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# Friction Measurements during Dry Compaction of Silicon Carbide

U. Klemm,[a] D. Sobek,[a] B. Schöne[a] & J. Stockmann[b]

[a]Technische Universität Dresden, Institut für Werkstoffwissenschaft, Mommsenstr. 13, 01062 Dresden, Germany
[b]Fraunhofer - Institut für Keramische Technologien und Sinterwerkstoffe, Winterbergstr. 28, 01277 Dresden, Germany

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## Abstract

*One of the most important problems in uniaxial die compaction of ceramics is the selection of suitable organic additives which have to ensure the production of defect-free homogeneous green bodies with high density and sufficient strength. In most cases the assessment of the efficacy of additives with respect to a reduction of friction occurs in an empirical way. For this reason an instrumented compacting tool was constructed which renders it possible to measure the friction conditions exactly.*

*The compaction behaviour of different granulates for the production of pressureless sintered SiC ceramics was characterized on the basis of an evaluation system consisting of measured frictional parameters, compaction energies, elastic redeformations, and stress distributions. Variations of the powder type are discussed as well as the influence of addition of different organic materials. The choice of the die material proved to be of fundamental importance for friction conditions during compaction of powders with a very fine particle size. © 1996 Elsevier Science Limited.*

*Die Auswahl geeigneter organischer Additive zur Herstellung fehlerfreier Grünlinge mit hoher und möglichst gleichmäßig verteilter Dichte sowie für die Handhabung und Bearbeitung ausreichender Festigkeit stellt eines der wesentlichsten Probleme bei der Verdichtung keramischer Materialien durch Trockenpressen in Matrizen dar. Die reibungsvermindernde Wirkung von Additiven wird überwiegend auf empirischer Basis charakterisiert. Aus diesem Grund wurde ein instrumentiertes Preßwerkzeug entwickelt, das eine exakte Messung physikalischer Kenngrößen ermöglicht.*

*Das zur Charakterisierung des Verdichtungsverhaltens verschiedener SiC - Granulate benutzte System schließt reibspezifische Parameter, Verdichtungsenergien, die elastische Rückdehnung sowie Spannungsverteilungen ein. Der Einfluß der Pulverqualität wird ebenso diskutiert wie die Veränderung des Preßverhaltens durch organische Additive. Für die Verdichtung von Pulvern mit geringer Teilchengröße scheint die Wahl des Matrizenmaterials von entscheidender Bedeutung zu sein.*

## 1 Introduction

Because of the general non-plasticity of the materials and the occurrence of different friction mechanisms, organic additives such as binders and lubricants are necessary components in the compaction of technical ceramics by uniaxial pressing. They have to ensure an excellent compressibility, i.e. a high density with a pressure as low as possible, and an adequate compactibility, i.e. production of a defect-free green body which can be handled or in special cases machined without problems. Commonly the selection of the organic additives occurs on the basis of empirically obtained knowledge and the measured parameters are limited to the properties of the green compact. But for a correct assessment of the efficacy of a binder or a lubricant a device is desirable to determine exactly the amount of friction by measuring forces respectively stresses during compaction, and from these deriving physical parameters such as friction coefficients. The purpose of the present study was to demonstrate the possibilities of such an instrumented compacting tool using as example an examination of the influence of different organic additives on the friction conditions during compaction of a SiC granulate.

## 2 Experimental Procedure

The aim of granulate production and characterization is the production of a compact with optimized properties. But in view of the variety of variables which influence the compacting process from the groups of material parameters, the broad field of organic additives and the press technology itself[1] it seems to be impossible, even in approximation, to predict the result of the pressing process in terms of density, strength, homogeneity and macroscopic integrity of the compact from measurements of a sum of individual properties and influences. A special difficulty exists in the fact that not all particle or granulate properties can be quantified with respect to the final results of pressing, especially if they change under the influence of the pressure. For this reason some effort is concentrated into measuring during all stages of the pressing process[2-4] immediately.

