MINERALOGIA POLONICA
Vol. 11, No 2 - 1980

Franciszek STERMA[x]

CHANGES IN THE HABIT OF PRESSURE SINTERED CORUNDUM GRAINS

UKD 549.517.1:548.54:666.345:539.89] :666.764.32

A b s t r a c t. Pressure sintering becomes a common technique in the manufacture of products from high-melting materials. This paper discusses changes in the grain habit, observed during pressure sintering of Al_2O_3 powder. It has been found that these changes have a different nature than those implied by the mathematical model of sintering of spheroidal grains.

INTRODUCTION

The general theory of sintering has been the object of extensive studies, the results of which have been presented in many papers /Geguzin 1973, Pampuch 1971, Samsonov /ed./ 1974/. Fewer publications, on the other hand, deal with a new sintering technology, i.e. pressure sintering /Bukat, Rutkowski 1974; Samsonov, Kovalczenko 1969; Uskokowicz *et al.* 1974/.

The majority of research works rests on the assumption that the grains being sintered are spheroidal in shape, whereas in actual powders this form of grains is very uncommon. The actual process of sintering differs as a rule substantially from the mathematical model of sintering of spheroidal grains, both in respect of the motion of matter and the structures obtained. The ability of obtaining materials with required structures is tantamount to the production of materials with required functional properties /Jakovlev, Sterma 1977/. Some interesting changes in the grain habit have been observed in the process of pressure sintering of Al_2O_3 powder. Because of its mechanical strength,

[x]Technical University, ul. Racławicka 15-17, 75-620 Koszalin.

thermal and chemical resistance, and electrical properties /Gibas 1970/, Al_2O_3 is a material of particular interest for modern technology.

Although there are several publications dealing with the structure of pressure sintered Al_2O_3 /Spriggs, Atteraas 1968; Vasilos, Spriggs 1963/, none of them gives a full description of the changes in the grain habit occurring in the process of compaction. The aim of the present studies was to investigate and describe the complete evolution of the shape of grains from the moment the powder was introduced into the mould till the sintered product was obtained.

EXPERIMENTAL

Al_2O_3 of an average grain size 0.3 μm, purity above 99.7% and the α phase content more than 95% was subjected to sintering. To expedite the process, 2 wt.% of AlF_3 was added in the form of acicular grains. The two components were mixed in a ball mill in the presence of water. The powder after drying and homogenizing sieving is shown on Phot. 1.

Technological tests were carried out in a SGP-1 pressure sintering apparatus operated at temperatures up to 1673 K /1400°C/, in which the unidirectional pressure applied could amount to 300 KN /about 30 t/ /Sterma 1975/. Sintering was carried out in graphite moulds, wherefore the pressure did not exceed 30.0 MN/m^2 /about 300 Kg/cm^2/. Al_2O_3 was poured into a graphite mould and cold pressed at a pressure of 15 MN/m^2 /about 150 Kg/cm^2/. The fracture of cold pressed powder is shown on Phot. 2. Then the mould was placed in the SGP-1 apparatus and heated at an average rate of 10 K/min at constant pressure.

Technological parameters of

Sample number	Sample composition	Cold pressing pressure /MN/m^2/	Pressure during sintering /MN/m^2/	Maximum sintering temperature /K/
1	98% - Al_2O_3 2% - AlF_3	15	16	1423
2	as above	15	16	1473
3	" "	15	22	1473
4	" "	15	22	1523
5	" "	15	22	1523
6	" "	15	22	1523

The technological parameters of sample preparation are given in Table 1. The choice of the parameters /temperature, pressure, sintering time/ recommended to obtain a material with the required density is discussed by Sterma and Jakovlev /1977/. The surface and internal structure of samples was investigated with a JEOL JSM-S1 scanning electron microscope. The samples were in the form of disks 24 mm in diameter and 5 mm in thickness. To study the internal structure, each sample was split. Before microscopic investigations, the samples were not subjected to any additional treatment.

