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Paper# 20

Shrinkage calibration method for µPIM manufactured parts

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Abstract

Five green and five sintered parts of a micro mechanical component, produced by micro powder injection moulding, were measured using an optical coordinate measuring machine. The aim was to establish a method for quality assurance of the final produced parts. Initially, the so called "green" parts were compared with the sintered parts (final products) calculating the percentage of shrinkage after sintering. Successively, the expanded uncertainty of the measured dimensions were evaluated for each single part as well as for the overall parts. Finally, the estimated uncertainty for the shrinkage was evaluated propagating the expanded uncertainty previously stated and considering green and sintered parts correlated. Results showed that the proposed method can be effective in stating tolerances if it is assumed that the variability on the dimensions induced by the shrinkage equals the propagated expanded uncertainty.

Keywords: Micro Powder Injection Moulding, Shrinkage, Shrinkage Uncertainty, Expanded Uncertainty.

1. Introduction

Micro-powder injection moulding (µPIM) is considered an interesting manufacturing process for complex micro parts or micro structured parts. A low cost miniaturised manufacture, chances to have mass production and, finally, assembly steps integrated into the process (co-injection and co-sintering) [1-3] make µPIM particularly attractive.

Nevertheless, there are some limitations related to this manufacturing technique [3] and quality control is among them. Past works already investigated the µPIM dimensions replication [2, 4, 5] and the attainable surface topography [2, 6-8]. Other works [2, 9] focused on the influence of moulding parameters on dimensional accuracy. Additionally, the effect of the production variability on acceptance of tolerances has deeply been considered (see, e.g., [10, 11]). Indeed, the vice versa, i.e., assigning possible tolerances examining the production variability, to our knowledge, has not been investigated similarly.

In this view, the current study deals with the impact of the shrinkage, due to the sintering process, on the dimensions stated in the drawing. Taking advantage from a specific study case of a micro powder moulded part, a method for evaluating the influence of the shrinkage on tolerance specification is proposed and described in this paper.

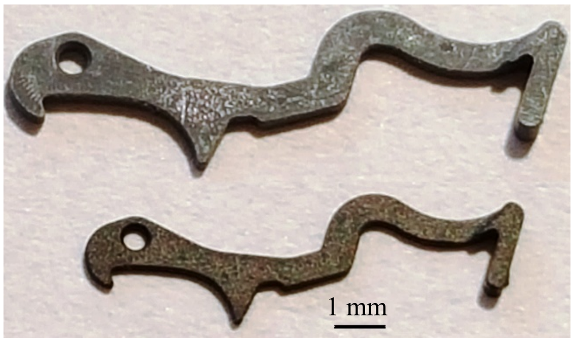

Fig. 1. Example of green (top) and sintered (bottom) micro mechanical parts.

2. µPIM manufactured parts

The investigated product is a micro mechanical part (Fig. 1), which is a critical functional component in a high-accuracy miniaturised mechanism. In particular, the size of the features of size of the micro component ranges from several millimetres to tens of micrometres. This is challenging when the measurements are to be performed, although, it is particularly useful for examining dimensions and tolerance chains, at different length scales, in the same process.

A sketch of the micro mechanical part, with a legend of the measured features of size, is shown in Fig. 2. The nominal values are summarised in Table 1. These values refer to the final dimensions, intended for the micro component after sintering.

Details about the production of the micro component and the materials used are also given in the next § 2.1. Even so, the study presented below deals with the metrological aspects and the quality assurance of the parts, considering the specific manufacturing process and, in particular, the accuracy of the sintering process and the mould repeatability.

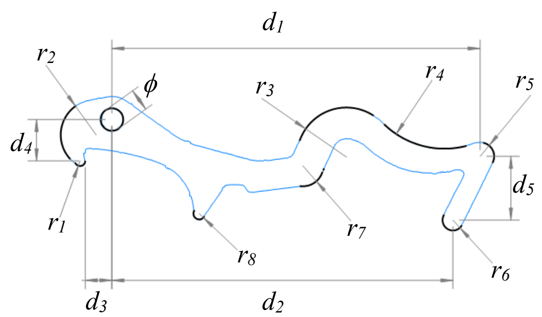

Fig. 2. Legend of the dimensions in Table 1.

