



Influence of surface morphology and roughness on water wetting properties of low temperature nitrided austenitic stainless steels



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ABSTRACT

Low temperature nitriding is known as a useful treatment to increase surface hardness and corrosion resistance of austenitic stainless steels, but little information is reported about its effect on surface wettability of the nitrided alloys. In this preliminary study the modifications of water wetting properties due to this treatment were investigated and they were related to the surface morphology and roughness changes. Samples of AISI 316L and AISI 202 austenitic stainless steels were nitrided using the glow-discharge technique, and modified surface layers consisting mainly of the so called S phase were produced. The treatments affected also the surface morphology and caused the formation of peculiar features, which were due to both ion bombardment during sputtering and nitriding, and local plastic deformations promoted by the high amount of solubilised nitrogen. As a consequence, roughness increased markedly on nitrided samples in comparison with untreated ones. Water wetting tests, performed with the sessile drop method, showed that the apparent contact angle values were higher on the nitrided samples in respect of the untreated ones, up to a hydrophobic behaviour. According to the Cassie–Baxter model, it may be supposed that the drops lie on solid and air pockets, formed thanks to the rougher surfaces. Additional tests suggest that this state is metastable: when the water drop was pushed on the nitrided surface, filling of almost all the cavities and valleys was obtained, and smaller contact angles were measured, also in respect of the untreated samples, suggesting that a state near to that hypothesised by Wenzel was achieved.

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1. Introduction

Low temperature nitriding has received increasing attention in the last decade for the surface modification of austenitic stainless steels [1, 2]. By using treatment temperatures lower than 450 °C it is possible to avoid the precipitation of large amounts of chromium nitrides and retain nitrogen in the austenite phase beyond the solubility limit. As a consequence, a supersaturated solid solution of nitrogen (up to about 10 wt.% [1]) in the expanded and distorted f.c.c. austenite lattice, known as S phase or expanded austenite, is obtained. As reviewed by Dong [1], many researches have been devoted to study the microstructure and crystal lattice characteristics of the S phase, and the treatment techniques which allow producing modified surface layers consisting of this phase. The remarkable increase of hardness and corrosion resistance in chloride-ion containing solutions, shown by the S phase, has focused the interest especially on mechanical [1,3–5], tribological [1,3,6,7] and corrosion resistance [1,3,8–10] properties of the low temperature nitrided austenitic stainless steels.

In the international literature less attention has been paid on the modifications of wetting characteristics due to low temperature nitriding. Wetting behaviour of a solid surface by a liquid is relevant for many

industrial applications and it is governed by the chemical composition and the roughness of the surface itself [11–15]. In respect of water, the contact angle (i.e., the angle between the gas–liquid and solid–liquid interfaces) of an ideal flat solid surface, ϑ_{flat} , can be less than 90° (hydrophilic surface) or higher than 90° (hydrophobic surface), depending on the chemical composition of the surface. When a real surface is considered, the surface roughness affects the measured (apparent) contact angle, which can increase or decrease in respect of the intrinsic contact angle, ϑ_{flat} , depending on the surface characteristics. The relationship between the intrinsic contact angle, ϑ_{flat} , of a flat surface, and the apparent contact angle, ϑ_{rough} , of a rough surface is usually described using two different models, proposed by Wenzel [16] and Cassie and Baxter [17]. According to the Wenzel model [16], the liquid drop has a complete contact with the rough surface, filling up the grooves. This situation is described by the relation:

$$\cos \vartheta_{\text{rough}} = r \cos \vartheta_{\text{flat}} \quad (1)$$

where r is the so called ‘roughness factor’, defined as the ratio of the actual surface to the geometric projected area on the horizontal plane. As a consequence, roughness causes a decrease of the contact angle when the surface is hydrophilic, so wetting is improved, while it causes an increase of the contact angle when the surface is hydrophobic.

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According to the Cassie–Baxter model [17], the contact between the drop and the rough surface is not complete. The drop, known as fakir drop, lies on a composite surface made of solid (the top of the asperities) and air pockets trapped underneath the liquid. The apparent contact angle is given by the relation:

$$\cos\vartheta_{\text{rough}} = \phi_s(1 + \cos\vartheta_{\text{flat}}) - 1 \quad (2)$$

where ϕ_s is the area fraction of the solid surface in contact with water. The apparent contact angle results to be larger than the contact angle for an ideal surface and it increases as ϕ_s is decreased.

Therefore, tailoring the surface roughness can be used to enhance hydrophilicity of a hydrophilic surface or hydrophobicity of a hydrophobic surface [11,13–15], or to produce hydrophobic behaviour on hydrophilic materials [12,14]. In particular, microtextures have been used to produce hydrophobic surfaces on austenitic stainless steels [18,19]. Since low temperature nitriding changes also the surface roughness of treated austenitic stainless steels [8], a change in the wetting behaviour is expected.

The aim of the present study was to preliminarily investigate the water wetting properties of austenitic stainless steels, subjected to low temperature nitriding by means of the glow-discharge technique. Two stainless steels of fairly large use were chosen, AISI 316L and AISI 202. The latter alloy, which is cheaper due to its lower nickel content, has a corrosion resistance significantly lower than that of AISI 316L. However, when AISI 202 is subjected to low temperature nitriding, a marked increase of corrosion resistance is observed [8,20,21]. As an example, when corrosion testing is performed in 5% NaCl aerated solution with the potentiodynamic method, the potential value, at which localised corrosion phenomena begin, is about +410 mV (Ag/AgCl) for untreated AISI 202 samples and +780 mV (Ag/AgCl) for untreated AISI 316L ones; when the alloys are nitrided at 380 °C at 500 Pa for 5 h, localised corrosion begins at potential values higher than +1000 mV (Ag/AgCl) for both the steels [21]. In the present research the treatment parameters were chosen on the basis of our previous studies [8,21], in order to produce, on the used stainless steels, modified surface layers consisting of S phase and avoid the precipitation of chromium nitrides, so that the corrosion resistance of the steels can be maintained or even increased. Microstructure, phase composition, surface roughness and water wetting characteristics of the nitrided samples were evaluated and compared to those of the untreated alloys.