The surface and internal structure of samples of various density, obtained using different technological parameters, is shown on Phot. 3 - 10.

RESULTS

The grains of Al_2O_3 powder are in fact aggregates. As mentioned earlier in this paper, grains of a size of 0.3 μm dominate in the Al_2O_3 powder used for investigations. Spheroidal aggregates consist of a few to a dozen or so elementary grains. While grains within an aggregate are relatively closely packed, the aggregates are rather loosely bound to one another. The powder behaves as if these aggregates were elementary grains. In fact, they make up lumps in the shape of spheres, which can be subject to deformation under the influence of external forces. This is clearly visible on Phot. 2 showing the fracture of powder cold pressed at a pressure of 150 MN/m^2. The deformed aggregates form layers and areas that undergo compaction and displacement, depending on

Table 1

samples preparation from Al_2O_3

Heating rate /K/min/	Time of holding at maximum temperature /min/	Cooling rate /K/min/	Relative density /% of theoretical density/	Sample shown on Phot.
10	90	5	69	3
10	30	5	76	4
10	90	5	86.0	5 and 6
10	50	5	97.0	7
10	70	5	98.0	8 and 9
10	90	5	99.3	10

the external forces acting upon them. Despite pressing, the boundaries between the individual deformed aggregates are distinct.

Phot. 3 shows a relatively far advanced stage of sample sintering. Clearly visible are flattened polyhedral lumps with a hexagonal outline. Lumps of similar size are predominant although smaller lumps also appear in lesser amounts. A comparison of Phots. 1 and 3 indicates that the polyhedral lumps are flattened and sintered equivalents of spheroidal aggregates. As appears from Phot. 3, the elementary grains within these aggregates /lumps/ have been sintered, whereas the polyhedral lumps are loosely bound.

It has also been noted that the polyhedra show different orientation. There are areas exhibiting ordered orientation /Phot. 4/ and ones with random orientation /Phot. 3/. These different modes of orientation are presumably due to the uneven compression of the sample, which is always the case with unidirectional pressing.

Polyhedral grains are sintered only later in the process of pressure sintering. Phot. 5 shows the surface, and Phot. 6 the fracture of a denser, better sintered sample. It is evident that the grains are better fitted, adhering to one another with whole surfaces. It is worth noting that the grain growth does not take place; on the contrary, the grains slightly diminish, which is indicative of further /internal/ sintering and contraction of lumps. Phot. 7 shows a still better sintered sample in which the lumps adhere closely to one another.

Worth noting is Phot. 5 showing the sample surface adjoining the end face of the mould stopper. Clearly visible is the flat orientation of lumps.

The surface of a highly sintered sample is shown on Phot. 8, and its fracture on Phot. 9. It can be seen that only in places the lumps do not adhere to one another and pores are present. The adherence of lumps at full compaction is shown on Phot. 10. It is a sample obtained at 1523 K, in which the process of diffusion has been very intense. Striving towards a decrease in free energy, the lumps change from flat into isometric bodies.

DISCUSSION

The present studies have revealed that the process of pressure sintering of actual, non-spheroidal, grains is different from the sintering of spheroidal grains. Initially grain aggregates are sintered into polyhedral lumps which, in turn, become sintered in a later stage of the process.

Sintered polyhedral lumps determine the final structure, and therefore the properties of the resulting material. The size of aggregates, and thereby the size of polyhedral lumps, depends both on the size of elementary /initial/ grains and the parameters of the processes of homogenization and sintering.

The results obtained contribute to the explanation of the evolution of the grain habit during pressure sintering of non-spheroidal grains. They also show new possibilities of controlling the structure of materials resulting from the sintering of powders.

REFERENCES

BUKAT A., RUTKOWSKI W., 1974 - Teoretyczne podstawy procesów spiekania. Wyd. Śląsk.

GEGUZIN J. E., 1973 - Physik des Sinterns. VEB DVG.

GIBAS T., 1970 - Korund i jego zastosowanie w technice. Wyd. Śląsk.

