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## PREPARATION AND CHARACTERIZATION OF STAINLESS STEEL

## FILTERS

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## ABSTRACT

Stainless steels are used as filters in corrosive environments and at relatively high temperatures. High porous filters are prepared by powder metallurgy process.

Stainless steel AISI 316L powders were separated in two granulometric fractions: 210 to 105  $\mu\text{m}$  and 74 to 44  $\mu\text{m}$ . Discs sized 40 mm in diameter and roughly 2 mm thick were obtained by uniaxial compacting under 100 to 300 MPa pressure and sintered in a reducing atmosphere (H<sub>2</sub>).

Geometrical and hydrostatic densities, median pore size and microstructure characteristics were determined in sintered filters. The relationship between these parameters is presented and discussed.

Filters characteristics are correlated to the processing parameters. It is shown that filters properties are partially controlled by the appropriate selection of powder size distribution and sintered density.

## I-INTRODUCTION

Metallic filters with controlled porosity can be produced by powder metallurgy techniques. These filters should be able to remove particles from a fluid with low pressure drop (permeability), maintaining their integrity in the environment (1,2).

Stainless steel sintered metallic powders provide a corrosion resistant material with enough strength for many applications. The particle retention and permeability of a filter depend on the pore characteristics, which in turn are dependent on the fabrication method (3,4). It was shown that several choices in the fabrication sequence are available to meet specific requirements.

Manufacturers normally quote a filtration rating (which is a function of porosity microstructure) and permeability (flow resistance). Filtration rating is an arbitrary value indicating a given percentage removal of particles of a specific size using a standard test procedure, e. g., 98 wt% removal of all particles larger than a specified size. Usually the flow resistance is given by two coefficients (5):  $\alpha$  -

viscous flow coefficient or permeability coefficient and  $\beta$ -inertial resistance coefficient. Permeability is directly related to the coefficients, i.e., lower coefficients means small permeability. Both, filtration rating and permeability coefficients are governed by selecting a very narrow powder fraction size. Moreover the porosity structure can be related to the permeability (6,7).

This paper deals with the filters processing parameters, their porosity and permeability.

## II-MATERIALS AND METHODS

### Specimen Preparation

Porous discs approximately 40 mm diameter by 2 mm thick were consolidated from water atomized 316L stainless steel powder. Two different size fractions, 210  $\mu\text{m}$  to 105  $\mu\text{m}$  and 74  $\mu\text{m}$  to 44  $\mu\text{m}$  were compacted with pressures from 100 to 300 MPa. Fig. 1 shows scanning electron micrographs of the powders. The discs were sintered in a commercial continuous furnace with H<sub>2</sub> atmosphere at 1250 °C for 1 hour.

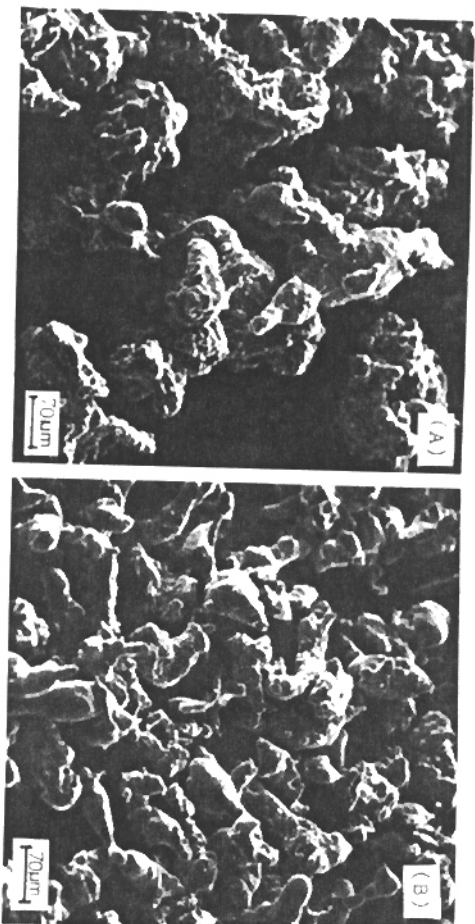


FIGURE 1.- Scanning Electron Micrographs.  
(a) 210 - 105  $\mu\text{m}$   
(b) 74 - 44  $\mu\text{m}$   
Porosity Measurements

Porosity contents were obtained from density determination. The sintered densities were calculated by measuring the mass and volume. Geometrical density ( $\rho_g$ ) was obtained from the geometrical dimensions and skeletal density ( $\rho_s$ ) from hydrostatic determination. Considering that the true 316L stainless steel density (7,96 g/cm<sup>3</sup>) it is possible to calculate the total volume fraction of porosity ( $\epsilon$ ) and the volume fraction of interconnected porosity ( $\epsilon_i$ ), as follows:

$$\epsilon = 1 - (\rho_g/\rho_s) \quad (1)$$

$$\epsilon_i = \epsilon \cdot F_i \quad (2)$$

$$F_i = \left[ \frac{(\rho_s - \rho_g)}{(\rho_s - \rho_g)} \right] \cdot (\rho_s/\rho_s) \quad (3)$$

