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Master Sintering Curve Constructing Techniques: A Review

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Abstract

Additive manufacturing (AM) is a quickly expanding technology that has various uses in different fields. Many AM-techniques involves stages in which diffusion processes play an important role. One of such stages is the sintering, which means consolidation of a part blank with low density to a dense final part. Because of this, it is precious to forecast a result of sintering. The master sintering curve (MSC) is a useful technique for calculating the final density of a part, in dependence on the process parameters. This paper reviews theoretical base of the technique and the ways to realize it. Existing realizations as a code are reviewed as well. The paper summarizes most approaches to build the master sintering curve and it aimed to be the base to construct a software and to enhance the technique as well.

Keywords: Master sintering curve, ceramics, additive manufacturing

1. Introduction

Sintering is a heat treatment process where atoms or particles of a solid material are fused together at high temperatures without melting to form a new, denser material. It is a crucial step in the production of many industrial products, including ceramics, metals, and composites.

The advanced ceramics is a class of materials that owing a set of outstanding mechanical and biocompatible properties, that made it very attractive to use in various areas, including implant constructions for reconstructive surgery due to good immune-acceptance [1]. It is hard, wear-resistant, brittle, prone to thermal shock, refractory, electrically and thermally insulative, intrinsically transparent, nonmagnetic, chemically stable and oxidation-resistant [2]. It finds use in biomedical applications, serving as a material for various implants. Because of properties a machining of ceramics is low productive. Thus, new technologies, like additive manufacturing, can produce implants of various shape, which allows to increase the personification of such products.

Additive manufacturing of ceramic materials involves several stages, including layer-by-layer photopolymerization, debinding and sintering [3]. The last of them is the most important as at the end of the stage we get the final geometry. During the process, a so-called green body, that results from the previous two stages, is heated to a high temperature and dwelled for some time. Diffusion phenomena lead to coalescence of ceramic particles and, as a result, to densification of a whole sample. As result of the process, the volume of a sample shrinks down to 70-80% of initial value. To get desirable shape, it is essential to have a way to predict such changes.

The kinetics of sintering are under study for a very long time. Because of this, several methods to calculate the value of shrinkage of a sample can be applied. Most of them are based on finding the activation energy value of the Arrhenius equation. Conception of a master sintering curve (MSC) is one of them. A master sintering curve is a graph that shows the relationship between the sintering temperature and the sintering time of a material. It can help in determining the optimal sintering parameters for the material, such as temperature and time, to achieve the desired properties. The master sintering curve theory is based on several works. DeHoff developed a stereological basis of the method [4,5]. Hansen et al. have derived a single equation for the sintering process [6]. Su and Johnson proposed the final shape of the method [7].

The analysis of publication activity shows that the method is actively used (see Fig.1) and for the 2023 year there are 13 papers already published (Figure 1). Currently, various researchers use the master sintering curve as a very handy tool to predict the resulting density of sintered samples.

Historically, the MSC approach developed for powder metallurgy needs and it still remains actual. Now it is used to simulate and optimize the sintering process of various metals and alloys: uranium dioxide [8-10], tungsten [11], dispersion strengthen alloys [12], nanocrystalline materials [10,13-15] and etc. The last one is very sensitive to temperature treatment and thus it is very valuable to choose appropriate heating modes that allow to keep nanocrystalline nature of a powder. MSC method allows to do this [13,15]. Some authors propose two-stage [12] and multi-stage [16, 17] approaches as well. This case considers constructing MSC for all specific temperature intervals separately. Such techniques allow to increase predictive ability of the method. Multistage MSC is a best choice for powder with dissimilar grains or materials especially.

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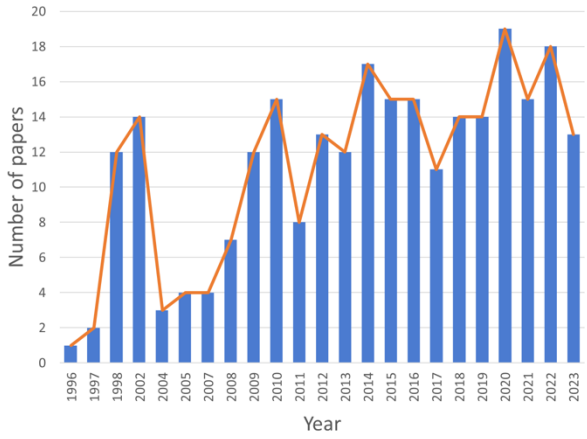

Fig. 1. The number of papers that mentioned the master sintering curve technique. Data were got by the ScienceDirect service.

