



INFLUENCE OF VARIOUS SURFACE CONDITIONS ON PITTING CORROSION

RESISTANCE OF STAINLESS STEEL TUBES TYPE EN 1.4404

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ABSTRACT

The pitting resistance of pickled, ground and electropolished surfaces of EN 1.4404 (AISI 316L) tubes has been tested using cyclic polarization and ASTM G150 for determination of the pitting potential and CPT, respectively. The materials were tested in chloride-containing solutions with 0.05 to 5.0% Cl⁻. Crevice corrosion was avoided using a flushed port cell with a specially cast gasket. Ground finish having low surface roughness shows no improvement over the coarse pickled finish, while electropolished finish provides considerably higher resistance but at the same time scattered results. The two testing techniques give almost identical CPTs that show only little dependence on chloride concentration. The variation in pitting resistance has been correlated to the surface morphology of the different surface conditions.

Keywords: EN 1.4404, tubes, surface condition, pitting potential, critical pitting temperature, ASTM G150

INTRODUCTION

Besides from choosing the right stainless steel grade providing sufficient corrosion resistance, it is equally important to specify the right surface condition of materials used in food and pharmaceutical processing plants. This parameter obviously influences the cleanability and hygienic properties of the equipment. Moreover, the surface condition affects the corrosion resistance to a certain extent, which means that requirements may possibly be met by specifying the proper finish rather than upgrading the chosen alloy. On this basis, the main objective of the current investigation has been to quantify the dependence between surface condition and corrosion resistance of the most commonly used stainless steel types in food and pharmaceutical industry, i.e. EN 1.4404 also known as AISI 316L. In this business the main corrosion form in question is localized corrosion, such as pitting and crevice corrosion, since the equipment is exposed to chloride containing and potentially aggressive products and cleaning agents.

The literature provides several data about mechanical and chemical surface treatments in respect to corrosion resistance. However, the way data is obtained varies (to great deal) due to the lack of standardized testing techniques and so direct comparison of different datasets is difficult. Ericsson et al have published pitting potential data for AISI 316 that emphasizes the importance of pickling subsequent to abrasive treatment in order to improve resistance [1]. Moayed et al have shown that the critical pitting temperature (CPT) of 904L increases with increasing surface smoothness obtained by abrasive treatment [2]. Data based on exposure in ferric chloride (ASTM G48) show a similar tendency [3]. The beneficial effect of decreasing surface roughness has furthermore been proven by quantifying the nucleation rate of metastable pitting and electrochemical noise measurements [4,5,6]. Another approach has been to characterize the dependence between chemical composition of the passive layer (i.e. Cr/Fe ratio) and abrasive and chemical surface treatment [7,8].

Based on the literature as a whole, it is commonly accepted that electropolished finish provides better resistance than pickled finish, whereas mechanically ground or polished finish fall in the least corrosion resistant group. Presumably, this ranking can be related to the resulting amount of free inclusions and surface roughness related to the different surface treatments.

A wide range of data is also available for stainless steel surfaces exposed to atmospheric conditions [9,10,11]. Generally the same ranking is reported as for the fully immersed exposure conditions above. However, other parameters are more decisive here, such as wetting properties and the ability to collect dirt.

The main approach of the current study has been to apply well-known testing methods to characterize the pitting resistance of various surface conditions. This has involved use of two testing methods, namely cyclic polarization and determination of the critical pitting temperature (CPT) according to ASTM G150. The cyclic polarization technique measures the pitting potential, but when testing a wide matrix of chloride concentrations and temperatures, this technique can also provide the CPT. This approach was originally introduced by Brigham et al [12,13] and has formed the basis for establishing the ASTM G150 standard [14].

EXPERIMENTAL PROCEDURES

The tested materials include tube materials in three different surface finishes: Pickled, ground and electropolished. The materials were supplied in accordance with standards EN 1.4404 and DIN 11850, BC [15]. Surface roughness and chemical composition are listed in Tables 1 and 2.

The critical pitting temperature (CPT) was obtained in two ways, either by performing a series polarization tests at fixed temperature or by using the ASTM G150 method. The test specimens (60x60 mm) were cut from sections of the tube without the longitudinal weld. Cleaning prior to exposure involved treatment in 2% NaOH and 2% HNO₃ to simulate conventional cleaning-in-place (CIP). The same set-up was used for both cyclic polarization and ASTM G150, Figure 1. To avoid crevice corrosion, a flushed port cell with a specially formed gasket was used. The exposed tube area was 5 cm².

Polarization was performed from the corrosion potential in anodic direction until 1 mA/cm² or +1000 mV SCE at a scan rate of 10 mV/min. The return rate was 20 mV/min. Prior to polarization, the specimen was conditioned at -200 mV SCE for 5 minutes. The chloride concentration of the solution ranged from 500 to 50,000 mg/l Cl⁻.

In the ASTM G150 test, the specimen was polarized to +700 mV SCE in a solution of 1 M NaCl. The temperature was raised from 0°C (or 1°C) at a rate of 1°C/min up to 95°C. The test was ended when the current exceeded 1 mA/cm².

All test solutions were prepared from demineralized water with an addition of NaCl. The pH was neutral (7-8) as the solutions were unbuffered. Deaeration was maintained by purging with nitrogen throughout the tests.

RESULTS

Cyclic Polarization

The cyclic polarization experiments have involved testing at several combinations of chloride concentration and temperature. Besides determining the dependence of the critical pitting potential, the tested levels were selected to obtain the CPT as precisely as possible. This implies narrowing of the test temperature in a certain region, which results in an asymmetric test matrix. Pickled tubes (A) have been tested at seven chloride levels. Since only a minor difference was observed within a decade of chloride concentration, the test matrix was reduced to three levels for the ground (B) and electropolished (C) tubes. The complete test matrix and obtained pitting potentials are listed in Tables 3 to 5.

