

## Recycling process of casting molds applying to precision castings

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### Manufacturing and processing

#### ABSTRACT

**Purpose:** The present work relates to investigations about the possibility of recycling and producing sinters from ceramic molds. These molds are applied for producing precise single crystal casts of nickel based superalloys to elements which are applied to the hot section of aircraft jet engines.

**Design/methodology/approach:** The processes of milling were carried out using the vibratory mill Herzog HSM 100H and the planetary grinder Pulverisette 7 - Fritsch Company. The measurements of all kinds of powders were conducted by using of the Kamika IPS U Analyzer and Malvern Nanosizer-ZS. To the purpose of analysis of the chemical composition an X-ray microanalysis technique was applied with the dispersion of the energy (EDS) using of Thermo and Noran equipment.

**Findings:** On the basis of examinations carried out on the powders before and after processes of milling and after processes of pressing and sintering of powders, it is possible to state that technological processes of reuse and further applying of used molds are possible. Processes of pressing were conducted applying different amounts of powders. Powders were pressed both without the addition as well as with the addition of modifiers which were added to basic powder in appropriate amounts. It was found that processes of pressing and sintering in both cases showed good results. Surfaces of pressed and sintered tablets were smooth and not-delaminated as well as did not shell.

**Research limitations/implications:** Results will be used for future researches among others concerning the research on mechanical and thermal properties.

**Practical implications:** Waste products and their reuse, recycling and especially consolidation of dangerous and hazardous compounds including of ceramic materials from foundry industry, will have an important place in the future on account of problems with their recycling and the storage.

**Originality/value:** Researches on new possibilities of the application and properties of waste materials.

**Keywords:** Recycling, Ceramic molds, Milling, Pressing, Sintering

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### 1. Introduction

Nowadays, the level of industrialization and the continued growth in consumption leads to increase in the amount of waste

materials and by-products. These materials often pose a big problem with the disposal, storage or reprocessing with very strict environmental standards especially in the countries of the European Union [1,7]. The rules and criteria for the protection of the environment sometimes even re-enforce their use as

feed/process stock in other branches of industries. For this reason, much attention is paid to problems of re-use of waste materials, such as municipal and ones coming from a variety of industrial processes. There are many on-going attempts to re-use all kinds of waste materials, even such as sewage sludge, river or sea ones [2], slag and sludge from metallurgical processes [3-4], which can be successfully processed into building materials, such as bricks or tiles. The above problem also applies to the waste materials in the foundry process, for example, foundry sands and ceramic molds. Globally, in the foundry industry, millions tons of waste per year are produced, of which approximately 70% are so called molding sands [5]. From analysis of the literature data [6-8], it can be concluded, that the ceramic molds represent about 62% of the waste mass generated in the production of precision castings, what, according to the French foundries, is about 600 000 tons per year. One of the re-management possibilities for used ceramic molds is regeneration in internal recycling. Another option is to use them outside the foundry, for example for road construction, building materials, ceramics, or to apply methods of recovering metals from waste molds, which, however, can create a big economic and technological problem.

The paper concerns a study on the possibility of producing sintered materials in the recycling process of used ceramic molds for investment casting of aircraft engine components.

## 2. Results and discussion

Ceramic molds are used inter alia in the aerospace industry, for the manufacture of precision castings components of hot air engines [9]. Preparation of such molds can be described briefly in the following steps: finished product wax model preparation in the specific matrix, ceramic mold wax model preparation on the product model by successively applying layers of a liquid sandmix followed by the molding granules and so on until the total dry. As the materials used to produce the molds, corundum powders, which are part of so-called fillers, are used. They are a part of molding fillers which form a matrix, containing mostly  $\alpha$ - $\text{Al}_2\text{O}_3$  and  $\beta$ - $\text{Al}_2\text{O}_3$  phases with varying grain between 15-50 microns in diameter, as well as mullite ( $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ ) with an average grains diameter of about 30-120 microns. This mullite is included in the fillers on so-called "backup" rear layer.

There are also powders used on first and rear layer with an average grain diameter of about 250 to 850 microns, and a binder of colloidal silica with an average  $\text{SiO}_2$  content of 25-30%. In the final stage of the production, ceramic mold is subjected to heat treatment.

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The analysis of chemical composition of the finished mold was carried out using scanning electron microscope HITACHI S-3400 NII, with EDS (Energy-dispersive X-ray spectroscopy). As shown in Figs. 1, 2.

