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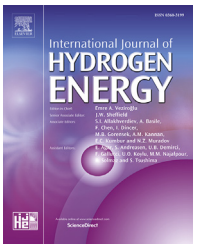

Impact of chemical inhomogeneities on local material properties and hydrogen environment embrittlement in AISI 304L steels

Gero Egels [a,*], *Lais Mujica Roncery* [b], *Robert Fussik* [c], *Werner Theisen* [a], *Sebastian Weber* [c]

[a] *Ruhr-Universität Bochum, Institut für Werkstoffe, Lehrstuhl Werkstofftechnik, 44780 Bochum, Germany*
[b] *Universidad Pedagógica y Tecnológica de Colombia, Grupo de Investigación en Materiales Siderúrgicos e INCITEMA, Tunja, Colombia*
[c] *Bergische Universität Wuppertal, Lehrstuhl für Neue Fertigungstechnologien und Werkstoffe, 42651 Solingen, Germany*

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ABSTRACT

This study investigated the influence of segregations on hydrogen environment embrittlement (HEE) of AISI 304L type austenitic stainless steels. The microstructure of tensile specimens, that were fabricated from commercially available AISI 304L steels and tested by means of small strain-rate tensile tests in air as well as hydrogen gas at room temperature, was investigated by means of combined EDS and EBSD measurements. It was shown that two different austenitic stainless steels having the same nominal alloy composition can exhibit different susceptibilities to HEE due to segregation effects resulting from different production routes (continuous casting/electroslag remelting). Local segregation-related variations of the austenite stability were evaluated by thermodynamic and empirical calculations. The alloying element Ni exhibits pronounced segregation bands parallel to the rolling direction of the material, which strongly influences the local austenite stability. The latter was revealed by generating and evaluating two-dimensional distribution maps for the austenite stability. The formation of deformation-induced martensite was shown to be restricted to segregation bands with a low Ni content. Furthermore, it was shown that the formation of hydrogen induced surface cracks is strongly coupled with the existence of surface regions of low Ni content and accordingly low austenite stability. In addition, the growth behavior of hydrogen-induced cracks was linked to the segregation-related local austenite stability.

Introduction

Austenitic stainless steels of the CrNi-type are frequently used for hydrogen storage and hydrogen conduction applications due to their high resistance against hydrogen environment embrittlement (HEE). HEE describes a phenomenon in which materials undergo a deterioration of their mechanical properties caused by the presence of hydrogen in the surrounding atmosphere. In applications such as

* *Corresponding author.*
E-mail address: egels@wtech.rub.de (G. Egels).

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hydrogen storage in fuel cell vehicles, there is a strong demand for cost-efficient materials that are compatible with high-pressure hydrogen gas, which moves the investigation of the hydrogen compatibility of low-Ni austenitic stainless steels such as AISI type 304L into the focus of current research. According to *Michler* et al. [1], literature data on the HEE resistance of austenitic stainless steels containing 8–10 wt.% Ni show a large scatter. Since they also proved that mechanical testing in a hydrogen atmosphere is highly repeatable and reproducible *Michler* et al. related the scatter to a high sensitivity of HEE to metallurgy and local variations in chemical composition of the tested material. *Martin* et al. [2–5], and *Weber* et al. [6,7], have contributed several studies to clarify the impact of microstructural aspects such as grain size, machining condition or segregation effects on the HEE of austenitic stainless steels. *Michler* et al. stated that segregations of Ni in AISI 316L steel can cause the formation of a duplex like austenite–martensite microstructure upon straining which results in a shape dependent susceptibility to HEE of the tested material [8]. Since the precise role of microsegregations has not yet been clarified, the present work focuses on the impact of different segregation states of commercial AISI 304L type steels on their susceptibility to HEE. As recently shown by *Man* et al. [9] and *Fussik* et al. [10], segregation effects of Ni show a substantial influence on the presence of stable and metastable regions in the material with respect to deformation-induced $\gamma \rightarrow \alpha'$ transformations. Since hydrogen can diffuse some orders of magnitude faster through the bcc lattice of martensite in comparison to the fcc lattice of austenite, once martensite has arisen, it can serve as a pathway for fast uptake and diffusion of hydrogen into the material [2,11,12]. Austenite stability can be quantified by thermodynamic as well as empirical approaches. A thermodynamic estimation of the austenite stability can be achieved by calculating the Gibbs free energies of both the fcc and the bcc phase, because the difference between these values represents the thermodynamic driving force for a $\gamma \rightarrow \alpha'$ transformation $\Delta G^{\gamma \rightarrow \alpha'}$. Alternatively, the calculation of empirical values, for instance M_S or M_{d30}, can be applied to estimate the austenite stability [13,14]. Calculated values of these parameters are frequently found in literature on HEE of austenitic steels. However, the calculations are always based on the nominal composition of the investigated alloys and neglect processing conditions and their impact on local element distributions [15–17].

