

**EVALUATION OF THE CORROSION RESISTANCE OF AISI 316 STAINLESS
STEEL FILTERS**

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Abstract

In this investigation, the corrosion resistance of AISI 316 SS filters prepared using powders of (74-44) μm granulometric range and compacted with pressures of 300 MPa and 400 MPa has been evaluated in naturally aerated 0.5 M H_2SO_4 solution at 25 °C. Weight losses related to the filters fabricated using a compacting pressure of 400 MPa were significantly larger than for the filters compacted at 300 MPa. The filter compacted at 400 MPa had larger carbon and nitrogen contents comparatively to that compacted at 300 MPa, and chromium rich precipitates and oxides were found in the grain boundaries of the first filter. Also, the pores of the filters compacted at 400 MPa were smaller than for that compacted at 300 MPa. Small pores favor the formation of concentration cells and consequently assist crevice corrosion.

Keywords: Sintered stainless steels; Filters; Corrosion; AISI 316

Introduction

Stainless steel (SS) filters produced by Powder Metallurgy (P/M) find many applications in various industries such as, chemical, petrochemical, nuclear and medical. These filters are used for solid-liquid separation, gas filtering, separation of antibiotics crystals, or any application where the contaminants are detrimental to the final product [1]. In many of these applications, the filters are exposed to corrosive environments, and therefore, they should have proper corrosion resistance in practical environments. Other important characteristics of filters are uniform distribution of porosity, high permeability to a specific pore size, and high regeneration ability.

The presence of porosity in materials produced by P/M decreases their corrosion resistance comparatively to similar materials produced by conventional metallurgy. Other factors that affect the corrosion resistance of austenitic SS filters are the carbon and nitrogen content that might cause sensitization. The presence of interconnected porosity in materials produced by P/M has been associated in the literature to inferior corrosion resistance, comparatively to materials produced by conventional metallurgy [2,3]. According to literature [4-8], the stagnant electrolyte in the interconnected pores leads to the development of hydrogen concentration cells between the external and the internal surfaces of the pores. According to Reen and Hughes [8], large potential differences are established between the small anodic areas, within the pores, and the outer and passive areas at the pores surroundings. This large potential difference is responsible for the continuation of corrosion inside the pores while the neighboring region acts as cathodic areas, being protected. Based on these premises, the smaller the percentage of interconnected pores the better the corrosion resistance of the material. However, interconnected porosity is an essential characteristic of filters.

The characteristics of porosity, such as mean pore size, pore size distribution, and percentage of interconnected porosity are dependent on the fabrication process parameters. The development of filters of satisfactory corrosion resistance therefore must regard optimization of the process parameters that can be controlled. For instance, characteristics of the metal powder used, such as size, geometry and surface, and other parameters, for instance, compacting pressure, sintering atmosphere and sintering temperature.

In this investigation, the corrosion resistance of AISI 316 SS filters prepared using powders size in the (74-44) μm and (210-105) μm granulometric ranges and compacted with pressures of 300 MPa and 400 MPa has been evaluated in naturally aerated 0.5 M H_2SO_4 solution at 25 °C.

EXPERIMENTAL

AISI 316L water atomized powders of (74-44) μm and (210-105) μm granulometric ranges were used for producing the stainless steel filters investigated. The filters were compacted in a cylindrical matrix of 40 mm diameter, with compacting pressures of 300 and 400 MPa. After compacting, the filters were sintered for one hour in a 75% H_2 -25% N_2 atmosphere. The total and interconnected porosity were determined by measuring the geometric and hydrostatic densities. A theoretic density of 7.96 g/cm^3 was adopted. The porosity mean diameter was determined by mercury porosimetry. The porosity characteristics of the filters studied are shown in Table 1.

Table 1 - Porosity characteristics of filters.

| Powder Granulometric Range (μm) | Compacting Pressure (MPa) | Porosity (%) | | Mean Pores Diameter (μm) |
|---|------------------------------|--------------|----------------|--|
| | | Total | Interconnected | |
| (74-44) | 300 | 27 | 21 | 9.2 |
| | 400 | 23 | 18 | 6.0 |
| (210-105) | 300 | 27 | 23 | 12.8 |
| | 400 | 30 | 24 | 8.1 |

The corrosion resistance of the filters was investigated by weight loss measurements, chemical analysis of the solutions where the specimens had been immersed by atomic absorption spectroscopy, and surface observation by scanning electron microscopy (SEM) and X-ray dispersive (EDX) analysis after corrosion test.

