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Determination of structural parameters of metallic foams from permeametry measurements

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An experimental technique, permeametry, is carried out in order to determine the dynamic specific surface area and the tortuosity of three nickel foams. A capillary-type model allows calculation of these structural parameters from pressure-drop measurements. Studying pressure drops of two different flow configurations also allows quantification of a third parameter due to the anisotropy of the material structure. The values of the parameters determined throughout this work are compared with those obtained in previous works using different experimental methods.

Nomenclature

A	experimental coefficient defined by Equation 3 (Pa s m^{-2})
A_{vd}	dynamic specific surface area, related to volume of solid (m^{-1})
A_{ve}	specific surface area, related to volume of porous medium (m^{-1})
B	experimental coefficient defined by Equation 4 (Pa s^2 m^{-3})
Cr	precision criterion
D	hydraulic diameter of the cell (m)
d	equivalent pore diameter (m)
f	friction factor
H	bed height or thickness of porous material (m)
J	coefficient defined by Equation 8 (m^{-1})
K	coefficient defined by Equation 9 (m^{-2})
l	pore length (m)
mre	mean relative error
$n+1$	number of pressure taps
ΔP	pressure drop (Pa)
R	anisotropy factor or shape anisotropy ratio
Re	superficial Reynolds number, $Re = \rho U_o d/\mu$
Re_i	interstitial Reynolds number, $Re_i = \rho U_o d/(\varepsilon\mu)$
T	tortuosity
U_o	superficial velocity (m s^{-1})
ε	porosity
μ	dynamic viscosity (Pa s)
ρ	fluid density (kg m^{-3})

1. Introduction

The structural parameters of a porous material, metallic foams, are determined using a permeametry method and then are compared with results obtained using other techniques. The nickel foams used in this experimental work are characterized by a reticulated structure (Fig. 1) and porosities close to unity. Consequently, particularly high specific surface areas are developed. The importance of the latter is interesting for industrial applications in the field of electrochemical engineering. In particular, optimizing catalyst recycling or organic electrosynthesis in an electrochemical reactor fitted with nickel foams as porous electrodes requires quantification of the active surface area of this porous material. This potentially reacting surface area is generally considered to be close to the dynamic specific surface area actually presented to the fluid. Its value also allows us to establish a mass transfer balance or to compare the performances of different porous electrodes.

Among the several methods available for measuring surface area [1], mercury porosimetry and gas adsorption measurements (BET method) are currently used in order to characterize cementing materials [2]. With the BET method, the surface area of micropores is

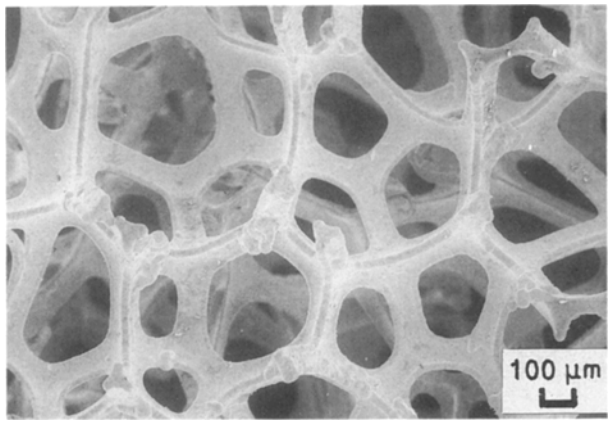

Figure 1 Scanning electron micrographs of nickel foam (Grade 60).

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taken into account as they are reached by gas molecules. The mercury porosimetry method is based on the intrusion or extrusion of mercury under pressure in the porous medium. The intruded volume and pressure data are related to the size of the pores by using the Whasburn equation. This method does not allow us to take into account the surface area of large pores (pore radius $\lesssim 100$ μm).

Another kind of method, permeametry, is based on the study of a fluid flow through the porous medium. The surface area presented to the flow is deduced from measurements of the mechanical energy dissipation in the flow. Using a suitable flow model, the determination of two structural parameters of the porous medium, respectively the specific surface area and the tortuosity, is possible.

Choosing a suitable method is conditioned by the nature of the specific surface area to be quantified, corresponding to the potential application.

