Mariusz A.WÓJCIK, Tomasz GAJDA*, Julian PLEWA, Horst ALTENBURG,
Ansgar LUTTERMANN**, Viliam FIGUSCH, Miroslav HAVIAR***

EFFECT OF SELECTED PARAMETERS ON GRINDING PROCESS OF ALUMINA IN THE ROTARY-VIBRATION MILL

The effect of selected parameters on grinding process of two types of alumina in the rotary-vibration mill (RVM) was presented. The effect of mutual relation between filling, media and suspension ratios as well as the size of grinding media, time and frequencies of chamber mill on grinding of alumina was investigated and the kinetics of grinding of this materials was shown. Investigations revealed that it is possible to obtain fine alumina powders of alumina characterizing with d_{50} approximately 3 µm and d_{90} close to 10 µm during about 60 minutes of grinding in RVM. Also, the effect of mentioned above parameters on physical properties of moulders prepared from ground alumina powders was shown. The relations between grinding time and green and fire density and shrinkage was also presented and discussed. The results revealed that longer grinding time inreased both green and fire density and shrinkage of moulders.

INTRODUCTION

All fabrication processes commonly used for the production of high alumina ceramics usually consists of blending of raw materials and additives, grinding and dispersing, spray-drying, pressing, sintering, hard machining and final inspection. The grinding and dispersing of a ceramic mass consisting with high quality low soda alumina and flux additives belong to very important unit operation in the technology of manufacturing of ceramics insulators for sparking plugs. Effective grinding provides the powder with the required small grain size distribution and with the necessary developed surface activity for further sintering process. The proper grain size distribution of alumina ceramic mass (ACM), usually monodispersed, makes an essential influence on forming and sintering of moulders at adequate temperatures (Bruch 1967, Frey, Halloran 1984, Wójcik et al. 1996). In particular, it depends on the proper size and shape of primary crystal of corundum usually agglomerated to one

————

   * University of Mining & Metallurgy, Faculty of Ceramics & Materials Engineering,
Al. Mickiewicza 30, A-3, 30-059 Cracow, Poland.
   ** Fachhochschule Munster, Fachbereich Chemieingenieurwesen Stegerwaldstrasse 39, D-48565 Steinfurt, Germany.
   *** Academia Istropolitana, Klariska 5, 810-00 Bratyslava, Slovakia.

particle (Guo et al. 1985). A decrease of the size of alumina particle has a favorable effect on the improvement of some chemical and physical parameters such as specific surface area, bulk density and total free surface energy. The improvement of these properties has also a favorable effect on powder compacting process and accelerates sintering. The compactness of powders is a kind of characteristics affected by size, distribution and shape of particles. The compactness of powders is reflected in the green density of the moulder. The green density influences the shrinkage factor which belong to the most important industrial parameters.

The qualification of the ground ACM into the further manufacturing of insulators is done on the basic test of grain size distribution of powders and measurements of the green density of moulders which has the most essential influence on following fire density and shrinkage. The ACM prepared for insulator cannot contain neither too much fine nor too much coarse particles, because such deviations lead to a great number of defective insulators. Therefore, there is a need to make an optimization of grinding process of ACM for the practical applications.

Considering the process of grinding of ACM in rotary mills, there is an interesting problem of the possibility of shortening the grinding time keeping hitherto parameters of grain size distributions as well as investigation of the influence of the quick grinding on grain morphology.

The rotary-vibration mill (RVM) belongs to the vibrating devices with low frequency of vibration (10–16) Hz and rotary-vibrating movement of the chamber. The fundamental investigations reveal that the RVM has great technological potential with considerably larger durability and lower destructiveness to the environment (Sidor 1987). In respect of above, several selected parameters were investigated in grinding of alumina in the RVM and their effect on grinding process was presented in this paper.

EXPERIMENTAL

Material

Two types of alumina (A) and (B) were used in the tests. Both materials were produced in the rotary kilns in two Polish different factories. Alumina A consists of primary crystals shaped like needles or spindles. The lengths of the crystals are about 7 μm and diameters 1 μm at both ends and 2 μm in the middle of the crystals. Alumina B consists of final bean-like crystals; their diameters ranged from 1.5 to 3 μm. Alumina A is a finer material then alumina B. The d_{50} and d_{90} of agglomerates of A powder were equal 67 and 102 μm while of B powder 188 and 243 μm, respectively.