2.1. Micro-powder injection moulding production of the micro component

The µPIM process was, specifically, a ceramic injection moulding (CIM) performed by an Arburg Allrounder 270 S 250-60, with a diameter of the reciprocating screw of 15 mm, a diameter of the nozzle of 2 mm and a maximum clamping force of 250 kN. Parameters and settings are summarised in Table 2. Material properties can be found in [12].

The ceramic feedstock used was Catamold® TZP-F 315 produced by BASF SE, i.e., a compound of polyoxymethylene (POM) binder and ZrO_2, Y_2O_3-stabilised. After sintering it became polycrystalline yttria-stabilised tetragonal zirconia, with typical composition of about

- 89 % of zirconium dioxide (ZrO_2)
- 5 % of diyttrium trioxide (Y_2O_3)
- 6 % of unspecified material(s) (not disclosed by the producer).

Table 1. Nominal values, given in millimetres, of the dimensions in Fig. 2 (sintered component).

d_1	d_2	d_3	d_4	d_5	ϕ
7.939	7.515	0.612	0.984	1.438	0.40

r_1	r_2	r_3	r_4	r_5	r_6	r_7	r_8
0.08	0.75	1.06	1.82	0.30	0.22	0.40	0.10

After a debinding process (at 110 °C by nitric acid), with a minimum loss of 17.5 %, a typical sintering cycle consisted of the following steps performed in air:

- Heating from room temperature to 270 °C with rate 3 K/min; hold on 1 hour.
- Heating from 270 °C to 1500 °C with rate 3 K/min; hold on 1 hour.
- Cooling from 1500 °C to 600 °C with rate 5 K/min.
- Furnace cooling.

The shrinkage subsequent to the sintering process can be taken into account oversizing the mould dimensions. To obtain the desired sizes in the final sintered part, the material producer specified an oversizing factor in the range 1.285 ÷ 1.292.

The specifications forthwith given are intended to better explain the context. Nonetheless, the proposed investigation has the ambition to be independent from specific process conditions because based on a statistical approach.

Hereafter, the purpose is to define a method for shrinkage calibration for high precision micro powder moulding applications.

3. Measurements

All the features of size in Fig. 2 were measured for five green parts and five sintered parts, using a DeMeet 220 optical Coordinate Measuring Machine (OCMM – magnif. 2×, lateral res. 4 µm). Sintered and green parts were chosen independently from each other. As a consequence, the investigation of this specific manufacturing process is to be considered in reproducibility conditions.

Table 2. Parameters and settings of CIM process.

Parameter	*Setting*
Material type	ceramic feedstock (ZrO^2)
Barrel temperature /°C	164-172
Mould temperature /°C	140
Injection speed /cm^3 s^{-1}	8
Switch-over pressure /MPa	152
Cushion /cm^3 s^{-1}	1.1
Packing pressure (pressure profile vs time)	0 s: 120 MPa, 1 s: 90 MPa, 2 s: 7.5 MPa
Total packing time /s	2
Machine	Arburg Allrounder 270 S 250-60 (15 mm)