Another big area for MSC using is ceramics [18-22]. As it is said before, traditional shaping methods, like machining, are not suitable for production of ceramic parts. Thus, casting and sintering are the main useful ways to produce ceramics with complex shape [2, 23]. But due to features of processes and material itself, compliance with geometric tolerance is tricky. Then as master sintering curve technique allow to predict geometric changes then, by solving the inverse problem, it allows to determine the shape of a casting mould or the dimensions of the green body before sintering. The MSC is very valuable for a new type of ceramic, which have been developed recently and due to this has a very brief experimental description yet [22].

Sintering process of modern complex composites which are mixture of ceramic and metals particles also can be described by MSC [24, 25]. For these materials more suitable multistage approach as a powder for a composite production contains dissimilar materials.

Then we can conclude, that master sintering curve technique has a wide range of application in producing parts of advanced materials. Due to this, it can be interesting to summarize basis, approaches and possible/existing realization of the method.

Our review presents the state-of-the art for the basics of the master sintering curve theory, and summaries the knowledge about the sintering curve construction. There are papers that try to summarize the master sintering curve approach [26-28] already. The last of them was published in 2018. As we can see form Figure 1, interest to the method has increased, then we suppose that a fresh review with summarizing new ideas can be interesting and maybe valuable for researchers.

2. Theoretical basis

To understand applicability and limits of the master sintering curve approach, we need to know its suggestions and assumptions. Below we provide brief theoretical description of the method.

Green-body is shaped sample, that got during additive manufacturing and processed in the oven to eliminate a binder agent. Green-body's powder grains contact to each other. As DeHoff has proposed [4], we can divide a sample into polyhedral cells that contain material (a grain of the powder), pores and contact areas of two grains. The last one DeHoff propose to describe as a plane (see Fig.2a). It is valuable to point out, that at the beginning a contact area is very small and will be increased during sintering process. Any polyhedral cells can be represented as a collection of pyramids with the common central vertex, that will be the center of a cell. The center of each cell can be chosen freely, but usually it is a centroid (see Fig.2b). Let us choose in two neighbor cells (a cell with number 1, and cell with number 2) pyramids (j) that has common base surface and join them into bipyramid. Then the change in volume of the pyramid in cell #1 that is associated with shared face j can be expressed:

$$dV_{1j} = A_j dp_{1j} \tag{1}$$

here V_{1j} – volume of the pyramid j of the cell 1, A_j – area of the base of the pyramid j, p_{1j} – distance from the centroid to the base of the pyramid j. In other words, change of a cell #1 volume (and all cells in a body) will be determined by moving of all pyramid' basis. The DeHoff theory is limited to a linear case only. It claims, that motions of faces that result in rotations or expansions contribute higher order differentials to the volume change. He supposed that vacancy annihilation contributes to the volume changes only. By summing volume changes of bipyramids, DeHoff derived well-known expression for changes of a whole sample volume:

$$\frac{dV}{dt} = -\Omega \bar{f}(D_V \overline{\nabla C}_s S + D_b \overline{\nabla C}_b bL) \tag{2}$$

here Ω is atomic volume; $\bar{f}$ is average efficiency factor; D_V and D_b are volume and grain boundary diffusion coefficients correspondingly; $\overline{\nabla C}_s$ and $\overline{\nabla C}_b$ are average concentration gradients of vacancies in a volume and at a boundary; b is the thickness of the zone of enhanced diffusivity, roughly of an atomic dimension; L is the length of triple line (grain$_1$–grain$_2$–pore).

The DeHoff model suggests that sintering temperature has a little effect on the path of structural changes, as long as sintering is performed near melting temperature [4]. And it has proposed: as purely geometric factors in Eq.2 are invariant, as a comparison of densification rates at different temperatures may be undertaken.

