

## Corrosion protection of AISI 304 stainless steel filters by a surface treatment

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**Abstract:** In this study a corrosion protection method was used with AISI 304 SS filters and its effect on their corrosion resistance was evaluated in a sodium chloride solution (0.5 mol/L). The corrosion resistance of the surface treated filters was investigated at increasing temperatures, from 22 °C to 100 °C. It was found that the treatment improved the corrosion resistance of the filters at all temperatures tested. The film formed was maintained on the filters surface during the whole period of test (3 weeks). Surface analysis of untreated and treated filters, after corrosion tests, supported the indication of the beneficial effect of the corrosion protection treatment evaluated in this study.

### Introduction

Stainless steel (SS) filters prepared by powder metallurgy (P/M) techniques, mainly with AISI 304 powders, find many applications in various industries, such as food, nuclear, medical and chemical industries. These filters are used for solid-liquid separation, separation of antibiotic crystals, and many other applications where the corrosion products could cause final product contamination. Consequently, these materials should have high corrosion resistance [1-10].

However, the presence of open porosity in materials produced by P/M has been associated to inferior corrosion resistance, comparatively to materials produced by conventional metallurgy, a feature of their porosity [11,12]. Inside pores, aeration conditions differ from that on external areas and this can change the corrosion mechanism of sintered SS comparatively to conventional types. Open porosities increase the area exposed to a corrosive environment and can induce crevice corrosion reducing the passivity of sintered stainless steels [13-15]. To increase the corrosion resistance of these types of steels, protective measures could be used.

The literature reports that the corrosion resistance of conventional 304 SS in sulphuric acid can be increased by the addition of benzotriazole (BTAH) to the acid solution, the BTAH acting as a corrosion inhibitor [16-18]. It was also found [16] that a surface film was formed on immersion of conventional 304 SS in the BTAH containing solution and this film increased the corrosion resistance of the steel upon immersion in a sulphuric acid solution without BTAH. According to literature [19] this film acts as a mixed inhibitor, although it hinders predominantly the cathodic process.

The surface treatment for film formation in acid solution containing BTAH has also been previously carried out with 304L SS filters [20]. The results showed that the surface treated filters had nobler corrosion potentials and were associated to lower passive current densities comparatively to the bare (untreated) filters. The results also indicated the stability of the surface film after immersion in acid solution without BTAH and no signs of corrosion were seen on the surface of the filters after periods up to 11 days of immersion.

Once BTAH is a corrosion inhibitor of conventional SS by formation of a surface film, and it also showed inhibiting properties for 304L SS filters at room temperatures. It was the purpose of this study to evaluate if the film formed would also effectively inhibit the corrosion of 304L SS filters at temperatures in the range from the room temperature to 100 °C. The formation of the surface film on sintered 304L SS filters and also its effect on the corrosion resistance of the filters in a chloride solution, at increasing temperatures, was investigated. The corrosion resistance of the filters, either bare or surface treated, was evaluated by Scanning Electron Microscopy (SEM) at increasing times of test and by potentiodynamic polarization curves after immersion test.

## Experimental

*Material.* 304L stainless steel (SS) filters prepared from powder of (35–50) mesh were used in this study. The filters were compacted in a cylindrical matrix of 14 mm diameter with a compacting pressure of 300 MPa, without lubricant addition. After compacting, the filters were sintered under vacuum for one hour at 1200 °C. Cylindrical filters of 14.0 mm x 3.8 mm were obtained. The chemical composition of the compacted filters is shown in Table 1.

Table 1. Chemical composition (weight %) of 304L stainless steel filters tested.

| Element | C     | Si   | Mn    | Cr    | Ni   | Mo    | P     | Cu    | S     |
|---------|-------|------|-------|-------|------|-------|-------|-------|-------|
| (wt. %) | 0.019 | 0.90 | 0.067 | 16.47 | 9.22 | 0.034 | 0.019 | 0.029 | 0.016 |

Fig. 1 shows the surface of the sintered 304L SS filter used in this study. The total surface area of the filters tested was measured by mercury porosimetry and a value of 173.65 cm<sup>2</sup> was obtained.

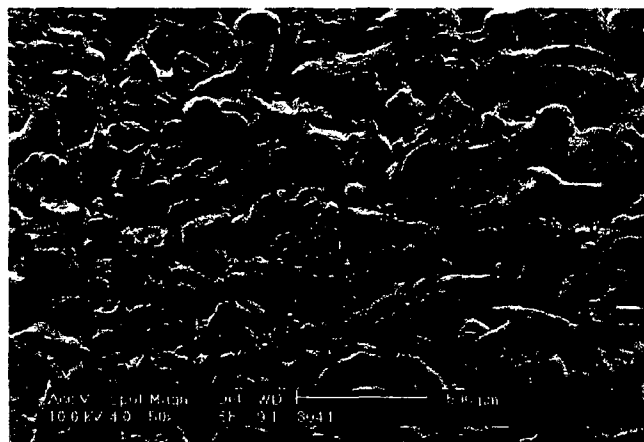


Fig. 1. Micrography by SEM of sintered 304L SS filter.