The aim of the grinding tests

The chief aim in presented investigations was to find the optimal grinding parameters. The next one was to determine the effect of grinding time of two types of

alumina in the rotary-vibration mill on physical properties of moulders such as green and fire density and shrinkage. To reach this aim it was needed to prepare alumina powders characterized with d_{50} approximately 3 μm and d_{90} close to 10 μm by grinding in the RVM. The grinding parameters are: size of chambers (0.5 and 1.0 l), frequencies (11 and 14 Hz), grinding media (balls 10 and 20 mm in diameter), times of grinding, changing amounts of grinding media, alumina and water.

Characteristic of RVM

The technological parameters of RVM LAMOW D1/0.5 used in all grinding tests are as follows: power 0.55 kW, frequencies of 11 and 14 Hz, plastic chamber of 0.5 l and 1.0 l, grinding media: alumina balls of 10 and 20 mm in diameter, feed: suspension of alumina in water.

The grinding in RVM consists of set of the composed motion of the mill chamber filled with balls and feed. The motion is collected with the rotary and vibration movements. The amplitude of the vibration movement is perpendicular to the axis of a chamber rotation. It gives quasilinear amplitude of a vibration. The schema of a basic work of RVM, i.e. chamber and charge motion as well as the work of RVM with circular trajectory of vibration is shown in Fig. 1. Particular description of RVM LAMOW-B-5/2 similar to LAMOW-B1/0.5 used in the investigations of grinding of alumina was shown in papers of Sidor and Wójcik (1994).
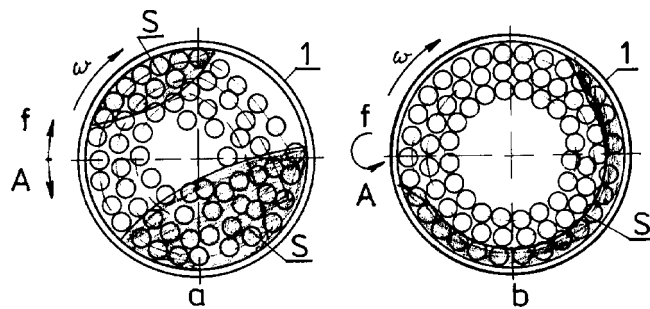

Fig. 1. Principle operation of RVM, a) LAMOW-B-1/0.5 with quasilinear trajectory of vibration,
b) RVM with circular trajectory of vibration:
1− chamber, *f* – frequency, *A* – amplitude, *ω*− angle velocity, *S* – intensive grinding

METHODS OF INVESTIGATIONS

Sample preparation

A particular amount (in grams) of alumina (A) or (B) used for grinding test at specified parameters is called a sample. Every time two chamber of RVM were

prepared using the same ratios and values for all parameters. Chamber 1 was always filled with alumina A (feed A) while chamber 2 with alumina B (feed B). Thus on every grinding two samples were obtained. For one grinding the first ratios were determined and due to those values for mass of grinding media (alumina balls), alumina and distilled water were calculated. Chambers were filled with feeds which were ground according to particular grinding time and amplitude. The ground suspension of alumina and water was spilled into plastic bottles and next balls were dried and weighted to determine the loss of balls using both weight before and after grinding.

To prepare samples for sieve analysis, the bottle containing suspension was vigorously shaken and a small part of the solution was spilled out on a clock glass while the rest was put into the porcelain dish. Both parts of the sample were dried in a laboratory drier. The bigger part was next passed through a 400 μm sieve to destroy agglomerates and was packed in a plastic bag for further tests of green and fire densities and shrinkage. The smaller, dried part was passed through a 60 μm sieve to prepare the powder for the measurement of grain size distribution and d_{50} and d_{90} using Sartorius balance.

Grinding procedures

To optimize the grinding process the best ratios such as: filling ratio (1), media ratio (2) and suspension ratio (3) should be determined in the first part of investigations and then the kinetic of the grinding should be determined in the second one. The following parameters should remain constant during part one: chamber – 0.5 l, ball
– 20 mm, frequency – 11 Hz, grinding time – 20, 40 or 60 min.

$$fr = \frac{V_{feed}}{V_{chamber}} = \frac{V_{alumina} + V_{liquid} + V_{balls}}{V_{chamber}} \tag{1}$$

$$mr = \frac{V_{alumina} + V_{liquid}}{V_{alumina} + V_{liquid} + V_{balls}} \tag{2}$$

$$sr = \frac{V_{alumina}}{V_{alumina} + V_{liquid}} \tag{3}$$

The smaller chamber was chosen to realize a higher number of possible filling ratios since the number of 20 mm balls was to low to realize every wanted ratio and 10 mm balls were not available at primary tests. The smaller frequency was applied because at a higher one better results were expected. The necessary time to reach representative results should be determined at first using ratios from before done tests.