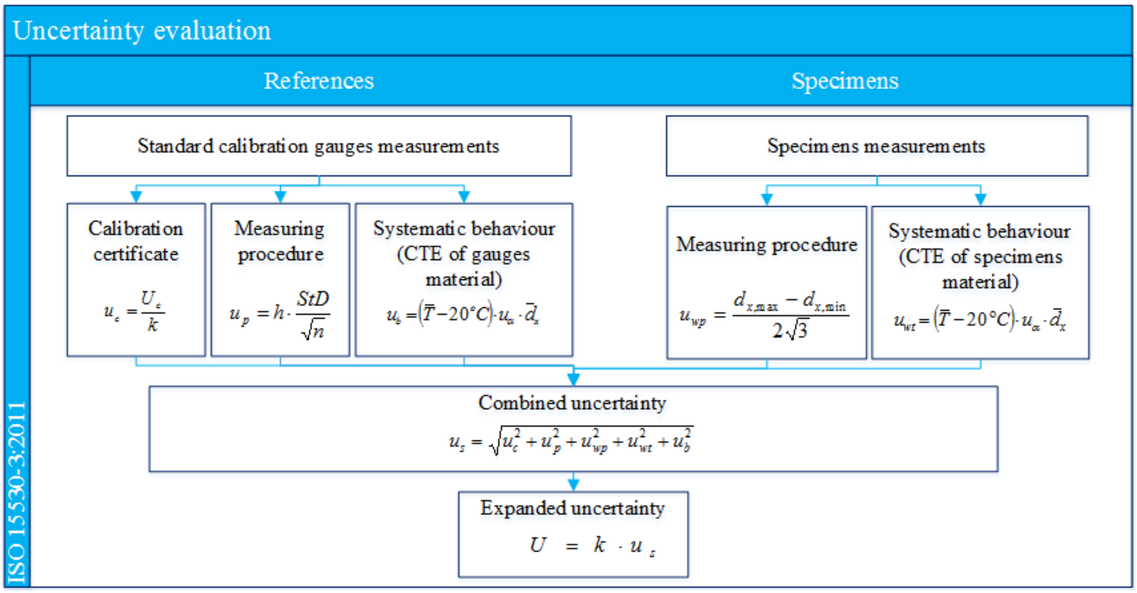

Fig. 3. Uncertainty model used in the analysis.

4. Shrinkage evaluation and uncertainty model

The shrinkage of the i^{th} green part $\delta L_{\%,i} = f\left(d_{x_i,g}, \bar{d}_{snt}\right)$ was estimated, for each dimension $d_{x_i,g}$, as the relative deviation from the average $\bar{d}_{snt}$ of all sintered parts.

The average of all sintered parts (stable parts) was, in fact, considered the reference for the achieved manufacture (see Eq. 1):

$$\delta L_{\%,i} = f\left(d_{x_i,g}, \bar{d}_{snt}\right) = \frac{d_{x_i,g} - \bar{d}_{snt}}{\bar{d}_{snt}} \times 100 \qquad (1)$$

where

- $f\left(d_{x_i,g}, \bar{d}_{snt}\right)$ is the shrinkage of the i^{th} green part, expressed in percentage;
- $d_{x_i,g}$ is a generic dimension of a generic green part;
- $\bar{d}_{snt}$ is the average value for a generic dimension considering all sintered parts.

For evaluating the uncertainty of the shrinkage due to the sintering process, the expanded uncertainty (see § 4.1 below) was propagated considering green and sintered parts correlated and, according to the truncated non-linear Taylor series of the shrinkage approximated to the first order, it is explicitly:

$$u_{\%,i} = \left(\frac{\partial f}{\partial d_{x_i,g}}\right)^2 \Delta d_{x_i,g}^2 + \left(\frac{\partial f}{\partial \bar{d}_{snt}}\right)^2 \Delta \bar{d}_{snt}^2 + $$
$$+ 2 \frac{\partial f}{\partial d_{x_i,g}} \frac{\partial f}{\partial \bar{d}_{snt}} \rho_{g,s} \Delta d_{x_i,g} \Delta \bar{d}_{snt} \qquad (2)$$

where

- $u_{\%,i}$ is the uncertainty of the shrinkage propagated for the i^{th} green part, expressed in percentage;
- $\frac{\partial f}{\partial d_{x_i,g}}$ and $\frac{\partial f}{\partial \bar{d}_{snt}}$ are respectively the partial derivative of the shrinkage with respect to the generic dimension of the i^{th} green part and the one with respect to the average of all sintered parts;
- $\Delta d_{x_i,g}$ and $\Delta \bar{d}_{snt}$ are respectively the variability of $d_{x_i,g}$ (i^{th} green part) and the variability of $\bar{d}_{snt}$ (all sintered parts) due to the measuring process.

It was assumed that these deviations equal, correspondingly, the expanded uncertainties for $d_{x_i,g}$ and for $\bar{d}_{snt}$ (conservative choice). They were evaluated considering, respectively, seven repeated measurements per each specimen and the repeated measurements of all the specimens.

4.1. Evaluation of the expanded uncertainty

The expanded uncertainty U was evaluated according to [13]. The uncertainty evaluation flow and the considered contributors are shown in Fig. 3.

A measurement result d_x and its expanded uncertainty U were, consequently, considered as $d_x \pm U$, where U was determined with a coverage factor $k = 2$, i.e., considering an approximated expanded interval of 95 %.

