

# Correlation between PIII nitriding parameters and corrosion behaviour of austenitic stainless steels

S. Mändl<sup>a,\*</sup>, D. Manova<sup>a</sup>, H. Neumann<sup>a</sup>, M.T. Pham<sup>b</sup>, E. Richter<sup>b</sup>, B. Rauschenbach<sup>a</sup>

<sup>a</sup>Leibniz-Institut für Oberflächenmodifizierung, Permoserstr. 15, 04303 Leipzig, Germany

<sup>b</sup>Institut für Ionenstrahlphysik und Materialforschung, Forschungszentrum Rossendorf, 01314 Dresden, Germany

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## Abstract

In this work, nitrogen plasma immersion ion implantation (PIII) treatment of austenitic stainless steels 1.4301 and 1.4571 was performed to investigate the influence of the process conditions on the corrosion properties. Short treatment, high voltage and high temperature result in a decreased corrosion potential while no correlation to layer thickness, nitrogen concentration or lattice expansion was found. Except for the possibility of small CrN agglomerates at high temperatures, no direct explanation for the results can be provided and it is argued that intrinsic stress accumulation and relaxation may be responsible.

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## 1. Introduction

Recent advances in computational material science nowadays allow the design of advanced materials, e.g., alloy steels [1,2]. Especially, the parallel optimization of several properties is possible, for instance hardness, ductility and corrosion resistance. At the same time, descriptions of macroscopic parameters in terms of the electronic band structure or related features are available [3,4]. Despite these advances in computation, material design is still hampered by missing structure–property relations on the quantum scale [5]. Recent calculations aiming to optimize the ternary Fe–Cr–Ni system, which includes important austenitic stainless steel alloys as AISI 304, 316, 317, with respect to hardness and corrosion resistance [6,7] are highly controversial [8] and can be regarded only as first attempts.

Despite the large commercial interest in nitrided austenitic stainless steels, no satisfying explanation for the outstanding results is known to the authors. A

hardness of up to 1200 HV is reported at nitrogen concentrations of 10–25 at.%, in conjunction with a lattice expansion of 5–10% at a process temperature of 350–380 °C [9,10]. Higher temperatures lead to the decomposition into CrN and ferrite and thus in destroying of the corrosion resistance [11]. Nevertheless, even at 380 °C and below, improved and degraded corrosion resistance is reported in the literature [12–14]. The present investigation focuses on studying the influence of process parameters on the corrosion resistance of nitrided austenite steel by plasma immersion ion implantation and attempts to find a correlation between the process parameters as voltage, time or temperature and the resulting corrosion resistance.

## 2. Experiment

Two different austenitic stainless steel grades – DIN 1.4301 (AISI 304) and 1.4571 (316Ti) – were used in these experiments. Nitrogen plasma immersion ion implantation was performed at different parameters simultaneously in samples from both steel grades. The experiments were made at a base pressure lower than  $10^{-3}$  Pa and working

\* Corresponding author. Tel.: +49 341 235 2944; fax: +49 341 235 2313.  
E-mail address: [stephan.maendl@iom-leipzig.de](mailto:stephan.maendl@iom-leipzig.de) (S. Mändl).

pressure of 0.2 Pa resulting from nitrogen flow rate of 50 sccm. The implantation parameters varied in a limited range to obtain representative sample encompassing the typical parameters used in energetic nitriding of austenitic stainless steel.

The process temperature between 340 and 380 °C was maintained by adjustment the pulse frequency and measured by an IR pyrometer. No external heating or cooling was employed so the samples were heated only by implanted ions. The pulse voltage was 10 or 20 kV with pulse length of 15  $\mu$ s. The process time was 1 or 2 h resulting in implanted dose between  $8.0 \times 10^{17}$  at./cm<sup>2</sup> and  $2.5 \times 10^{18}$  at./cm<sup>2</sup>. Additionally, samples from both steel grades nitrided for 1 h at 340 °C were post-implanted with oxygen at 15 kV for 15 min at 300 °C.