2. The flow model used to calculate structural parameters

The dissipation of mechanical energy occurring in the flow through porous media can be expressed by the following Forchheimer-type equation

$$\frac{\Delta P}{H} = AU_o + BU_o^2 \tag{1}$$

where ΔP is the pressure drop through the porous material of bed height H, and U_o is the superficial velocity (ratio of the flow rate over the cross-sectional area of the porous medium). The first term of Equation 1, proportional to the flow velocity, is due to viscous resistance at the walls of the pore. The second, proportional to the square of the flow velocity, is due to inertial resistance, and corresponds to kinetic energy losses due to direction changes of streamlined paths.

Laser velocimetry and flow visualization studies of liquid flow through porous structures [3] have proposed the existence of different flow regimes in porous media. These regimes are characterized by an interstitial Reynolds number, Re_i which corresponds to the ratio of inertial forces to the viscous ones

$$Re_i = \frac{\rho U_o d}{\mu \varepsilon} \tag{2}$$

where ρ is the fluid density, μ is the dynamic viscosity of the fluid, d is the pore equivalent diameter, and ε is the porosity of the porous medium. The different flow regimes are defined below.

(a) The Darcy or creeping flow regime is observed when the flow is dominated by viscous forces ($Re_i < 1$).

(b) The inertial flow regime initiates between $Re_i = 1$ and $Re_i = 10$ where the boundary layers become more pronounced and an "inertial core" appears. The development of these "core" flows outside the boundary layers is the reason for the non-linear relationship between pressure drop and flow rate. This steady non-linear laminar flow regime persists to $Re_i \simeq 150$.

(c) An unsteady flow regime occurs in the Reynolds number range 150–300.

(d) For $Re_i > 300$, a highly unsteady and chaotic flow regime observed by Dybbs and Edwards [3] is seldom reached in fluid experiments.

Our experimental study corresponds to the creeping flow and the inertial flow regimes.

Comiti and Renaud [4] proposed a capillary-type flow model where the porous medium is described as a bundle of tortuous pores of diameter d and length L. In this model, terms A and B in Equation 1 are expressed as a function of the fluid characteristics and of the structural parameters of the porous medium: ε the porosity, A_{vd} the specific surface area and T the tortuosity. A_{vd} is the ratio of the surface area presented to the flow over the volume of solid and T is the ratio of the mean distance covered by the fluid, L, over the thickness of the crossed porous medium, H.

Wall effects due to the experimental cell are taken into account. The expressions for parameters A and B of Equation 1 are

$$A = 2\mu T^2 A_{vd}^2 \left[1 + \frac{4}{A_{vd} D(1-\varepsilon)}\right]^2 \frac{(1-\varepsilon)^2}{\varepsilon^3} \tag{3}$$

$$B = \left\{\left[1 - \left(1 - \frac{4\varepsilon}{(1-\varepsilon)A_{vd}D}\right)^2\right] 0.0413 + 0.0968\left[1 - \frac{4\varepsilon}{(1-\varepsilon)A_{vd}D}\right]^2\right\} \times T^3 \rho A_{vd} \frac{(1-\varepsilon)}{\varepsilon^3} \tag{4}$$

where D is the hydraulic diameter of the cell.

Structural parameters cannot be calculated from the whole experimental data obtained with different fluid characteristics. Therefore, a second equation (7) was derived in order to calculate general correlations. Let us define a friction factor, f, and a Reynolds number, Re, by

$$f = \frac{\Delta P}{H U_o} \frac{\varepsilon^3}{(1-\varepsilon)\rho U_o} \tag{5}$$

$$Re = \frac{\rho U_o d}{\mu} \tag{6}$$

$$\frac{f}{d} = J + K \frac{1}{(Re/d)} \tag{7}$$

with

$$J = \left(\left\{1 - \left[1 - \frac{4\varepsilon}{(1-\varepsilon)A_{vd}D}\right]^2\right\} 0.0413 + 0.0968\left[1 - \frac{4\varepsilon}{(1-\varepsilon)A_{vd}D}\right]^2\right) T^3 A_{vd} \tag{8}$$

and

$$K = \left[1 + \frac{4}{A_{vd}D(1-\varepsilon)}\right]^2 [2A_{vd}^2 T^2 (1-\varepsilon)] \tag{9}$$

f/d is a linear function of d/Re, the intercept and the sloop are independent of the physical properties of the fluid.