The specimens for weight loss measurements were cut with an external surface area of approximately 7 cm^2 . Subsequently, they were degreased with acetone, dried in an oven at $200 \text{ }^\circ\text{C}$ for two hours, and then weighed. After weighing, the specimens were immersed in the test solution (naturally aerated $0.5 \text{ M H}_2\text{SO}_4$). After certain periods, the specimens were removed from solution, washed with acetone, rinsed with de-ionized water in an ultrasonic bath, dried in an oven at $200 \text{ }^\circ\text{C}$ for two hours, and then weighed.

Fig. 1 (a) and (b) shows filters compacted at 300 MPa using powders of granulometric ranges corresponding to $(74-44) \mu\text{m}$ and $(210-105) \mu\text{m}$, respectively.

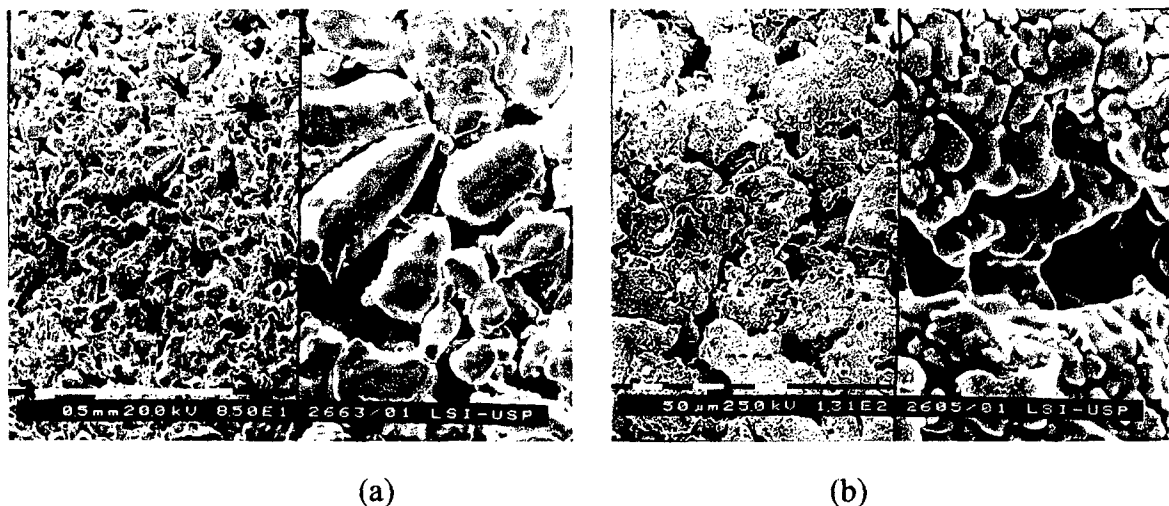


Fig. 1. Filters produced with powders of (a) (74-44) μm and (b) (210-105) μm , and compacted at 300 MPa, before corrosion test.

RESULTS AND DISCUSSION

The results of the weight loss measurements with immersion time are shown in Fig. 2. It can be seen that for filters prepared with powders of (210-105) μm and compacted at 400 MPa, there is an initial period, approximately 24 hours, during which their weight does not change with time. After this initial period, the kinetics of gravimetric losses was similar for the filters produced with the larger powders, (210-105) μm , for both compacting pressures. For the filters prepared with powders of smaller granulometric range, (74-44) μm , reduced weight losses were obtained for filters compacted at 300 MPa than at 400 MPa, during the whole test period. For this last filter, a large increase in weight loss occurred between 40 hours and 60 hours of immersion, but from 60 hours until the end of the test (180 hours), its weight did not change significantly. All filters showed stable weight values from 100 hours onwards.