Figure 2 shows examples of obtained polarization diagrams. The pitting potential is read at 10 μA/cm², but the curves also provide information about passive current, repassivation potential and exchanged charge ascribed to pitting. At temperatures below or near CPT there may be no clear transition from passive to active state below +1000 mV SCE. In this case the pitting potential is read as +1000 mV SCE. In cases of doubt we assume a charge of the hysteresis above 100 mC/cm² to verify persistent pitting. The charge related to pitting is in the range of 600 – 38.000 mC/cm² where values increase with test temperature.

The technique to avoid crevice corrosion using a flush port proved to work well. In most cases the pits are located within the exposed area of 26 mm in diameter as shown in Figure 3. Otherwise the test was discarded, which ensures a reasonable reproducibility within the tests.

The amount of pits on the exposed specimens varies within the test matrix. For materials A and B, the number generally increases from few pits at temperatures near CPT to 30-50 pits at higher temperature. The electropolished tubes deviate from this behavior by showing only few pits (often just one).

The overall dependence between pitting potential and temperature is illustrated for the pickled tube in Figure 4a. A sharp increase in pitting potential is observed at low temperatures where CPT is read. At higher temperatures the decrease in pitting potential is less pronounced and remains within a band of 250 mV for tested chloride levels from 0.05% to 5.0%.

When comparing the different surface conditions, the electropolished finish shows by far the highest pitting potentials although some scatter is observed at 0.05% and 0.5% Cl⁻, Figure 4b and Table 5. The pickled and ground tubes are somewhat comparable both showing considerably lower pitting potentials.

ASTM G150

The ASTM G150 method is perhaps the only standardized electrochemical method that allows comparison of pitting data between testing laboratories. Consequently, it was decided to include this relatively new method in the study. The method is originally developed to cover stainless steel grades from EN 1.4404 up to “six-moly” types; therefore testing is made at high chloride concentration (1 M NaCl) and high potential (+700 mV SCE) to allow distinguishing between a wide range of alloys within the temperature range from 0°C to 100°C.

These test conditions imply rapid activation of the tested EN 1.4404 grade, which sometimes compromises the reading of a clean CPT, Figure 5. However, from the data presented in this graph and Table 6 it is clear that the as-delivered electropolished tubes provide much higher CPT than the pickled and ground tubes.

Since the different tubes originate from different alloy charges, there are differences in composition that are in favor of the electropolished tube. This difference is described by the pitting resistance equivalent of each tube in Table 6. In order to evaluate the effect of this difference, samples of each tube were electropolished using a conventional commercial treatment. In this way, effects from the original surface condition were reset thus allowing quantification of the alloy effects in a second series of G150 tests. From Table 6 and Figure 6 it appears that the effect of the additional electropolishing brings the CPT of the originally electropolished tube to a level comparable to that of the pickled and ground tubes, which both show little effect of the additional electropolishing.

Surface and Pit Morphology

In order to correlate the above pitting results with surface morphology, both unexposed and exposed materials were examined in a scanning electron microscope. The surfaces as received from the pipe mill are shown in Figures 7a-7c. The pickled material (Figure 7a) shows the usual grain boundary ditches and scattered open pits from surface-breaking inclusion. The ground material (Figure 7b) shows the usual smearing with a lot of laps and possible occluded cell sites. The electropolished surface (Figure 7c) is extremely flat and smooth at this magnification, with just a hint of twin and grain boundaries revealed

and some open depressions, possibly former surface breaking inclusions that have been removed and the hole further opened.

The characteristics of pit initiation are best observed with the specimens from ASTM G150 testing because the test is terminated as soon as significant corrosion is observed. The specimens exposed to an entire cyclic polarization have drawn a much larger amount of charge, and the pits have generally developed into the well-known spheroid shape. The three surface conditions tested show markedly different initiation behavior, Figures 7d-7f.

The pickled surface (Figure 7d) develops a number of spheroid pits. The ground surface (Figure 7e) develops several spots, where the ground surface starts to reveal the grain structure of the un-deformed material below the surface. The depth of attack in the initiation stage is quite shallow. The electropolished surface (Figure 7f) develops a pattern that appears to be associated with rolled-out inclusion particles. Just one or perhaps a few such sites develop on the entire test area (5 cm²).

Because the pitting resistance of the mill-electropolished tube was so remarkably better than the pickled or ground, a number of specimens from all three tube types were treated at a commercial electropolisher. The electropolishing bath used is of the phosphoric-sulfuric acid type; the procedure, current and polishing time applied are based on the experience of the operator. The acceptance criterion is basically that the specimen gets a reflecting surface, and if this is not obtained in the first go, the rack is immediately given additional treatment. This did not result in any significant improvement of the pickled or ground specimens, while it greatly reduced the CPT of the mill-electropolished tubes; see Table 6 and Figure 6.

SEM investigation of the specimens after the commercial electropolish before corrosion testing shows that all three types of the original surface have become flat with only a shadow of twin and grain boundaries, Figures 8a-8c. However, the originally pickled tubes (Figure 8a) now show the same type of etched-out inclusions as the mill-electropolished after ASTM G150 test, albeit more numerous.

The originally ground specimens (Figure 8b) now show a different type of etched-out inclusions, much more narrow and deep. The slightly darker spots adjacent to the etch-holes seen on the SEM micrograph indicate the presence of lighter elements.

The originally mill-electropolished (Figure 8c) now shows a number of etched-out inclusions, not very different from those observed after G150 test, but more numerous.

