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# Structure of sintered porous materials for gas diffusion

I. VIDA –SIMITI*, N. JUMATE, T. BOLOG
*Technical University of Cluj – Napoca, Romania*

Porous disks were fabricated by sintering stainless steel powder type 316L and bronze (Cu - Sn10) having different grain sizes. The influence of the particle size distribution, temperature and sintering time on the structural and functional characteristics (porosity, pore size) are studied. A porous structure with small-sized pores and a uniform distribution of the pore sizes is obtained for a narrow range of small particle size distribution.

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*Keywords*: Porous diffusers, Sintering, Sintered porous materials

## 1. Introduction

An uniform porous structure with small sized pores ensures the main conditions required for gas diffusers [1]. The permeable elements should have a uniform porous structure with as small as possible pores. Small sized pores ensure an uniform and efficient distribution of fluids in different media due to the large specific surface of gas bubbles thus formed. Structural parameters (porosity and size of pores) are influenced by the technological processing parameters as the powder size range, compacting pressure, sintering temperature and sintering time [2 - 9].

As an on going work on the matter we analyze in this paper the influence of sizes of free stainless steel and bronze (Cu-Sn10) powders poured over a permeable porous structure by varying the sintering time.

## 2. Materials and experimental method

Porous samples under the form of disc tablets, having 25 mm in diameter and approximately 2 mm thick, were made by freely spreading and then sintering stainless steel powder (316 L) and bronze (Cu-Sn10) powder. The powder size range obtained by sieving were: -40 μm; (+40 -63) μm; (+63 -80) μm; (+80 -100) μm; (+100 -125) μm; (+125 -160) μm. The sintering was performed in a vacuum furnace ($5\times10^{-5}$ torr) at a temperature of 1150 °C for stainless steel powders, and for bronze powders at relatively low temperature of 750 °C.

The sintering times were changed from 30 to 180 minutes for stainless steel powders and 30 to 60 minutes for powders bronze.

The maximum and minimum pore sizes were determined by using the method of fluid dislocation from pores (the bubble method), according to the international standard EN 24003. Isopropanol was used as an impregnation fluid. Micrographs of the porous structure were observed by using scanning electron microscopy (JEOL 5600 LV microscope). The porosity of regularly shaped samples was determined from volume and weight of the sample.

## 3. Results and discussions

The sintering time at a temperature $T_s$=1150 °C influence little the porosity, for different grain sizes, as shown in Fig. 1. The initial poor contact between the particles of the freely spread powder cannot ensure intense mechanisms for the development of material transport in the course of the sintering process, which would result in a more marked densification by sintering.

A marked reduction in porosity in the case of the powder having mean grain size -40 μm was attributed to the higher initial packing degree of small sized particles. The apparent (initial) compaction was higher in powders with smaller sizes and also in the case of a larger powder size, like that below -40 μm. The variation in the size of the largest pores, and of the mean pore sizes respectively as function of sintering time are given in Figs. 2 and 3. The pores undergo small reductions, even insignificant with increasing sintering time. The mechanism of transfer of material around the interparticle neck during sintering is not particularly intense. Consequently, the size of pores decreases significantly with sintering time.

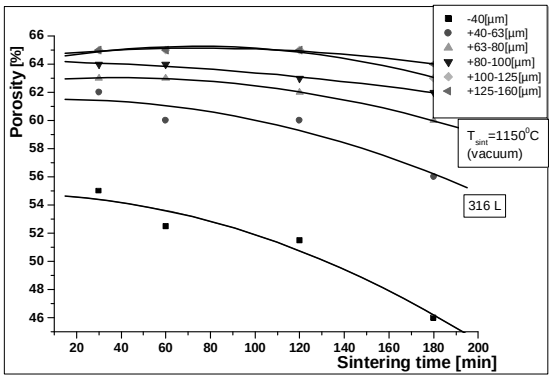



*Fig. 1. Influence of the sintering time and grain sizes on porosity.*

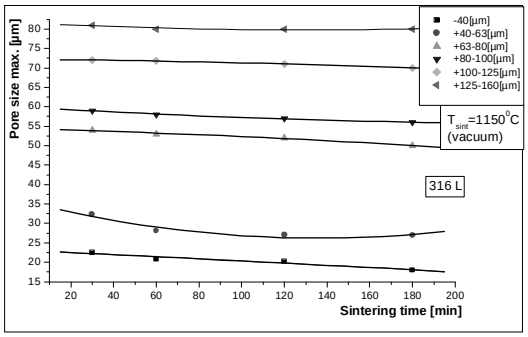



*Fig. 2. Influence of sintering time and grain size on the maximal pore size.*

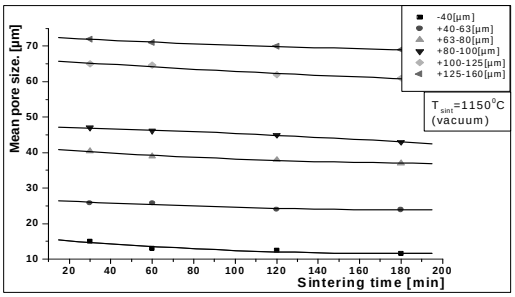
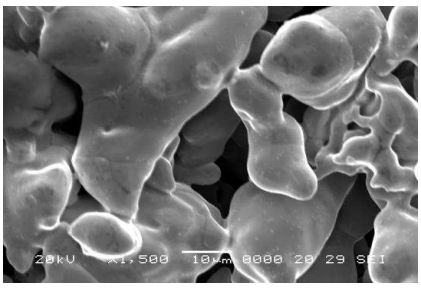