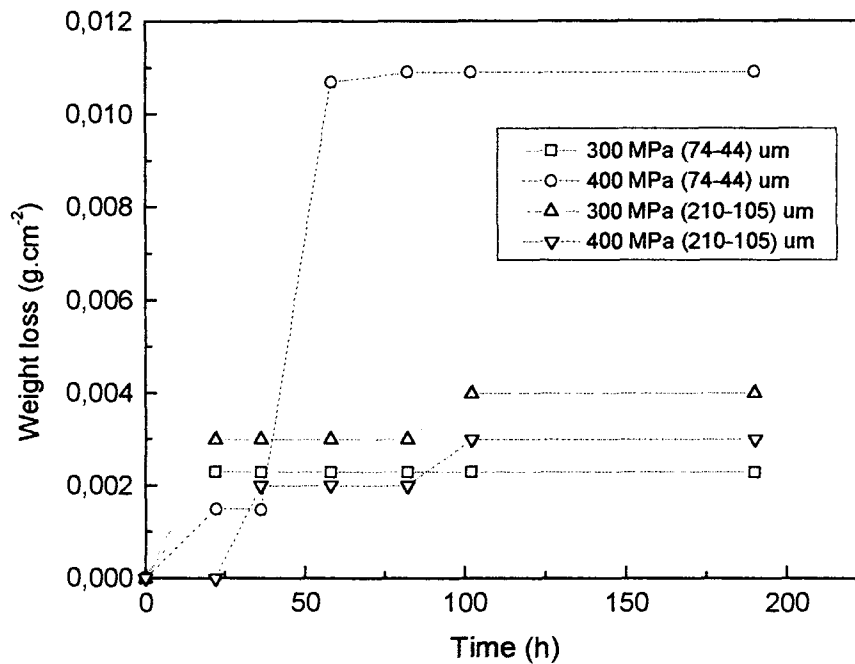


Fig. 2. Weight losses of filters at increasing times in 0.5M H₂SO₄ naturally aerated solution.

Despite of the decreased porosity of the filters prepared with finer powders (74-44) μm and compacted at 300 MPa comparatively to that compacted at 400 MPa, larger weight losses were associated to this last filter during the whole test period. One of the possible reasons for this apparent discrepancy is the enlarged pores associated to the filter compacted at lower pressure. The easier access of solution and oxygen into the inner areas of the broaden pores must assist the formation and/or reparation of a protective layer, resulting in a decreased corrosion rate. On the other hand, the more difficult renewal of solution inside the narrower pores, and the slower diffusion of oxygen through these pores, might prevent restoration of the protective layer at local defects. Additionally, the formation of concentration cells in the filters prepared with finer powders and compacted at 400 MPa, must have led to decreased corrosion resistance. For the filters compacted at 400 MPa, the very low weight losses in the first hours of immersion, could be indicative of an incubation

period. It is believed that after this incubation period, the inner areas of the pores work as anodic sites whereas the external surface, as cathodic regions.

Another possible reason for the decreased corrosion resistance associated to the filter prepared with finer powders and compacted at 400 MPa was its highest carbon contamination, comparatively to the other filters tested. The carbon and nitrogen contents of the starting powders were 0.024 % and 0.096 %, respectively, and the results in Table 3 show that contamination of the filters with these elements occurred during the fabrication process.

Table 3 - Carbon, oxygen e nitrogen contents in the 316 SS filters used in this study.

| P _{comp.} | Carbon content (wt %) | | Nitrogen content (wt %) | |
|--------------------|------------------------|------------|--------------------------|-----------|
| | (210-105)µm | (74-44) µm | (210-105) µm | (74-44)µm |
| 300 MPa | 0.10 | 0.12 | 0.56 | 0.25 |
| 400 MPa | 0.05 | 0.31 | 0.44 | 0.44 |

The highest carbon contents in the filters prepared with finer powders and compacted at 400 MPa, favors the precipitation of chromium carbides, and this in fact occurred, as it will be shown later.

The results of the chemical analysis of the main alloying elements (Fe, Cr, Ni and Mo) released into the test solution during a 180 hours period, obtained by atomic absorption spectroscopy, are shown in Table 2.

Table 2 – Corrosion products leached from AISI 316 filters into 0.5 M H₂SO₄ naturally aerated solution, during a period 180 hours . Results obtained by atomic absorption spectroscopy.

| Filter | Elements | | | |
|----------------------|---------------|---------------|---------------|---------------|
| | Fe (mg/mL) | Cr (mg/mL) | Ni (mg/mL) | Mo (µg/mL) |
| 300 MPa (74-44) µm | 0.004 | 0.024 | 0.004 | - |
| 400 MPa (74-44) µm | 2.93 | 0.46 | 0.62 | 45.38 |
| 300 MPa (210-105) µm | 0.003 | 0.16 | 0.003 | - |
| 400 MPa (210-105) µm | 0.009 | 0.005 | 0.001 | - |

The chemical analysis of the solution supported the decreased corrosion resistance associated to the filter prepared with finer powders and compacted at 400 MPa as compared to that compacted at 300 MPa. In fact, this last filter presented the best corrosion performance among the filters tested. Therefore, the high corrosion rates associated to the first type of filter must be likely due to the combination of its highest contamination and narrowest porosities.

SEM observation of the filter compacted at 400 MPa after approximately 180 hours of immersion in the test solution showed intense intergranular corrosion attack associated to it, as Fig. 3 shows.