The next step was made by Su and Johnson [7, 29]. They developed an isotropic sintering model and derived the equation for the shrinkage rate:

$$-\frac{1}{L}\frac{dL}{dt} = \frac{\gamma\Omega}{k_B T}\left(\frac{D_V \Gamma_V}{G^3} + \frac{bD_b\Gamma_b}{G^4}\right), \tag{3}$$

here L is a linear size of the sample, γ is a surface energy, k_B is Boltzmann's constant, D_b, D_V are a grain-boundary diffusivity and a volume diffusivity correspondingly, G is a powder grain diameter. Some other parameters are illustrated by Figure 3. The isotropic nature of a shrinkage was described as:

$$\frac{1}{V}\frac{dV}{dt} = 3\frac{1}{L}\frac{dL}{dt} = -\frac{1}{\rho}\frac{d\rho}{dt}, \tag{4}$$

here V is a volume of a sample that is equal to the sum of all pyramids' volumes, ρ is the bulk density (or relative density).

As we can see, Eq.2 and Eq.3 are very similar. But we still need to explain values Γ_b and Γ_V. They are cumulative values, that summaries dependences of various parameters on grain diameter, and this is one of the point of the model. (Full derivation of coefficients in Eq.5 you can found at work [7]).

$$\Gamma_b = \frac{\alpha C_K C_b}{C_\lambda C_h C_a}, \qquad \Gamma_V = \frac{\alpha C_K C_V}{C_\lambda C_h C_a}. \tag{5}$$

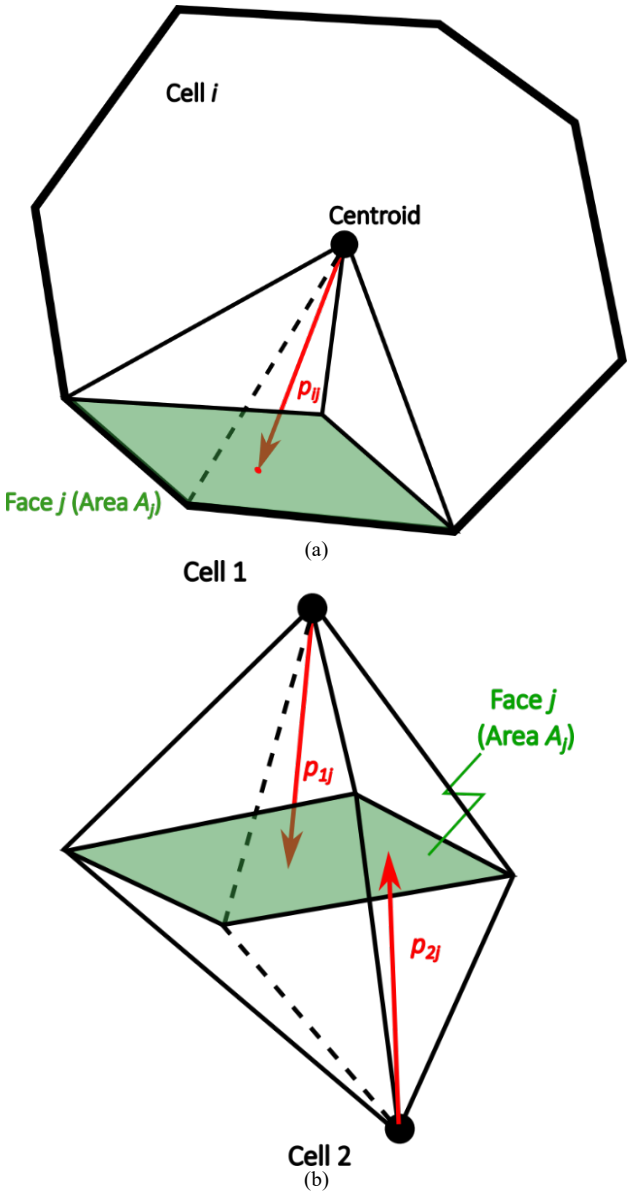

Fig.2. (a) cell of a sample volume and (b) bipyramid construction associated with each face in the cell structure [2].

Here C_i are coefficient that determine dependence of following values on grain diameter (G):

$$\begin{aligned} K &= -\frac{C_K}{G}, & \lambda &= C_\lambda G, \\ \frac{1}{2}L_b &= C_b G, & h &= C_h G, \\ A_V &= C_V G^2, & A_b &= C_a G^2. \end{aligned} \tag{6}$$

Here K is a curvature of a pore surface, λ is the distance over which material is drawn to the pore, L_b is the total grain-boundary–pore intersection length, h is the mean centroid-to-base distance, A_b is the grain-boundary area of the cell, A_V is the area of volume diffusion of the cell. The expression in Eq.6 just describe that most values have linear or quadratic dependence on grain size. As it is well-known, real powder has grains with size that corresponds to some distribution (generally Gaussian distribution), but the model suppose that all grains are same size, usually it is equal to average grains' size. This is simplification of the model, that leads to non-universality of MSC in connection to size of particles.