Then only three parameters should change during the first part of the tests: amount of grinding media, amount of alumina, amount of water. Every time the d_{50} of the ground powder should be determined and then a decision should be made about the next changes.

Starting the second part of the investigation, the determination of the kinetic of grinding, the ratios of the samples that shown the best result in part one should be used for further tests. Now amount of: grinding media, alumina and water should remain constant while the before constant parameters should be changed using the following values: ball 10 and 20 mm, frequencies 11 and 14 Hz, chamber 0.5 and 1.0 l and grinding time 20, 40, 60, 80 and 100 min.

Determination of d_{50} and d_{90}

The grain size distribution measurement was carried out only by sedimentation method using Sartorius SEDIGRAPH 4610. The used liquid was a solution of water with small addition of sodium pyrophosphate to reduce free surface energy of the alumina particle to avoid reagglomeration of the grains. Based on calculations done on the measurement paper grain size distribution was established and due to that it was possible to determine d_{50} and d_{90} of the powder.

Moulders tests

The green and fire density and shrinkage were calculated by measurements of the dimensions of moulders and weighing its masses. The starting diameter of moulders was 20 mm. The pressing pressure was constant in all tests.

Results and discussion

The starting values including filling ratio and volume of grinding media, alumina and water were set up basing on previous grinding technology elaborated for an older type of RVM differing with bigger chamber. The aim of experiments was to determine the grinding time for all samples of the part one of the investigations to reach representative results for both powders. The starting values were 50% of filling ratio, 106 ml of volume of grinding media, 38 ml of alumina, 106 ml of water. Other, above mentioned, parameters were constant. Both aluminas were ground for 20, 40 and 60 min.

The first results (not presented in this paper) of the sieve and sedimentation analysis looking on d_{50} and d_{90} showed, although the values decreased with increasing time of grinding, that the change is not high as necessary when the short time are used. As expected the loss of balls increased with increasing grinding time. Looking on these results it was not possible to decide, which time should be applied at further tests. Thus, another practicable parameter had to be found. The residue of the 60 μm sieve analysis was useful to describe the progress of grinding. In view of all facts,

residue, d_{50}, d_{90}, the decision was taken to grind all further samples of part one of the investigations during 60 minutes.

For the next tests both the volume of alumina and water was reduced while the number of balls, respectively amount of grinding media was not changed. Thus the all ratios were changed as follows: filling ratio decreased from 50 to 33.5%, media ratio decreased from 57.6 to 37.5% and suspention ratio increased from 26.4 to 32.5%.

The results of the grain size distribution measurements showed that the changes had a favorable effect on the decrease of d_{50}, d_{90} and R_{60} (means the residue of the 60 μm sieve analysis). D_{50} was 8 μm for alumina A and 7 μm for alumina B. Because of the success of the changes the same receipt was used again. The filling ratio was hold constant at 33.5% while media ratio was again decreased to 23.6% and suspention ratio was increased to 35%. Again better powders are obtained as before. Tne next sample gave d_{50} equal 3 μm.
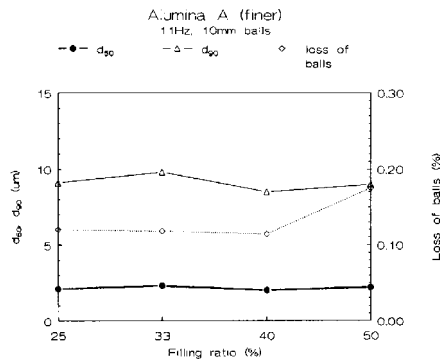

Fig. 2. Influence of filling ratio
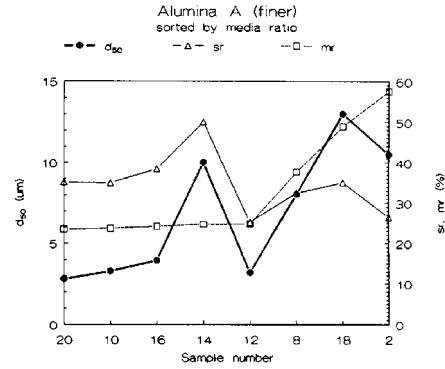on d_{50}, d_{90} and loss of balls

