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Temperature effect on strength evolution under sintering

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Abstract

The evolution of strength at the initial stages of sintering is considered both theoretically and experimentally. Theoretically, the strength properties are assessed accounting for hardening due to the growth of the interparticle necks during sintering and softening due to the increase of the temperature. Experimentally, the strength properties are determined for steel powder specimens using a three-point bending test simultaneous to sintering. The calculated and the experimental results are compared and possibilities of the elaboration of a criterion of the engineering strength under sintering are discussed. Both the model and the experiments for sintering under bending yield values for material strength that should be intermediate between the strength of a powder compact during free sintering and the strength of a finished sintered product. © 2001 Elsevier Science Ltd. All rights reserved.

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1. Introduction

1.1. Engineering strength of sintered materials

The evolution of the strength of a particulate material during sintering is one of the most important characteristics in powder processing. While a great number of works have been dedicated to the analysis of the strength of the finished sintered product [1], the strength of

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powder components during the on-going sintering process has been studied to much less of a degree. Despite the intensive development of sintering models, including the recently elaborated continuum approach [2,3,4,5,6,7], presently, the main information, which a technologist can obtain from modeling, is the data on dimensional change, stress-strain conditions and porosity (density) distribution. The engineering strength properties of powder components during sintering are also of considerable importance, including the analysis of crack formation, various defects nucleation, *etc.* In general, the prediction of failure and damage is of utmost significance in sintering practice*.

Thus, one of the actual problems of modern theories of sintering is the development of criteria for engineering strength (“in-sintering strength” of material). These criteria should supplement the existing models and computer codes for a more detailed description of certain processes of sintering. The elaboration of such criteria is a multi-task problem, because material strength is determined by a large number of factors such as regularities of contact and interparticle neck formation, the evolution of material properties, influence of heterogeneities, *etc.*

In the present work, an analysis of an intermediate problem is proposed: an investigation of the strength accumulation under sintering. From the experimental viewpoint, the main idea is an application of an external destructive load to a powder specimen during sintering and the consequent measuring of the failure stress. From the theoretical viewpoint, the analyzed strength properties are intermediate between the after-sintering and the in-sintering strength, because the damage is obtained under an external load, which is absent during free sintering; along with this, the damage is obtained in temperature conditions which are close to those in sintering.

As it is shown in the present work, a combination of the experiment and the model enables a comparison of various concepts for neck formation and damage.

1.2. A brief review of works related to the effect of temperature on strength of sintered materials

The effect of sintering atmosphere [1,8], time of sintering [1,9], powder alloying [1] on the strength of sintered parts have been widely investigated. In contrast, the temperature influence on the accumulated strength of sintered materials [8,10,11] was studied in a limited number of publications. Dunmore and Smith [8] analyzed the dependence between the temperature, structure characteristics and mechanical strength in tension of sintered copper powder specimens. It was indicated that sintering in the temperature range of 300–500°C led to an increase of the tensile strength. However, the microhardness decreased. The authors related this effect to a simultaneous strengthening of bonds between particles on the one hand, and the recrystallization and softening of the individual particles on the other hand. After about 600°C the tensile strength decreased, and the authors related this phenomenon to grain growth and the expansion of gas trapped within closed pores.

* Here it is pertinent to determine the main purpose of most of sintering processes in the increase of the strength rather than density of a powder component.

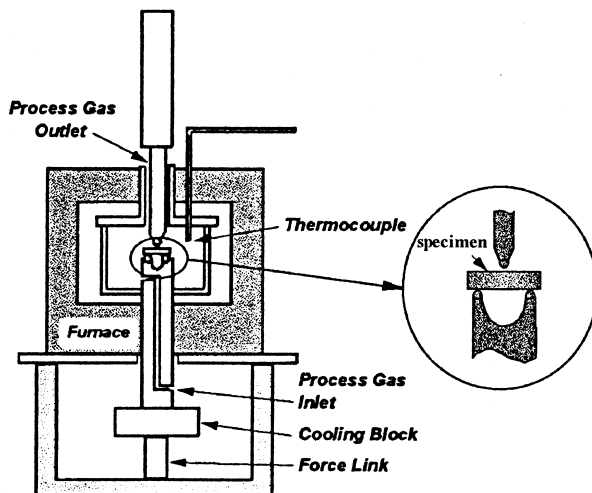


Fig. 1. Flaming tensile tester.