DISCUSSION

The ASTM G150 method was developed with a view to ranking of different stainless steels. However, the very concept that below a certain temperature pitting simply does not occur has attraction from an engineering application as well. Though ASTM G150 specifies testing in 1 molar NaCl solution, the test method is directly useable for other environments as well. We expected that this CPT would depend on the composition of the environment, i.e. the chloride content. However, this dependency is remarkably small, as can be seen from Figure 4 and Table 7: In the range of chloride concentration from 0.05 to 5% all the CPT results fall within a band less than 10°C wide. The actual position of this band depends on alloy composition and surface treatment.

From an engineering point of view, the CPT value for 1.4404 type alloys is, unfortunately, so low that economical constraints dictate the use at temperatures above the CPT, meaning that too harsh combinations of oxidation power, chloride content, and temperature will cause pitting attack. For this purpose, engineering diagrams like our Figure 4a have found widespread use.

With a temperature somewhat above the CPT, the lines in Figure 4a appear straight enough to be represented by a straight line. It has been suggested that E_{pit} may depend linearly on $\log[\text{Cl}^-]$. We do not claim that such a simple, linear and additive model represents any fundamental law of nature, but for engineering applications, a mathematical formulation may be useful. Linear regression on the data sets for A and B specimens give the following:

For pickled material A:

$$E_{\text{pit}} = 456 (\pm 21) - 4.6 (\pm 0.4) \times \text{Temp} - 159 (\pm 13) \times \log[\text{Cl}^-] ; R^2 = 0.91 \quad (1)$$

For ground material B:

$$E_{\text{pit}} = 543 (\pm 29) - 5.0 (\pm 0.4) \times \text{Temp} - 180 (\pm 14) \times \log[\text{Cl}^-] ; R^2 = 0.96 \quad (2)$$

E_{pit} in mV (SCE), Temp in °C, $[\text{Cl}^-]$ in g/L. ($\pm XX$) reports one standard error.

The R^2 values are not too convincing, but knowing the magnitude of the standard errors allows statement of an indication of the risk involved when comparing observed operating conditions with the data set.

CONCLUSIONS

Two techniques, cyclic polarization and ASTM G150, have been used for determining the pitting resistance of three surface conditions of EN 1.4404 stainless steel tubes.

Ground finish having little surface roughness shows no improvement over the coarser pickled finish when taking the small differences in alloy composition into account. In comparison, the electropolished finish shows much higher CPT, but at the same time scattered results, which has been ascribed to the reduction of possible pit initiation sites.

The chloride concentration (within an interval of 0.05% to 5%) has only little effect on the potential independent CPT and so the pitting potential should preferably be used for material selection in this range.

The obtained matrix of CPT and pitting potential data enables proper specification of the surface condition of stainless steel tubes to suit the corrosive conditions in food or pharmaceutical processing equipment.

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TABLE 1.

Characteristics of tested materials. All materials were longitudinal welded tubes according to EN 1.4404, DIN 11850, BC in the dimension: 63.3x1.6 mm, ODxWT.

| Material | Production route | Ra, μm^a | Rz, μm^a |
|--------------------|---|---------------------|---------------------|
| A. Pickled | Cold rolled, annealed, pickled | 0.48 | 4.6 |
| B. Ground | Cold rolled, annealed, pickled, polished | 0.19 | 1.7 |
| C. Electropolished | Cold rolled, annealed, pickled, electropolished | 0.12 | 1.0 |

a. Average roughness (Ra) and mean roughness depth (Rz), average of 4 measurements in longitudinal direction.

TABLE 2.

Chemical Composition (% wt) and PRE of tested tube materials.

| Material | %C | %Si | %Mn | %P | %S | %Cr | %Ni | %Mo | %Ti | %N | PRE ^a |
|-----------------|-------|------|------|-------|-------|------|------|------|-------|-------|------------------|
| Pickled | 0.024 | 0.38 | 1.40 | 0.040 | 0.005 | 17.2 | 10.7 | 2.13 | 0.007 | 0.035 | 24.8 |
| Ground | 0.014 | 0.50 | 1.85 | 0.036 | 0.012 | 16.8 | 10.9 | 2.15 | 0.009 | 0.022 | 24.2 |
| Electropolished | 0.021 | 0.60 | 0.94 | 0.032 | 0.006 | 18.1 | 11.6 | 2.21 | 0.009 | 0.036 | 26.0 |

a. Pitting Resistance Equivalent, $\text{PRE} = \text{Cr} + 3.3 \text{ Mo} + 16 \text{ N}$

TABLE 3.

Pitting potentials determined by using cyclic polarization. Pickled tube.

| T, °C | Chloride Concentration, mg/l | | | | | | |
|-------|------------------------------|-------|-------|-------|----------|--------|----------|
| | 500 | 1,000 | 2,000 | 5,000 | 10,000 | 20,000 | 50,000 |
| 5 | -- | -- | -- | -- | -- | -- | 923 |
| 7.5 | -- | -- | -- | -- | -- | -- | 1000 |
| 10 | 1000 | 1000 | 1000 | 1000 | 1000 | 1000 | 774, 718 |
| 12,5 | -- | -- | -- | 882 | -- | -- | 698, 583 |
| 15 | 980 | -- | -- | 837 | 618, 623 | 714 | -- |
| 17.5 | 1000 | -- | -- | 542 | -- | -- | -- |
| 20 | 622 | 569 | 549 | 542 | 429 | 439 | 446 |
| 30 | 516 | 398 | 416 | 365 | 289 | 196 | 184 |
| 40 | 448 | 381 | 355 | 294 | 202 | 174 | -- |
| 50 | 395 | 378 | 297 | 259 | 186 | 166 | -- |
| 60 | 318 | 312 | -- | 259 | -- | -- | 70 |
| 70 | -- | 294 | -- | -- | -- | -- | -- |
| 80 | 284 | 255 | -- | 183 | -- | -- | 2 |
| 95 | 252 | -- | -- | 133 | -- | -- | -39 |

TABLE 4.

