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Influence of grain size on nitrogen diffusivity in austenitic stainless steel

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Abstract

Ion nitriding of austenitic stainless steel with the aim to improve the tribological properties while retaining the excellent corrosion resistance is a well-established method. At the same time, strongly varying microstructures can be obtained depending on the pretreatment. In this work, the influence of prior heat treatment in the temperature range between 950 and 1200 °C on the microstructure, especially the grain size, and the corresponding observed nitrogen diffusivity in austenitic stainless steel DIN 1.4301 (AISI 304, X6CrNi18.10) after nitrogen plasma immersion ion implantation (PIII) is studied. Cross-section and plan view samples were prepared and investigated. With increased annealing temperature, both larger grains and slower diffusion was observed, despite diffusion ranges much smaller than the average grain size. Another, still hidden effect of dislocation densities or other defects on both secondary parameters is suggested.

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Keywords: PIII; Expanded austenite; Diffusion; Grain size; SIMS

1. Introduction

Ion nitriding of austenitic stainless steel in the temperature range from 350 to 380 °C is a widely used industrial method to obtain modified stainless steel surfaces with outstanding hardness and wear resistance while the corrosion resistance is similar to that of the base material or even improved [1–5]. These properties are attributed to the formation of the so-called “expanded austenite” as the expanded lattice and concentration-dependent nitrogen diffusion are the main characteristics of this new phase. Despite of many studies devoted to ion nitriding of austenitic stainless steel, the exact formation mechanism of expanded phase is still unknown and is the subject of current investigations.

It is well known that pretreatment of stainless steel such as alloying, cold working and heat treatment can strongly change its properties [6]. In general, alloying and cold working determine the stress level and yield strength of the material, while the heat treatment influences the microstructure and thus the grain size, and consequently diffusivity. A decrease in the grain size of steel from 1000 μm to 13 nm results in 1000 times faster diffusion in ion nitriding, as was shown by another group [7]. However, cold working is required for a very long time in this study, hence no industrial application can be envisaged. Additionally, different data sets are reported in the literature concerning nitrogen diffusion in austenitic stainless steel without providing any information about the grain size [8–10]. There is no model trying to explain these strongly diverging results. No systematic study on nitrogen diffusion in steel in connection with microstructure and thus pretreatment performed on steel is performed so that the published diffusion values could be misleading.

The aim of this presentation is to investigate the influence of a prior heat treatment on the nitrogen diffusion in austenitic stainless steel after nitrogen plasma immersion ion implantation (PIII). This study attempted to find a correlation between the microstructure of the base material and the diffusion rate during ion nitriding.

2. Experiment

Samples of austenitic stainless steel DIN 1.4301 (AISI 304, X5CrNi18.10) cut from a cold-rolled rod (ϕ15 mm), as obtained from a distributor of commercial steel products, were used as substrates in this investigation. After cutting, some samples were

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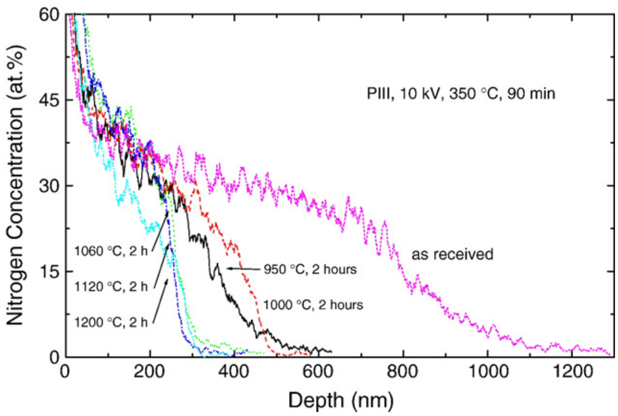

Fig. 1. Nitrogen depth profiles from SIMS measurements for five samples annealed at different temperatures together with one sample in the as-received condition after nitrogen PIII with 10 kV pulses at 350 °C for 90 min.

heated in a conventional furnace at temperatures in the range from 950 to 1200 °C for 2 h to start recrystallisation and were cooled down in air. Prior to PIII treatment, the samples were grinded and polished down to a final roughness of $R_a < 50$ nm (after the implantation, R_a values between 500 and 1000 nm were measured). PIII implantations were carried out in an HV chamber equipped with a 40.68 MHz ICP helix source operating at 150 W at a base pressure better than 10^{-4} Pa. 10 kV high voltage pulses with a rise time less than 0.5 µs and 15 µs pulse length were applied to the samples while the current density during the experiment was adjusted to maintain the 350 °C process temperature without additional heating. The implantation time was 90 min resulting in an incident nitrogen fluence of about 1.5×10^{18} cm^{-2}. After the treatment, the samples were left in the vacuum until their temperature was lower than 100 °C.