Fors [10] analyzed the tensile strength of electrolyte iron powder as a function of sintering temperature. A non-monotonous character of this curve has been pointed out. This was explained by an allotropic transformation in iron. In such a case interparticle bonds developed more rapidly in α -iron than in γ -iron. Goetzel [11] noted an increase in tensile strength and the nonmonotonic behavior of the microhardness with temperature for copper powder specimens sintered in the temperature range of 500–900°C.

It is noteworthy, that all the above-mentioned works included studies of strength of sintered specimens *after the sintering process had been completed*. Also, almost no quantitative theoretical analysis of the question has been developed.

In the present work, the damage stress applied *in-situ* during sintering is analyzed. This enables the consideration of the material properties as close to technological (in course of sintering) properties.

2. Experimental

2.1. Equipment

For the destructive evaluation of the strength of powder specimens, the flaming tensile tester (FTT) was used (see Fig. 1). The construction details have been described in Refs. [12,13]. As shown in Fig. 1, a prismatic powder specimen rested on two cylindrical supports in a retort. The furnace was controlled by a K-type thermocouple located coincident with the specimen. This permitted very accurate control of the sintering thermal profile. For the experiments, all heating cycles were performed at 10°C/min. The process atmosphere was one hundred percent hydrogen. The damage stress was measured *in-situ* by performing the standard 3-point bend test (Fig. 1) at the specified test condition during the sintering cycle.

Table 1
Expressions for tensile strength σ_c as a function of porosity θ

Eudier [15]	Zheng <i>et al.</i> [16]	Ryshkewitch [17]
$\sigma_c = \sigma_{co} \left(1 - 1.21 \cdot \theta^{\frac{2}{3}} \right)$	$\sigma_c = \sigma_{co} \left[\left(\frac{0.381 - \theta}{0.381} \right)^{1.3} \left(1 - \theta^{\frac{2}{3}} \right) \right]^{\frac{1}{2}}$	$\sigma_c = \sigma_{co} \exp(-m \cdot \theta)$

In accord with the MPIF Standard 41 [14], the distance between the supporting rods was $L = 25.4$ mm (1 inch), the radius of the supporting rods and the radius of the indenter contact surface were $r_s = 3.1$ mm.

2.2. Materials

The prismatic specimens (rectangular transverse rupture bars) were compacted from the stainless steel SS304L powders with an average particle size of $70 \mu\text{m}$. The specimens were compacted to approximate relative densities of 0.68. Zinc stearate die wall spray was the only lubricant. In conformity to the MPIF Standard 41 [14], the test specimens had the dimensions of $a = 31.7$ mm \times $b = 12.7$ mm \times $c = 6.35$ mm.

3. Model concepts

The model approach developed here for the description of the damage of stainless steel powder specimens assumes the combination of two factors: evolution of the interparticle contact areas and softening of the particle material due to the increase of the temperature.

3.1. Tensile strength criterion

A large number of models have been developed for the description of the strength of powder materials as a function of porosity. The expressions for the tensile strength derived by Eudier [15] (using a geometrical analysis), Zheng *et al.* [16] (using a percolation theory), and Ryshkewitch [17] (using experimental data) are given in Table 1 (σ_{co} is the tensile strength of a fully-dense material). The relationships given in Table 1, are plotted in Fig. 2 in the form of the relative strength (σ_c/σ_{co}) dependence on porosity θ . The constant m in the Ryshkewitch model is assumed to be $m = 4$.

It should be noted that the above-mentioned models do not include the interparticle neck radius which is an important microstructure parameter. At the earlier stages of sintering, the strengthening of a powder specimen can occur only due to the increase of the interparticle contact area whereas porosity is almost unchanged [18]. The mechanism of surface diffusion, which dominates at the initial stage of sintering, does not cause any substantial shrinkage of the powder specimen. This known phenomenon is confirmed by the results of the present