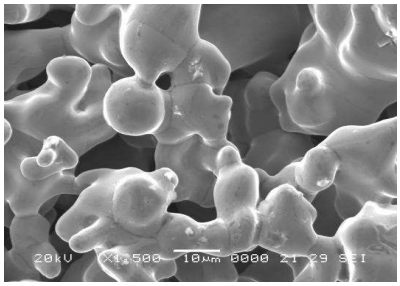

*Fig. 3. Influence of sintering time and grain sizes on the mean pore dimensions.*

SEM images of the porous structures obtained confirm the previous findings, Fig. 4. The size of pores and the diminution of interparticle necks are not significantly changed. However, the surface of particles is found to be smooth, as a consequence of the transport of material on the surface layer. Thus, surface defects and rugosity disappear following the mechanisms occurring in the course of sintering. Images show interparticle necks, formed following sintering, only in the area of initial small contacts between freely spread particles.

In the case of the freely spreading powder, the conditions favouring high sintering such as cold - hardening in the contact area between particles due to pressure were absent. This fact reduced the increase of the sintering necks, the rounding and the decrease of pore sizes.




a

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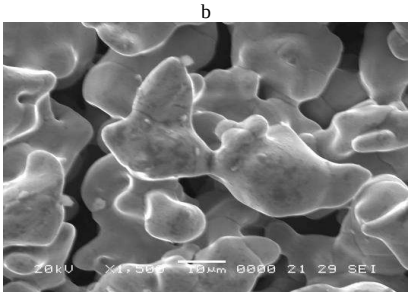
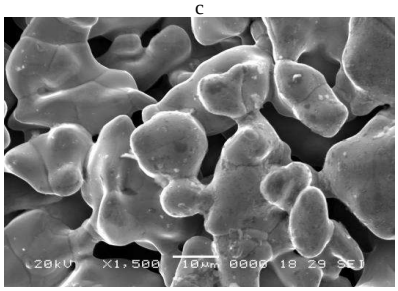



c




d

*Fig. 4. - SEM images (x1500) of the porous structure (- 40 µm): a) $\tau$ = 30 min; b) 60 min; c) 120 min; d) 180 min.*

The surface layers having austenitic crystalline grains can be seen on the surface and were attributed to the preferential evaporation of atoms at the grain limit during sintering proces. An uniform structure with interconected pores can be observe in case of the narrow powder size range.

For smaller pore sizes a reduced compacting pressure may be applied in order to prevent the closure of the pores. The absence of cold - hardening as a mechanical activation of the sintering process of the freely spread powder reduces the development of sintering necks and maintains the porosity and the sizes of pores relatively constant. Marked necks may be noted especially between some large and small particles or between small irregularly shaped particles, due to their increased surface energy.

*Fig. 5. Bronze powder samples sintered at 850 °C.*

*Fig. 6. Bronze powder samples sintered at 750 $^{o}$C.*

Experimental sintering tests for bronze powders were performed at 850 $^{o}$C, [7]. The powder samples having greater sizes were melted and underwent marked contractions (Fig. 5). The sintering was performed for 30, 45 and 60 minutes, at 750 $^{o}$C. For more than 45 min sintering time, the powder size range (100-125 µm; 125-160 µm) were highly contracted (Fig. 6).

The heat transfer in case of vacuum sintering is realized mainly by thermal radiation. Due to the high porosity of the powder having a grain sizes between (+100 – 125) µm and (+125 -160) µm, the "absorbent black body" phenomenon occurs. In this case a part of the thermal radiation is absorbed by the sample material, a part is reflected by the sample surface and another part penetrates into the pore cavity where heat accumulates, leading to the local temperature increase. The diffusion process at the level of the sintered bridges is enhanced. The increase of the temperature by 20 $^{o}$C practically increase twin the diffusion coefficient and triggers the transfer of material around the bridges between particles. Consequently, temperature there is a reduction of porosity and increase of contractions. This accounts for the high contraction in the powder samples having particle sizes between 100 – 160 µm.

The influence of the sintering time on the total contraction of samples at a $T_s$=750 $^{o}$C is shown in Fig. 7. High contractions may be noted in the size ranges (+100 - 125) µm; (+125 -160) µm. A more marked reduction of the porosity in the 40 µm powder size range may be accounted for by the initially higher degree of packing of the small particles. The same behavior can be seen in Figs. 8-10. The pore size undergoes small, even insignificant changes when sintering time increases. The mechanism of material transfer around the inter-particle bridges during sintering is not very intense. Consequently the pore size does not decrease too much with sintering time.