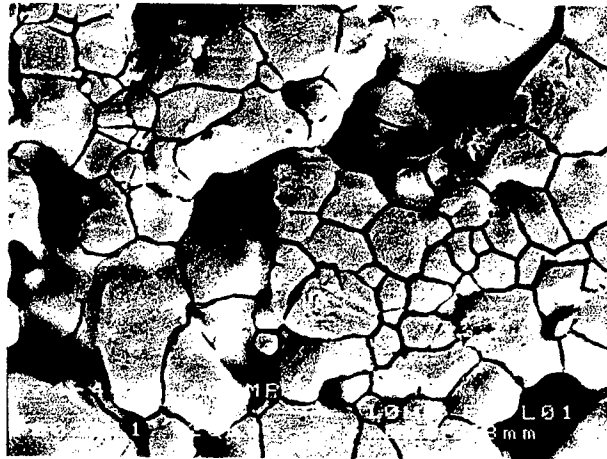


Fig. 3. SEM of filter prepared with powders of (74-44) μm compacted at 400 MPa after 180 hours in naturally aerated 0.5M H_2SO_4 solution.

SEM and EDX analysis of the filter compacted at 400 MPa showed chromium rich precipitates and oxides located at the grain boundaries (Fig. 4). X-ray diffraction of precipitates, obtained after matrix dissolution, showed that chromium nitrides and carbides are the main types present in the filters tested.

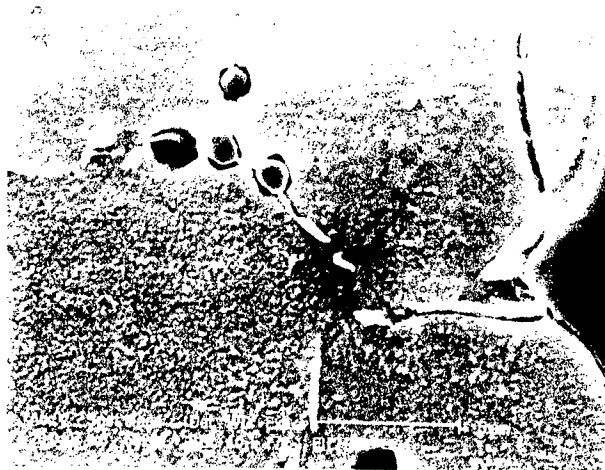


Fig. 4. SEM of filter fabricated with powder of (74-44) μm compacted at 400 MPa.

Precipitates at the grain boundaries are chromium rich and the round particles are aluminum silicates.

Contamination of the filters with carbon and nitrogen occurred during the fabrication process and led to the precipitation of chromium rich nitrides and carbides. Nitrogen contamination was caused by the sintering conditions used in this study (atmosphere: 75% H₂-25% N₂, temperature: 1250 °C) that was carried out in an industrial furnace. Carbon contamination was likely caused by reaction products of the binder (polyethylene glycol) and lubricant (zinc stearate) used, during the sintering process. These compounds form carbon at temperatures above 540 °C, and must be removed at low temperatures, before the sintering process, using oxidant gases, such as carbon dioxide. The highest contamination of the filter fabricated with finer powders and compacted at higher pressure could therefore be related to a hindered removal of reaction products originated by the binder/lubricant during sintering. Nitrogen and carbon contamination lead to chromium nitrides and/or carbides precipitation and consequently to steel sensitization. Sensitization works synergistically with the porosities, decreasing the corrosion resistance of the sintered steels [9].

The pores mean diameter of the filters fabricated with finer powders and compacted at 400 MPa was smaller (6.0 μm) than that of the filters compacted at 300 MPa (9.2 μm). As mentioned previously, narrow pores act as crevices favoring the formation of concentration cells and leading to intense localized corrosion. The corrosion mechanism of high density specimens must be different from that related to highly porous ones. For this last type of porous material, the easy access of electrolyte to the pores lead to a more uniform attack of the surface layer.

On the other hand, narrow pores act as pits/crevices and the stagnant electrolyte leads to a corrosion mechanism typical of crevice corrosion. By this mechanism, concentration cells are created between the external surface and the inner region of the pores [6]. Porosities behave as crevices if they are sufficiently large to let electrolyte in,

and, at the same time, are narrow enough to maintain it stagnant [7]. The stationary electrolyte inside the pores becomes increasingly acid with time, causing the attack of the passive layer on the porosities surface [4], and consequently, promotes corrosion inside the pores. This observation was supported by the results of this study. Surface observation of the filters after immersion test showed that the porosities in the filters produced with finer powders were more attacked comparatively to that in filters fabricated with coarser powders, mainly in those compacted at 400 MPa.

CONCLUSIONS

AISI 316 filters fabricated with finer powders (74-44) μm and compacted at higher pressure (400 MPa), and consequently with narrow porosities, were associated to decreased corrosion resistance. This filter was also associated to higher carbon/nitrogen contamination during sintering. It has been proposed that the low corrosion resistance of such a filter was due to a combination of electrolyte stagnation inside the pores, creating conditions for crevice corrosion, and consequently to intense corrosion attack, and to the higher contamination that led to sensitization and consequently to intergranular corrosion.

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