Fig. 3. Influence of media
and suspension ratios on d_{50}

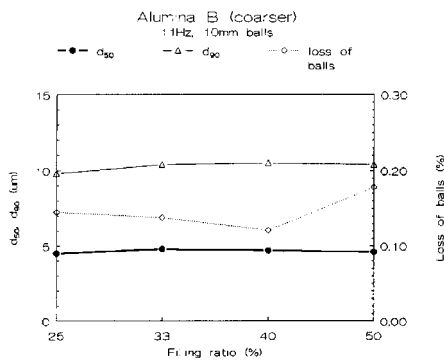
Fig. 4. Influence of filling ratio on d_{50}, d_{90}

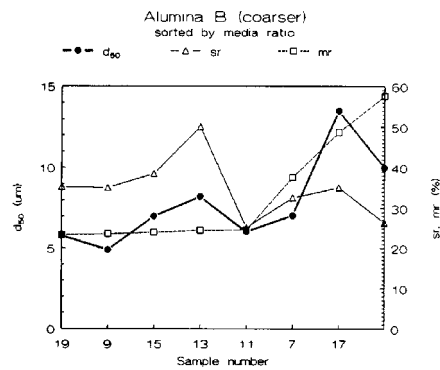
Fig. 5. Influence of media

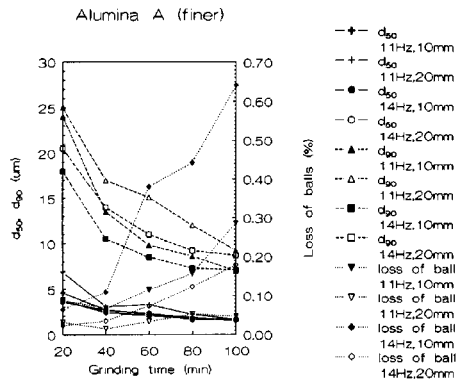and loss of balls   and suspension ratios on d_{50}

Fig. 6. Effect of grinding time on d_{50}, d_{90}
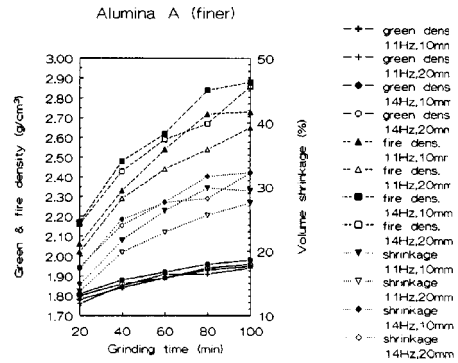and loss of balls

Fig. 7. Effect of grinding time on green and fire
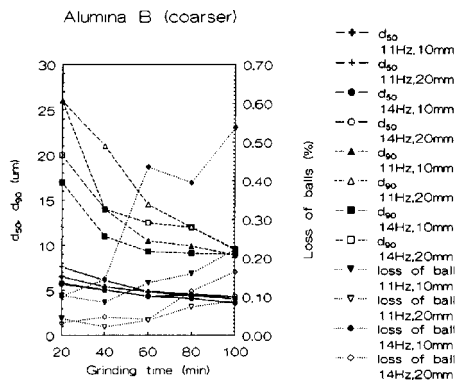density and shrinkage of moulders

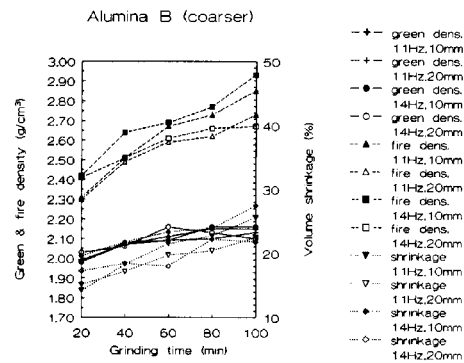Fig. 8. Effect of grinding time on d_{50}, d_{90}
and loss of balls

Fig. 9. Effect of grinding time on green
and fire density and shrinkage of moulders

The next changes had to be chosen. The filling ratio and the media ratio should not be further increased to avoid the low yield of alumina from every test. Thus the only suspension ratio left to be changed. For next samples the filling ratio and the media ratio were hold constant. The suspension ratio varied in a range from 25.2 to 50%. For next samples also the filling ratio was lowered to about 25% and the media ratio increased to about 48.8%. For the suspension ratio the same value, about 35%, was chosen at previous samples. The results showed that a high influence of ratios on

the grain size distribution was received. D_{50} was 3.2 µm for alumina A and 6 µm for alumina B. For the next test, the filling ratio was reduced to about 16.5% while the media and suspension ratio were 23.6 and 35%, respectively. Results showed a very low influence of the filling ratio.