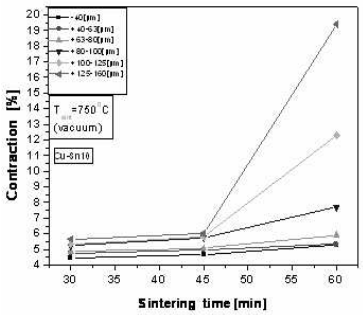



*Fig. 7. Influence of sintering time and powder sizes on the contraction.*

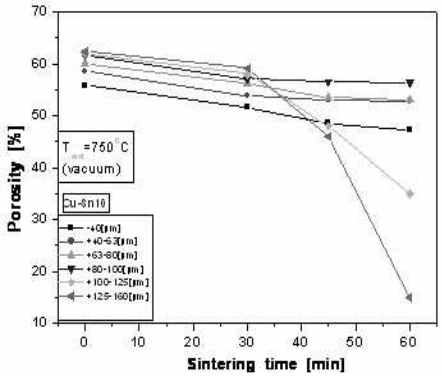



*Fig. 8. Influence of sintering time and powder sizes on the porosity.*

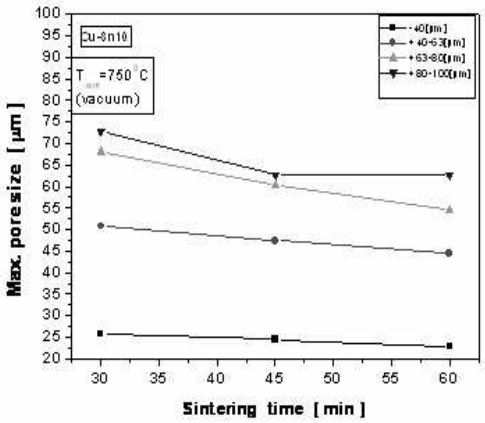



*Fig. 9. Influence of sintering time and size range on the maximal pore size.*

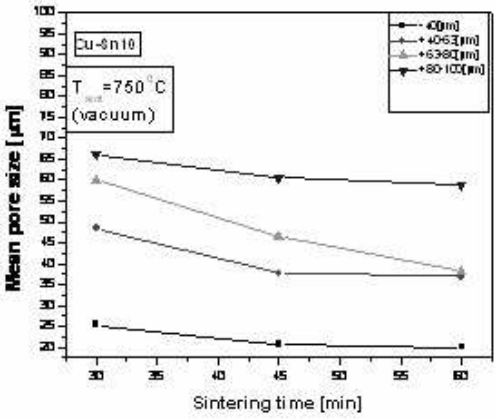
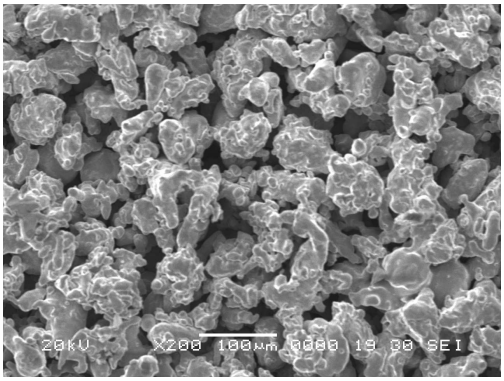



*Fig. 10. Influence of the sintering times and size range on the average pore size.*

The SEM images (Fig. 11) evidence the fact that the pore sizes and the inter-particle bridges reduction are not significantly changed. However, it may be noticed that the particle surface is smooth, as a result of the material transport into the surface layer. The surface defects and the roughness disappear following the sintering mechanisms. The SEM patterns evidence the inter-particle necks formed

after sintering only in the small areas of initial contacts between the free powder particles (Fig. 11).



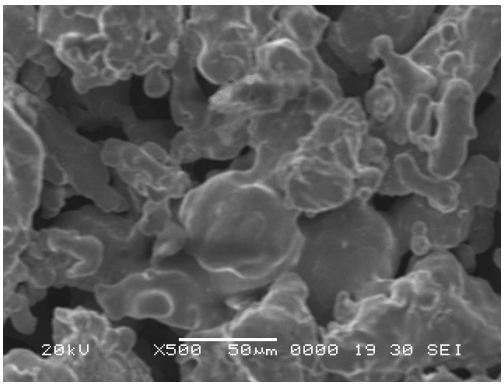




*Fig. 11. SEM image (x 500) of the bronze porous structure (45 min.).*

The narrow powder size ranges, formation by free spreading into the mould as well as the relatively low sintering temperature (750 $^{o}$C) ensures a uniform porous structure with intercommunicating pores, favourable to fluid flow and filtration.

## 4. Conclusions

Sintered materials for gas diffusers were prepared.

By sintering, the surface of the powder particles becomes smooth as a consequence of the transport of substance on the surface layer.

The sintering time of bronze powder has a marked influence on the porosity and pore sizes when using powder sizes between +100 and -160 μm. The sintering time does not influence significantly the porous structure parameters (porosity, pore size) in the samples obtained from powder size (+40 - 100) μm.

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*Corresponding author: ivida@personal.ro