerical simulation of pressing and sintering it is possible to reduce these distortions by calculation of the volume which must be removed by green machining to achieve a minimised density gradient.

The question is how the quality of ceramic components is influenced by green shaping. Problems arise from cracks induced by the gripping during machining, from the surface roughness and from flaws at the edges. These types of damage must be quantified and the effect on the quality of the sintered component must be evaluated. Depending on the machining method it is possible to evaluate the damage on green compact surfaces by different strength tests. Dry pressed samples were found to obey fairly well the classical laws (Weibull's statistics, the relationship between stress intensity and fracture energy).[2] Suitable test methods must be found to characterise the ceramic components as well as to determine the materials data.

A further possibility for cost reduction is the recovery of the powder removed during green machining and the repeated use of this powder for fabrication of green bodies. The possibility of repeated use arises if no alterations in primary grain size and chemistry are present. Then a repeated green compaction is useful if there is no significant decrease of properties like the strength of the green compacts or the possibility to increase the strength by alterations in the green compaction technique. Lower strength values lead to an impossibility of further green machining. Furthermore, it is necessary to avoid impurities introduced by shaping or handling. To investigate this possibility green compacts made of recovered oxide powders were tested.

2 Experimental Procedures

The ceramic powders used in this study were Al_2O_3, Al_2O_3 + 10% ZrO_2 and Mg-PSZ with average grain sizes of 1·0 μm, respectively 2·5 μm for the Al_2O_3. Each powder was available in a granulated form and as recovered products from drill machining. The green compaction was done by uniaxial pressing with pressures between 5 and 250 MPa including the usual industrial way of green compaction. Strength tests published in this paper were performed using the Brazilian disc test and the C-ring test. The sample dimensions for the Brazialian disc test were a diameter of 16 mm and a thickness of about 10 mm. For the C-ring test an outer diameter of 60 mm and an inner diameter of 20 mm with a thickness of 15 mm were chosen. Both tests were performed with a time to rupture within 10 s according to the rules for the testing of dense ceramics. Surfaces, microstructures and powders were inspected by SEM investigations. The surface conditions of the green compacts were characterised by a laser profilometer, whereas the surfaces of the sintered samples were characterised by conventional roughness measurements.

3 Results and Discussion

3.1 Numerical simulation of pressing and sintering

The methodology of the numerical simulation is described in Refs 3 and 4. The aim is to predict the density distribution in the green state, and the resulting shape distortions after sintering. This allows a correction of the shape of the green part either by machining or by modification of the pressing tools, such that the part has the desired shape after sintering. Here, the production of a typical ceramic component with various kinds of machining is simulated. The part is pressed as a solid cylinder, machined to the desired shape in the green state, and sintered. The material used in this study is a dispersion ceramic consisting of a mixture of Al_2O_3 with 10% ZrO_2. Pressing is simulated using the Drucker-Prager cap model for granular materials, which is available as a standard option in ABAQUS®. A detailed description of the model is given in Ref. 5. The friction coefficient between powder and die wall was measured in a special device.[6] Figure 1a shows the inhomogeneous green density distribution, which occurs as a consequence of the die wall friction. The removal of material during green machining is simulated by projecting the density distribution on the machined geometry (Fig. 1b).

After the pressing of the powder and the machining of the green body the sintering of the compact is simulated. In the sintering model grain boundary diffusion is considered to be the dominant mechanism for material transport responsible for the shrinkage of the body. A linear relationship

