Qualification Methods of Al₂O₃ Injection Molding Raw Material

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Abstract. Nowadays, the sophisticated ceramic industry and technologies require higher and higher assumptions against quality, volume and yield of end product. [1,2] In the illuminant industry, for producing arc tube parts for High Intensity Discharge lamps the applied method is the ceramic injection molding. The ceramic arc tube parts are made of high purity alumina powder. By producing ceramic parts, one of the most critical step is to optimizing the injection molding process, determining and controlling the influential machine parameters, which have effect on the quality of end product. [3] Nevertheless, before we fall to doing the optimization of injection molding process, we need to know the properties of injection molding raw material, because later the molding process will be optimized for this material, to decrease the amount of cracked ceramics. [4]

For producing ceramic arc tube parts (plugs), there are used two different major components for producing injection molding raw material (feedstock): high purity alumina powder as the main component, and an organic paraffin wax as a binder material. It is expressly important to know the material, physical and chemical properties of these components, since mainly these have effect on the homogenity of feedstock, and therefore on the quality of end product. [5]

In this research, both of the main components and the moldable raw material was investigated by visual, physical, and thermal methods. As most important and main statement, the researchers found that the dynamic viscosity of raw material depends more on the applied temperature, than on the deformation speed gradient.

Applied analitical methods were laser granulometry, sieve analysis, differential thermal analysis and rheology analysis.

Keywords: alumina, wax, injection molding, laser granulometry, thermal analysis, rheology analysis

1. Introduction

Powder injection molding (PIM) is a technology for manufacturing complex, precision, netshape components from either metal or ceramic powder. The potential of PIM lies in its ability to combine the design flexibility of plastic injection molding and the nearly unlimited choice of material offered by powder metallurgy, making it possible to combine multiple parts into a single one. Furthermore, PIM overcomes the dimensional and productivity limits of isostatic pressing and slip casting, the defects and tolerance limitations of investment casting, the mechanical strength of die-cast parts, and the shape limitation of traditional powder compacts. [2,5]

In the illuminant industry for producing ceramic arc tube parts (plugs) for the High Intensity Discharge lamps, there are used two different major components for producing injection molding raw material (feedstock): high purity alumina powder as the main component, and an organic paraffin wax
as a binder material. Producing the feedstock, the wax is heated in a sigma-blade mixer, and the alumina powder is sequentially dosaged to the molten wax material during mixing. The given, properly homogenized mixture is cooled down, and broke in jaw crushe to get granules from it. This granules now is usable for the ceramic injection molding process. (figure 1.)

**Figure 1.** Granules raw material, injection molding green- and end product (arc tube)

To know the physical, chemical and thermal properties of the given feedstock granules is necessary, if we would like to set up an efficient injection molding process, which results a quality end product. The most commonly occurring failures in the end product are the cracks, voids and different material discontinuities, and to avoid these failures, the best way is the qualification of raw materials. [1,6]

### 2. Experimental
To qualify the alumina powder and feedstock material, the following measuring methods were tested:

#### 2.1. Laser granulometry
By qualifying the alumina powder, absolutely necessary to use laser granulometry method to determine the grain size distribution of the chosen powder. In this way, we can predestine the quality of end product. [7,8] In addition, using this method, if we know the grade of polydispersity, and the given grain distribution (figure 2.), we can conclude to the compaction and volume filling properties of the powder, and thus the density of the end product. [9, 10, 15,17,18,19,20,21,22]

**Figure 2.** Schematic of polydisperse alumina powder
From: Tamás F 1970 Szilikátipari laboratóriumi vizsgálatok Műszaki könyvkiadó Budapest

#### 2.2. Sieve analysis
The sieve analysis is the one of the oldest method to classify powders and granules based on its particle size distribution. Using woven screen material, the sieving classifies the particles according to its middle dimension, namely the width. The mechanical sieving is the most suitable in the case, when the most of the particles larger than 75 μm. In the case of sieving smaller particles its slight weight does not ensure the proper force to overcome the cohesion and adhesion forces. [11,12,13,]

In our case, the prepared feedstock granules can be classified using sieve analysis. This qualification method is very important, because during the injection molding the too small grains can cause feeding difficulties, they can stick on the feeder and so abort the injection molding process. In addition, the too large grains can cause inhomogenous material melting, and therefore material incontinuities, voids, cracks can be in the end product. [4,5]

Using the sieve analysis, the securely usable grain size of granules is determinable, and we can identify and eliminate the improper grain fractions from the material.
2.3. Differential thermo analytics
The effect of heat occurring endotherm and exotherm reactions in solid materials can be measured using the well known differential thermo analytics method. The investigated material heated together with a comparative material (inert material). In the inert material does not occurs any endotherm or exotherm process or transformation in the used temperature range. Applying this measurement, the temperatures of phase transformations are determinable in different materials, like the melting, evaporation or inflammation temperatures. [14,16]

In our case the method seems to be very important, when we would like to identify the injection molding work temperature of the used raw material. Since the process have to be adjusted to the proper temperature, which is above the melting temperature, but it is under the intensive evaporation temperature, to avoid the material loss during the injection molding process.