TABLE I Characteristics of nickel foams

	Grade		
	G 100	G 60	G 45
Porosity, ε	0.973	0.975	0.978
Thickness of sheets (mm)	1.65 ± 0.04	2.57 ± 0.06	2.54 ± 0.10
Dimensions of sheets (mm × mm)	750 × 150	750 × 150	750 × 150

3. Experimental procedure

3.1. Apparatus and experimental technique

3.1.1. Porous material

Most foams, metallic ones as well as organic ones, are characterized by their grade, i.e. the number of pores present in the structure per inch (p.p.i.). Nickel foams used in this work are presented in the form of thin sheets whose dimensions and characteristics are given in Table I. Two flow configurations were tested:

(i) the flow-through configuration for which the mean direction of the flow is perpendicular to the plane of the sheets;

(ii) the flow-by configuration for which the mean direction of the flow is parallel to the plane of the sheets.

3.1.2. Apparatus

Measurements of pressure drops occurring during the flow of a fluid through nickel foams was performed with the equipment shown in Fig. 2, completed with inclined or vertical tubular differential manometers. The test cell used for the flow-through configuration is a cylindrical column of diameter 0.06 m packed with discs of nickel foams, while a parallelepipedal cell of side 0.055 m packed with rectangular sheets of foams allows pressure drop measurements in flow-by configuration. Both cells are fitted with pressure taps. The distance between two pressure taps is 0.05 m, the maximum height of a porous medium available for measurements is 0.20 m for the cylindrical cell and 0.25 m for the parallelepipedal one. Calming sections precede and follow the measurement section, consisting of a bed of glass beads and stacked foams upstream, stacked foams downstream. The height of the calming sections is large enough to allow an established flow in the test section.

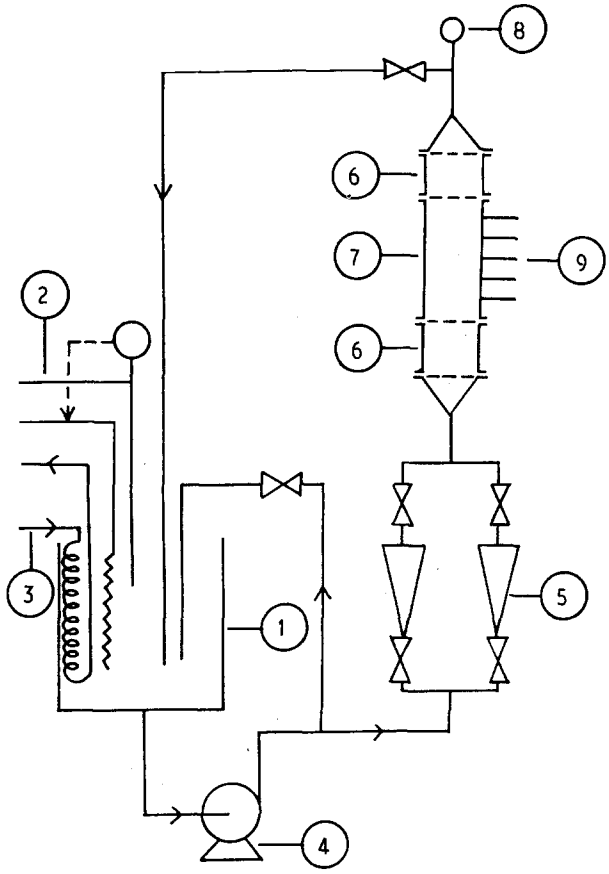

Figure 2 Experimental equipment (1, storage tank; 2, contact thermometer coupled with a regulated heating resistance; 3, water refrigerant; 4, centrifugal pump; 5, flowmeters; 6, calming sections; 7, bed packed with foam; 8, temperature probe; 9, pressure taps).

3.1.3. Experimental technique

The experimental work consists in measuring pressure losses as a function of the flow rate, for a given bed height, in order to determine experimental constants of Equation 1.

A preliminary study of the homogeneity of each bed was carried out. The criterion of precision is defined as

$$Cr = \frac{\Delta P_{(H/n)} - [\Delta P_{(H)}/n]}{\Delta P_{(H)}/n} \quad (10)$$

where $\Delta P_{(H)}$ is the pressure drop measured for the bed height H, $\Delta P_{(H/n)}$ is the pressure drop measured between two pressure taps and $(n + 1)$ is the total number of pressure taps. For all experiments presented in this work, Cr is less than 5% in the range of Reynolds numbers investigated. Experiments were carried out with different Newtonian liquids, respectively demineralized water and an aqueous solution of glycerol, in order to cover a large range of Reynolds numbers. A few complementary experiments were performed with air so as to verify that the values of structural parameters do not depend on the nature of the flowing fluid. Experiments carried out with liquids needed drastic precautions, because air bubbles are easily confined in the nickel foams when filling the bed. Further details on experimental conditions are given in Table II.