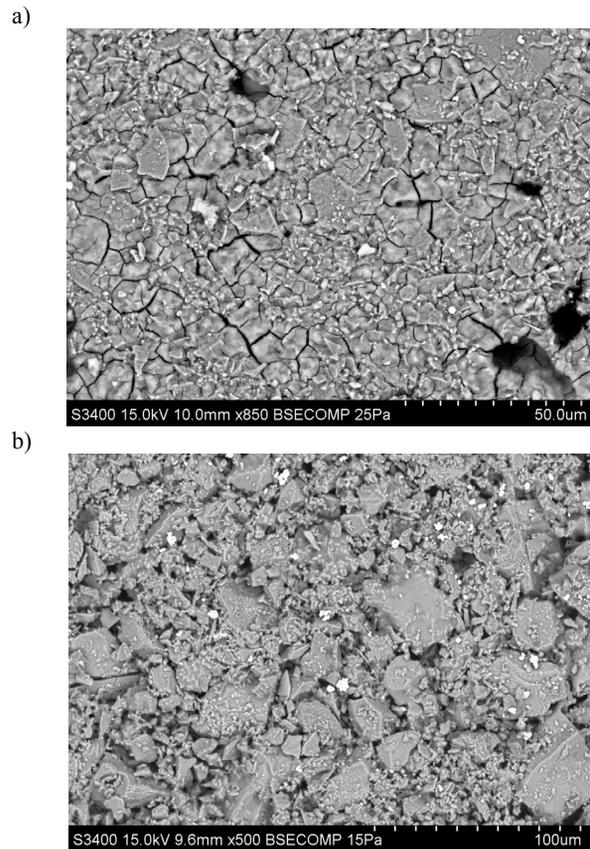


Fig. 1. Ceramic mould a) after the full sintering, b) after the preliminary sintering - SEM photo

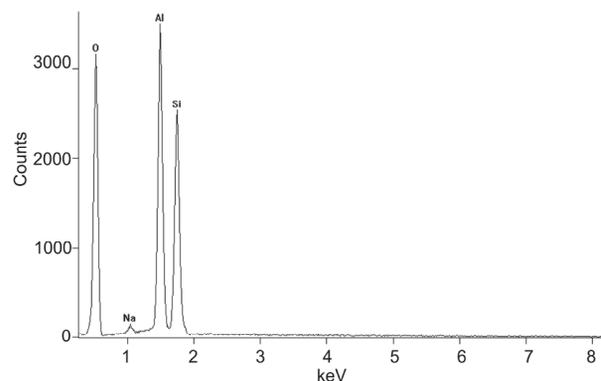


Fig. 2. Characteristic X-ray spectrum of the mold

In the first stage of study, ceramic mold and modifiers as nickel oxide, metallic nickel and aluminum compounds were subjected to crushing and grinding. These processes consist in the reduction of particle size of the starting material. Crushing and grinding take place under the external forces what leads to formation of grain stresses beyond their strength. Grinding methods give more massive products than chemical methods, which, however, allow better quality of the powders which

expresses a better chemical homogeneity. Moreover the powder particles are also smaller and have smaller size variation. The grinding process was done on Herzog HSM 100 H vibration mill and on Pulverisette 7 planetary mill by Fritsch. In order to determine the average grain diameter of milled mold analyzer IPS U by Kamika was used. IPS analyzer measurement method is complex and consists of measuring smallest moist particles bonded from 0.5 to 1000 nm with regard to the effect of laser diffraction, for the larger particles to move gradually and continuously, to measure changes in radiation flux scattered by the moving particles. Infrared radiation flux not only identifies the particle size, but also allows accurate count of them in the entire measurement range. Each particle corresponds to an electrical signal proportional to the particle size. A collection of particles is initially measured with

division in 4096 size classes and converted (calibrated) to 256 size classes available to the user. For the aluminum compounds powders the measurement was performed on **Nanosizer-ZS** device by **Malvern Instruments**. Measurement of particle size, in this device, is based on the Brownian phenomenon of motion and *Dynamic Light Scattering* technique (DLS) where zeta potential measurement is based on a combination of electrophoresis and *Laser Doppler Velocimetry* technique. Measured particles size range given by the manufacturer is from 0.6 nanometer to 6 microns and the measurement can be carried out both in water and aqueous solutions. Use of an ultrasonic disintegrator during sample preparation allows effective breaking of the agglomerates and the actual measurement of particle size. The measurement results are shown in Figs. 3-5.