JAKOVLEV B., STERMA F., 1977 - Influenza dei parametri di pressatura su alcune proprieta di materiali ceramici di alumina pressati a caldo. *Ceramurgia Anno VII, no 3.*

PAMPUCH R., 1971 - Podstawy inżynierii materiałów ceramicznych. PWN.

[SAMSONOW G. W., KOWALCZENKO H. S.] Самсонов Г.В., Ковальченко Х.С., 1969: Горьячие прессование. Г.И.СССР.

[SAMSONOW G. W. ed.] Самсонов Г.В., 1974: Теория и технология спекания. Изд. Научн. Думка. Киев.

SPRIGGS R. H., ATTERAAS L., 1968 - Ceramic microstructures. New York.

STERMA F., 1975 - Urządzenie do spiekania pod ciśnieniem Al_2O_3. *Mat. El. No. 4/12/.*

STERMA F., JAKOWLEW B., 1977 - Badania procesu zagęszczania i własności spiekanej pod ciśnieniem ceramiki korundowej Al_2O_3. *Mat. El. No 1 /17/.*

[USKOKOWICZ D. P., SAMSONOW G. W., RISTICZ M. M.] Ускокович Д.П., Самсонов Г.В., Ристич М.М., 1974: Активованное спекание. Ниш.

VASILOS T., SPRIGGS R. M., 1963 - Pressure sintering, mechanisms and microstructures of alumina and magnesia. Ibid 46/10.

Franciszek STERMA

EWOLUCJA POKROJU ZIARN KORUNDU Al_2O_3 SPIEKANYCH POD CIŚNIENIEM

S t r e s z c z e n i e

Przy wytwarzaniu wyrobów z wysokotopliwych materiałów coraz częściej stosowana jest technika spiekania pod ciśnieniem. W artykule niniejszym opisano zmiany pokroju ziarn, zaobserwowane w procesie spiekania proszku Al_2O_3, pod ciśnieniem. Stwierdzono inny typ ewolucji pokroju ziarn, niż to wynika z matematycznego modelu spiekania ziarn kulistych.

OBJAŚNIENIE FOTOGRAFII

Fot. 1. Proszek Al_2O_3 stosowany w badaniach. SEM, x 3000
Fot. 2. Przełam próbki z proszku Al_2O_3 sprasowanego na zimno. SEM, x 3000
Fot. 3. Przełam próbki 1. SEM, x 7000
Fot. 4. Przełam próbki 2. SEM, x 7000
Fot. 5. Powierzchnia próbki 3. SEM, x 2000
Fot. 6. Przełam próbki 3. SEM, x 2000
Fot. 7. Przełam próbki 4. SEM, x 3000
Fot. 8. Powierzchnia próbki 5. SEM, x 2000
Fot. 9. Przełam próbki 5. SEM, x 2000
Fot. 10. Przełam próbki 6. SEM, x 10 000