For the investigation of the relationships between basic material properties, densification technique, and organic additives a computerized compacting tool according to Fig. 1 was constructed. Within the cycle compaction–pressure holding–pressure removal and ejection independent online measurements of forces, stresses and displacements are possible. From typical compaction graphs such as force–time- and force–way-graph[5] a great variety of parameters can be derived which can be used for the assessment of the compaction process. These include

- Frictional parameters:
  Wall friction coefficient, powder friction coefficient, friction force, coefficient of force transmission, ejection force, coefficient of radial stress.
- Compaction energy and its individual parts:
  Total compaction energy, energy of elastic redeformation, friction energy, real compaction energy.
- Geometrical parameters:
  Density of the compact under pressure using continuous recording of the displacement of the upper punch, bulk elastic redeformation of the green compact and its parts inside the die, respectively, at and after ejection from the different length of the compact.
- Distribution of compressive stresses, shear stresses and densities according to a modified model of Thompson.[6]

Beyond these quantifiable parameters some other important observations such as the appearance of stick-and-slip mechanisms, residual forces and stresses after unloading, dependence of the efficacy of binders or lubricants on the pressure, mobility of the organics during pressure holding, and the ratio between static and dynamic friction during ejection can be derived.

The experiments were generally carried out on a universal testing machine with 400 kN capacity. For measurements of friction conditions the experiments were performed naturally by single-action pressing in the pressure range up to 300 MPa in each case. The special instrumented dies were made from hard metal or hardened steel both with nearly the same roughness of the inner surface.

Starting material for the investigation was a silicon carbide which was selected as one of four tested powders with a medium grain size of 0·39 μm and a specific surface of 16·1 m$^2$/g. The aqueous slurry of powder, a special resin and selected additives from the groups of polyethylene glycols,

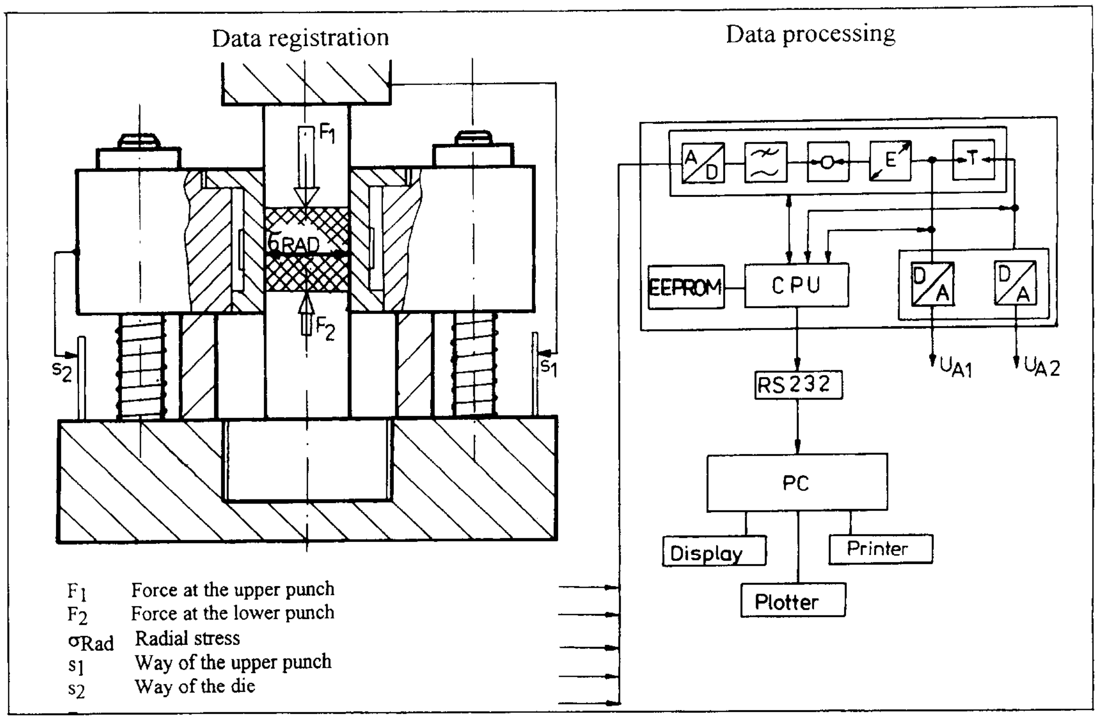


**Fig. 1.** Instrumented compacting tool with integral radial stress measurement.

emulsions of waxes with different viscosities, various polyethylene-based compositions, interfacial active esters, aqueous emulsions of fatty acids and their salts, and polyvinyl alcohol was dried under laboratory conditions by a sieve granulation or in selected cases by a semi-industrial spray granulation process. The granules were dried to an equilibrium moisture content and subsequently examined and stored under constant climatic conditions.