Two terms in brackets of Eq.2 or Eq.3 describe grain-boundary diffusion (subscript b) and volume diffusion (subscript V). Many authors suppose that at a moment of a sintering process, one of two mechanisms is prevailed. Then the Eq.3 can be simplified by eliminating one of two terms. By using the Arrhenius equation for the diffusion, we can rewrite Eq.3 as [7]

$$\frac{1}{3\rho}\frac{d\rho}{dt} = \frac{\gamma\Omega}{k_B T(t)} \cdot \frac{\Gamma(\rho) D_0}{\left(G(\rho)\right)^n} \exp\left(-\frac{Q}{RT(t)}\right) \tag{7}$$

where Q is the apparent activation energy, R is the gas constant, $D_0 = (D_V)_0$ and $n = 3$ for the volume diffusion and $D_0 = (bD_b)_0$ and $n = 4$ for the grain-boundary diffusion. We can see that the equation allows to divide variables:

$$\frac{k_B}{\gamma\Omega D_0} \cdot \frac{1}{3\rho} \frac{\left(G(\rho)\right)^n}{\Gamma(\rho)} d\rho = \frac{1}{T(t)} \exp\left(-\frac{Q}{RT(t)}\right) dt \tag{8}$$

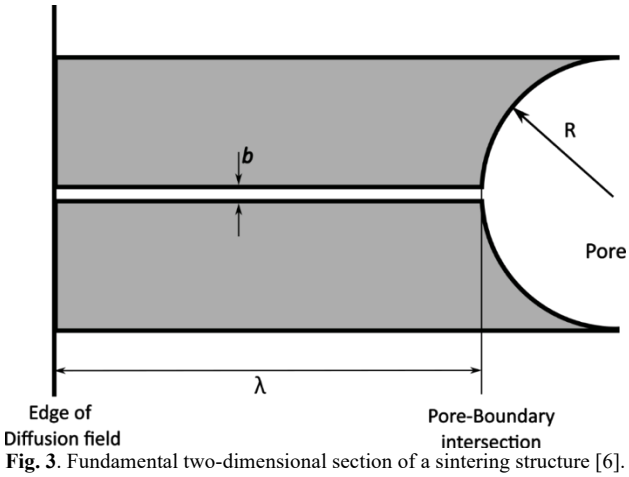

Fig. 3. Fundamental two-dimensional section of a sintering structure [6].

The left-hand side of the Eq.8 represents changes of density of a sample during sintering and independent on time. The right-hand side describes the history of the process and depends on temperature and on the value of the activation energy. The more general form of the Eq.8 is:

$$\begin{aligned} \Phi(\rho) &= \Theta\big(t, T(t)\big), \\ \Phi(\rho) &\equiv \frac{k_B}{\gamma\Omega D_0} \int_{\rho_0}^{\rho} \frac{1}{3\rho} \frac{\left(G(\rho)\right)^n}{\Gamma(\rho)} d\rho, \\ \Theta(t,T) &\equiv \int_0^t \frac{1}{T(t)} \exp\left(-\frac{Q}{RT(t)}\right) dt \end{aligned} \tag{9}$$

Here $\Theta(t,T)$ is the work of sintering. The relation $\rho(\Theta)$ for the optimal value of Q is defined as a master sintering curve (MSC) [7].

Summarizing the above mentioned theory, we can write all assumptions that lies in base of the MSC theory:

1. all grains are same constant size;
2. only one diffusion mechanism is prevailed;

3. grain boundaries are moving along inter-centroid lines only.

These assumptions consider valuable simplification of the model. We can see, that MSC is built for specific material and grain size only. In case of using same material powder but with different average grain size, MSC should be built from a scratch. Thus, MSC has a problem of transferability of results.

3. Algorithms of the master sintering curve constructing

Let us see variations in master sintering curve construction.

First, the master sintering curve approach demands experimental data. We need results of a minimum of two (4 or 5 will be better) experiments [7] of samples sintering with different heating rates using dilatometry. Usual table with data contains three columns: density (ρ), temperature (T) and time (t). On the base of these data, we should build dependence $\rho(\log\Theta)$, that gives us sintering curves. Thus, to construct the MSC, we have to choose two expressions: an approximation of MSC shape and a minimization criterion. This section reviews the known variants of them.