Францишек СТЭРМА

ЭВОЛЮЦИЯ ГАБИТУСА ЗЕРЕН КОРУНДА, СПЕКАЕМЫХ ПОД ДАВЛЕНИЕМ

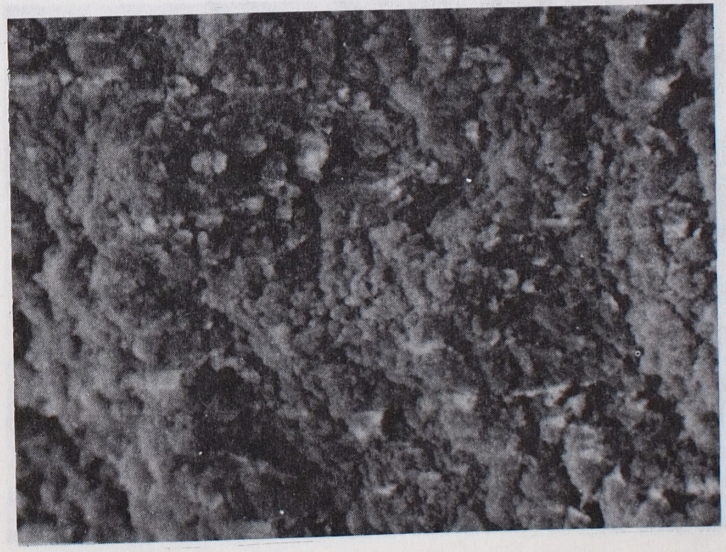
Р е з ю м е

В производстве продуктов из тугоплавких материалов все чаще применяется технику спекания под давлением. В настоящей статье описаны изменения габитуса зерен, наблюдаемые в процессе спекания порошка Al_2O_3 под давлением. Констатирован иной тип эволюции габитуса, чем это следовало бы из математической модели спекания шаровидных зерен.

ОБЪЯСНЕНИЯ К ФОТОГРАФИЯМ

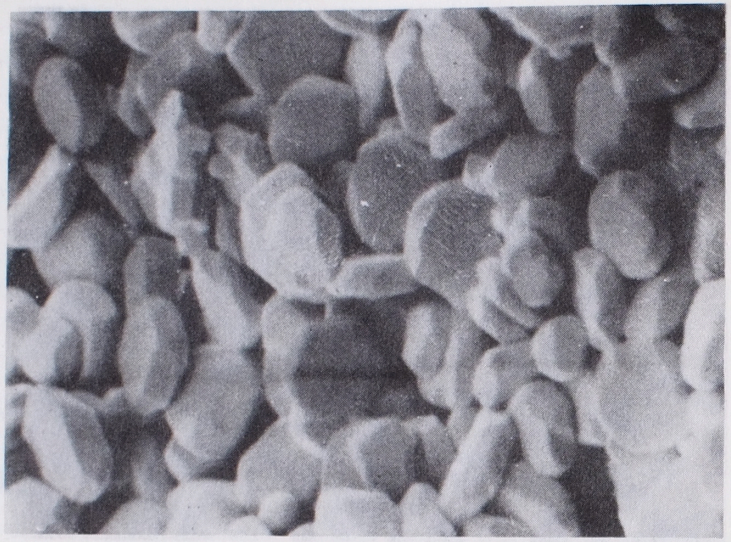
Фото 1. Применяемый в исследованиях порошок Al_2O_3. МЭВ, х 3000
Фото 2. Излом образца из порошка Al_2O_3, спрессированного в холодном состоянии. МЭВ, х 3000
Фото 3. Излом образца 1. МЭВ, х 7000
Фото 4. Излом образца 2. МЭВ, х 7000
Фото 5. Поверхность образца 3. МЭВ, х 2000
Фото 6. Излом образца 3. МЭВ, х 2000
Фото 7. Излом образца 4. МЭВ, х 3000
Фото 8. Поверхность образца 5. МЭВ, х 2000
Фото 9. Излом образца 5. МЭВ, х 2000
Фото 10. Излом образца 6. МЭВ, х 10 000