Choice of parameters

Comparing the values of d_{50}, d_{90}, residue and loss of balls of all samples of part one of the investigations, the best results were reached when filling ratio was 33.5%, media ratio was 23.6% and suspension ratio was 35.0%. The yield was also sufficient.

Therefore, the values of those samples were used in the determination of the grinding kinetic.

Grinding kinetic

Four sets of grindings had to be made to elaborate the grinding kinetic. The changeable parameters were ball sets, times of grinding and the frequency. The size of chamber (0.5 l), grinding media, alumina and water were constant during all grindings.

Set I   11 Hz,   bigger balls (20 mm diameter, 36 balls per chamber),
Set II   14 Hz,   bigger balls,
Set III 14 Hz,   smaller balls (10 mm diameter, 273 balls per chamber),
Set IV   11 Hz,   smaller balls,

DISCUSSION

Results revealed that the best ratios between alumina, water and grinding media where achieved for sample 9 and 10 with respect of d_{50}, d_{90}, residue on 60 µµ sieve. For those samples the values for filling ratio where 33.8 and 33.6%, for media ratio 23.6 and 23.7% and for suspension ratio 35% for both, coarser and finer alumina. Influence of filling ratio on d_{50}, d_{90} and loss of balls is presented on Fig. 2 and 3. The changes of filling ratio where realized without changing media and suspension ratio. As it can be seen from this Figs. the influence of filling ratio on these parameters is low. Therefore it can be expected to find a high influence of media and suspension ratios od d_{50}-values. In fact, looking on Figs. 4 and 5 it is possible to observe such relations when one of the ratios is approximately constant. The d_{50} is obviously much more influenced by media ratio then by suspension ratio. The suspension ratio can be a measure of the fluidity of suspension. If the suspension ratio is to high the suspension is like clay and pastes on balls and surfaces of chambers (samples 13, 14 when $s_r = 50\%$). If the ratio is to low, the amount of alumina in the suspension influences the transport of the feed in the process of mechanical grinding. The media ratio can describe the ratio between grinding area and amount of suspension and due to that the thickness of the layer of alumina on the surface of balls. The thickness of

the layers influences the propagation of energy and thus the result of every event of use.

Figs. 6 and 7 show, that the best grain size distribution was reached using 10 mm diameter balls and a frequency of 14 Hz. The best values at these adjustment was reached after 100 minutes of grinding. D_{50} of 1.6 μm was obtained for finer alumina A while d_{50} of 3.5 μm was obtained for coarser alumina B. Also using the bigger balls it was possible to reach comparable results for d_{50}. Using the lower frequency the yielded d_{50} were clear worse. The maximum d_{90} exists at approximately 9 μm for the coarser alumina and about 7 μm for finer one. The hierarchy of the different kinetics, looking on d_{50}, d_{90} values put in order of frequencies and ball diameter starting from the best to worse function is as follows: 1) 14 Hz and 10 mm, 2) 14 Hz and 20 mm, 3) 11 Hz and 10 mm, 4) 11 Hz and 20 mm.

Looking on the same Figs. 6 and 7 the kinetic of the loss of balls is drawn. The highest looses had got using the higher frequency of 14 Hz and smaller balls of 10 mm. Grinding longer then 60 minutes seems to be not profitable in respect of the loss of ball. The energy is spent rather to grind down the balls then grind alumina material. The loss is much less if we also use the higher frequency, but the bigger diameter of ball (20 mm). The results of loss of balls looking on d_{50} and d_{90} stay in opposite order. Loss of balls can be also used as a helpful factor for controlling the progress of alumina grinding in RVM. Thus, a practical conclusion can be drawn from the kinetics of alumina grinding that the optimal time is close to 60 minutes in respects of d_{50} and d_{90} values and loss of balls.