Pitting potentials of ground tube determined by using cyclic polarization.

| T, °C | Chloride Conc., mg/l | | |
|-------|----------------------|----------|--------|
| | 500 | 5,000 | 50,000 |
| 5 | 1000 | 911 | 896 |
| 7.5 | 1000 | 737 | 872 |
| 10 | 855 | 734, 675 | 516 |
| 15 | 789 | -- | -- |
| 20 | 668 | 504 | 381 |
| 40 | 593 | 318 | 164 |
| 60 | 539 | 246 | 153 |
| 80 | 383 | 148 | 12 |
| 95 | 282 | 148 | -44 |

TABLE 5.

Pitting potentials of eletropolished tube determined by using cyclic polarization.

| T, °C | Chloride Conc., mg/l | | |
|-------|----------------------|----------|--------|
| | 500 | 5,000 | 50,000 |
| 10 | 1000 | 1000 | 1000 |
| 20 | 1000 | 1000 | 1000 |
| 40 | 1000 | 1000 | 1000 |
| 50 | -- | 666, 545 | 896 |
| 60 | 1000 | 944 | 509 |
| 70 | 729 | -- | -- |
| 80 | 525 | 345, 928 | 260 |
| 95 | 885 | 406 | 133 |
| | -- | -- | -- |

TABLE 6.

Critical Pitting Temperatures determined using ASTM G150.

| Material | PRE ^a | CPT, °C | |
|-----------------|------------------|------------------------------------|-----------------------------|
| | | As supplied from tube manufacturer | Commercial electropolishing |
| Pickled | 24.8 | 8.6 | 18.6 |
| | | 17.7 | 15.2 |
| | | 16.7 | 15.8 |
| Ground | 24.2 | 7.0 | 5.2 |
| | | 4.6 | 5.3 |
| | | 8.2 | 7.4 |
| Electropolished | 26.0 | 68.0 | 23.3 |
| | | 64.4 | 20.1 |
| | | 57.4 | 21.6 |
| | | (38.7) | -- |

a. Pitting Resistance Equivalent, $PRE = Cr + 3.3 Mo + 16 N$

TABLE 7.

Comparison of obtained critical pitting temperatures (CPT, °C) using either polarization or ASTM G150.

| CPT in °C | Polarization | | | ASTM G150 |
|-----------------|------------------------|-------|--------|-----------|
| | Cl ⁻ , mg/l | | | |
| Material | 500 | 5,000 | 50,000 | 35,500 |
| Pickled | 17 | 14 | 9 | 14 ± 5 |
| Ground | 14 | 9 | 6 | 7 ± 2 |
| Electropolished | 65 | 45 | 45 | 57 ± 13 |

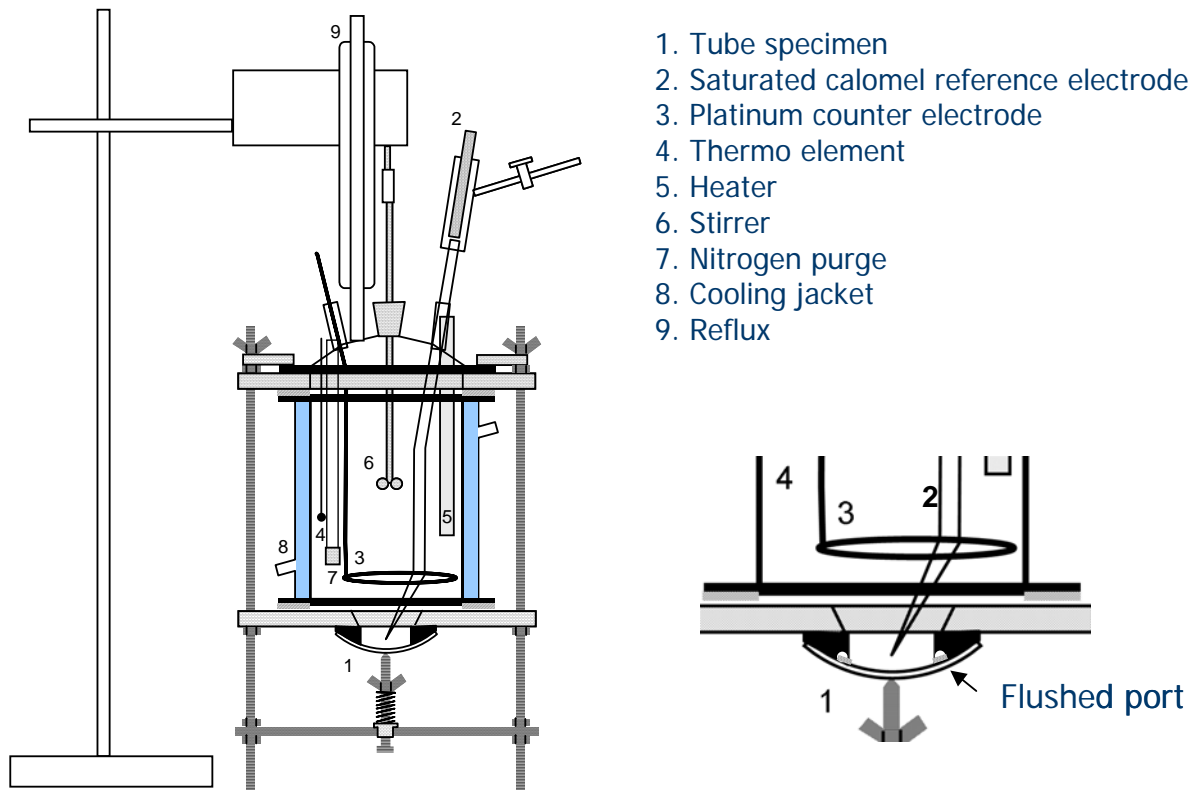


FIGURE 1 - Schematic of the test setup. A specially cast silicone gasket was used to form the flushed port.