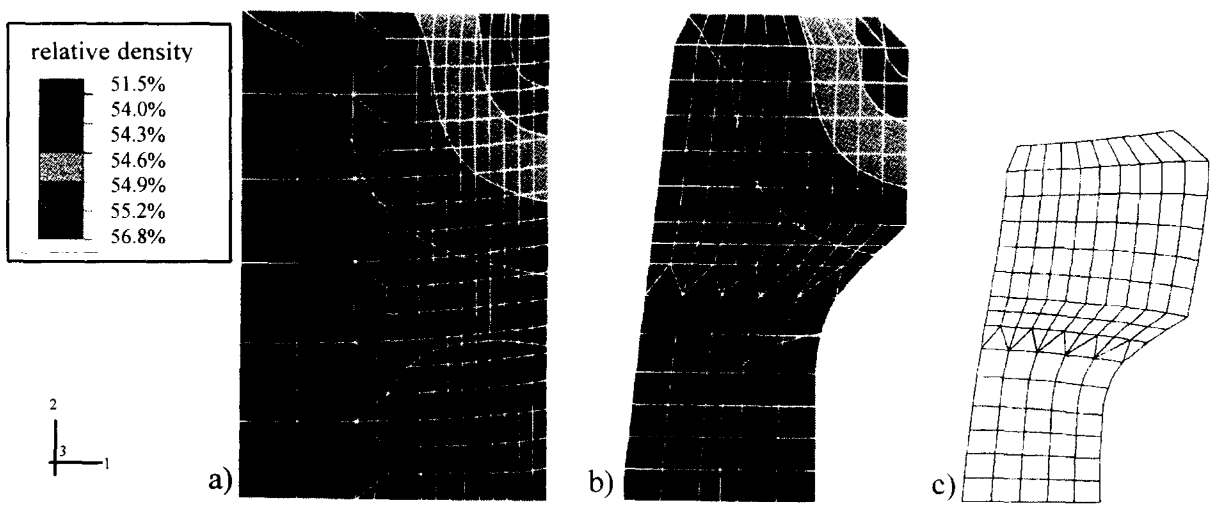

Fig. 1. (a) Density distribution in upper right quarter of a green cylinder after uniaxial die pressing. (b) Density distribution in the 'machined' green part. (c) Shape distortions after sintering (exaggerated by a factor of 5).

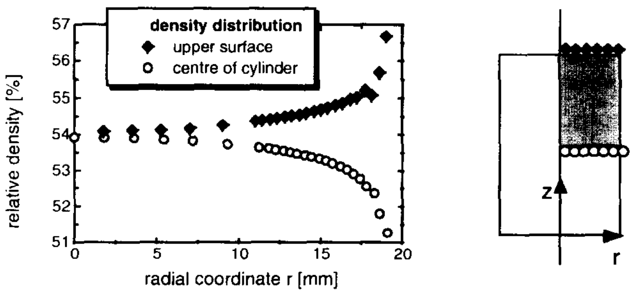

Fig. 2. Relative density versus radius in the green cylinder after uniaxial pressing. Values for the surface and in the centre of the cylinder are shown.

between strain rates and stresses is obtained. The sintering model[3,7] is implemented in the finite-element program ABAQUS®. Using the density distribution of the green part as input it calculates the stresses in the body as well as shrinkage rates and shape distortions after completion of the sintering process. The deformed finite-element mesh is shown in Fig. 1c with the displacements multiplied by a factor of 5 in order to illustrate the shape distortions.

The numerical simulation of uniaxial pressing in a cylindrical die allowing for friction between the die wall and the powder predicts high density gradients near the die walls. Figure 2 shows the calculated relative density as a function of the distance from the axis. A higher densification can be observed in the corner under the punch face, whereas the centre of the cylinder shows lower densification than the average. The density gradients exceed ± 1% per mm in regions near the wall. Machining of the green cylinder by turning removes the highest density gradients. This is the first step to minimise shape distortions after sintering caused by density gradients.

3.2 Strength of green compacts

For the practical part of this study it is possible to divide the four investigated strength testing methods by their specific properties and their advantages and drawbacks. The Brazilian disc test and the tensile test are sensitive to volume defects, while the C-ring test and the concentric ring test are suitable for evaluating the machined surface because the maximum stress occurs at the surface.

Among the volume-sensitive tests, the Brazilian disc test is simpler and cheaper than the tensile test. Therefore, the Brazilian disc test was chosen for the evaluation of the uniaxial pressed specimens with small geometries. If the machining is performed by milling, the surface can be tested by the concentric ring test. If the machining is performed by inner or outer turning, the C-ring test will be suitable. Considering the fact that turning is a frequently used machining method the first investigations were focused on the C-ring test.