3.1. Approximations shape of the master sintering

Dependence of the density of a sample on temperature usually has S-like shape (see Fig.4). Then we have to choose the expression to approximate it. Then, by fitting the activation energy value in order to overlap all these curves, we can get a master sintering curve.

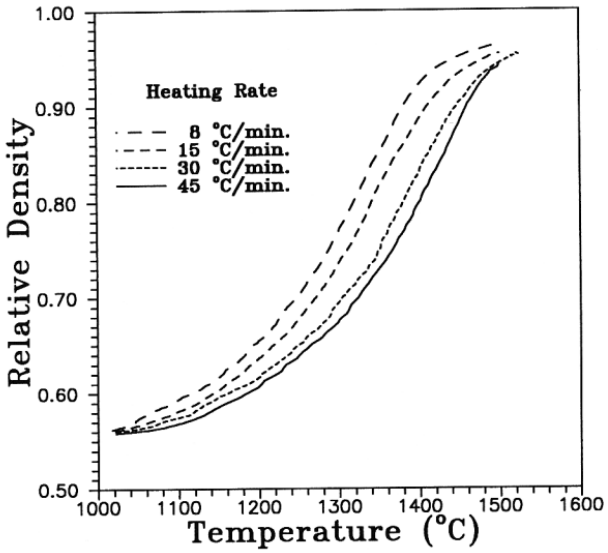

Fig. 4. Basic sintering results from the model experiments [7].

In the original work [7], authors proposed to use polynomial approximation. At most other works to approximate MSC authors used sigmoid function, but with a various set of parameters.

Authors of works [30-33] are used the expression

$$\rho = \rho_0 + \frac{a}{\left[1 + \exp\left(-\frac{\log(\Theta) - \log(\Theta_0)}{b}\right)\right]^c} \quad (10)$$

here ρ_0 is the density of a green-body (initial density), Θ_0 is the value of Θ at the point of inflection of the curve. Parameters a, b and c are determined for each experimental set of data by the Levenberg-Marquardt method.

At works [34, 35] another expression was used

$$\rho = \rho_0 + \frac{1 - \rho_0}{\left[1 + \exp\left(-\frac{\log(\Theta) + \alpha}{b}\right)\right]}, \quad (11)$$

here α and b are fitting parameters as well. We can see that Eq.10 can be reduced to Eq.11 by assuming $\log(\Theta_0) = -\alpha, c = 1, a = 1 - \rho_0$. Then these two functions have the similar nature. Such approach is very popular. Many researchers use the custom form of Eq.10 to represent their own shape of a fitting function by rewriting constants in Eq.10.

At work [36] one more expression was introduced:

$$\rho = \rho_0 + \frac{\rho_{fin} - \rho_0}{\left[1 + \left(\frac{\Theta(t)}{\Theta_m}\right)^{-h}\right]^c} \quad (12)$$

here ρ_{fin} is the final density after sintering; h, c and Θ_m are fitting parameters. We can conclude, that authors try to consider, that the final relative density is not equal to 1.0, as it stated in the Eq.11. Then, by assuming $\Theta_0 = \Theta_m, h = 1, a = \rho_{fin} - \rho_0$, we can establish a strong connection between Eq.10 and Eq.12.

On the base of above mentioned, we can conclude that Eq.10 is the most general form of approximation of the density of a sample dependence on the work of sintering.

Thus, the sigmoid representation of the master sintering curve is a good choice, but it is not an exclusive way. Kiani et al [37] proposed new piecewise representation of the MSC that is very suitable for finite element analysis. By their method, MSC has divided into many sections, that called elements (see Fig.5). Then we can choose two or more neighbor nodes (it is determined by approximation order) and construct piecewise representations of a MSC region. Experimental data and shape functions ($N_i(\Theta)$) are used for this in accordance with the classic finite element method.

For example, for the highlighted region at Fig.4 we can write the following [37]:

$$\rho = \sum_{i=1}^{3} \rho_i N_i(\Theta)$$

As we use three nodes as a quadratic approximation is used. Then shape functions can be written:

$$N_1 = \frac{(\Theta - \Theta_2)(\Theta - \Theta_3)}{(\Theta_1 - \Theta_2)(\Theta_1 - \Theta_3)},$$

$$N_2 = \frac{(\Theta - \Theta_1)(\Theta - \Theta_3)}{(\Theta_2 - \Theta_1)(\Theta_2 - \Theta_3)},$$

$$N_3 = \frac{(\Theta - \Theta_1)(\Theta - \Theta_2)}{(\Theta_3 - \Theta_2)(\Theta_3 - \Theta_1)}$$

On next step, an initial value for the activation energy should be chosen and then the sintering work values Θ_i (see Eq.9) are calculated using any numerical integration method. And by iterative method, we can find the optimal value of activation energy.