$F_i$ =relative fraction of interconnected porosity

The pore size were estimated by mercury intrusion porosimetry measurements. In the porosimetry, mercury is intruded in the volume of the interconnected porosity as a function of pressure. The pore size (assuming cylindrical geometry) is calculated from the equation:

$$D = - 4 \gamma \cos\phi / P \quad (4)$$

where  $D$  = pore diameter (cm)

$\gamma$  = 485 dyne/cm=surface energy of mercury

$\phi$  = 130 =contact angle between mercury and 316L.

$P$  = intrusion pressure (dyne/cm<sup>2</sup>)

By this technique the volume distribution of pores can be estimated and the median pore size volume ( $D_p$ ) based on the pressure required to intrude 50 % volume of porosity.

The specimens were subsequently prepared by metallography. Besides qualitative observation the pore surface area per unit specimen volume ( $S_v$ ) was measured.

### III-RESULTS AND DISCUSSION

Optical micrographs of filters are shown in Fig. 2. These micrographs show sintered specimens with high and low porosity. Table 1 presents the measured parameters.

The interconnected porosity ( $\epsilon_i$ ) is the effective porosity in filtering. Therefore it would be desirable if this porosity approached the total porosity ( $\epsilon$ ), providing  $F_i$  (relative fraction of interconnected porosity) nearest to one. There are two sources of closed porosity (not connected to the surface), the internal porosity of powder and the geometric isolation of porosity during the compacting and sintering. In these experiments  $F_i$  changed from 0,80 to 0,90. The value of  $F_i$  decreases at higher compacting pressure due to geometric isolation of the pore network. So it would be more appropriate to manufacture filters with low compacting pressures in order to have higher  $F_i$  values.

Specimen	Powder (μm)	Pressure Compaction (MPa)	e	e <sub>i</sub>	F <sub>i</sub>	$\bar{D}_p$	$S_v \times 10^4$ (m <sup>-1</sup> )
L1	210 - 105	100	46.3	42.2	0.91	32.13	3.01
L2	210 - 105	200	40.9	35.0	0.86	25.08	2.91
L3	210 - 105	300	32.4	26.9	0.83	17.97	2.68
L4	74 - 44	150	39.7	33.3	0.84	14.29	4.68
L5	74 - 44	200	38.8	32.8	0.85	13.60	4.50
L6	74 - 44	300	32.5	26.4	0.81	10.63	3.33

e = Total Porosity  
e<sub>i</sub> = Interconnected Porosity  
F<sub>i</sub> = Fraction of Interconnected Porosity  
 $\bar{D}_p$  = Median Pore Size  
S<sub>v</sub> = Surface / Volume

Table I - Experimental Data

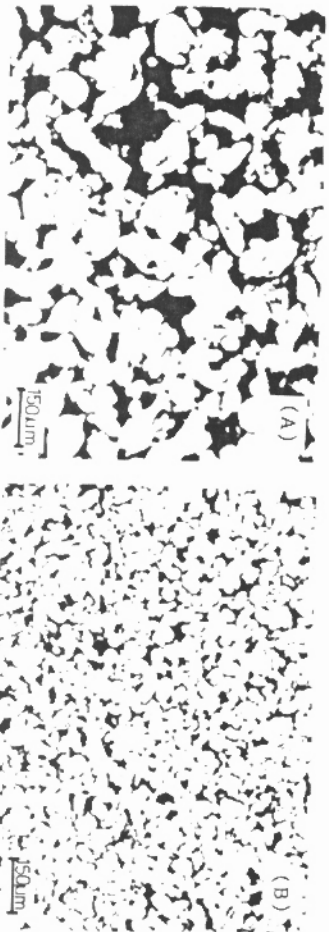


FIGURE 2 - Optical Micrographs of High and Low Porosity Specimens.  
(a) 210 - 105 μm, 100 MPa  
(b) 74 - 44 μm, 300 MPa

The median pore size ( $\bar{D}_p$ ) measured shows smaller values for higher compaction pressure and finer particles. These trend was expected. In a previous paper it was shown that  $\bar{D}_p$  could have roughly the same value as mean pore size determined by bubble point testing (6), considering 140° as the contact angle instead of 130° used in this work.  $\bar{D}_p$  can be used to evaluate the filter retention capacity but it should be compared with the effective filter retention capacity. Usually the effective retention capacity is smaller than  $\bar{D}_p$ .