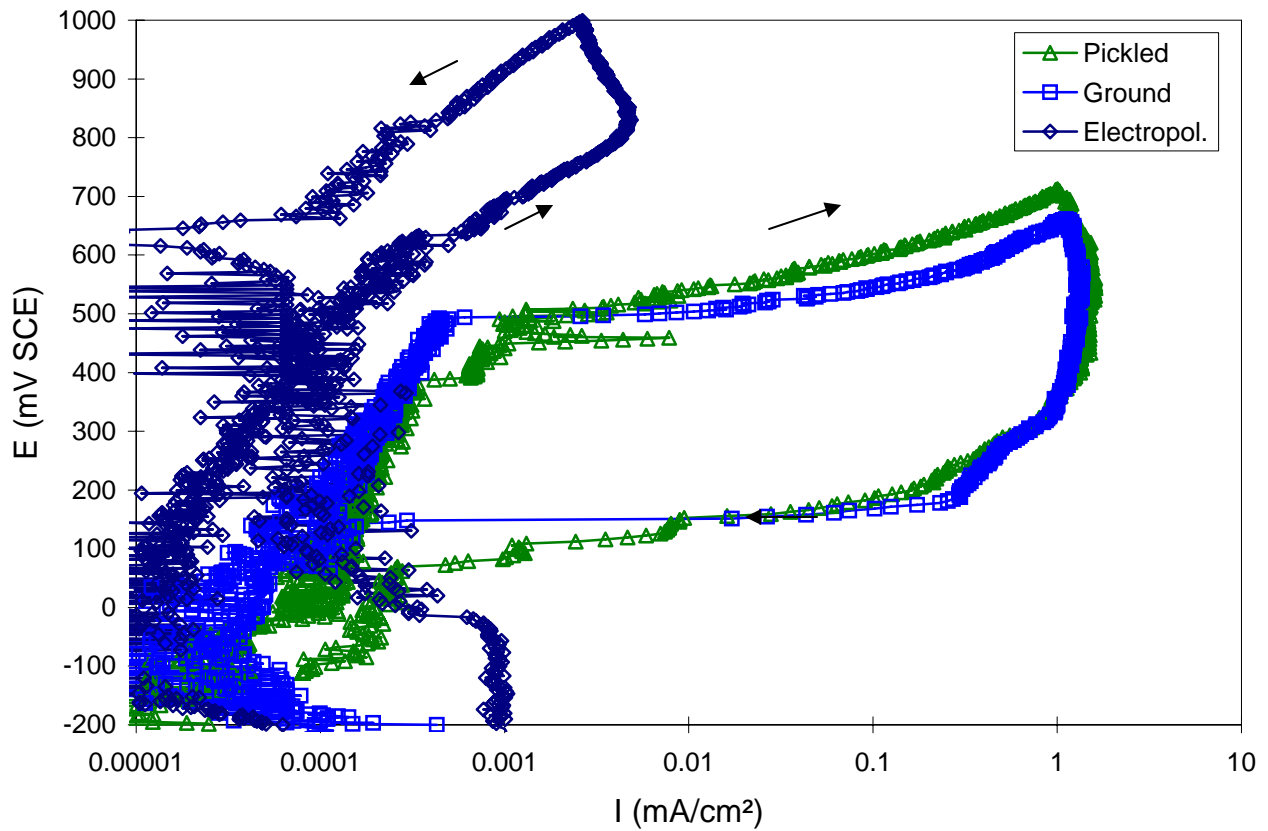


FIGURE 2 – Examples of obtained polarization diagrams obtained at 20°C in 0.5% Cl₂.

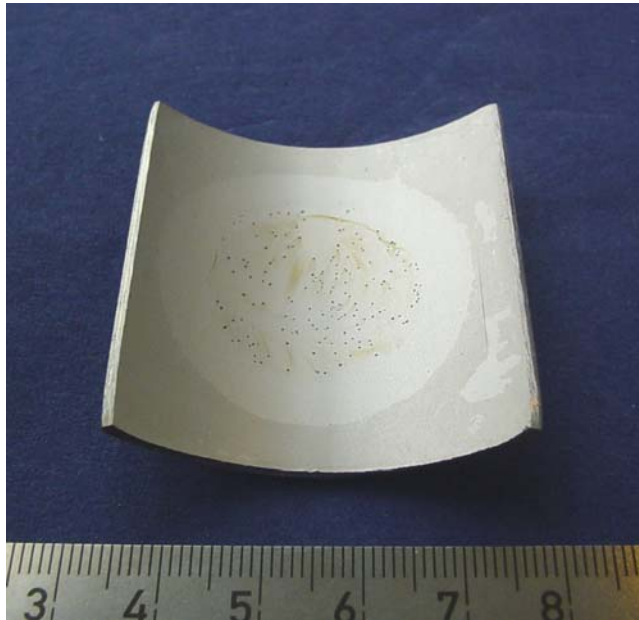
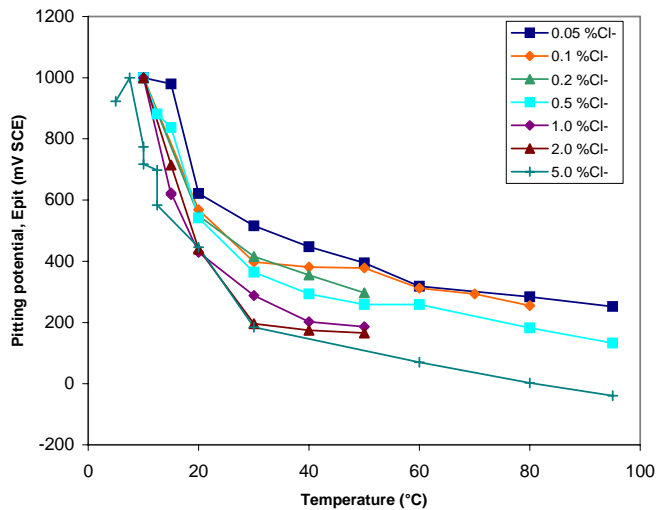
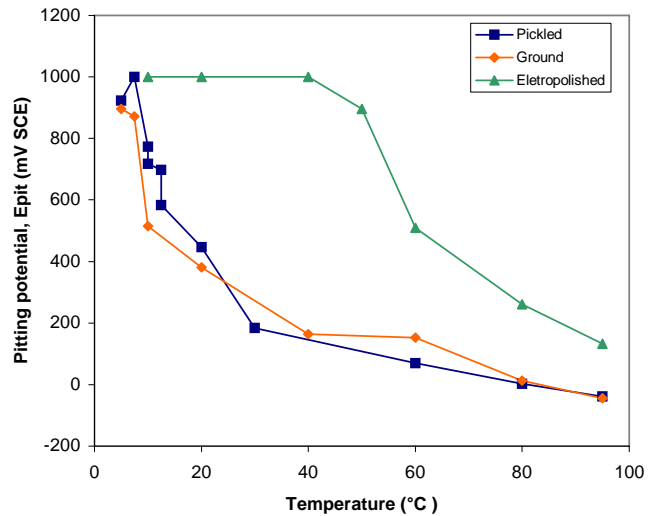