The large pores seen in the filter investigated in this study are due to the fairly low compacting pressures used (300 MPa), and to the large grains of the powder employed for filter preparation.

*Surface treatment.* A surface treatment to develop a film on 304L SS filters was carried out by immersion of 304L SS filters in a solution made of 3 mol/L H<sub>2</sub>SO<sub>4</sub> and 0.1 mol/L BTAH for 1 day. These filters were washed with water and then immediately immersed in the test solution.

*Test solution.* The corrosion resistance of the filters, either treated in BTAH containing solution or untreated, was evaluated by immersion in a 0.5 mol/L NaCl at increasing temperatures, 22 °C, 75 °C, 90 °C and 100 °C. The solution used in the immersion tests was quiescent and naturally aerated. The experimental conditions used in this study are shown in Table 2.

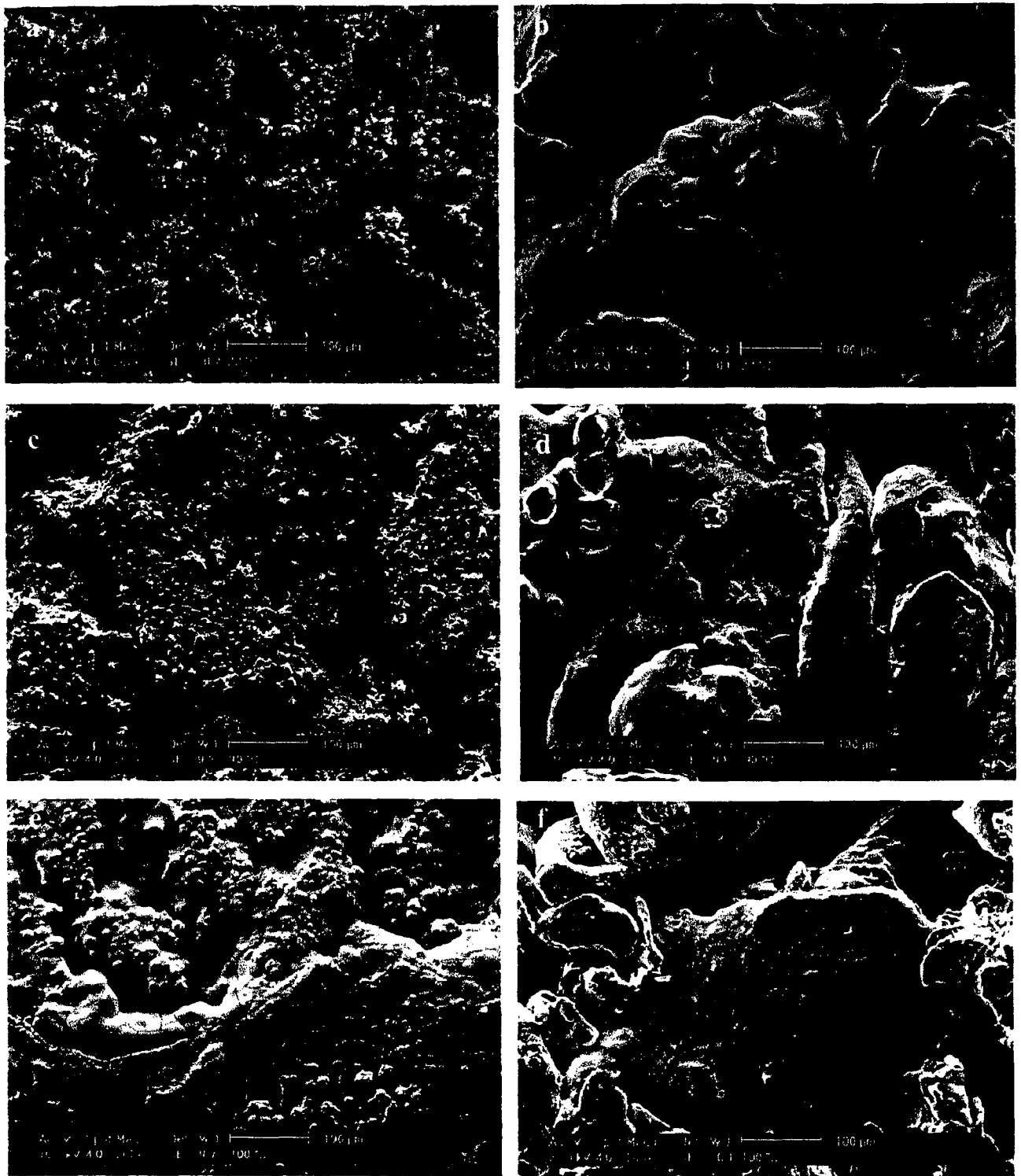


Fig. 2: SEM of 304L SS filter after corrosion test in naturally aerated 0.5 mol/L NaCl solution. (a) untreated filter after 1 week of immersion at 22 °C, (b) treated filter after 1 week of immersion at 22 °C, (c) untreated filter after 1 week at 75 °C followed by 1 week at 90 °C, (d) treated filter after 1 week at 75 °C followed by 1 week at 90 °C, (e) untreated filter after 1 week at 75 °C followed by 1 week at 90 °C and 1 week at 100 °C, (f) treated filter after 1 week at 75 °C followed by 1 week at 90 °C and 1 week at 100 °C.

Table 2. Experimental conditions of immersion test in 0.5 mol/L NaCl solution.

| Sample | 22 °C for 1 week | 75 °C for 1 week | 90 °C for 1 week | 100 °C for 1 week |
|--------|------------------|------------------|------------------|-------------------|
| A      | X                |                  |                  |                   |
| B      |                  | X                |                  |                   |
| C      |                  | X                | X                |                   |
| D      |                  | X                | X                | X                 |

For instance, sample B was immersed in 0.5 mol/L NaCl solution at 75 °C for 1 week, sample C was immersed for 2 weeks (1 week at 75 °C followed by 1 week at 90 °C) and sample D was tested by immersion in 0.5 mol/L for 3 weeks (1 week at 75 °C followed by 1 week at 90 °C and 1 week at 100 °C). After the immersion tests, the different samples were transferred to 0.5 mol/L NaCl at 25 °C, and then polarized to determine the pitting potential.

*Experimental.* Potentiodynamic polarization measurements were carried out in chloride media at 25 °C by means of a Solartron 1287 potentiostat coupled to a computer, with the scan rate of 1 mV/s. A three-electrode cell arrangement was used, with a large area platinum foil and a saturated calomel electrode (SCE) as counter and reference electrodes, respectively.

A Philips XL-30 scanning electron microscope (SEM) was used for surface observation before and after corrosion test.