Another crucial value in the context of the deformation behavior of austenitic stainless steels is the stacking fault energy (SFE). Since the SFE shows a distinct dependency on the chemical composition, segregations may influence SFE values locally and therefore cause local differences in the material's deformation behavior, which possibly impacts HEE [4]. High SFE values typically promote dislocation glide, whereas twinning and martensite formation are known to be predominant deformation modes in low SFE alloys [18]. In austenitic steels for hydrogen applications, high SFE values are favorable on the one hand in order to promote uniform deformation instead of localized deformation and on the other hand to avoid martensitic transformations [19].

Experimental

Sample production and tensile testing

The microstructural investigations presented in this study were performed on specimens that were produced and tensile tested in the framework of earlier investigations by *Martin* [3]. The tensile specimens investigated in this study were produced out of three different commercially available materials in the compositional range of AISI type 304L steel provided by Deutsche Edelstahlwerke (DEW, Germany). The chemical compositions of the materials were measured by optical spark emission spectrometry (OES) and are presented in Table 1. All materials were produced by continuous casting. The material designated as W11 was cast with a 265 mm square cross-section and subsequently hot-rolled to a bar shape with a diameter of 30 mm. The materials designated as W11-ESR and W11-Ni-ESR were refined by electroslag remelting (ESR) after continuous casting. The remelted bar material with a diameter of 160 mm was subsequently hot-forged to a diameter of 50 mm. For all materials, cylindrical tensile samples with a gauge length of 30 mm and a diameter of 5 mm were machined out of the center of the bars, parallel to the forging direction, by wet turning. After machining, all samples were solution annealed in an industrial vacuum furnace for 30 min at 1050 °C and quenched with argon gas at a pressure of 200 kPa. This solution-annealing step after machining avoids a possible impact of machining-induced martensite on HEE [2]. The average grain sizes of the materials in the solution annealed state were measured with the linear intersection method and are listed in Table 2.

Tensile tests were carried out in air and in pure hydrogen gas (≥99.9999% H_2) at room temperature. The tensile tests in air were performed at ambient pressure, whereas 40 MPa pressure was applied in case of testing in a hydrogen atmosphere. Under both sets of environmental conditions, two tensile tests were performed for each material. The tensile tests were performed according to the ASTM G129 standard with an initial strain rate of $5.5 \times 10^{-5} s^{-1}$. The properties measured in the tensile tests were the yield strength ($R_{p0.2}$), ultimate tensile strength (R_m), and elongation to rupture (A). In addition, the reduction of area (RA), which is known to be very sensitive to hydrogen embrittlement [20], was quantified ex situ by measuring the specimen's initial and final diameter at the necking circumference with a digital caliper. Before and after tensile testing, the volume content of the magnetic bcc phase (ferrite and/or α'-martensite) was determined by the magnetic induction method using a Feritscope (Helmut Fischer GmbH, Sindelfingen, Germany). The Feritscope measurements were made on the surface of the tensile specimens in the area of uniform elongation.

Microstructural analysis

For the microstructural analyses, longitudinal sections of the tested tensile specimens were prepared metallographically by cutting and embedding, followed by several steps of manual grinding and polishing down to 1 μm. In order to obtain

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