Phot. 1. Al_2O_3 powder used in studies. SEM, x 3000

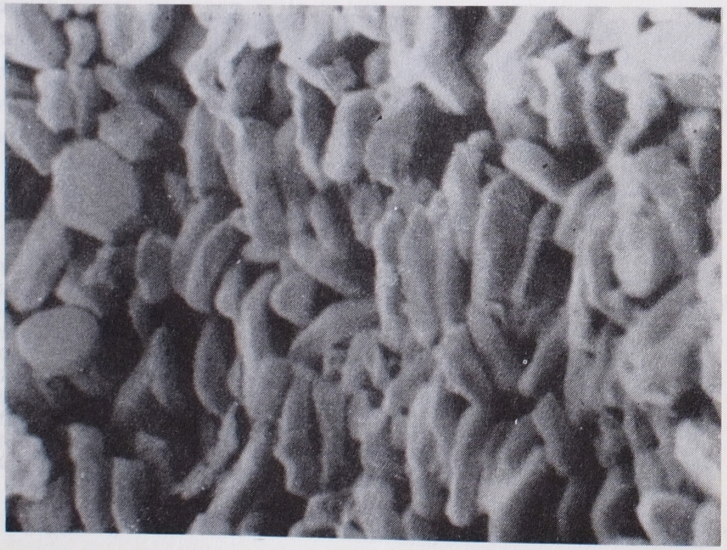


Phot. 2. Fracture of Al_2O_3 powder cold pressed. SEM, x 3000

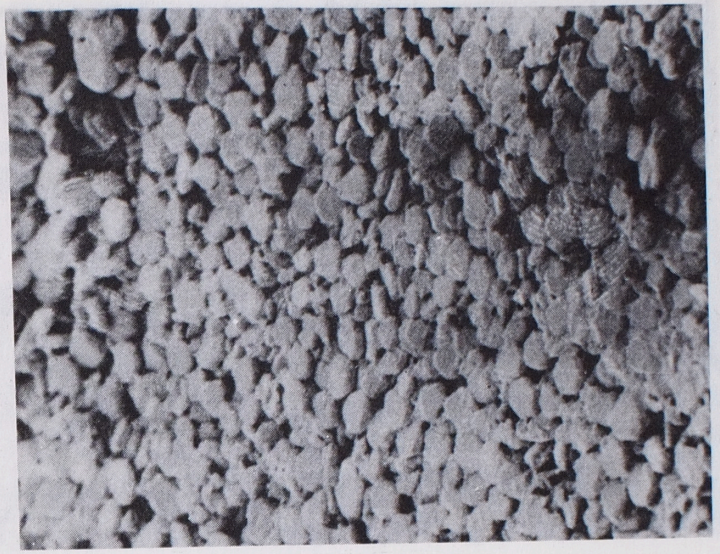
Franciszek STERMA - Changes in the habit of pressure sintered corundum grains



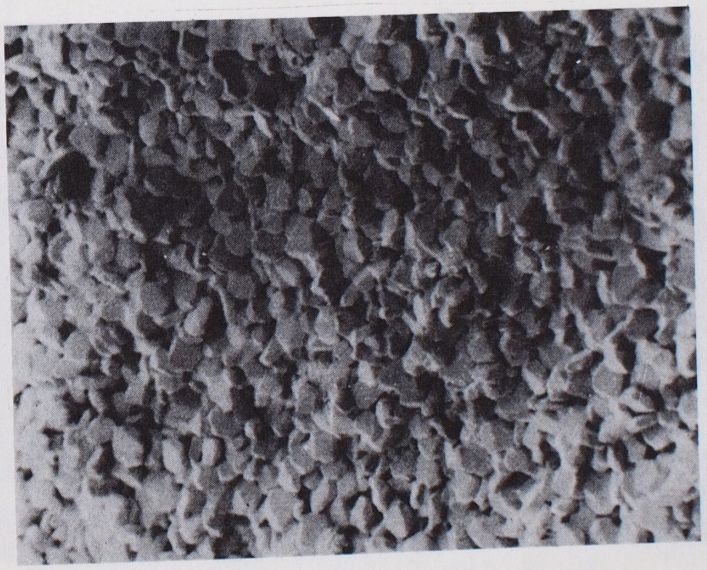
Phot. 3. Fracture of sample 1. SEM, x 7000