The phase composition was investigated by X-ray diffraction (XRD) in Bragg–Brentano geometry in the angular range from 30° to 100° with 0.05° step width and 60 s measuring time per step. The nitrogen depth profiles were obtained from time-of-flight secondary ion mass spectroscopy (SIMS) measurements. 15 keV Ga^+ ions for analysis and 2 keV O_2^+ ions for depth profiling at an approximate sputter rate of 1 nm/s were used with a linear sputter rate extrapolated from the final crater depth. The results obtained from these measurements were checked against and calibrated with glow discharge optical spectroscopy (GDOS). Dynamic hardness measurements using Vickers nanoindenter at a maximum load of 100 mN were performed without correction for elastic recovery, so the real hardness values are expected to be 15–20% higher.

For the investigation of the microstructure, metallographic cross-sections of non-implanted but annealed samples (parallel to the original rod axis) were prepared and together with plan view samples (along the rod cross-section) were chemically etched with aqua regia and analyzed with optical microscopy.

3. Results and discussion

Fig. 1 presents the nitrogen depth distribution obtained from SIMS measurements for five samples annealed at different temperatures together with one sample in the as-received condition after nitrogen PIII with 10 kV pulses at 350 °C for 90 min. Diffusion far beyond the implantation range – which is

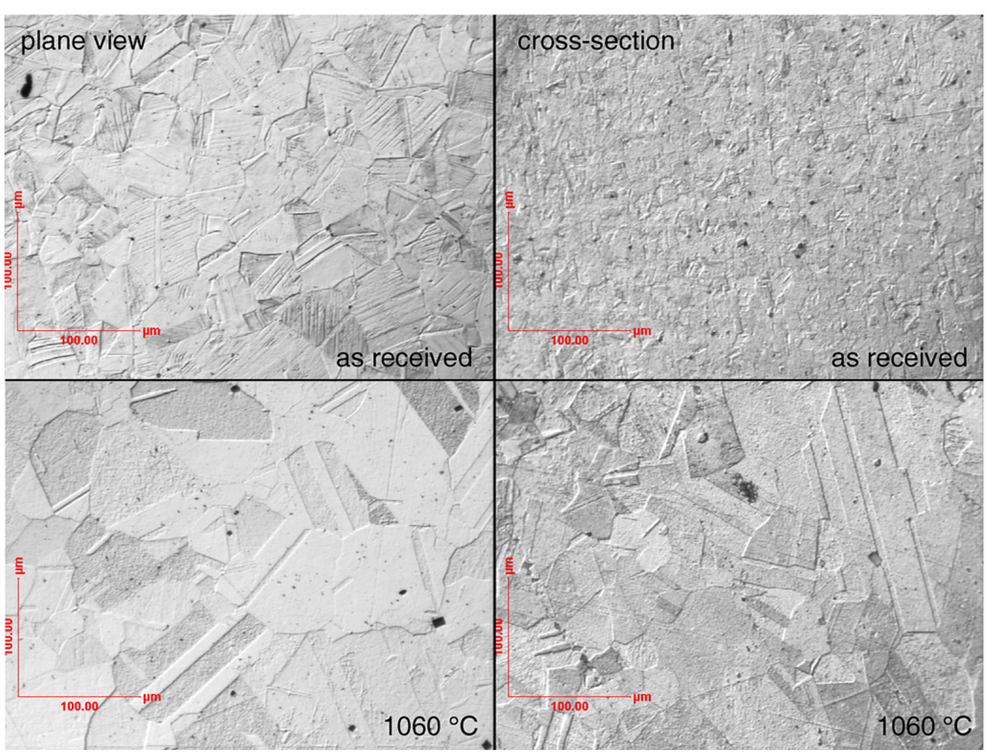

Fig. 2. Optical micrographs in plan view (parallel to sample surface across the original rod) and cross-section (parallel to the diffusion direction and the rod axis) of two samples – as-received and after annealing at 1060 °C for 2 h.