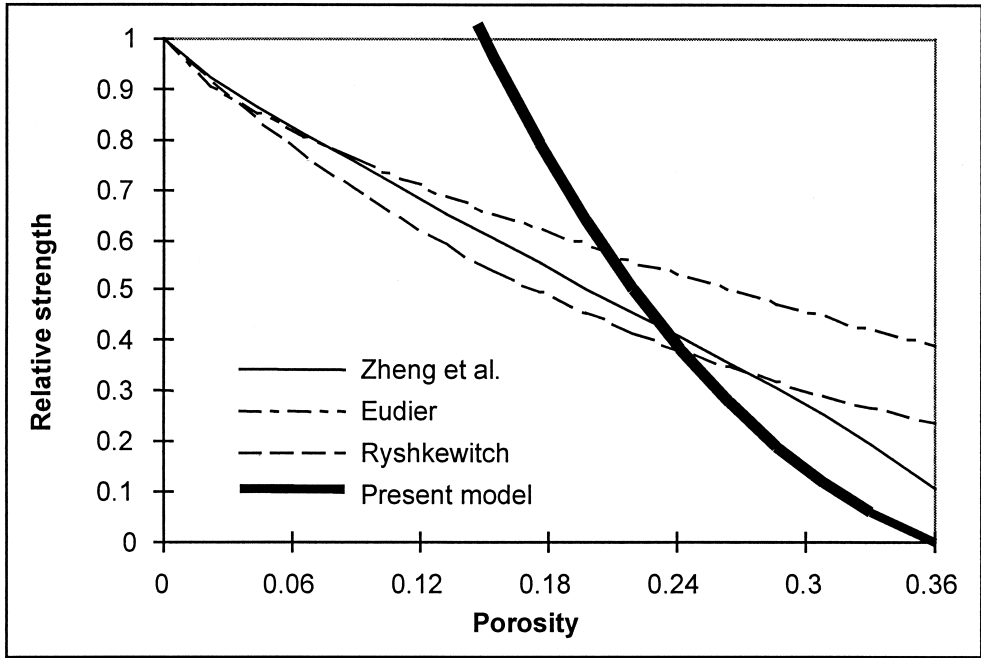


Fig. 2. Relative strength σ_c/σ_{co} of powder materials as a function of porosity θ .

experiments which indicate only a small change of porosity of about 4%. In this connection, at the early stages of sintering, change of porosity cannot be the only governing factor which determines the evolution of the strength of powder material.

An appropriate model should explicitly include interparticle contact characteristics (e.g. the relative neck radius r/R , where r is the neck radius and R is the particle radius). The above-mentioned strength criteria (Table 1) cannot be used for evaluation of the possibility of damage at the initial stage of sintering (when failure is highly probable) as they describe properties of *finished sintered* products.

Apparently, the only group of models, which explicitly include interparticle contact characteristics, are the models developed for the evaluation of the strength of agglomerates [19,20,21]. Here we use the Rumpf [19] relationship for the tensile strength of the powder compact:

$$\sigma_c = \frac{9(1 - \theta)Z}{32\pi R^2} f \quad (1)$$

where Z is the coordination number.

The unknown parameter f is the interparticle force. This value can be obtained using the solution of Davidenkova and Spridonova [22] for the field of stresses in the neck area of an isotropic material. This solution can be represented in terms of the particle (R) and the neck (r) radii and the neck curvature radius (r_p) as follows:

Table 2

Dependence between the relative interparticle neck radius and porosity

Skorohod (initial stages of sintering) [24]	Skorohod (all stages of sintering) [24]	Helle <i>et al.</i> [23]
$\frac{r}{R} = 2\sqrt{\frac{1}{3}\ln\left(\frac{\theta_o}{\theta}\right)}$	$\frac{r}{R} = \sqrt{1 - \left(\frac{\theta}{\theta_o}\right)^{4/3}}$	$\frac{r}{R} = \sqrt{\frac{1}{3}\left(1 - \frac{\theta}{\theta_o}\right)}$

$$f = \frac{\pi r^2}{2} \frac{4 + r/r_p}{2 + r/r_p} \sigma_{\max} \quad (2)$$

where σ_{\max} is the maximum normal stress in the interparticle neck, and it can be assumed that $\sigma_{\max} = \sigma_{co}$ if neck damage occurs.

The neck curvature radius r_p can be determined at the initial stages of sintering by the relationship derived by Helle *et al.* [23]:

$$r_p = R(\theta_o - \theta) \quad (3)$$

where θ_o is the initial porosity.

