



## Letter

# The effect of annealing on the corrosion behaviour of 444 stainless steel for drinking water applications



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## ABSTRACT

In this study, the corrosion behaviour of annealed and not annealed AISI 444 ferritic stainless steel in tap water with and without addition of selected concentrations of chloride ions was investigated. Cyclic potentiodynamic macro (large area) and micro (small area) polarization measurements (CPP), salt spray test, SEM and EDS analysis were employed to evaluate the pitting and crevice corrosion susceptibility of annealed and not annealed AISI 444. The results obtained indicate that annealing does not improve the resistance to pitting and crevice corrosion. Moreover, micro CPP indicates local susceptibility to pitting on both annealed and not annealed materials; such susceptibility was not evident from macropolarization tests.

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## 1. Introduction

The wide use of austenitic stainless steel in several industrial sectors is due to the combination of mechanical properties and corrosion resistance [1]. Recently, owing to the market competition, a substantial increase in the austenitic stainless steels price has been observed, with price per ton increasing from 2.500 to 13.200€ over the last five years [2]. This substantial price increase is attributed to the development of the Asian and South American economies, contributing to an increased demand for Ni and Mo, key alloying elements for the production of austenitic stainless steel.

A possible route to minimize the impact of increased Ni and Mo cost on stainless steel price is the reduction of Ni and Mo content. Thus the replacement of austenitic stainless steels with ferritic steels, such as the AISI 444, is an option for some application. However, it is important that the ferritic stainless steel provides a solution of similar quality both in terms of mechanical properties and corrosion resistance. Areas in which the applications of ferritic stainless steels could increase in the near future range from industrial plants to the transport of drinking water. While the pitting corrosion resistance of the austenitic stainless steels 304 L and 316 L is widely studied [3,4], less attention has been paid to the corrosion behaviour of AISI 444 [5,6].

Tests performed with mixtures of hydrocarbons (immersion in heavy oil and cyclic reactivation EPR-DL), at temperatures typical

of a distillation tower of crude oil (200, 300 and 400 °C) showed that AISI 444 is not substantially more susceptible to corrosion compared to the AISI 304 typically adopted for such applications [7]. On the other hand, tests performed at temperatures typical of distillation towers of the petrochemical industry (400, 475 °C) [6], indicate that at these temperatures the maximum precipitation of  $\alpha$  phase occurs, resulting in an improvement of the mechanical properties but a decay of the corrosion resistance. Additional tests, performed to evaluate the effect of annealing at temperatures of 955, 980 and 1010 °C [8], revealed precipitation of titanium and niobium carbides and nitrides, which improve the mechanical properties and make the AISI 444 suitable for deep drawing. However, no tests were carried out to characterize the corrosion behaviour after annealing.

Recently, due to new restrictions on the use of materials that might potentially release Ni ions, AISI 444 has been indicated as a potential substitute of 304 and 316 stainless steels, traditionally employed for drinking water applications [2]. Often, a press fitting system is used to join reliably and economically pipelines of diameter ranging from 15 to 108 mm for various type of industrial, residential and commercial applications. Press fittings have, at each end, a toroidal groove containing a synthetic rubber o-ring. During assembly, an appropriate tool induces plastic deformation of the toroid in order to provide the tightness to the coupling. Pipes and fittings can be made of both austenitic and ferritic stainless steel. If the plastic deformation of the fitting is not optimal, regions of stagnant fluid might develop under the o-ring or between the contacting metals. In these regions, a classical differential aeration

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cell can develop and chloride concentration increases, leading to potential crevice corrosion problems.

The pitting corrosion behaviour of steel AISI 444 (not annealed) was examined in [9] with the aim of investigating the possibility of replacing the drinking water tanks walls, currently made of AISI 304 and AISI 316L. From the tests conducted in synthetic drinking water, it was revealed that AISI 444 does not have a better resistance to pitting compared to the austenitic steels. However, corrosion resistance could be improved up to the levels offered by the austenitic steels by performing a surface treatment of passivation. On not annealed AISI 444, tests were conducted to evaluate the susceptibility to crevice corrosion [10]. It was shown that AISI 444 might suffer crevice corrosion, similarly to AISI 304 and AISI 316L [11,12] but, by using a suitable surface treatment with a mixture of HNO<sub>3</sub> and HF, the corrosion resistance can be improved [9].

A recent study was conducted on not annealed steel AISI 444 [13], according to test described in the Japanese standard JIS G0592 (equivalent to the ASTM G192-08). This is an electrochemical method for the determination of the potential for crevice corrosion. The results showed that the presence of chloride ions has a detrimental effect on the crevice corrosion resistance of AISI 444.