Eventually, it should be noted that we consider the proposed method independent on the used uncertainty model. In our opinion, other approaches for uncertainty evaluation can be used, e.g., PUMA method or the so-called frequentist approach. In the last case, a larger number of repeated measurements are required to evaluate the expanded uncertainty and a lower uncertainty interval may be expected.

5. Measurements capability

Being the average of all the sintered parts the reference, their variability can affect the estimated variability of the green parts.

If the variability of the sintered parts is intrinsic to the production, the results can be considered an acceptable estimation. Conversely, if it is due to the measuring process the results cannot be reliable [14]. For this reason, in order to understand if the evaluation could rely on the measurement process, it was convenient to consider the evaluated expanded uncertainty such as separated in two contributions: one for the instrument *U(instr)* and another for the production *U(μPIM)*.

Considering *U(instr)* as the average of the expanded uncertainties, each related to a single part, and *U* the one evaluated on all parts, *U(μPIM)* was estimated, for both green and sintered parts, as (see Eq. 3):

$$U^2(\mu PIM) = U^2 - U^2(instr) \qquad (3)$$

Eventually, an indication of the measuring process capability was given as the ratio (see Eq. 4) [10, 11, 14]:

$$\frac{U(instr)}{U(\mu PIM)} < (10 \div 20)\,\% \qquad (4)$$

The estimation of the influence of the measuring process may be useful for tolerances specification. When an evaluation satisfies a fixed limit for the ratio in Eq. 4, the supplier conformity specifications can be stated, according to [15], as propagated uncertainty for the shrinkage $u_{\%,i}$ (see § 7 below).

6. Results

Results of the evaluated expanded uncertainties and the ratio in Eq. 4 are given in Table 3 for the green parts and in Table 4 for sintered parts. *U* is related to all samples. *U(instr)* is the expected value for the instrument contribution, i.e., the average of the expanded uncertainties related to each single sample (U_{gi} and U_{si}). *U(μPIM)* is given by Eq. 3.

In Table 5, instead, the estimated shrinkage, for each green part and for the overall ones, is specified. The corresponding propagated uncertainties are in Table 6.

7. Tolerances estimation

Tolerances can be estimated by the expanded uncertainties propagated to the shrinkage.

Two sets of contributions can be propagated: $u_{\%,i}$, which is related to the shrinkage of each single sample, and u_{Ave}, which is the propagated uncertainty of the average shrinkage, i.e., considering all the samples. Equivalently to what stated in § 5, the Eq. 3 applied to u_{Ave} and $u_{\%,i}$ can be an estimation of the process variability due to the shrinkage. Nonetheless, conversely to what considered there, the average of the uncertainties was considered a too weak assessment of the samples variability. Instead, the quadratic average was performed.

For each sample under evaluation, the influence of the measuring process was eliminated according to the following formula

$$u^2_{proc,\%,i} = u^2_{Ave} - u^2_{\%,i} \qquad (5)$$

When this difference was a negative number (an explanation may be seen in the approximation established by the truncated Taylor series), it was considered $u^2_{proc,\%,i} = u^2_{Ave}$.

Successively, the variability (expected expanded uncertainty) of the overall process was estimated as the quadratic average of the propagated uncertainties per each sample, considered as expected standard uncertainties, according to the following equation

$$U_{proc,\%} = h_s \times 2 \times \sqrt{\sum_1^n u^2_{proc,\%,i} \Big/ n} \qquad (6)$$

where n is the number of samples considered and $h_s \geq 1$ is a safety factor.