Substituting (2) and (3) into (1), we have:

$$\sigma_c = \frac{9(1 - \theta)Z(r/R)^2}{64} \frac{4(\theta_o - \theta) + r/R}{2(\theta_o - \theta) + r/R} \sigma_{co} \quad (4)$$

The latter expression can be used as an accumulated strength criterion for the initial stages of sintering. It satisfies the limiting transition $r/R \rightarrow 0$. Formula (4), which explicitly includes the relative neck radius r/R and porosity θ as independent parameters, does not satisfy the limiting transitions $r/R \rightarrow 1$ and $\theta \rightarrow 0$. This is understandable, because the relative neck radius r/R and porosity θ should be interrelated at the later stages of sintering when the shrinkage-causing mechanisms (such as volume or grain-boundary diffusion) act. Table 2 includes expressions for the dependence between the relative neck radius r/R and porosity θ derived by Skorohod [24] (for the initial and for the entire range of sintering) and by Helle *et al.* [23].

The relationships from Table 2 are plotted in Fig. 3 (the initial porosity θ_o is assumed to be $\theta_o = 0.36$ which corresponds to the dense packing of identical spherical particles). The relationship of Skorohod (initial stages of sintering) overestimates the relative interparticle neck radius at the late stages of sintering, and the relationship Helle *et al.* underestimates the relative interparticle neck radius at the late stages of sintering (these relationships do not satisfy the limiting transition $r/R \rightarrow 1$ when $\theta \rightarrow 0$, which is valid for isomeric spherical particles).

In order to compare the strength criteria presented in Table 1 and Fig. 2 with the strength criterion given by Equation (4), the expression derived by Skorohod (for all stages of sintering) for the dependence between the relative interparticle neck radius r/R and porosity θ is substituted in Eq. (4) and also plotted in Fig. 2. One can see that Eq. (4) provides comparable results for initial stages of sintering when $\theta > 0.25$.

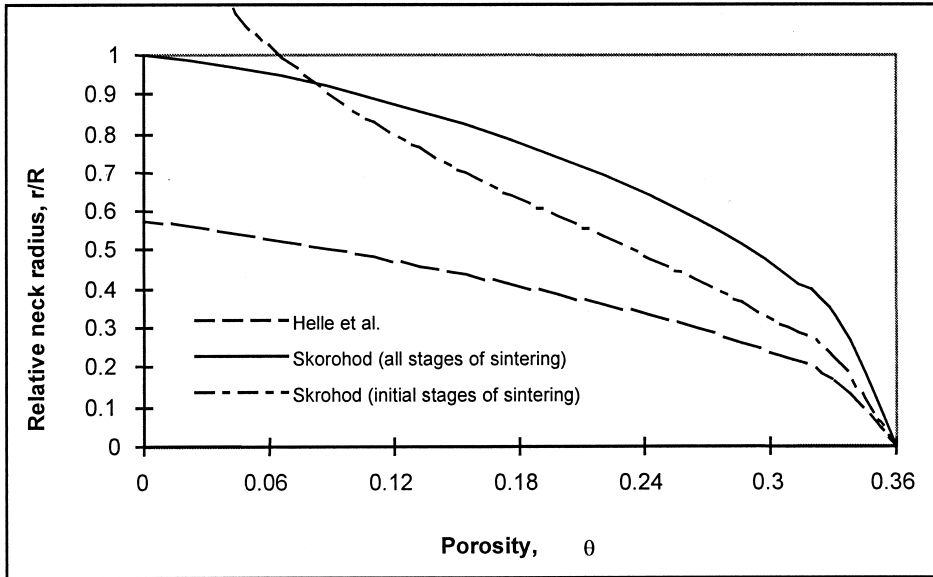


Fig. 3. Dependence of the relative interparticle neck radius on porosity.

3.2. Calculation and experimental data for the three-point bending test

In accordance with the MPIF Standard No. 41 [14], the measured value of the axial stress in the indenter which corresponds to the transversal tensile stress in the specimen during the three-point bending test can be given as follows:

$$\sigma_1 = \frac{2\pi bc^2 r_s^2}{3L} \sigma_c \tag{5}$$

here the indenter contact area is assumed to be equal to πr_s^2 .

Substituting Eq. (4) into Eq. (5) we can compare the experimental data of the three-point bending test with the results of the modeling.

Two parameters in Eq. (4) should be considered as time-dependent. These are the relative interparticle neck radius r/R and the failure stress of the fully dense material σ_{co} which depends on temperature and thereby (due to the non-isothermal character of sintering) on time. The dependence of the tensile failure stress for the stainless steel SS304L on temperature (adopted from [25]) is plotted in Fig. 4.