## 3 Results and Discussion

Typical results of compaction experiments with different organic additives are summarized in Table 1. It can clearly be seen that each additive which was selected as representative for a larger group may affect the pressing of the granulated material significantly. In all cases the density is naturally raised by additives. In this the addition of a special ester wax and a polyethylene glycol with a molecular mass of 10000 show the largest effect. In contrast, granulates with polyvinyl alcohol need a higher pressure to reach the same density. The strength of the green compacts is changed in different way but in all cases according to the magnitude of the absolute amount of the compaction energy. This connection is also demonstrated in Fig. 2 for polyethylene glycols with different molecular masses. With increasing molecular mass of the additive a higher amount of compaction energy is consumed followed by a rising diametral compressive strength. In a comparison of the properties of green compacts produced for instance from granulates with addition of an emulsion of a fatty acid or a polyethylene-based composition there seem to be only negligible differences. But the measured frictional parameters and typical compaction graphs show remarkable differences in the compaction behaviour. An example is given in Fig. 3 for the course of the lower punch force during loading and unloading. While the compaction curve of granulates with addition of an emulsion of a fatty acid takes a normal course without inhomogeneities, the granulate with addition of a polyethylene-based composition compacts with some variations with the character of a typical stick-slip-mechanism. Such a mechanism can occur in very different appearances, for example a stick-slip-process with a high frequency and a low amplitude is in the same way possible as in the combination of high amplitude and low frequency. A stick-slip-mechanism definately gives rise to a high die wear and especially causes the appearance during the ejection stage of a deterioration of the surface quality of the green compact. Such mechanisms require a very

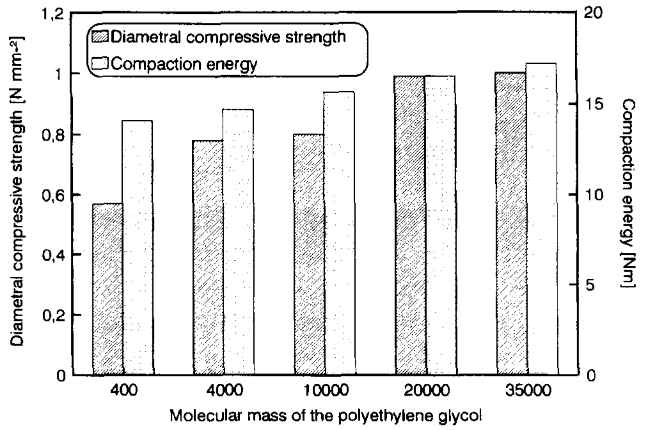


**Fig. 2.** The effect of different molecular masses of polyethylene glycols on the compaction energies and strength of the green compact.