This does not involve hard optimization techniques such as Levenberg-Marquardt, it is easy and compatible with finite element analysis.

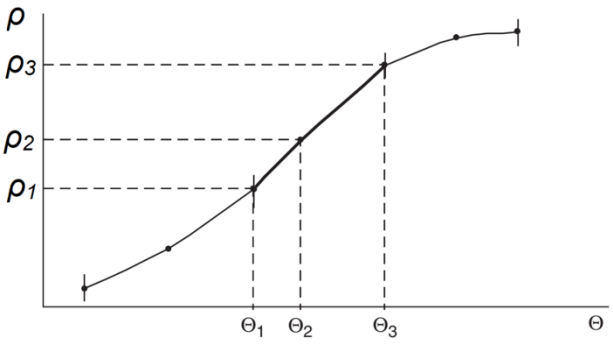

Fig. 5. Piecewise approximation for a master sintering curve [19]

We can conclude that at this stage there are only two well-known approaches to approximate master sintering curve shape: sigmoid and linear piece-wise representation. There are some other methods [35,38] that allow to use linear approximations, but they realize approach that differs from master sintering curve.

3.2. Minimization criterions

As it said before, the finding the optimal value of an activation energy is an iterative process. Because of this, we need to choose a criterion to stop iterations. Most using approach is to use mean residual square (MRS) for this [13,30,31,39]:

$$MRS = \sqrt{\frac{1}{\rho_{fin} - \rho_0} \cdot \frac{1}{n} \int_{\rho_0}^{\rho_{fin}} \sum_{i=1}^{n} \left(\frac{\Theta_i}{\Theta_{avg}} - 1 \right)^2 d\rho}$$

Here ρ_0, ρ_{fin} are initial and final relative density correspondingly, Θ_{avg} is the average value of all Θ_i for a given density, n is a number of heating profiles used.

Pouchly and Maca [40,41] argued that due to the sigmoid shape of the master sintering curve, the distance between curves can be overestimated, especially at the start and end (Fig.5a). Then they proposed another criterion – Mean Perpendicular Curve Distance (MPCD). The criterion can be expressed as:

$$MPCD = \frac{1}{\rho_{fin} - \rho_0} \int_{\rho_0}^{\rho_{fin}} \sum_{i=1}^{n} PD_i(\rho)\, d\rho$$

where the perpendicular distance (PD) of individual curves is evaluated (Fig. 6b) instead of horizontal ones (Fig.6a). This approach is more complex from the calculation's point of view, but can produce more accurate results.

Fig.7 shows an example of an evolution of both criteria during the iterative process of the optimal value of activation energy finding. We can see that MPCD has more expressive minima.

Thus, we can conclude that existing minimization criterion realize to evident ideas:

- to use constant value of density and thus reduce calculation costs but obtain unstable values at curves plateaus;
- to use shortest way between curves at each point, that increases calculation costs but stabilizes results.

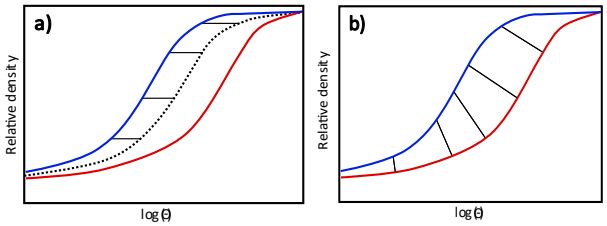

Fig.6. Comparison minimization criterions: a) MRS, b) MPCD [21].