Table 3. Green parts: overall expanded uncertainty *U*, *U(instr)* and *U(μPIM)*. Results of the ratio in Eq. 4 are also given. All values are expressed in micrometres.

	d_1	d_2	d_3	d_4	d_5	ϕ
U_g	67	35	33	29	75	10
U(instr.)	16	12	14	12	15	1
U(μPIM)	65	33	30	26	73	10
ratio	24 %	36 %	47 %	45 %	21 %	15 %

	r_1	r_2	r_3	r_4	r_5	r_6	r_7	r_8
U	15	24	89	19	83	27	73	12
U(instr.)	4	3	7	9	10	9	3	3
U(μPIM)	14	23	89	19	82	26	73	12
ratio	28 %	14 %	8 %	5 %	12 %	33 %	5 %	27 %

Table 4. Sintered parts: overall expanded uncertainty *U*, *U(instr)* and *U(μPIM)*. Results of the ratio in Eq. 4 are also given. All values are expressed in micrometres.

	d_1	d_2	d_3	d_4	d_5	ϕ
U_s	99	23	16	40	38	3
U(instr.)	10	5	11	11	11	2
U(μPIM)	99	23	12	39	37	2
ratio	10 %	20 %	97 %	28 %	31 %	78 %

	r_1	r_2	r_3	r_4	r_5	r_6	r_7	r_8
U	21	77	21	16	46	51	20	19
U(instr.)	3	8	6	6	7	4	4	3
U(μPIM)	20	77	20	16	46	50	19	19
ratio	15 %	10 %	33 %	4 %	16 %	9 %	23 %	17 %

Table 5. Shrinkage for each of the 5 green parts and average shrinkage.

	d_1/%	d_2/%	d_3/%	d_4/%	d_5/%	ϕ/%
sh_{g1}	28	29	28	30	28	23
sh_{g2}	29	29	30	29	29	27
sh_{g3}	28	29	30	26	30	26
sh_{g4}	29	29	35	27	33	26
sh_{g5}	28	29	32	28	29	26
Ave	28	29	31	28	30	26

	r_1/%	r_2/%	r_3/%	r_4/%	r_5/%	r_6/%	r_7/%	r_8/%
sh_{g1}	57	26	20	44	68	32	20	35
sh_{g2}	53	23	23	32	39	34	16	51
sh_{g3}	79	22	29	27	58	24	44	35
sh_{g4}	58	21	19	36	31	28	36	39
sh_{g5}	63	23	16	32	63	30	25	37
Ave	62	23	21	34	53	29	28	39

Table 6. Shrinkage propagated uncertainty for each of the 5 green parts and for the average shrinkage.

	d_1/%	d_2/%	d_3/%	d_4/%	d_5/%	ϕ/%
$u_{\%,1}$	1.6	0.4	2.7	5.1	3.5	0.6
$u_{\%,2}$	1.6	0.4	2.7	5.0	3.5	0.7
$u_{\%,3}$	1.6	0.3	2.7	4.9	4.3	0.7
$u_{\%,4}$	1.6	0.3	2.8	4.9	3.6	0.7
$u_{\%,5}$	1.6	0.4	2.7	4.9	4.2	0.7
u_{Ave}	1.7	0.4	4.2	5.0	5.8	2.0

	r_1/%	r_2/%	r_3/%	r_4/%	r_5/%	r_6/%	r_7/%	r_8/%
$u_{\%,1}$	44	12	2.5	13	29	30	6.5	25
$u_{\%,2}$	42	12	2.6	12	28	29	6.2	28
$u_{\%,3}$	51	12	3.0	12	27	27	9.9	25
$u_{\%,4}$	44	12	2.8	12	23	28	9.8	25
$u_{\%,5}$	46	12	2.6	12	30	28	6.9	25
u_{Ave}	51	12	9.7	18	41	28	23	26

Finally, the estimated variability due to sintering was

$$U_{proc} = \bar{d}_{snt} \cdot U_{proc,\%} / 100 \quad (7)$$

The safety factor h_s was chosen such that the average of a generic dimension $\bar{d}_{snt}$ of the sintered samples satisfied the following inequality, with a certain margin

$$\bar{d}_{snt} - U_{proc} + U_s < \bar{d}_{snt} < \bar{d}_{snt} + U_{proc} - U_s \quad (8)$$

where U_S is the overall expanded uncertainty of the sintered parts.

Eq. 8 takes into account the conformity zone defined in [15]. Henceforth, the two tolerance limits are

$$\Delta d_L = \bar{d}_{snt} - U_{proc} + U_s \quad (9a)$$

$$\Delta d_U = \bar{d}_{snt} + U_{proc} - U_s \quad (9b)$$

Consequently, tolerance limits were estimated according to the variability of the samples, i.e., such to be mainly due to the µPIM process, provided the chosen sets of samples were representative enough of the entire production.