3.2. Experimental results

3.2.1. Determination of the structural parameters

First, for a given fluid and grade, structural parameters are deduced from flow model (Equations 1, 3 and 4). A comparison of the obtained values shows that the nature of the fluid has no significant effect. Then, for each grade, Equation 7 is used in order to determine the structural parameters A_{vd} and T from all the experimental data concerning water, aqueous solutions of glycerol and air by minimizing a mean

TABLE II Experimental conditions

Fluid	Temperature (K)	$\mu(10^{-3}$ Pa s)	U_o (m s^{-1})	Flow rate (l h^{-1})
Demineralized water[a]	295	0.958	0.005–0.15	50–1 570
Aqueous solutions of glycerol[a]	303	7.8–10.3	0.0008–0.11	8–1 100
Air[a]	297–298	0.0184	0.47–2.01	4 700–16 000
Demineralized water[b]	297	0.914	0.0075–0.15	80–1 680

[a] Flow-through; [b] flow-by.

TABLE III Experimental results

	G 100		G 60		G 45	
	Flow-through	Flow-by	Flow-through	Flow-by	Flow-through	Flow-by
K (10^8 m^{-2})	190	152	45.6	41.9	24.3	27.4
J (10^4 m^{-1})	10.1	7.34	3.92	3.61	3.37	4.04
Number of experimental data	98	64	34	67	69	83
Mean relative error (%)	2.0	0.8	1.5	1.6	2.1	0.7
A_{vd} (10^3 m^{-1})	441	439	255	249	187	186
Tortuosity, T	1.34	1.20	1.17	1.15	1.24	1.32

relative error criterion, mre

$$\text{mre} = \sum_{i=1}^{n} \left| \frac{y_i - y_i}{y_i} \right| \qquad (11)$$

where $y_i = f/d$ measured experimentally and $y_i = f/d$ calculated from Equation 7. The values obtained are reported in Table III.

Experimental data for the flow-through configuration are plotted in Fig. 3 as the friction factor, f, versus the Reynolds number, Re. A variation of flow regime is noticeable in the Reynolds numbers range 5–10. The creeping flow is progressively replaced by the inertial flow regime.

3.2.2. Proposal of an anisotropy factor

The results appearing in Table III show that for a foam of a given grade, the value of the specific surface area does not depend on flow configuration, which is a logical result. On the other hand, tortuosity is a parameter sensitive to flow configuration, which implies that the covered path, for the same medium thickness, is different in either configuration. Therefore, the existence of a certain anisotropy in the structure of nickel foams may be admitted.

The ratio between the tortuosity measured in flow-through configuration and that measured in parallel configuration constitutes an anisotropy factor (Table IV). It explains the difference between the pressure drop, for a given velocity, measured in flow-through configuration, and that measured at the same velocity in parallel configuration (Table IV).

In reality, it must be emphasized that the anisotropy factor not only integrates the anisotropy of the porous medium structure, but also, to a lesser extent, the effect of foam sheet stacking.

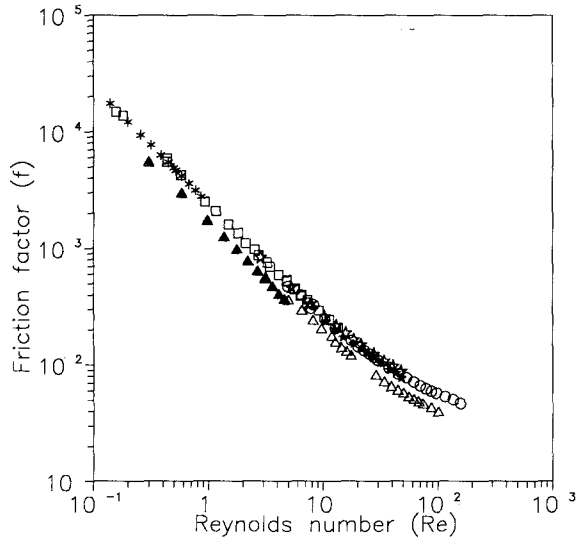

Figure 3 Experimental data concerning flow-through configuration. Foam g 45: (○) water, (□) glycerol. Foam g 100: (☆) water, (✳) glycerol, (★) air. Foam g 60: (Δ) water, and (▲) glycerol.