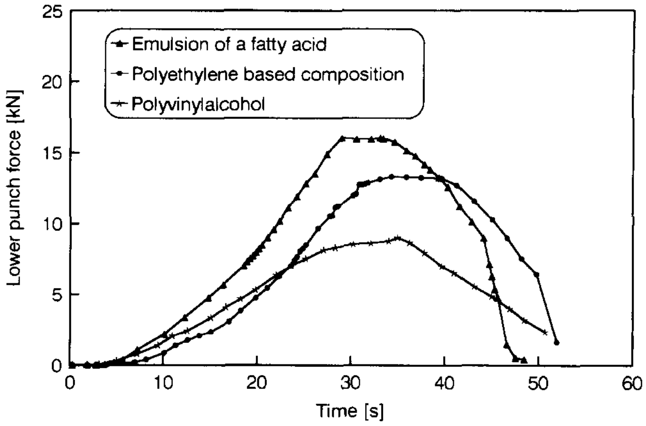


**Fig. 3.** Typical course of the lower punch force during loading and unloading.

**Table 1.** Typical compaction results for SiC granulate with different additives

| | | *Without addition* | *Ester wax* | *Polyethylene glycol* | *Emulsion of a fatty acid* | *Polyvinyl alcohol* | *Polyethylene-based composition* |
|---|---|---|---|---|---|---|---|
| Green density | (g/cm³) | 2·26 | 2·46 | 2·45 | 2·40 | 2·31 | 2·42 |
| Diametral strength | (N/mm²) | 1·49 | 0·90 | 0·80 | 0·77 | 2·68 | 0·56 |
| Force transmission | (%) | 51 | 62 | 62 | 76 | 41 | 63 |
| Wall friction coefficient | | 0·334 | 0·248 | 0·252 | 0·140 | 0·426 | 0·243 |
| Powder friction coefficient | | 0·411 | 0·405 | 0·402 | 0·399 | 0·426 | 0·420 |
| Ejection force | (kN) | 3·4 | 2·6 | 2·4 | 0·8 | 3·2 | 1·9 |
| Compacting energy | (N m) | 21·9 | 14·9 | 15·6 | 17·7 | 33·9 | 15·0 |
| Part of inner redeformation | (%) | 89 | 90 | 87 | 100 | 85 | 86 |

thorough analysis of all measured parameters because they can lead to substantial falsifications of all results.

A comparison of the influence on friction coefficients of the organics reveals the dominant character of wall friction processes for the selected geometry. The lowest wall friction coefficients were obtained by addition of specially emulsified fatty acids; the type from Table 1 showed the best results of four types examined. An addition of polyvinyl alcohol results in a very high wall friction coefficient which is even higher than for an additive-free granulate.

The absolute amount of the axial redeformation and the distribution of individual parts, inside the die or after ejection, is an important criterion for the development of macroscopic or end-capping defects. For granulates with added fatty acid emulsions this redeformation takes place at 100% already inside the die during the unloading stage. For bad lubrication conditions very high residual forces are left on the lower punch, respectively, large residual stresses remain on the die wall (Fig. 4). Despite the essential higher force at the lower punch at the maximum pressure the residual force after unloading for a granulate with an emulsion of a fatty acid is much lower compared to the materials with a polyvinyl alcohol or without any addition. This is followed by the lowest ejection forces and the already mentioned complete elastic redeformation inside the die. In extreme cases the share of elastic redeformation at or after ejection may amount to 50% of the total. The consequences for the quality of the green compact with respect to the macroscopic integrity do not need to be discussed.

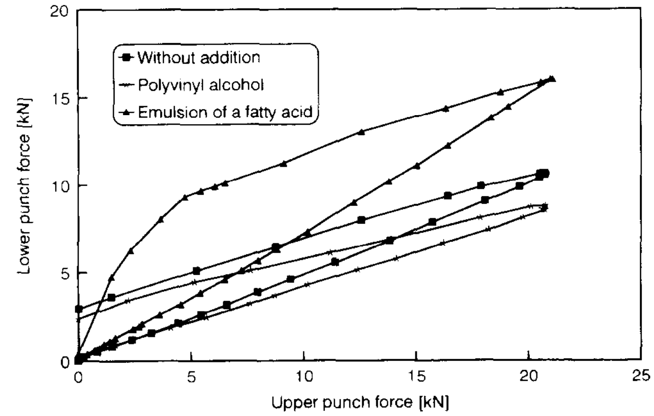
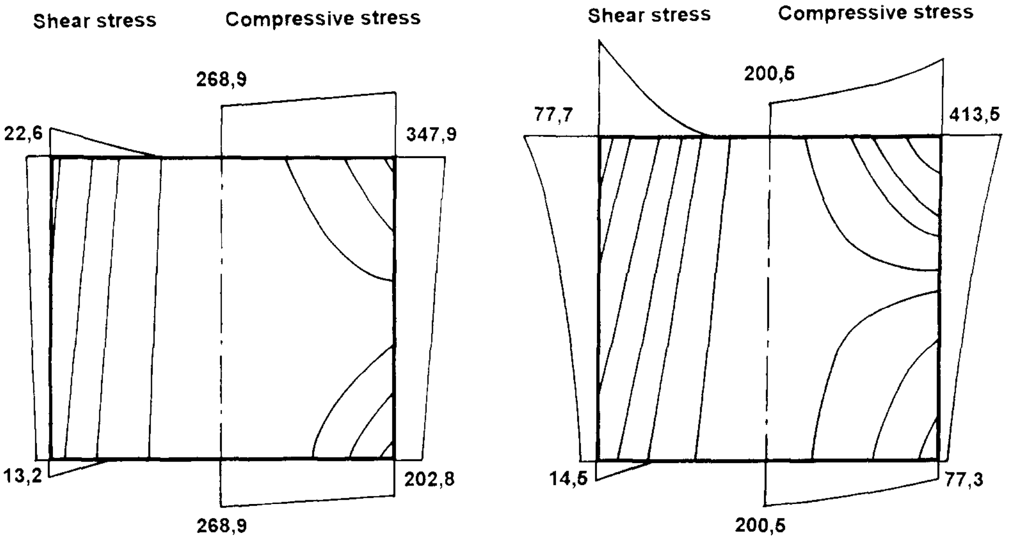


