DETERMINATION OF PORE SIZE OF FILTER MATERIALS FROM NONSPHERICAL POWDERS

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Gatushkin and co-workers [1-3] have carried out, using mainly porous stainless steel, a comprehensive study of the filtration properties of porous materials from nonspherical powders. In that study, the effects were determined of specimen thickness, porosity, and powder particle size on the permeability, filtration fineness, sludge-holding capacity, and regenerating power of filters. However, to solve the basic problem of formulation of porous materials having predetermined properties, it is necessary to explore more fully the relation between the macrostructure of porous bodies and their properties.

The present work forms the first phase of investigations in this direction. It was undertaken with the aim of comparing pore diameters determined by various techniques and correlating them with filtration fineness (the minimum size of contaminant particles stopped by the filter with an efficiency of 100%).

Pore sizes were determined metallographically (Dmicr), by the method of forcing a gas bubble through a porous specimen impregnated with a liquid (Dmax) [4], and by calculation, from the gas-permeability coefficient of porous materials (Dhydr) [4]. As a result of their work on filters from spherical powder, Andrievskii [5] and Shibriyaev [6] have shown that the maximum size of particles passing through a filter is approximately half the pore size determined by the technique of forcing a gas bubble through the filter (Dmax). No relation of this kind has been reported for filters from nonspherical powders. Lev [7] and Strizhevs'kii [8] merely cite some data yielded by a determination of Dmax for porous specimens from reduced stainless steel powder, while Konev [9] describes the determination of Dhydr of porous stainless steel; Korovin [10] notes that Dmicr for porous nickel electrodes is much greater than the maximum pore size determined by mercury porosimetry.

It is true that Belov [12] has determined the mean pore sizes of materials from nonspherical powders by the techniques of metallography, gas-bubble passage, and calculation based on permeability. However, in view of the limited scope of this investigation (a porosity limit of 55%, use of powders with particles of a single size, and, most important, failure to find a correlation between pore size and filtration fineness), the work of Belov [12] cannot be regarded as sufficiently comprehensive. Furthermore, it is impossible to agree with Belov's claim that the mean metallographic pore diameter is equal to the mean diameter determined by the liquid displacement technique, as this is in contradiction with our own findings. Thus, the object of the present work was to carry out a detailed comparative study of Dmicr, Dmax, and Dhydr and to find a correlation between these diameters and the filtration fineness of porous materials from nonspherical powders.

Experiments were carried out on two typical examples of porous materials from nonspherical powders:

1) a high-porosity material sintered from a fine powder with the aid of fillers;
2) a material sintered from a coarser nonspherical powder without a filler.

The former material was a Ni-30% Mo alloy of 64-83% porosity, prepared by sintering a mixture of carbonyl nickel and fine molybdenum powders with 20-40 wt.% of NH₄Cl filler. The NH₄Cl filler powder used was of three fractions: < 0.07, 0.07-0.1, and 0.18-0.3 mm. The method of preparation of such a porous
material has been described elsewhere [11]. The other material was a stainless steel of 30-60% porosity, sintered from reduced powders with particles < 0.04, 0.07-0.15, and 0.16-0.3 mm in size. The investigation was carried out on disk-shaped specimens 20 and 30 mm in diameter and 3 mm thick. Determinations were made of the gas permeability of specimens, and their gas-permeability coefficient was calculated [4]. \( D_{\text{micr}} \) was taken as the maximum pore size measured on micrographs of porous specimens, \( D_{\text{max}} \) was determined by the gas-bubbling method described by Berkman [4], and \( D_{\text{hydr}} \) was calculated with the formula recommended by Berkman [4]:

\[
D_{\text{hydr}} = 565 \sqrt{\frac{K}{\theta}} \,
\]

where \( K \) is the gas-permeability coefficient, in darcies, and \( \theta \) is the specimen porosity, in fractions of unity.

Data on the filtration fineness of porous stainless steel were taken from [2]. Figure 1 shows that the pore size grows with increase in both filler particle size and porosity. From Fig. 2 it can be seen that, with increase in porosity, the pore size of the material sintered with the filler almost invariably increases approximately linearly. It is interesting that the different methods of pore diameter determination give widely differing values. For specimens sintered with different filler fractions, the following relation is valid: \( D_{\text{micr}} > D_{\text{max}} > D_{\text{hydr}} \). Thus, for example, for Ni–30% Mo specimens of 75% porosity, the relevant ratios are as follows:

<table>
<thead>
<tr>
<th>Filter fraction, mm</th>
<th>( D_{\text{micr}} : D_{\text{max}} : D_{\text{hydr}} )</th>
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<tbody>
<tr>
<td>0.18–0.3</td>
<td>10:5:2:1</td>
</tr>
<tr>
<td>0.07–0.15</td>
<td>6.9:2:1</td>
</tr>
<tr>
<td>&lt;0.07</td>
<td>5.8:2:1</td>
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The difference between the metallographic pore diameter and the diameter determined by the method of forcing through a gas bubble appears to be due to the variable cross section of pore passages and to the presence in these passages of constrictions. Since capillary pore passages have a variable cross section, to expel a liquid from them completely it is necessary to apply the pressure required for freeing the narrowest capillary throat from the liquid. Thus, the diameter determined by the method of forcing through a gas bubble characterizes the diameter of the narrowest parts of all the passages present in the specimen [4]. It is therefore entirely understandable that the metallographic pore diameter is greater than \( D_{\text{max}} \) because a typical specimen cross section, such as is obtained when a metallographic microsection is prepared, must inevitably contain also some wider portions of pore passages, whose diameter is, of course, greater than the diameter of the most constricted opening. It must also be noted that, as can be seen from Fig. 2 and the data quoted above, the difference between pore diameters determined by different techniques diminishes with decrease in the particle size of the filler used in the preparation of the porous Ni–30% Mo material.

Let us consider now the reasons why the calculated diameter \( D_{\text{hydr}} \) is less than \( D_{\text{max}} \). To do this, a certain assumption will be made. The mean hydraulic pore diameter \( D_{\text{hydr}} \) must be compared with the mean porosimetric diameter \( (D_p) \) determined by the method of mercury porosimetry [4]. For the Ni–30% Mo material, \( D_{\text{max}} \) may be identified with \( D_p \) on the following grounds. Firstly, Korovin [10], in an investigation of porous carbonyl nickel sintered with a filler (a material analogous to the porous Ni–30% Mo material used in the present work), experimentally demonstrated that the pore diameter determined by mercury porosimetry was similar to that obtained by the technique of forcing through a bubble. Secondly, in our case, during the pumping of air through a specimen impregnated with water, the pressure at which the first bubble appeared was close to that which gave rise to the appearance of the bulk of bubbles. This suggests that the maximum pore size of the Ni–30% Mo material must have been close to the mean pore size.

The mean hydraulic pore diameter \( (D_{\text{hydr}}) \) was calculated starting from the permeability coefficient and porosity. Such a calculation is based on the assumption that all the pore passages are cylindrical and of the same cross section and that they run through the material at right angles to its surface. The calculation yields the so-called equivalent size, i.e., the size of round-section pores whose effect on permeability is the same as that of the real pores [4]. The value of \( D_{\text{hydr}} \) is affected by tortuosity, which determines the length of the pore passages. The tortuosity coefficient (the ratio of the length of a pore passage