For the description of the evolution of the relative interparticle neck radius r/R caused by the mechanism of surface diffusion (when the surface diffusion is the fastest mechanism of material transport which is almost always the case in early stages of sintering), the expression obtained by Svoboda and Riedel [26] is used:

$$\left(\frac{r}{R}\right)^{\frac{9}{2}} \frac{d\left(\frac{r}{R}\right)}{dt} = (2\sqrt{2\phi})^3 \frac{\alpha\Omega}{kR^4} \frac{\delta D_s(T)}{T(t)} \tag{6}$$

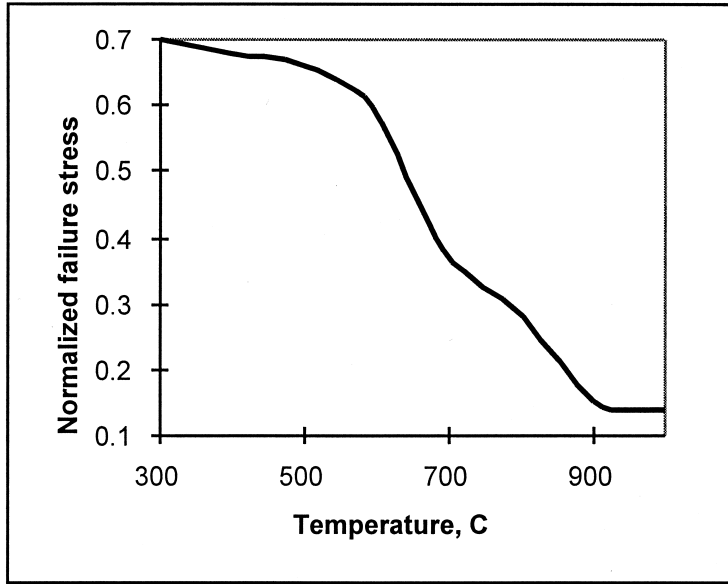


Fig. 4. The dependence of the tensile failure stress for the stainless steel SS304L on temperature.

where ϕ is the dihedral angle, α is surface tension, Ω - atomic volume, T - temperature, k - Boltzman's constant, δD_s is the coefficient of surface diffusion, t is time.

The differential equation (6) should be solved taking into account the relationship for the change of the temperature T :

$$T = T_o + ut \quad (7)$$

where $T_o = 298^\circ\text{K}$ is the initial temperature, $u = 1/6^\circ\text{K/s}$, and t - time (s).

The material parameters used in calculations for the stainless steel powder in accord with Eq. (6) are given in Table 3.

The initial relative interparticle neck radius was calculated in accord with the Skorohod expression for all stages of sintering (see Table 2).

The results of the calculations for the relative interparticle neck radius r/R (Eqs. 6 and 7) and the relative strength σ_c/σ_{co} (Eqs. 4, 6, 7, and Fig. 4) are presented in Fig. 5. The coordination number Z is calculated as a function of porosity θ [18]:

Table 3

The material parameters used in calculations [18]

Surface energy, α J/m^2	atomic volume, Ω , m^3	Activation energy for surface diffusion, kJ/mol	Frequency factor for surface diffusion, m^2/s	Dihedral angle, ϕ	Initial porosity, θ_o	Particle radius, R , μm
2.2	$1.53 \cdot 10^{-29}$	220	0.5	$\pi/2$	0.32	70