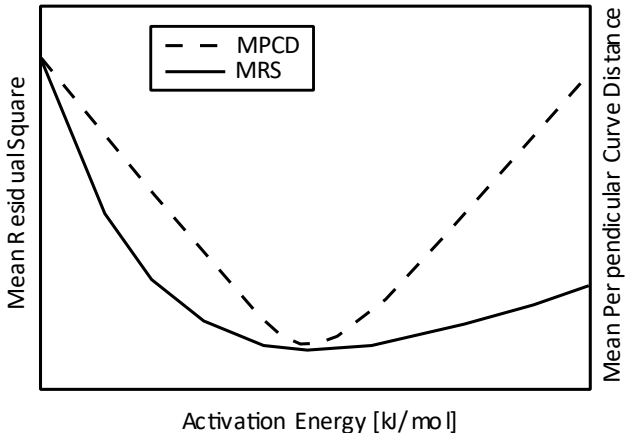

Fig. 7. Comparison evolution of two criterions with activation energy value [21].

3.3. Software for MSC calculations

The Master sintering curve is a good technique to predict shrinkage of a green-body during sintering. We have shown that there are some approaches to building it. And all of them consider sufficient calculations. Then it is obvious that researchers tried to build a tool to make such calculations in a quick and convenient manner.

The first such software was described in paper [30]. Authors use Microsoft Excel as a Table processor and code, that realizes MSC technique, in Visual basic for Applications (VBA). They used sigmoid approximation for the $\rho(\log \Theta)$ and MRS to find optimum of apparent activation energy. As a result of calculations with this simple tool, a user get value of Q and two plots: MSC itself and MRS(Q). As the final result, a user can build the contour map of the prediction of a sintering time. The paper has flow charts of algorithms and examples of a simulation as well. But the tool is inaccessible now.

Seong Jin Park published the description of software [39,42] for constructing a master sintering curve at the ResearchGate portal. To find an apparent activation energy, it uses two approaches: minimization of MRS and regression-based statistics. As an additional case, the software uses the linearized variant of the MSC. It enables the prediction of relative density and grain size. The software allows to optimize sintering cycle, and the paper contains examples of such a process. The software allows to construct master sintering surface as well. Authors propose to use the software results in FEM simulations of the sintering process.

Unfortunately, we couldn't to find any mentions for realizations of the software and the software itself. It is sad, as the software is very promising and can be a useful tool in numerous areas of additive manufacturing.

To sintering of ThO_2 pellets, Aditi Ray et al. developed software with FORTRAN programming language [43]. The paper contains a very detailed description of the algorithm and some generalization of the technique. But as in previous cases the software is unavailable.

One of the most advanced software was developed by V. Pouchly and K. Maca [40]. The application uses two different approaches to find an apparent activation energy value (MRS and MPCD) and the last version [44] incorporates Wang and Raj model [38] to find it. The software has friendly and intuitive interface and allow to export results to Microsoft Excel. And we couldn't find any possibility to download the software also.

R.M. Batista et al. developed software, *sintering fit*, using Matlab platform [45]. Above constructing the master sintering curve, the software allows to build sintering map and analyze evolution of a density with varying parameters of heating cycle. Happily, the source code of the software is accessible at GitHub (https://github.com/morgadoph/SinteringFit).

This short review of existing tools that use the master sintering curve approach shows that all of them are created in a certain research group as an internal software. Some of them can be found on GitHub as a source. But there is no handy tool that is ready to use from the box. And more important, there is no software that can work with CAD-models and allow predict shape changes during sintering. Such a tool will be precious for both researchers and a real production.

4. Conclusion

The paper reviews the master sintering technique to determine an apparent activation energy value in order to predict final density of a sample at the end of a sintering process. The main conclusions can be summarized as follows:

1. The Master sintering curve (MSC) approach has some limitations that should be considered in a research activity. Same time, accurate using the approach allow to predict shrinkage of a sample with adequate accuracy.
2. Sigmoid function (Eq.10) describes the most general case to approximate the S-like shape of a dependence of a blank density on a sintering work (or time). For FEM simulations a piece-wise representation is used as well.
3. There two general minimization criteria are used: Mean Residual Square (MRS) and Mean Perpendicular Curve Distance (MPCD). The first one is quicker and simple in realization as a code, but the second one is more accurate at the beginning and ending of the sintering process, when density changes with time or with temperature very slowly.
4. Existing software are tools that were created and used for a certain research group and only one of them is accessible. Most of code, that were described in existing papers, is inaccessible. We have found only one realization and it is accessible as a source code without documentation. Then there is no any software that use MSC approach and ready to use from a box.

As a result of the work, we plan to develop new software that will incorporate the most valuable and fruitful variants of the MSC theory and other approaches. This new tool should allow to predict not only the change in density, but the shape of bodies as well.

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