The impact of the measuring process on the overall evaluation can be assessed as indicated in § 5. If the ratio in Eq. 4 (Tables 3 and 4) is not satisfactory, a different choice of the measuring instrument or a more accurate measuring session may be required.

Table 7. Green parts: average values in millimetres and experimental oversizing factors.

	d_1	d_2	d_3	d_4	d_5	ϕ
$\bar{d}_g$	10.210	9.703	0.783	1.233	1.779	0.605
$O.F.$	1.283	1.291	1.308	1.280	1.300	1.256

	r_1	r_2	r_3	r_4	r_5	r_6	r_7	r_8
$\bar{d}_g$	0.119	0.942	1.361	2.412	0.415	0.279	0.509	0.137
$O.F.$	1.619	1.230	1.213	1.341	1.525	1.293	1.282	1.394

The average values for all the green parts $\bar{d}_g$ and the ones of the oversizing factors, experimentally evaluated for each feature of size, are in Table 7. The average values $\bar{d}_{snt}$, related to the sintered parts, are in Table 8 together with the tolerance limits of Eq. 9a and Eq. 9b. The safety factors used are also reported in Table 8.

Eventually, it should be noted that the contributions due to the measuring process might also have been separated before the propagation of the uncertainties. Nonetheless, we believe that the suggested procedure is a more stringent choice. Eq. 3 and Eq. 5 are approximated estimations. Such approximations may have been enlarged by the propagation of the uncertainties.

Table 8. Sintered parts: average values, tolerance limits (in millimetres) and safety factors h_s.

	d_1	d_2	d_3	d_4	d_5	ϕ
Δd_L	7.939	7.504	0.577	0.942	1.287	0.466
$\bar{d}_{snt}$	7.958	7.517	0.599	0.963	1.369	0.482
Δd_U	7.978	7.530	0.621	0.984	1.450	0.497
h_s	1.10	1.00	1.00	1.00	1.00	1.00

	r_1	r_2	r_3	r_4	r_5	r_6	r_7	r_8
Δd_L	0.060	0.677	0.934	1.487	0.152	0.156	0.245	0.086
$\bar{d}_{snt}$	0.073	0.765	1.122	1.799	0.272	0.215	0.397	0.098
Δd_U	0.087	0.854	1.311	2.111	0.392	0.275	0.549	0.110
h_s	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.30

8. Discussion

The shrinkage of the linear dimensions (Table 5) seemed to be constant and the related propagated uncertainties (Table 6) confirmed this trend. Hence, lower tolerances could be specified (see Table 8) comparing to curved features (see below). Nonetheless, the impact of the measuring process on the current evaluation could not be considered negligible as can be seen in Table 3 and Table 4, from the uncertainty contributors and, in particular, from the ratio specified in Eq. 4.

An explanation of this contradiction might be that linear dimensions were indirectly measured using the centres of the curvatures of curved features as inputs, amplifying the measuring errors. The shrinkage non-uniformities, in fact, increased when considering curved features (Table 3 and Table 4), perhaps because of an anisotropic sintering of two-dimensional features. The Eq. 4 ratio was less critical but, again, the measuring process could not be neglected in many cases.

The effect of the improper measuring session can result in wrong estimated tolerances in which, i.e., the variability is mainly affected by the measuring process that overhangs the one related to the production. Eq 4 was used to evidence such behaviour.

To conclude, comparing the average results in Table 8 with the nominal values expected in Table 1, the effort for allocating the correct sizes to the green parts can be understood. Therefore, analysing the results calculated according to the proposed method, a clear understanding of the specific μPIM process was possible.

9. Conclusions

The proposed method aims to estimate possible tolerance specifications for a μPIM production, in a systematic approach.

The measuring and the production processes can be separated, reducing the complexity of analysing the overall μPIM process and, concurrently, evidencing limitations and advantages of the selected measuring system.

Consequently, an oversize factor can be determined for each feature of size, allowing for a more accurate dimensioning of the sizes of the green parts, in order to obtain the correct dimensions after sintering.

Finally, tolerances can be estimated propagating the expanded uncertainty to the shrinkage, i.e., according to the variability of the samples used as representative model of the entire production.

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