The analysis of results presented on Figs. 8 and 9 show that the green density of alumina A depending on the grinding time varies from 1.78 to 1.96 g/cm^3 while of alumina B from 1.99 to 2.16 g/cm^3. The fire density obtained after sintering at 1650 °C during 1h, varies from 2.06 to 2.88 and from 2.31 to 2.93 g/cm^3, respectively. The volume shrinkage is higher for alumina A then for alumina B and varies from about 15 to about 32 and from about 14 to 27%, respectively. Obtained values of these three parameters characterizing alumina powders are strictly combined with grain size distribution represented with d_{50} and d_{90}. The longer grinding time giving smaller values of d_{50} increased the green and fire densities and shrinkage of moulders for both tested alumina. Figs. show that range of changes of these parameters is higher for finer alumina A then for coarser one. Nevertheless, all considered parameters have higher values for coarser alumina B. Taking into the consideration the best d_{50} values for both alumina obtained as well as for the optimal grinding parameters as: filling ratio (fr), media ratio (mr) and suspension ratio (sr) and frequency (14 Hz) and balls (10 mm) it can be ascertained that obtained values for parameters characterizing moulders as green, fire density and shrinkage fit well each together.

CONCLUSIONS

Results of presented investigations allows to draw the following conclusion that the optimal parameters for alumina grinding in rotary-vibration mill is filling ratio 33.5%, media ratio 23.6%, suspension ratio 35%, frequency 14 Hz, balls diameter 10 mm. Using above parameters it is possible to produce alumina powders having d_{50} approximately 3 μm and d_{90} close to 10 μm during about 60 minutes of grinding in rotary-vibration mill. Longer grinding time increase green, fire density and shrinkage of moulders for both types of alumina.

REFERENCES

BRUCH C.A., 1967, *Problems in die pressing submicron size alumina powder*, Ceramic Age., 83 (10) 44.

FREY R.C. AND HALLORAN J.W., 1984, *Compaction bahaviour of spray-dried alumina*, J.Amer.Ceram. Soc., 67, (3), 199–203.

GUO A., BEDDOW J.K., VETTER A.F., 1985, *A simple relationship between particle shape effects and density, flow rate and Hausner ratio,* Powder Technology 43, 279–284.

SIDOR J., 1978, *The optimization of the work of rotary-vibration mill, Optymalizacja pracy m³yna obrotowo-wibracyjnego*, Ph.D. Thesis, Academy of Mining and Metallurgy, Cracow, Poland.

SIDOR J., WÓJCIK M.A., 1994, *Disintegration of fly ashes in the rotary-vibration mill*, WASCON Conference, Environmental Aspects of Construction with Waste Materials, J.J.J.M.Goumans (Publisher), H.A. van der Sloot and Th.G.Aalbers (eds.), Elsevier Science B.V. 1994.

WÓJCIK M.A., SIDOR J., KABA³A J., PA³YZ J., PURSKA E., 1996, *Effect of morphology and grains size distribution of alumina ceramic mass on the green and fire density and shrinkage of moulders*, Light Metals, 49–54.

Wójcik M. A., Gajda T., Plewa J., Altenburg H., Luttermann A., Figusch V., Haviar M., Wpływ wybranych parametrów na proces mielenia tlenku glinu w młynie obrotowo-wibracyjnym. *Fizykochemiczne Problemy Mineralurgii*, 31, 115–124 (w jęz. angielskim)

W artykule przedstawiono wpływ wybranych parametrów na proces mielenia dwóch typów tlenku glinu w młynie obrotowo-wibracyjnym. Badano zależności między ilością mielonego materiału, mielników, stopniem napełnienia komory młyna mediami oraz wpływ wielkości mielników i częstotliwości drgań komory młyna na rozdrobnienie tlenku glinu. Pokazano również kinetykę mielenia tych materiałów. Badania pokazały, iż jest możliwe otrzymanie drobnych proszków tlenku glinu charakteryzujących się wielkością ziaren d_{50} około 3 μm i d_{90} około 10 μm w ciągu około 60 minut. W artykule przedstawiono również wpływ powyższych parametrów mielenia na końcowe właściwości fizyczne wyprasek wykonanych ze zmielonych w różnych warunkach tlenków glinu. Badano zależności między czasem mielenia a gęstością objętościową przed i po spiekaniu wyprasek oraz skurczem. Stwierdzono, iż dłuższy czas mielenia powoduje zarówno wzrost gęstości objętościowej przed i po wypaleniu, jak i skurcz wyprasek.