**Fig. 4.** Force transmission during loading and unloading for granulates with different additives.

A survey of the distribution of compressive and shear stresses in the green compact can be calculated from the modified model by Thompson.[6] Examples for the extreme cases of additions of the emulsion of the fatty acid and polyvinyl alcohol are given in Fig. 5. It can clearly be seen that the differences in compressive stresses in axial and radial directions in a green compact with polyvinyl alcohol are much larger compared to the lubricant addition. Subsequently, a more inhomogeneous distribution of the green density combined with a very uneven shrinkage during sintering will follow. In most cases the absolute amount of maximal shear stresses will be more important for the macroscopic integrity of the green compact, especially high shear stresses give rise to the development of typical end-capping defects. In green compacts with polyvinyl alcohol this danger is compensated by the outstanding binding effect of this additive in combination with the added resin.

It is well-known that compaction in hard metal dies should be preferred because it ensures higher



**Green compact with addition of a fatty acid** **Green compact with addition of polyvinyl alcohol**

**Fig. 5.** Distribution of calculated compressive and shear stresses for green compacts with addition of a fatty acid or polyvinyl alcohol.

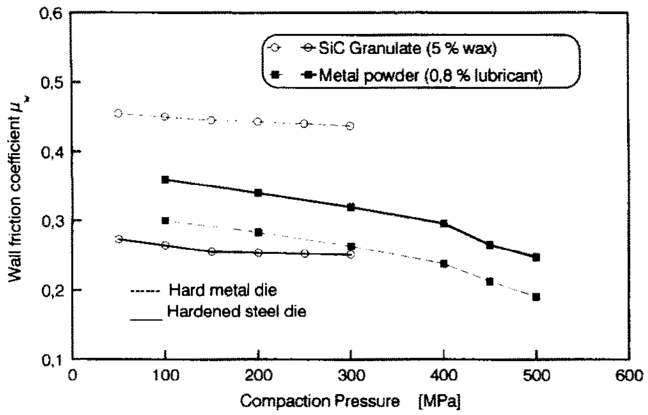


**Fig. 6.** Variation of the wall friction coefficient at compacting with different die materials.

wear resistance and more favourable friction conditions in comparison to other materials. For the compaction of metal powders this assertion has been proved to be correct (Fig. 6). But the experiments with different dies and the SiC granulates showed some unexpected results. All parameters which are connected in any way with wall friction processes — besides the wall friction coefficient, the force transmission to the lower punch, the ejection forces, the friction energy and the residual forces — have changed in an unfavourable direction if compared to measurements with a hardened steel die. Because the roughness of the dies was in both cases nearly the same it cannot be the reason for such a different friction behaviour. Moreover, these differences are not restricted to a selected additive, they also have been found with a granulate free of additives and only a lubrication of the die wall with various lubricants, for example pressing oils or solutions of stearic acid in a high concentration. These unfavourable friction conditions, connected with large differences of the compressive strains in axial and radial directions as well as high shear stresses, caused a high percentage of damaged green bodies compacted in the hard metal die.

## 4 Conclusions

The results of this study showed that it is not sufficient to characterize the complicated process of compacting very fine ceramic powders in a die only on the basis of the properties of the green body. An evaluation system that includes also frictional parameters, compaction energies, measured elastic redeformations, and stress distributions makes the selection of proper additives easier and points out ways for the improvement of the compaction, for instance by changing the die material.

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