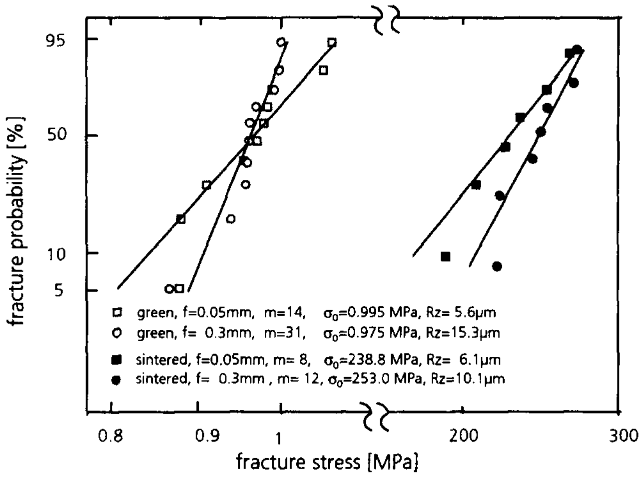

Fig. 3. C-ring test for green and sintered specimen (green machined with varied turning feed, outer turning, material Al_2O_3).

Figure 3 shows the Weibull statistics for different machining conditions in the green and in the sintered state for an Al_2O_3 ceramic. Because of an insufficient number of tests for an exact Weibull statistic these results can only show a tendency. Although a higher turning feed results in a higher roughness, a higher Weibull modulus was determined for similar values of the fracture stress. After sintering, the difference in roughness persists. This is not an absolute value because the determination of roughness for the green bodies was done by laser profilometer measurement and the roughness for sintered bodies was determined by a conventional method. This difference in roughness does not result in a significant lower fracture stress or a lower Weibull modulus for higher roughness. In contrast, the Weibull modulus for a lower turning feed is lower than that for the higher feed. These values of fracture stresses may not be exact materials strength data, since the formulas used here are valid only for thin dense specimens but not for the thick specimens. However, there is a tendency for a correlation between the quality of the green bodies and the sintered bodies as seen by the Weibull moduli.

The Brazilian disc test was investigated for experiments with recovered powders. Industrially granulated powders and powders recovered from drill machining were pressed uniaxially. Recovered powders show slightly higher densities at lower pressure as expected. An example of the densification behaviour for the two kinds of powders is given in Fig. 4. Pre-densified volumes as present in the recovered powders can lead to a higher density at lower pressure, however the packing of these powders is not optimised like that of the granulated powder. At a pressure higher than 100 MPa nearly the same densities are present for the two kinds of powder quality.

SEM-micrographs in Figs 5a and b for the green body show the boundaries for the granulated powders. Lower fracture stress than for the recovered materials is expected. After sintering, the boundaries are still visible (Figs 5c and d) and the same fracture stress behaviour is expected.

The Brazilian disc test (Fig. 6) for the green compacts was performed without any further machining. The fracture stresses for recovered materials are lower than those for specimens with granulated powders in spite of higher green densities (Fig. 7). The Weibull statistics (Fig. 8) for tests with specimens of same densities (56% T.D.) show high Weibull moduli. This illustrates the potential of the Brazilian disc test for the characterisation of homogeneity of the green body. The value for the density was chosen because this was the value of the industrial pressed green compacts tested by the C-ring method. Due to economic demands the green compaction is optimised by the density and the further densification behaviour during sintering. The lowest possible pressure corresponds to a minimum cost for the pressing tools. An earlier study has shown that the most important parameter determining the strength of a green compact is not density but the compaction pressure.[8] The results presented here show a tendency to confirm this opinion because of higher compaction pressure for the granulated powders in comparison to the recovered powders at 56% T.D. (compare with Figs 4 and 7). This dependency is clearly understandable if we look at fracture mechanisms for dense ceramics. Higher compaction pressure leads to a reduction of the defect size. So it is shown that the Brazilian disc test is a suitable method for evaluating the homogeneity of density for a powder compact.

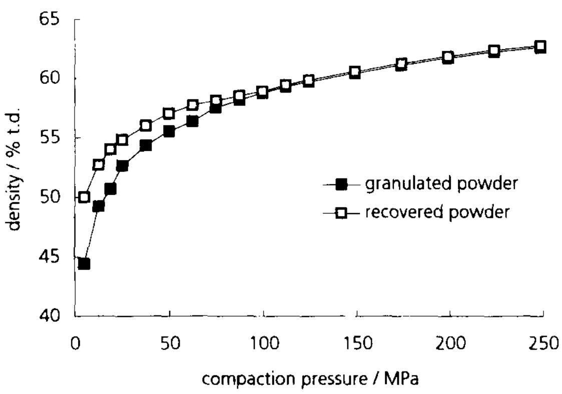

Fig. 4. Compaction behaviour of recovered and granulated Al_2O_3 powders.