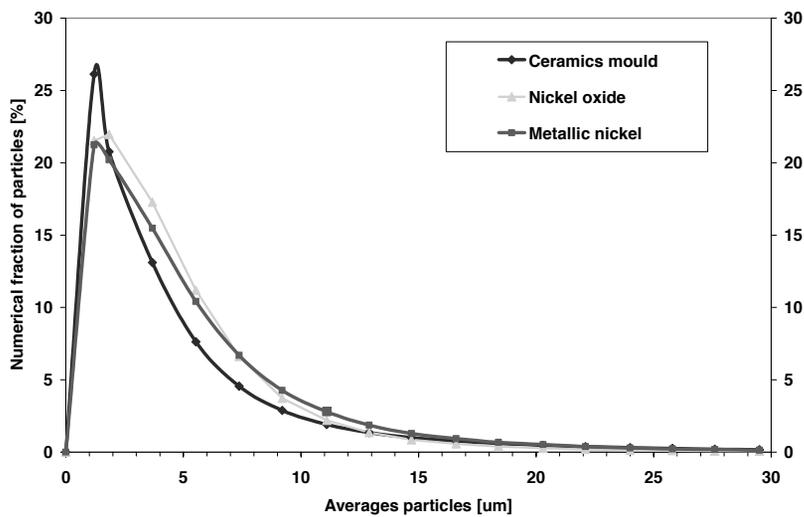


Fig. 3. Numerical fraction of particles of chosen powders

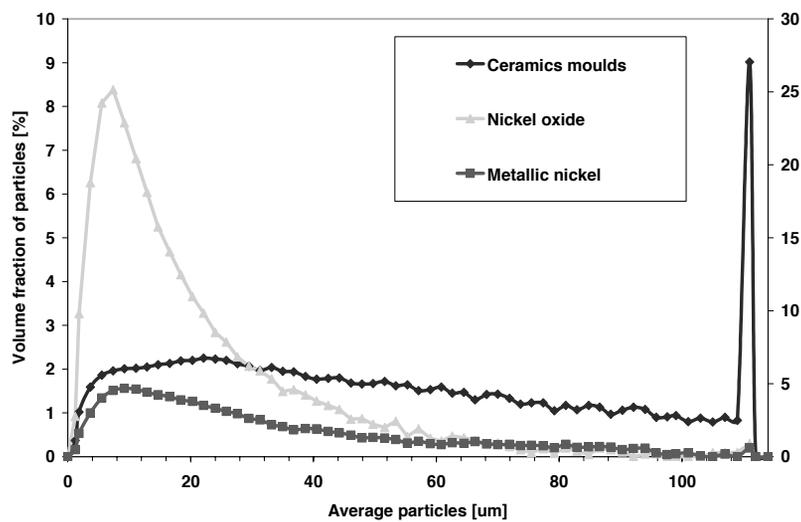


Fig. 4. Volume fraction of particles of chosen powders

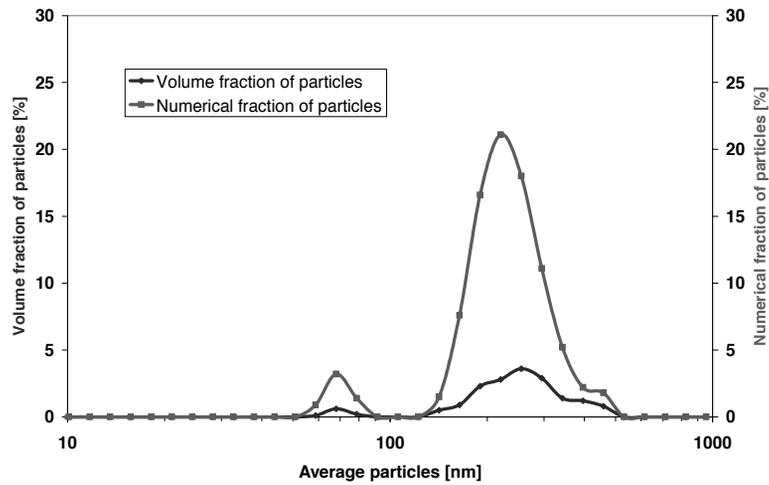


Fig. 5. Volume and numerical fraction of particles of aluminum compounds

Table 1. Chemical analysis of pure metallic nickel

Point	wt %	at %
1	100	100

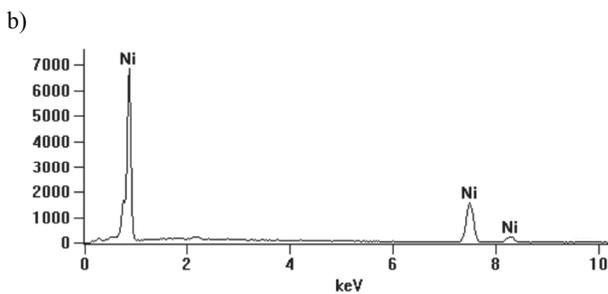