FIGURE 3 – Tested specimen. Pits are located within the exposed area of 26 mm in diameter. There are no signs of crevice corrosion along the flushed gasket.



a.



b.

FIGURE 4 – Pitting potential vs. temperature measured by polarization. a) Pickled material at all chloride levels. b) Comparison of pickled, ground and electropolished finish in solution with 5.0% Cl⁻.

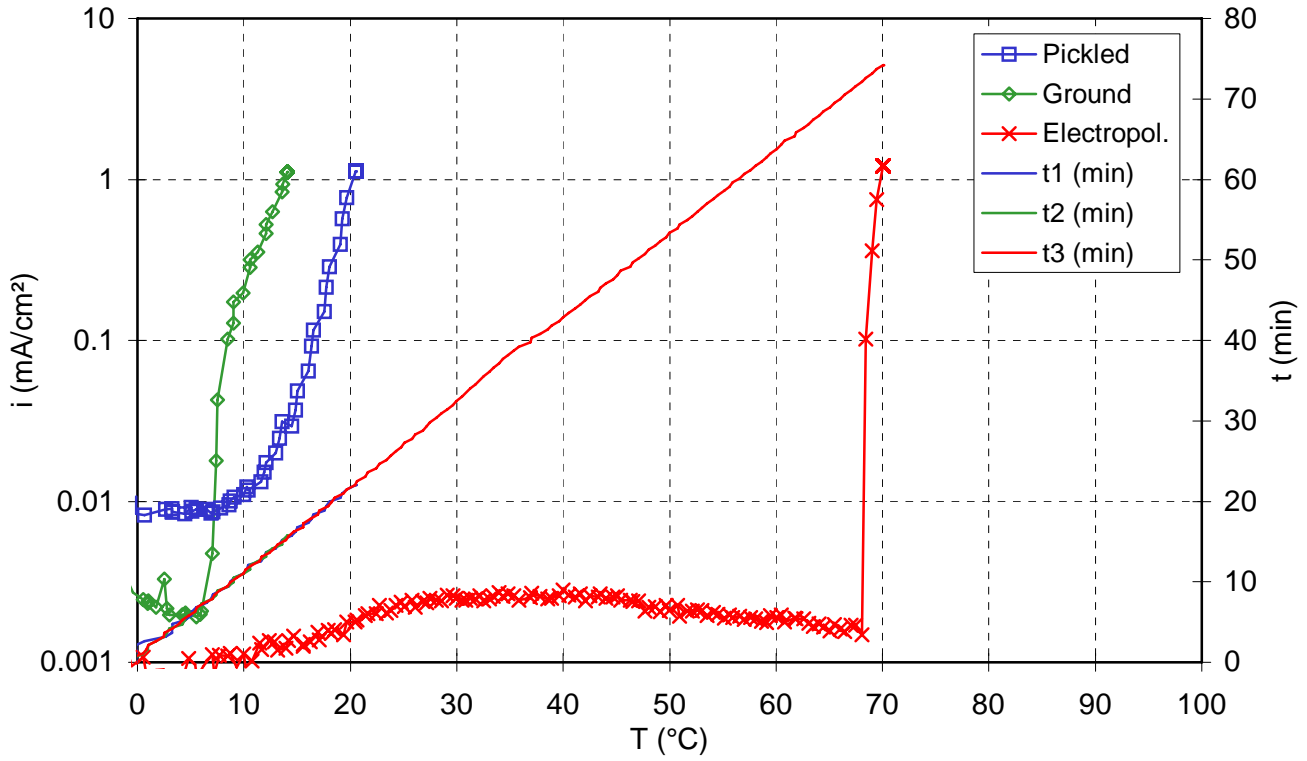


FIGURE 5 – Examples of obtained CPT curves using the ASTM G150 technique.