2.4. Rheological test
By injection molding it is absolutely necessary to know the reological properties and behavior of raw materials, especially its viscosity. Mainly, this property specifies the flow behavior in the injection molding tool, thus it can have effect on the quality of end product. [23,24,25]

To define the viscosity of feedstock, dynamical rotation rheometer was used, the principle of operation can be seen on figure 3.

![Figure 3. Principle of operation of dynamic rotation rheometer](image)

During the test, the rheological parameters are counted from the values of torque and angle using built-in software, with the application of optical encoder. [26,27]

Besides of the raw material, to know the rheological properties of wax binder material can be also important, since mainly the binder material defines the reological behavior and viscosity of feedstock.

3. Results and discussion

3.1. Laser granulometry
The first measurement to qualify the properties of alumina powder is the laser granulometry to investigate grain size distribution.

In the following, in my experiment I compared a raw, unmilled and a 10 minutes in vibration ball mill milled alumina powder. On figure 4 we can see the histogram of unmilled powder, where we can observe the two peaks on different grain sizes, which refers to the earlier mentioned polidisperse grain distribution (figure 2.). The polidisperse grain distribution - namely the small grains between the larger grains – results more effective volume filling, than the unified grain distribution.

The shape of histogram of milled powder is clearly different (figure 5.). We can see, that the second peak is lower, which refers to the less larger particles and less polidisperse properties of powder, but better compaction properties and higher density, thanks to the more small particles.
3.2. Sieve analysis

The injection molding process is very sensitive for the grain size of granules, because the not proper grains can cause dosage and compaction difficulties during injection molding. Therefore it is necessary to qualificate the mixed raw material according to its grain size, using sieve analysis. In this way, before usage we can eliminate the improper grains from the material.

In the experiment, the feedstock material was classified using this method, where the amount of usable fraction was ~55% between 0.1 and 1.5 mm, the smaller grains had to be rejected, the larger grains had to be regrinded.

3.3. Differential thermo analytics

The next measurement, what can be used for the IM raw material is the well known differential thermo analytically. This method is usable to investigate the behavior of injection molding raw material in the function of temperature. For the played out processes we can conclude from the weight changing of material. The feedstock material was experimentally tested, the derivatograph of thermal analysis can be seen on the figure 6.

Figure 4. Grain distribution of unmilled alumina powder

Figure 5. Grain distribution of 10 minutes milled alumina powder

Figure 6. Derivatograph of injection molding raw material
We can observe the melting point ($T_m$) on the first negative peak, the evaporation point ($T_e$) on the first positive peak and the flashpoint ($T_f$) on the second positive peak of the material on the DTA curve.

3.4. Rheological test

The rheological properties of raw material we can investigate using dynamical rotation rheometer to define the flow properties of material on different temperatures and shear velocities. This measurement is very important because the flow properties of the material have influence on the injection molding process, and thus on the quality of the end product. The principle of the dynamic rotation rheometer can be seen on the schematic picture (figure 3). The feedstock material was investigated using this method, the registered viscosity curves can be seen on the figure 7.

![Figure 7](image)

**Figure 7.** The viscosity of injection molding raw material depending on the deformation speed and the applied temperature

On this plot we can see how the viscosity of raw material depends on the applied deformation speed and temperature. The viscosity exponentially decreases with the increasing deformation speed, and decreases with the increasing temperature. We can observe, that the temperature have a huge effect on the viscosity.

4. Conclusions

Using the demonstrated investigations, the quality of the raw materials are determinable, and possible the classification before usage. Testing these methods, very important statements were determined in the optimization of injection molding process point of view.

Measuring the alumina powder using laser granulometry it is possible to determine the grade of polydispersity of the powder, and in addition, the grade of polydispersity can be reduced in the aluminum-oxide powder using vibration ball mill, to enhance volume filling.

It is necessary to qualify the raw material according to its grain size, to eliminate the inappropriate powder fractions from the granules, which can cause injection molding difficulties.

The injection molding material is well-characterized by DTA, to establish evaporation, melting and inflammation point, and to determine the temperature work point during injection molding of the usable material.

The dynamic viscosity of the feedstock exponentially decreases with the increasing deformation speed and decreases with the increasing temperature. In addition, it was observed, that the rheological behavior of raw material depends more on the applied temperature, than on the deformation speed gradient.
References

[1] Mutsuddy 1995 B C Ceramic Injection Molding Chapman and Hall,
[8] Gitzen W H 1970 Alumina as a ceramic material The American Ceramic Society Ohio
[22] Cotica L F Paesano 2005 High energy ball-milled (Fe2O3) (Al2O3) system: A study on milling time effects Journal of Alloys and Compound
[27] Cima M J Lewis J A 1989 Binder Distribution in Ceramic Greenware during Thermolysis