Fig. 6. Typical EDS analysis of the chemical composition of metallic nickel powder a) area of analysis, b) characteristic spectra of x-ray radiation

In order to complete the grain analysis, microscopic observations on the morphology of the particles were made using

Hitachi S-3400 NII SEM. The results of these observations are shown in Figs. 6-11.

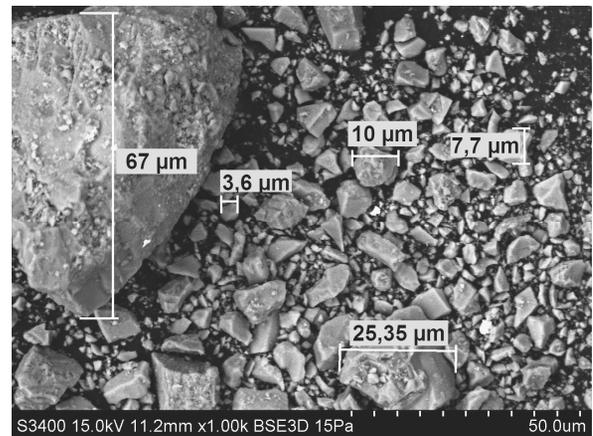


Fig. 7. The surface morphology of the crumbled mold particles

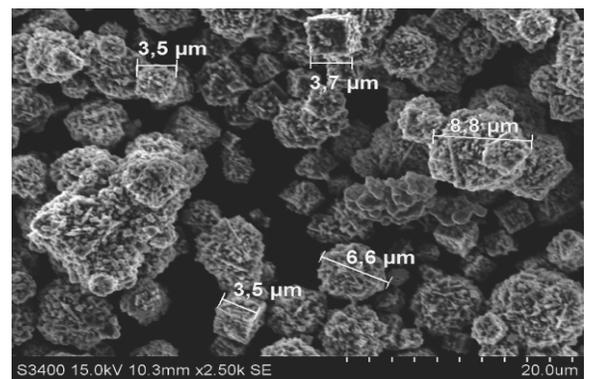


Fig. 8. The surface morphology of the cuboidal particles of metallic nickel powder