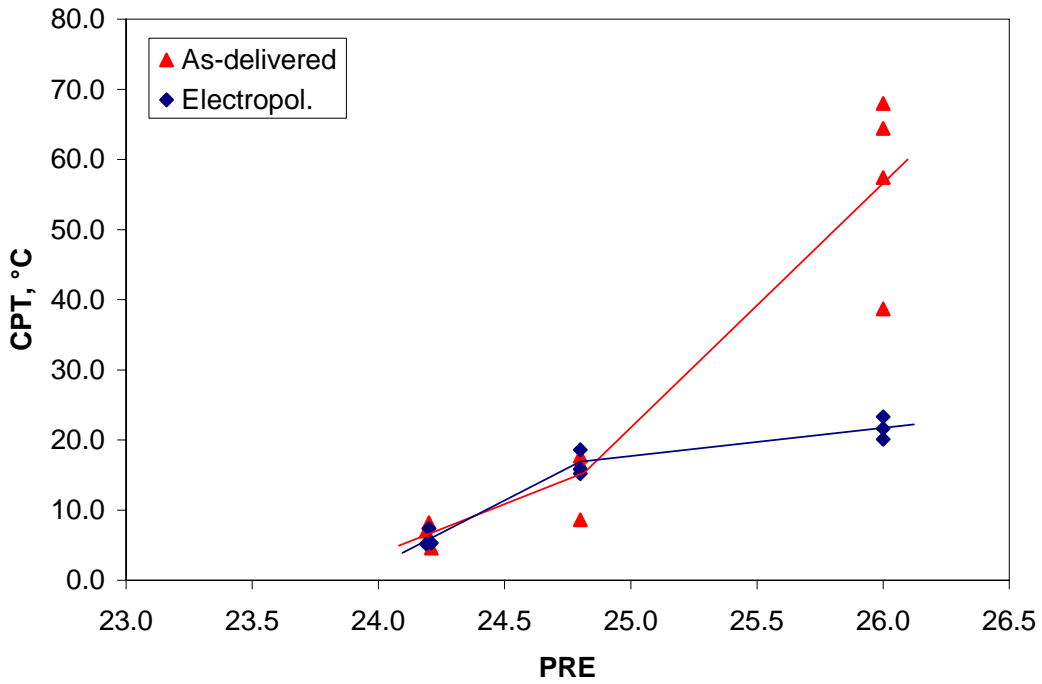
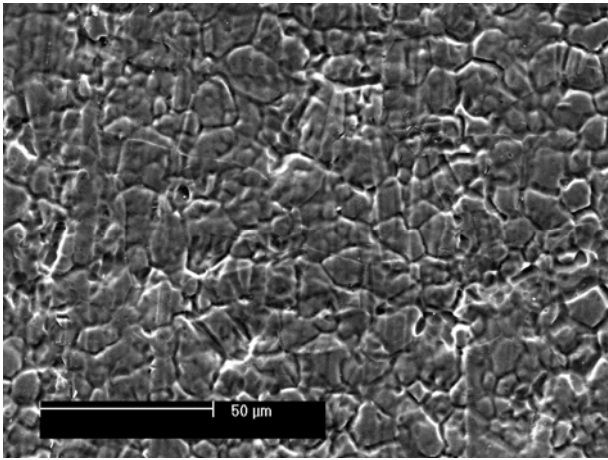
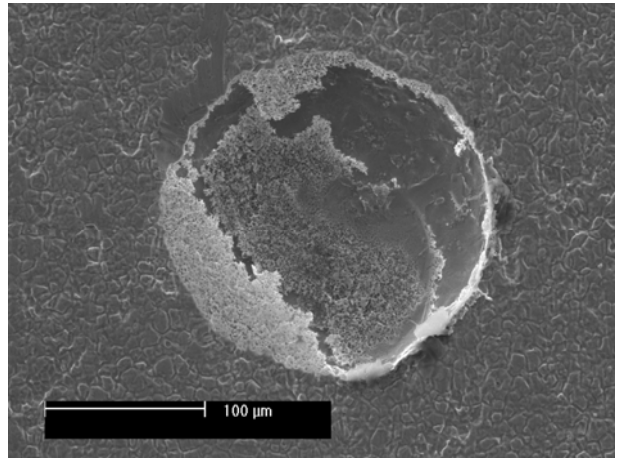


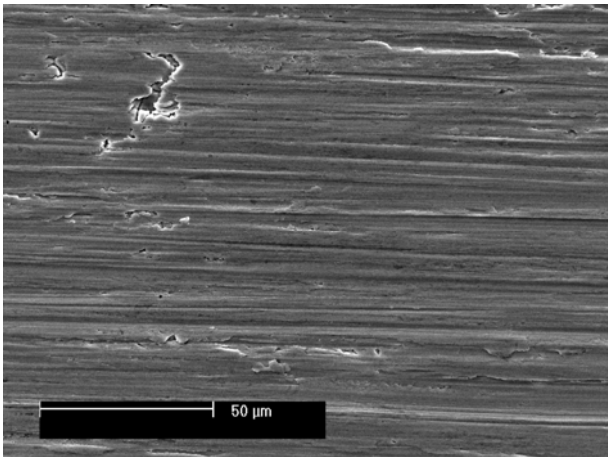
FIGURE 6 – Dependence between CPT and Pitting Resistance Equivalent (PRE) of tubes in as-delivered and electropolished condition.