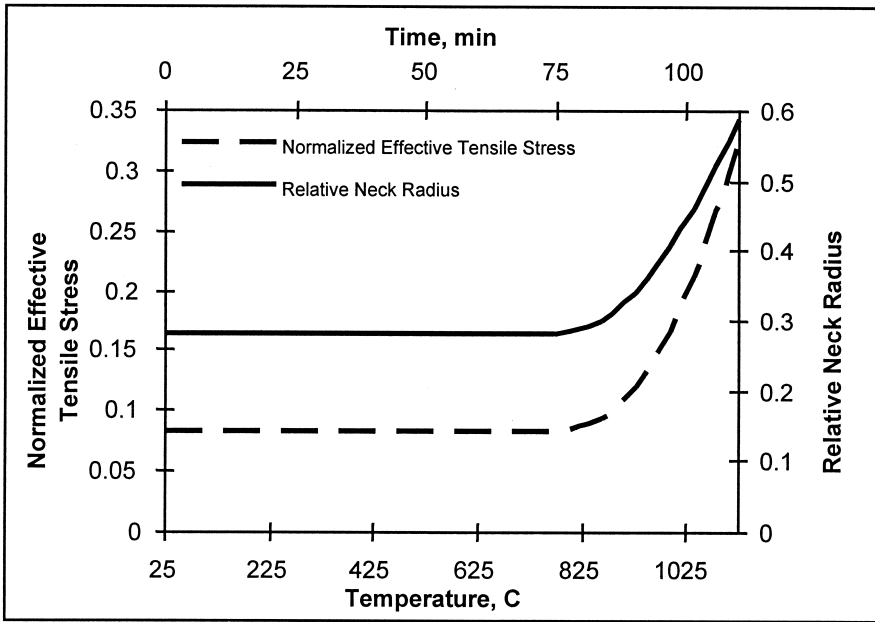


Fig. 5. The evolution of the relative interparticle neck radius r/R and the relative strength σ_c/σ_{co} for the stainless steel powder.

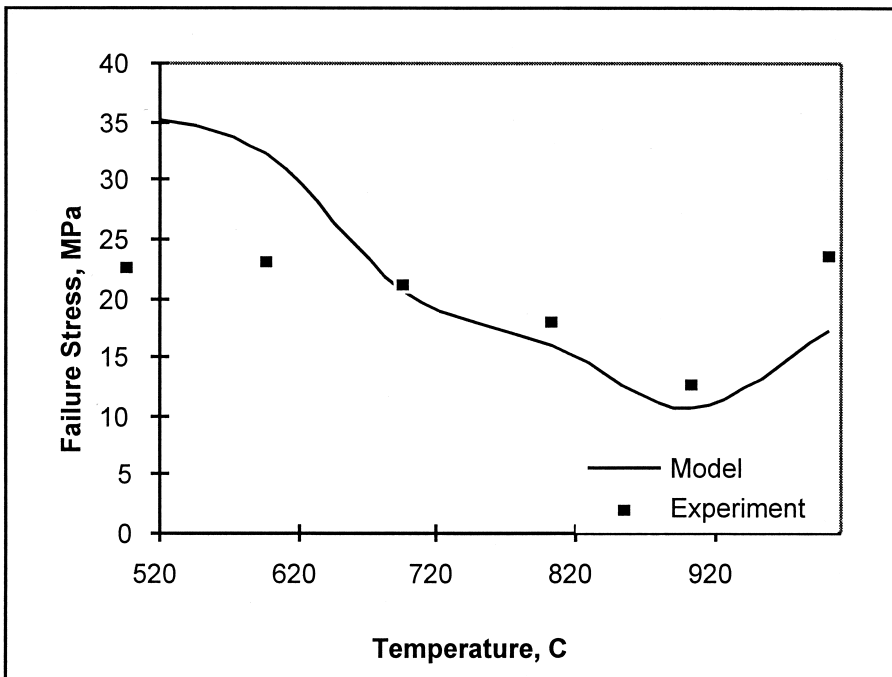


Fig. 6. Axial damage stress in the three-point bending test for the stainless steel powder.

$$Z = 7 + 17.5 \cdot (0.36 - \theta) \quad (8)$$

The calculation and experimental data of axial damage stress in the indenter are compared in Fig. 6. The nonmonotonous behavior of the dependence between the damage stress and the final temperature of sintering is the result of the interplay of three factors: the strengthening due to the increase of the interparticle contact areas and the increase of the total time of sintering, on the other hand, the softening of the particle material (Fig. 4) due to the increase of the temperature. Here time can be considered as an independent factor, because the experiments have been conducted under the same heating rate. Therefore, for achieving different temperatures, different times had to be spent.

The large deviation of the experimental and the calculated data for lower temperatures can be explained by the influence of factors different from sintering. For example, at these pre-sintering levels of temperature rearrangement of particles under the bending load can be significant.

4. Conclusions

1. A criterion for the accumulated strength under initial stages of sintering is elaborated.
2. The criterion includes the relative interparticle neck radius and porosity as two governing parameters along with the tensile strength of the corresponding fully dense material.
3. The results of the calculations based upon the developed strength criterion agree well with the experimental data on three-point bending test of stainless steel powder compacts.

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