The same test procedure was performed for the sintered bodies. The fracture stresses for all mate-

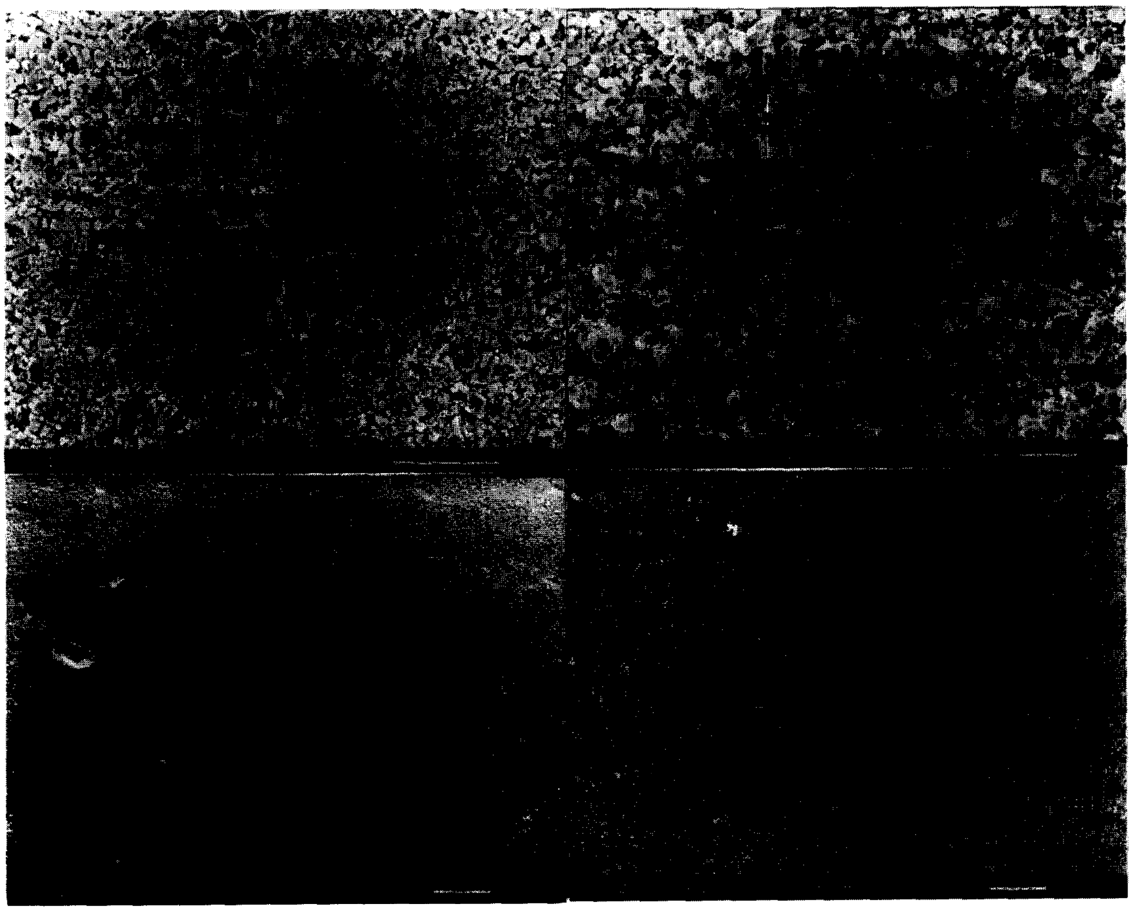

Fig. 5. Microstructure of green compacts and sintered bodies from granulated and recovered Al_2O_3 powder with 10% ZrO_2; (a) granulated green, (b) recovered green, (c) granulated sintered, (d) recovered sintered, compaction pressure 249 MPa.

rials made from recovered powders were higher than for those made from the granulated powders. The results are given in Table 1. These values should not be considered as materials strength data, since the specimens were not machined after sintering. This has great influence in the loading area, where due to inhomogeneous shrinkage the loading is not well defined. Nevertheless, one criterion for the test validity, fracture along the diametral plane, was fulfilled for all tested specimens.