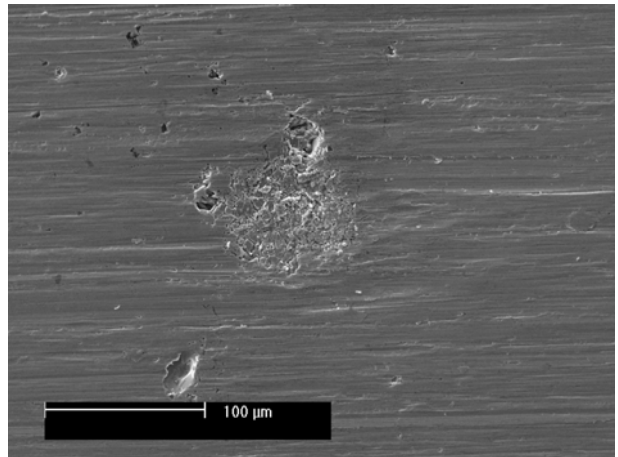
a) Pickled tube



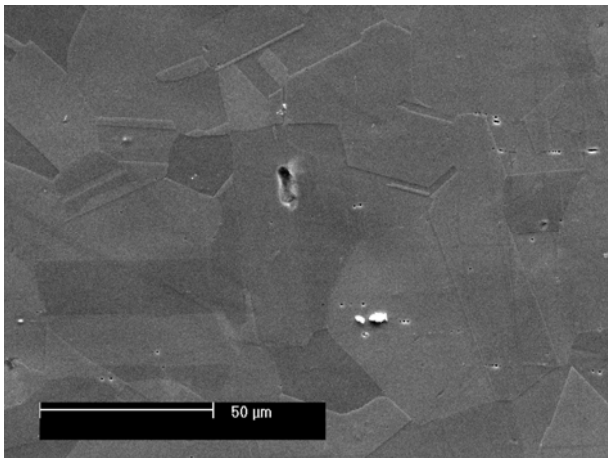
d) Pit in pickled tube



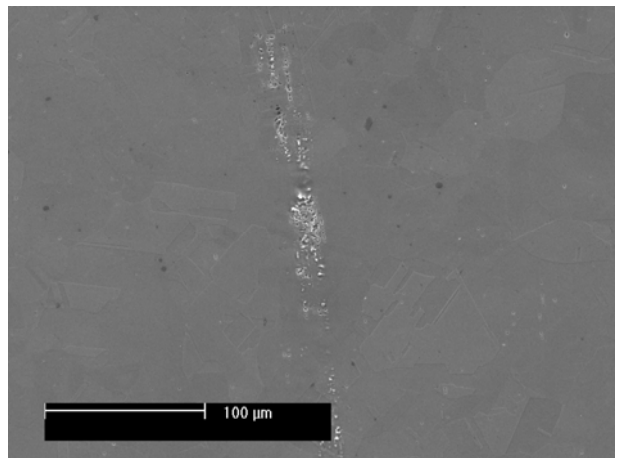
b) Ground tube



e) Pit in ground tube

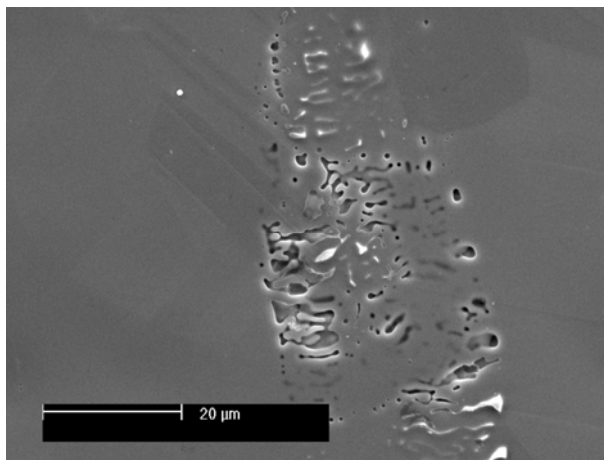


c) Electropolished tube

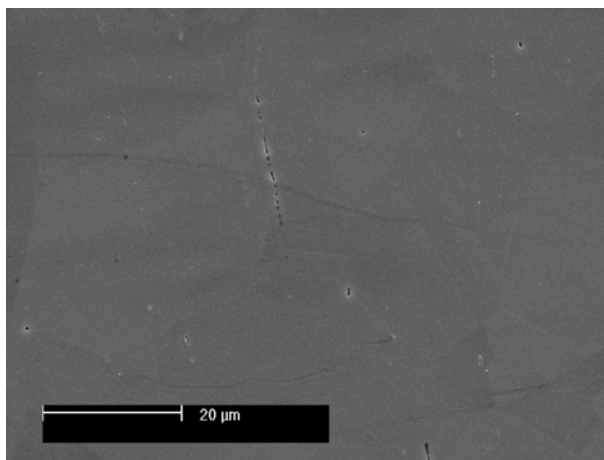


f) Pit in electropolished tube

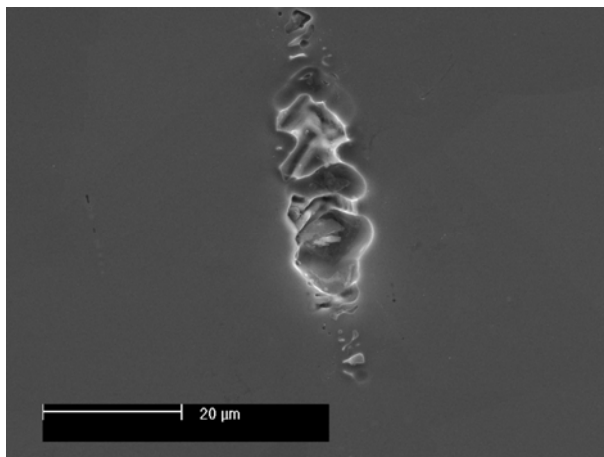
FIGURE 7 – Scanning electron micrographs of tube surfaces as-delivered from tube supplier (a-c) and after ASTM G150 testing (d-e).



a) Pickled and electropolished tube



b) Ground and electropolished tube



c) Tube electropolished twice

FIGURE 8 – Scanning electron micrographs of tubes electropolished by commercial process. The pictures show pits in the surface after ASTM G150 test.