4. Discussion

Langlois [5] measured the specific surface area of the nickel foams with the BET technique. Krypton was

TABLE IV Calculated anisotropy factors, R, and mean pore diameters from pressure drops measurements. Variations between pressure drops measured with water in flow-by and in flow-through configuration are expressed in per cent

	Grade		
	G 100	G 60	G 45
Mean pore diameter, d (10^{-3} m)	0.33	0.62	0.96
Anisotropy factor, $R = T_t/T_b$	1.11	1.02	0.94
$\frac{\Delta P_t - \Delta P_b}{\Delta P_t}$ (%)	25–30	8.6–8.8	(– 14)–(– 18)

[t] Flow-through; [b] flow-by.

TABLE V Comparison of specific surface areas measured with BET method [5] and permeametry

	Grade		
	G 100	G 60	G 45
A_{vd} (10^6 m^{-1})			
BET method	1.52	1.20	0.864
permeametry	0.440	0.252	0.186

TABLE VI Measured shape-anisotropy ratios and nominal cell size with elastomeric axisymmetric polyurethane foams, data from [6]. (l_i is the mean intercept length of the foam in the X_i direction and $R_{ij} = l_i/l_j$)

Nominal cell size (10^{-3} m)	$R_{21} = l_2/l_1$	$R_{32} = l_3/l_2$	$R_{31} = l_3/l_1$
0.33	1.13 ± 0.06	1.15 ± 0.03	1.18 ± 0.11
0.62	1.04 ± 0.04	1.26 ± 0.04	1.31 ± 0.04
0.82	0.91 ± 0.02	1.26 ± 0.12	1.21 ± 0.13
1.95	0.91 ± 0.10	1.26 ± 0.11	1.25 ± 0.04

the adsorbate gas. Table V presents the values of the specific surface area obtained with this method as well as the values obtained with permeametry (present work). Specific surface areas measured by gas adsorption are approximately 3.5–5 times as large as those measured with permeametry. In the BET method, by enveloping each part of the porous material sample in an adsorbed film, the method of gas adsorption can probe the surface irregularities and pore interiors even at the atomic level [1]. Such pervasiveness is not available with the presently implemented permeametry because flow measurements investigate only the external superficial area of the material, micropores and microroughnesses being ignored by the flow. This comparison points out the importance of the choice of a suitable method. It obviously depends on the nature of the specific surface area to be measured for a given application. A scale of area must be defined as some methods explore surface irregularities and micropores and others do not.

Huber and Gibson [6] have studied the anisotropy of a few kinds of foams such as axisymmetric elastomeric polyurethane foams with open cells. These elastomeric foams are characterized by a reticulated structure which is particularly similar to those presented by nickel foams; they are characterized by their "nominal cell size". The evaluation of anisotropy consisted in measuring mean intercept lengths using digital image analysis of scanning electron micrographs. The ratio of the mean intercept lengths measured in opposite directions is a shape anisotropy ratio. In Table VI the values of the shape anisotropy ratio obtained by Huber and Gibson are presented. These results may be compared with the values of the tortuosity ratio we have calculated. One can observe that the values of the shape anisotropy ratio, R_{21}, R_{23}, R_{31} obtained by Huber and Gibson with scanning electron micrography measurements have the same order of magnitude as the values of the anisotropy factor obtained by pressure-drop measurements. Moreover, the variation of R_{21} as a function of the nominal cell size is similar to that of the anisotropy factor as a function of the mean pore diameter. In both cases, values lower than unity are observed for large pore diameters. In our results, the stacking-up effect of the sheets of foams seems to have no particular effect.

5. Conclusion

Using permeametry, determination of the dynamic specific surface area of a highly porous material, such as metallic foams, is possible. This technique, combined with a suitable flow model, provides a second structural parameter: tortuosity. As tortuosity depends on the path covered by the fluid, this parameter is particularly sensitive to the structure of the material. The ratio of the tortuosities measured in two different flow configurations gives an interesting indication of the anisotropy of the porous medium.

This technique has the advantage of being easy to carry out and also of allowing evaluation of the surface area actually offered to fluid flow, which is different from the BET measurements. This surface area is of particular interest for engineering applications because it corresponds to the surface area where momentum, heat or mass transfers occur during fluid flow.

The results obtained in this work indicate that nickel foams constitute a nearly isotropic material which present a large potentially active surface area per unit volume, involving low pressure drops.

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