2. Materials and Methods

Prismatic samples (40 × 17 × 0.7 mm) of AISI 316L and AISI 202 austenitic stainless steels were cut from cold rolled, annealed and pickled plates, and then they were ground and polished up to 6- μ m diamond suspension. The chemical composition of the steels is reported in Table 1.

Glow-discharge nitriding treatments were performed in a laboratory plasma equipment, previously described [20]. Before the nitriding treatments, the samples were heated up to 310 °C by means of a cathodic sputtering performed at 130 Pa with 80 vol.% N₂ and 20 vol.% H₂. The following treatment conditions were chosen: a set of samples (type A) was nitrided at 380 °C and 500 Pa (current density: 1.6 ± 0.1 mA cm⁻²; voltage drop: 150 ± 10 V). Another set of samples (type B) was nitrided at 380 °C at a pressure of 130 Pa (current density: 0.8 ± 0.1 mA cm⁻²; voltage drop: 235 ± 10 V). Systematically higher voltage drop values were observed when AISI 316L samples were treated, in respect to those

measured during nitriding of AISI 202 samples. A set of AISI 316L samples was nitrided also at 400 °C and 500 Pa (current density: 1.7 ± 0.1 mA cm⁻²; voltage drop: 165 ± 10 V) (type C). It was chosen not to nitride AISI 202 samples, since with these treatment parameters sensible amounts of nitrides are able to form on this alloy [21]. For all nitriding conditions the duration was 5 h and the treatment atmosphere was 80 vol.% N₂ and 20 vol.% H₂.

The surface morphology and microstructure of untreated and nitrided samples were studied by means of light and scanning electron (SEM) microscopy and energy dispersion spectroscopy (EDS) analysis. In order to evaluate the thickness of the modified surface layers, nitrided samples were cross-sectioned and analysed by means of metallographic techniques. The phases present in the surface layers were identified by means of X-ray diffraction analysis (Cu K α radiation). Diffraction patterns were collected both in Bragg–Brentano configuration and using a constant incident angle of 15°; in this latter configuration, the mean penetration depth (i.e. the depth at which the intensity drops by a factor of *e*) was 0.8 μ m.

Roughness evaluation was performed by using a stylus profilometer. The measurements were carried out using a 2- μ m radius stylus with a 1-mN contact force; the cut-off length was 0.25 mm. Ten measurements were taken at different locations on each sample. The average surface roughness Ra (arithmetical mean deviation of the roughness profile from the mean line), the maximum height of profile Rz (sum of the largest profile peak height and the largest profile valley depth within a sampling length, according to EN ISO 4287–2009 norm) and the mean height of profile elements Rc (mean value of the profile element heights within a sampling length) were recorded.

The apparent static contact angle of water was measured according to the sessile drop method. Bi-distilled water was used as test liquid, and drops of 0.5 or 3 μ l were used. Before the measurement, the samples were sonicated in acetone for 5 min and then freely dried in air. For each sample the contact angle was measured at least 15 times across the sample surface. All measurements were taken under ambient laboratory conditions. The contact angle values are given as the average value ± standard deviation.

Additional non-standard measurements were carried out on selected samples. Drops of 0.5 or 3 μ l were allowed to fall on the surface from about 3-mm height, instead of depositing them carefully as in the sessile drop method. The contact angle was measured after the drop has attained a stable configuration. Other measurements were performed pushing down drops of 0.5- or 3- μ l size on the surface of the samples with a force of about 2 N and measuring the contact angle after the achievement of a stable configuration.

3. Results

3.1. Surface Morphology and Microstructure

The untreated samples of both steel types show a fairly smooth surface, with shallow grooves due to the grinding and polishing procedures. X-ray diffraction analysis shows that, besides austenite, γ -Fe (f.c.c.), a very small amount of α -Fe (b.c.c.) is also present in both the untreated alloys (Figs. 1a, 2a).

The characteristics of the nitrided surface depend on both the treatment parameters and the steel type. The sputtering pre-treatment is able to produce a slight surface etching, which outlines the austenitic microstructure with the characteristic twins, as shown in Fig. 3 for an AISI 202 sample. When both the sputtering step and the nitriding treatment are performed, the ion bombardment produces a more intense surface etching and its effects depend on treatment conditions.

When nitriding is performed at 380 °C and 500 Pa (type A), plasma etching well delineates the austenitic microstructure of the AISI 316L samples (Fig. 4a). The grooves due to the grinding and polishing procedures are still observable. On X-ray diffraction patterns, obtained with a 15°-constant incident angle, the peaks characteristic of the S phase are

Table 1
Chemical composition (wt.%) of AISI 316L and AISI 202 austenitic stainless steels.

Steel type	C	N	Si	Mn	Cr	Ni	Mo	Fe
AISI 316L	0.029	0.049	0.34	0.90	16.6	10.3	2.01	Bal.
AISI 202	0.065	0.15	0.40	7.7	17.0	4.1	–	Bal.

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