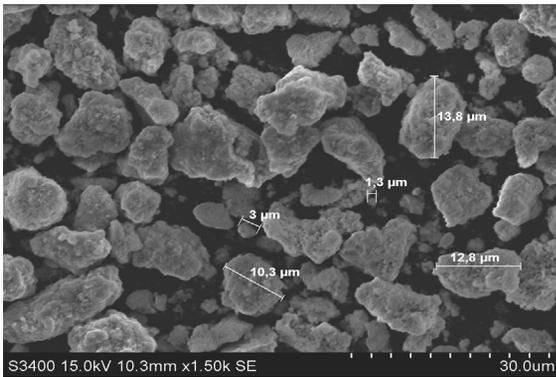


Fig. 9. The surface morphology of the irregular particles of nickel oxide powder

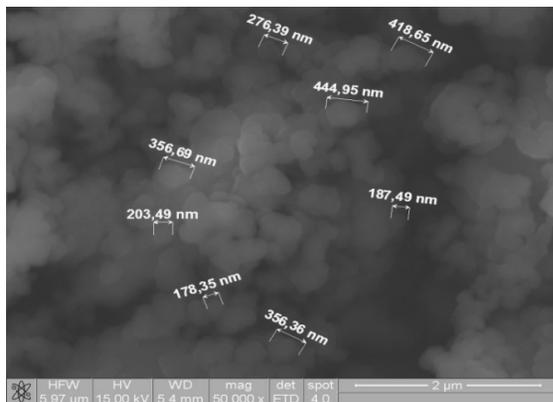


Fig. 10. The surface morphology of the nano-particles of aluminum compounds

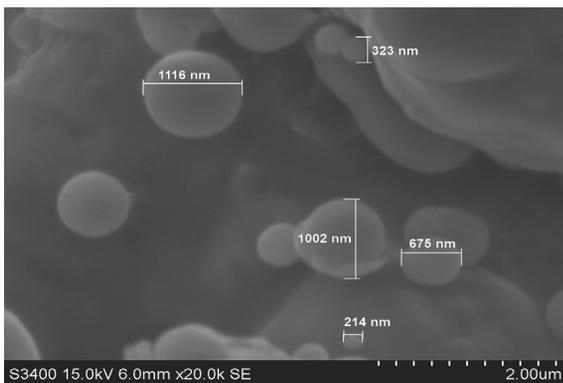


Fig. 11. The surface morphology of the spheroidal particles of metallic nickel powder

Analyses show that the molecules of ceramic mold and metallic modifiers as metallic nickel and nickel oxide are characterized by a mean particle size distribution in range to 7 μm. Aluminum compound powder has a particle size distribution in range of 200-600 nm. The next stage of the research was compaction of milled ceramic molds.

The compaction process was carried out on a hydraulic press TP Herzog's 40. Analytical samples of 20 g mass were used not only consisting of pure mold but also with addition of modifier compounds in various proportions shown in Table 2. Thus prepared sets were subjected to compression on a hydraulic press at a pressure of 100 MPa. As a result of compression, samples in cylindrical form of 40 mm base diameter and a height of about 10 mm were obtained. Determined by the following tests, proper sintering temperature was determined by subjecting them to heat treatment at 1350°C. In Figs. 12-15, selected die stampings after sintering process are shown.

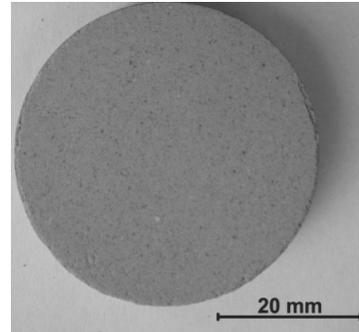


Fig. 12. Macrophotography of sample after sintering appointed in Table 1 as I.2

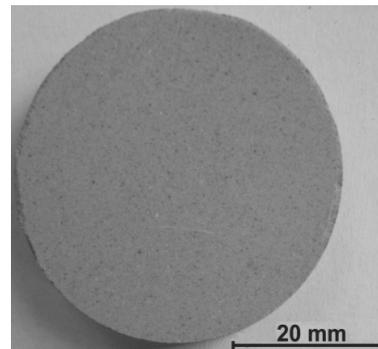


Fig. 13. Macrophotography of sample after sintering appointed in Table 1 as III.2

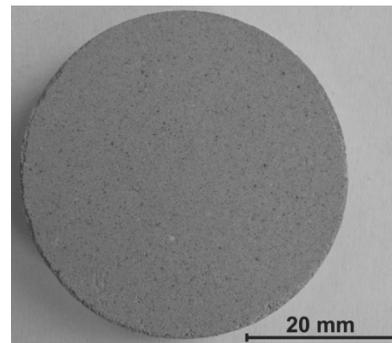


Fig. 14. Macrophotography of sample after sintering appointed in Table 1 as I.3

Table 2.

The profile of the mixtures of powders applied to pressing the tablets

	Mass of the mixed powders [g]							
	I.0	I.1	I.2	I.3	III.1	III.2	III.3	III.4
Ceramic mould	20	19	19	19	18	18	18	17
Metallic nickel	-	-	-	1	1	-	1	1
Nickel oxide	-	-	1	-	-	1	1	1
Albumina compounds	-	1	-	-	1	1	-	1

Table 3.