X-ray diffraction (XRD) was employed in Bragg–Bretano geometry to determine the phase composition while the elemental depth profiles were obtained from glow discharge optical spectroscopy (GDOS) measurements with an accuracy of 0.1–0.2 at.% and a reproducibility of better than 0.1 at.% [15]. The corrosion properties were studied by carrying out potentiodynamic polarization measurements in 1% NaCl solution with sweep rate of 10 mV/s. The potential was measured against saturated calomel electrode (SCE). At the same time, corrosion salt bath tests in 3% NaCl solution at an elevated temperature of 40 °C were conducted for several days or until the onset of corrosion was observed.

### 3. Results and discussion

Fig. 1a shows the nitrogen depth profiles of two samples from 1.4301 steel grade implanted with 10-kV acceleration voltage at 340 °C for 2 h and at 380 °C for 1 h, respectively. The near surface concentration differs between 40% and 25% for sample at 340 and 380 °C, respectively. In both samples, a slow initial concentration decrease starting just below the surface is followed by a steeper decrease beyond 1.6  $\mu$ m, respectively at approximately 25 and 15 at.% nitrogen, indicating a concentration dependent diffusion coefficient [16]. The layer thickness, defined here as 1.5 at.% nitrogen, obtained from the depth profiles is approximately 2.2  $\mu$ m for both samples.

XRD spectra for the same samples, presented in Fig. 2, show different peak intensities as well as a different lattice expansion, despite the quite similar layer thickness. The relative lattice expansion estimated from XRD peaks is 4.7% and 6.5% for the (111) and (200) peaks for the sample at 340 °C, respectively, 3.3% and 4.3% for the (111) and (200) peaks at 380 °C. These two samples show typical results of the whole series, where the layer thickness is between 1 and 3  $\mu$ m while the nitrogen surface concentration varies between 18 and 42 at.% and the relative lattice expansion, averaged over the (111) and (200) peaks is in the range of 2.4 to 5.5%.

The corrosion current as a function of the applied potential, obtained from potentiodynamic polarization

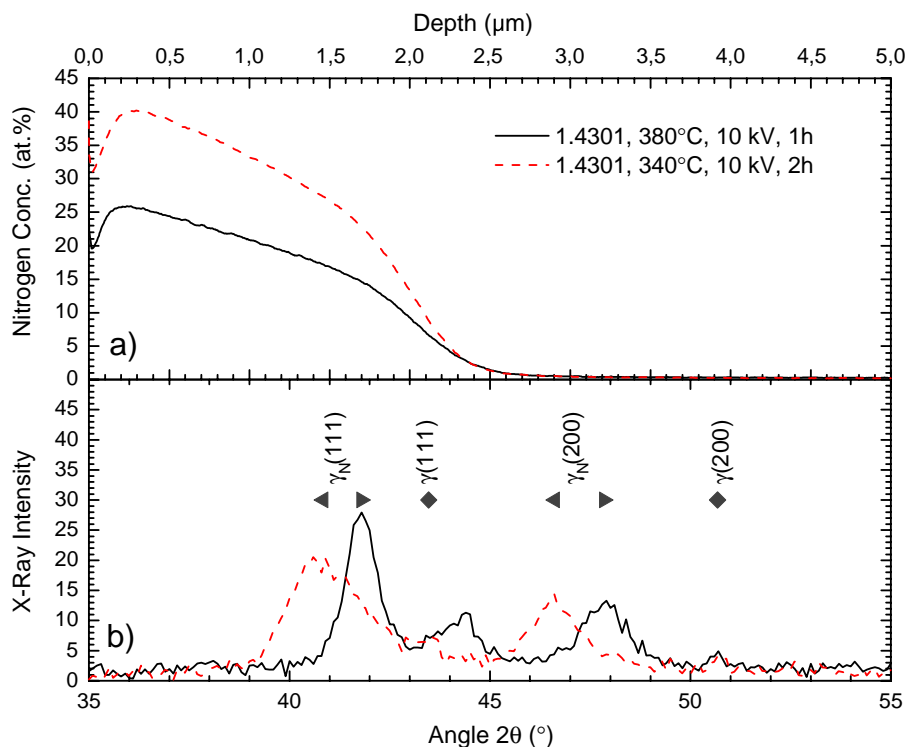


Fig. 1. (a) Nitrogen depth profile after PIII into steel 1.4301 at 340 °C and 380 °C for 2 and 1 h, respectively; (b) XRD spectra for the same samples with the peak positions for the austenite base material and the expanded austenite marked.

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