Previous papers (2,3,5 and 6) showed empirical relationship of the permeability coefficient ( $\alpha$ ) with porosity, pore surface area, pore size and particle size, as follows:

$$\alpha = 4.6 \times 10^{-11} (d)^{0.73} (e)^{6.8} \quad (2) \quad (A)$$

$$\alpha = 4.8 \times 10^{-13} (d)^{1.3} (e)^{4.8} \quad (2) \quad (B)$$

$$\alpha = 0.0048 \times (e_i \cdot \bar{D}_p)^{1.85} \quad (5) \quad (C)$$

$$\alpha = 1.97 (e^2 / S_v)^{2.13} \quad (6) \quad (D)$$

where d = mean particle size

The permeability coefficient ( $\alpha$ ) can be evaluated by these equations. In the equations A,B and D e is used instead of e<sub>i</sub>. In this work e<sub>i</sub> was used, because it is the effective filtration porosity. The mean particle size used in the equations above were 59 and 157 μm. The values calculate by these equations are presented in Table II.

The equations A and B involve the same parameters: porosity and mean particle size, both giving similar results. The calculations by equation C provide a good agreement with that calculated by equations A and B. This accounts for the fact that mean particle size affects the mean pore size. So there is a reasonable agreement between the results of equations A,B and C. It must be remembered that these equations were developed using different powders.

The results of  $\alpha$  calculated by equation D are systematically higher than the other calculated values. In the case of equation D it is also considered the particle and porosity shape in the S<sub>v</sub> term.

When the coefficient of permeability  $\alpha$  is not available, it can be evaluated by one of the above equations and this can be used as an indication of the true permeability.

Considering the permeability coefficient (calculated  $\alpha$ ) and median pore size  $\bar{D}_p$  (as indicator of filtration rating) it can be inferred that different powder particle sizes and compaction pressures can be used to obtain filters with similar characteristics. As an example, the  $\alpha$  and  $\bar{D}_p$  of the specimens L3 and L4 have similar values and different particle size range. Thus, from the manufacturing viewpoint there are many fabrication options or filter choices.

#### IV-CONCLUSIONS

Interconnected porosity accounts for most of the total porosity in the sintered filters. Care must be taken in selecting the fabrication parameters to avoid a significant decrease in the interconnected porosity.

By selection of the fabrication sequence (powder sizes and porosity content) filters with specific properties can be produced.

Specimen	Powder ( $\mu\text{m}$ )	Pressure Compaction (MPa)	A ( $\text{m}^2$ ) ( $10^{-13}$ )	B ( $\text{m}^2$ ) ( $10^{-13}$ )	C ( $\text{m}^2$ ) ( $10^{-13}$ )	D ( $\text{m}^2$ ) ( $10^{-12}$ )
L1	210 - 105	100	52.3	54.8	47.4	14.3
L2	210 - 105	200	14.8	22.5	21.3	6.98
L3	210 - 105	300	2.42	6.27	7.01	2.67
L4	74 - 44	150	5.09	4.90	6.82	2.03
L5	74 - 44	200	4.59	4.55	6.05	2.07
L6	74 - 44	300	1.06	1.61	2.57	1.61

- (A)  $\alpha = 4.6 \times 10^{-11} (D)^{0.73} (e)^{6.8}$   
 (B)  $\alpha = 4.8 \times 10^{-13} (D)^{1.3} (e)^{4.8}$   
 (C)  $\alpha = 0.0048 \times (e \cdot D_p)^{1.85}$   
 (D)  $\alpha = 1.97 (e^2 / S_v)^{2.13}$

Table II - Calculated Permeability Coefficient  $\alpha$ , With  $e$  Instead  $\epsilon$  in Equations A, B and D

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#### EFFECT OF MICROSTRUCTURE ON THE MECHANICAL RELIABILITY OF P/M STEELS

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#### ABSTRACT

The statistical analysis, carried out by means of the Weibull statistics, on the distribution of rupture strengths, can provide suitable data about the mechanical reliability of sintered materials [1,2]. In the present work this method is applied to the study of the mechanical characteristics of P/M steels produced using diffusion-bonded Distaloy-type powders. These materials are widely used and are characterized by a composite heterogeneous microstructure. The study shows that these materials possess a good mechanical reliability in the as-sintered and in the as-quenched and tempered states, whereas they are unreliable in the as-quenched state. A linear relationship exists between Weibull modulus and elongation. This relationship however, is different to that manifested by Fe-C-P alloys, studied in a previous work [3]. This was interpreted on the basis of the different mechanisms of deformation and fracture which are linked to the different characteristics of the microstructures.

#### INTRODUCTION

The concept of mechanical reliability of porous P/M steels was proposed in previous publications [1-3] with the objective of studying their mechanical behaviour in the light of structural applications. It was observed that, in spite of the low elongation and impact energy values, these materials present some typical features of ductile materials (ductile morphology of the surface fracture, collaboration factors higher than unity [4]). This emphasised the necessity to assess a method for the objective evaluation of the true mechanical characteristics of these materials. A new approach to investigating the mechanical behaviour of porous steels was therefore tried, based on the