Density and porosity measurements results

	Sintering temperature 1350°C							
	I.0	I.1	I.2	I.3	III.1	III.2	III.3	III.4
Actual density [g/cm <sup>3</sup> ]	3.2040	3.1596	3.2281	3.2027	3.2554	3.2972	3.3258	3.3240
Pressed density [g/cm <sup>3</sup> ]	2.1667	2.2009	2.2392	2.2456	2.2528	2.2490	2.3027	2.3013
Porosity [%]	38	27	27	25	31	31	31	31

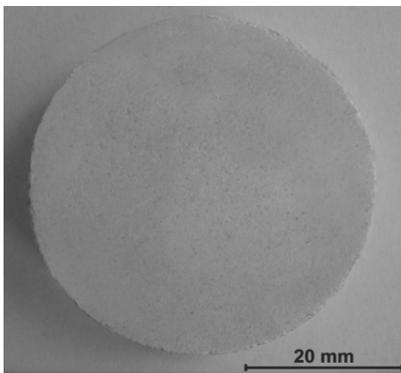


Fig. 15. Macrophotography of sample after the process of sintering (ceramic mold)

Sintered profiles were examined in the field of physical properties, such as actual density carried out on helium pycnometer AccuPyc 1300 by Micromeritics, the principle of which is based on gas usage to precisely determine the volume of the sample. Solid volume is that portion of previously calibrated measuring chamber which has not been occupied by the gas. Specimen weight is determined by weighing on an analytical balance and transferring the data to the operator software in the device. The test is fully automated, fast and accurate. Measurements are carried out in an atmosphere of helium, but for specific applications, the analyzer can be also used to measure in the dry air, carbon dioxide and nitrogen atmospheres. Helium use allows analysis of porous bodies, and much expanded areas. Pressed density and porosity of sintered profiles were determined on *GeoPyc 1360* analyzer by Micromeritics.

Mentioned device performs a precise measurement of the sample volume. This measurement is carried out in a semi-liquid medium called DryFlo, based on volumetric substitution of this substance by analyzed specimen placed in in a measuring cylinder. Results of these measurements are shown in Table 3.

Further, the internal structure was defined using optical microscopy. The results of microscopic observations are shown in Figs. 16, 17.

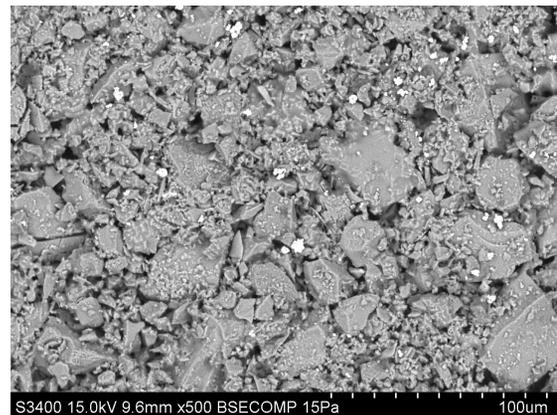


Fig. 16. Microphotography of the surface of specimen from milled mold after sintering process

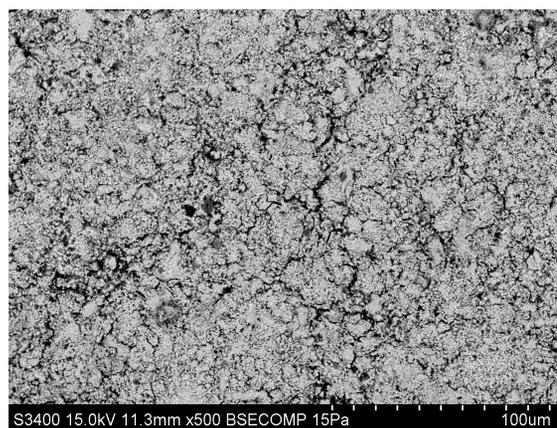


Fig. 17. Microphotography of the surface of die stamping III.3

### 3. Conclusions

Based on the powders studies before and after milling and on results of compression and sintering processes, it can be concluded that the recycling process of used ceramic molds in investment casting of aircraft engines components is possible. Compaction of powders was performed with and without modifiers addition. In both cases, good results in the process of compacting and sintering were obtained. The surfaces of die stampings were smooth and did not undergo stratification. Additionally, they did not show any edges crumbling which remained sharp without clear signs of chipping. Defects visible in the specimens in particular on the edges result from mechanical damage caused by transportation.

It can be assumed that the ceramic industry can re-use a large amount of used molding materials without special treatments for modification. On the other hand, there are assumptions, that the heat treatment can stop and bind harmful substances in the ceramic matrix.

### Acknowledgements

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