The results of the fracture experiments obtained by the Brazilian disc test show a potential for the repeated use of recovered powders. In this study the powders were used a second time without further recycling methods, like milling or sieving. This implies a maximum of cost reduction. The question of the purity of the sintered specimens must be answered in further investigations.

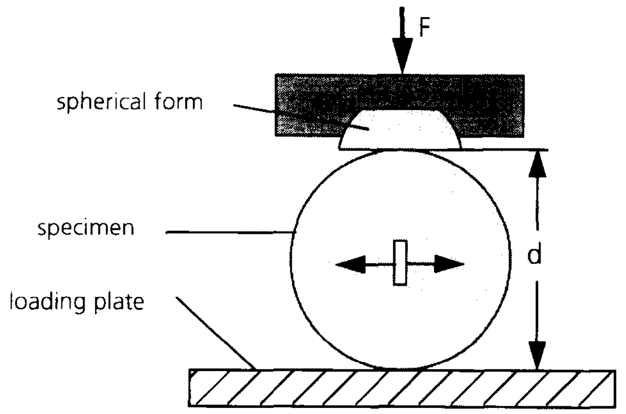

Fig. 6. Brazilian disc test; the stress state is tensile along the diameter joining the loading points with a maximum in the middle.

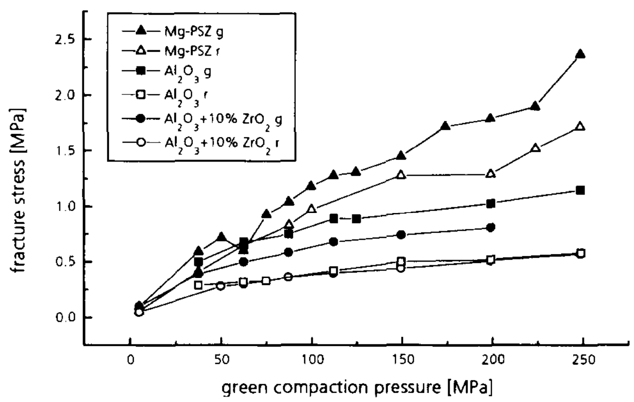

Fig. 7. Fracture stress versus compaction pressure for different powders.

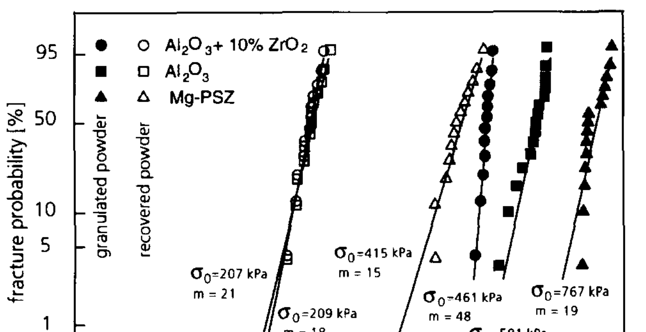

Fig. 8. Distribution of fracture stress obtained in Brazilian disc test for green parts.

4 Conclusions

It is shown that numerical simulation in combination with strength tests for green compacts can be useful tools for cost reduction and quality assurance concerning shaping and machining of ceramic components. Further developments of the simulation models are necessary. The experimental determination of the tensile strength by diametral compression in the so-called Brazilian disc test for ceramic green compacts was successfully performed, but an examination of the validity of strength tests as used here is necessary. A comparison with the fracture stress obtained by a conventional tensile testing is in progress.

The investigation of green and sintered parts made of recovered powders has shown that there is a potential for cost reduction by the repeated use of ceramic powders. Further investigations of the purity and the quality of the sintered ceramic parts are necessary.

Acknowledgements

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Table 1. Results of Brazilian disc test for sintered specimens

Material	*Al_2O_3+10%ZrO_2 granulated powder*	*Al_2O_3+10%ZrO_2 recovered powder*	*Al_2O_3 granulated powder*	*Al_2O_3 recovered powder*	*Mg-PSZ granulated powder*	*Mg-PSZ recovered powder*
σ_0 (MPa)	81	145	72	109	168	182
m	6·5	11·7	5·3	9·3	19·9	18·1

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