Nowadays, AISI 444 stainless steel is available on the market as annealed (more expensive) and not annealed. In this work, pitting and crevice susceptibility of annealed and not annealed AISI 444 stainless steel in tap water, contaminated with chloride ions, at room temperature was evaluated by using micro and macrocyclic potentiodynamic polarizations (CPP). The results from electrochemical testing were complemented by salt spray tests, scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS), in order to reveal the differences in corrosion behaviour between annealed and not annealed AISI 444 stainless steels.

## 2. Materials and methods

The composition of the alloy used in this investigation is reported in Table 1. The annealing treatment was carried out industrially by heating the material at a temperature of about 1000 °C for 60 s as indicated by the supplier.

In order to evaluate the corrosion behaviour of AISI 444, and the surface composition of the corroded areas, the following measurements were undertaken: (i) free corrosion potential (OCP), (ii) macroscopic cyclic potentiodynamic polarization (CPP), (iii) micropotentiodynamic polarization, (iv) metallographic test with EDS analysis, and (v) salt spray test. All the electrochemical tests were performed in tap water of the city of Naples whose hardness, pH and chemical composition is reported in Table 2.

To simulate more aggressive environmental conditions, selected concentrations of NaCl were added to the tap water, to obtain a solution with low (0.02 wt% NaCl), medium (0.1% NaCl) and high (3.5 wt% NaCl) aggressiveness.

### 2.1. Electrochemical tests

Aerated and deaerated CPP measurements were carried out on annealed and not annealed specimens of AISI 444 at room temperature in uncontaminated (pure tap water) and contaminated tap water with added NaCl. The measurements were performed in according to ASTM G3 and G5 using a 3 electrodes electrochemical

**Table 2**  
Hardness, pH and chemical composition of Naples tap water.

Total hardness (°F)	39
Residual at 180 °C (mg/l)	569
pH	7.3
Nitrates (mg/l of NO <sub>3</sub> )	12
Nitrites (mg/l of NO <sub>2</sub> )	<0.05
Ammonium (mg/l of NH <sub>4</sub> )	<0.03
Fluorides (µg/l of F <sup>-</sup> )	400
Chlorides (mg/l of Cl <sup>-</sup> )	53

cell, with a saturated calomel (SCE) as reference electrode (RE), a platinum foil as counter electrode (CE) and the stainless steel specimen as the working electrode (WE). Cell arrangement and procedure have been reported elsewhere [14].

The polarization curves were recorded using a Solartron SI1287 potentiostat connected with a Solartron SI1260, function generator, which was interfaced with a personal computer by a National Instruments GPIB-USB-HS data logger. Before the test, the corrosion potential ( $E_{corr}$ ) was monitored until it was steady. The ohmic drop between the working electrode and the reference electrode was not compensated.

All the anodic potentiodynamic curves were initiated 30 mV below the corrosion potential and performed with a scan rate of 0.166 mV/s (10 mV/min). The direction of polarization was reversed at 0.1 mA/cm<sup>2</sup>. After reversal, the polarization was interrupted either when repassivation was observed, or when the original corrosion potential was reached.

From the experimental polarization curves the corrosion current density,  $i_{corr}$ , the pitting,  $E_{pit}$ , and the protection,  $E_{prot}$  potentials were obtained. The  $i_{corr}$  values were estimated by extrapolating the linear region of the anodic potentiodynamic response to the corrosion potential. In this paper the pitting potential is taken as the critical potential at which a dramatic increase in current is observed in the anodic polarization scan. All the specimens were observed by optical microscopy before and after polarization, in order to exclude the occurrence of crevice corrosion and to investigate the morphology of pitting. Selected specimens were also examined by SEM and EDS analysis (Hitachi TM3000).

### 2.2. Potentiodynamic micropolarization

Potentiodynamic micropolarization measurements were carried out by using a microcapillary cell, described in detail elsewhere [15,16], and schematically represented in Fig. 1. On each specimen eight micro-areas were identified in two rows of four. This configuration allows to obtain a map of the electrochemical reactivity of both annealed and not annealed AISI 444 surface. With the micro-capillary cell setup, the test electrolyte is contained in a pipette tip, positioned on the area to be tested. The pipette is connected to a solution reservoir where both reference electrode (SCE) and counter electrode (platinum wire) are accommodated. For all the tests performed, a pipette tip size of 0.35 mm<sup>2</sup> was used. Micro CPP tests were carried out according to the procedure outlined above for macro CPP, with the only difference that the scan rate used was 1 mV/s, due to the small volume of the solution available in the microcapillary [16]. All measurements were carried out on triplicate coupons and the average values are reported in this paper.

**Table 1**  
Percentage chemical composition of AISI 444 used in this work.

Grade	C	Si	Mn	P	S	N	Cr	Mo	Ni	Ti	
AISI 444	X2CrMoTi18-2	0.014	0.279	0.216	0.029	0.002	0.0148	17.859	2.074	-	0.4122

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