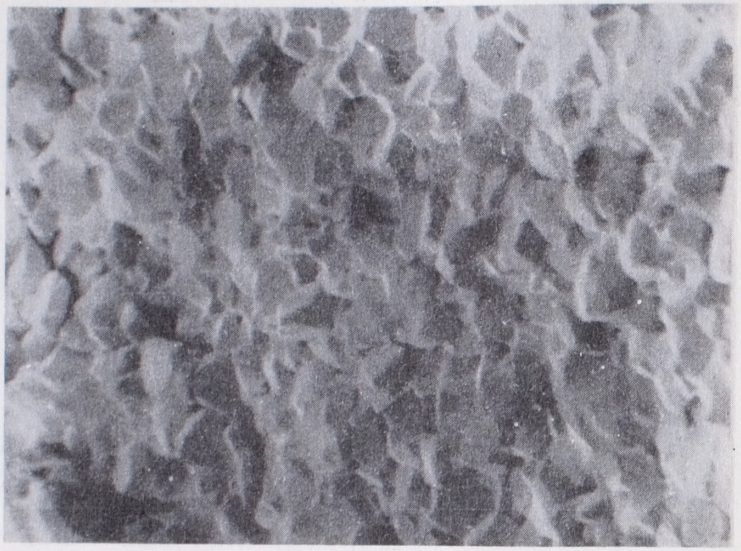
Phot. 4. Fracture of sample 2. SEM, x 7000



Phot. 5. Surface of sample 3. SEM, x 2000

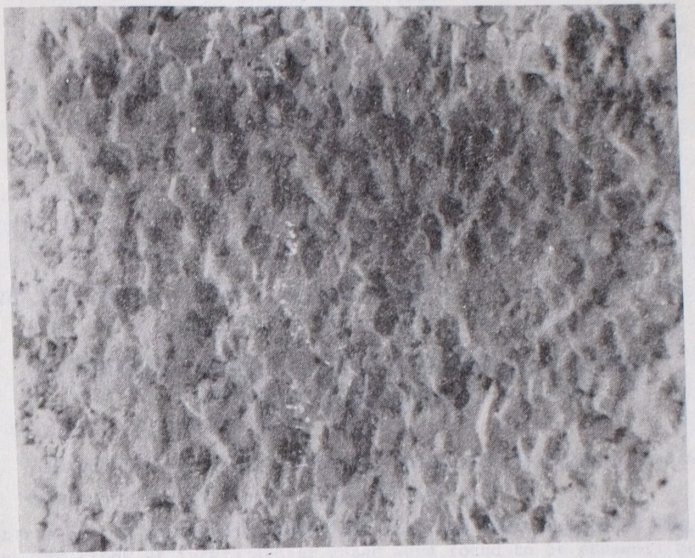


Phot. 6. Fracture of sample 3. SEM, x 2000

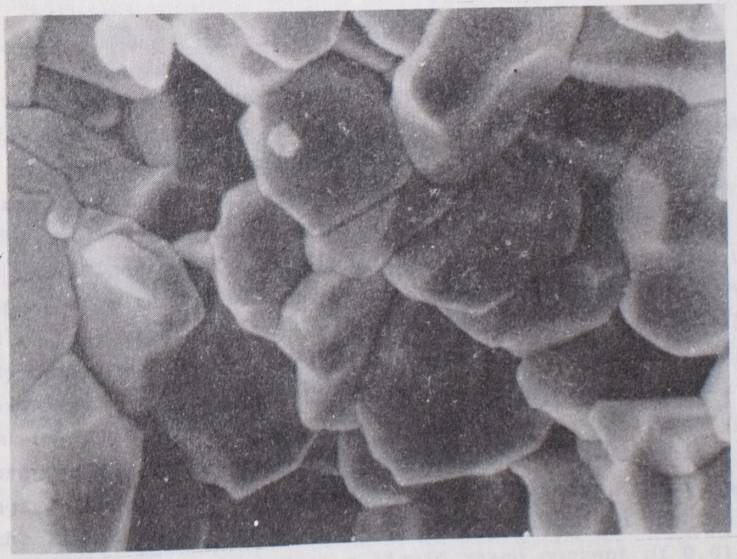


Phot. 7. Fracture of sample 4. SEM, x 3000

Phot. 8. Surface of sample 5. SEM, x 2000



Phot. 9. Fracture of sample 5. SEM, x 2000



Phot. 10. Fracture of sample 6. SEM, x 10 000