## Results and discussion

The pitting potentials obtained from the polarization curves for the various specimens are shown in Table 3.

Table 3. Pitting potential for various samples of 304 SS filter in 0.5 mol/L NaCl at 25 °C.

| Sample | Pitting Potential / mV/SCE  |                         | $\Delta E = E_1 - E_0$ |
|--------|-----------------------------|-------------------------|------------------------|
|        | $E_0$ , (without treatment) | $E_1$ , surface treated |                        |
| A      | 130                         | 210                     | 80                     |
| B      | 158                         | 202                     | 44                     |
| C      | 217                         | 243                     | 26                     |
| D      | 278                         | 300                     | 22                     |

The results in Table 3 show nobler pitting potentials associated to the filters that were treated in BTAH solution, for the whole range of temperatures used in this study. The increase in temperature of test solution caused a decrease in the pitting potential difference ( $\Delta E$ ) between the treated and untreated filters. This result suggests that temperature affects the corrosion inhibiting performance of the film. Although the surface film formed on the BTAH treated filters caused only a small increment in the pitting potential, it in fact protected the substrate from corrosion, as it could be clearly seen by visual inspection after immersion test.

The surfaces of the filters, after corrosion test, were also examined by SEM. Fig. 2 shows the surfaces corresponding to treated and untreated filters, after immersion test. A significant difference between these two types of filters is clearly seen. The untreated filter (Fig. 2a) was highly corroded after only 1 week of immersion at 22 °C, whereas the treated filter, after the same test period, showed no corrosion on its surface, Fig. 2b.

It can be seen that for untreated filters the corrosion in chloride solution was of a generalized type, whereas for the treated filters, the surface was not attacked even after immersion for long periods (3 weeks) at fairly high temperatures (100 °C).

The results presented indicate that the BTAH film formed on the 304L SS filters is resistant up to temperatures of 100 °C (Figs. 2b, 2d and 2f). Corrosion was inhibited by the presence of this film even after 3 weeks of exposure to a sodium chloride solution, at increasing temperatures.

## Conclusions

A surface treatment of 304L SS filters in BTAH containing acid solution was carried out and it produced the formation of a surface film that remained on the surface after removal from this solution. This film increased the corrosion resistance of the filter in chloride solutions at temperatures up to 100 °C, and also decreased slightly the filter susceptibility to pitting.

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