



## Evaluation of hydrogen trapping mechanisms during performance of different hydrogen fugacity in a lean duplex stainless steel



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### ABSTRACT

Hydrogen trapping behavior in a lean duplex stainless steel (LDS) is studied by means of thermal desorption spectrometry (TDS). The susceptibility of a metal to hydrogen embrittlement is directly related to the trap characteristics: source or sink (reversible or irreversible, respectively). Since trapping affects the metal's diffusivity, it has a major influence on the hydrogen assisted cracking (HAC) phenomenon. It is known from previously published works that the susceptibility will depend on the competition between reversible and irreversible traps; meaning a direct relation to the hydrogen's initial state in the steel. In this research the trapping mechanism of LDS, exposed to different hydrogen charging environments, is analyzed by means of TDS. The TDS analysis was supported and confirmed by means of X-ray diffraction (XRD), hydrogen quantitative measurements and microstructural observations. It was found that gaseous charging (which produces lower hydrogen fugacity) creates ~22% higher activation energy for hydrogen trapping compared with cathodic charging (which produces higher hydrogen fugacity). These results are due to the different effects on the hydrogen behavior in LDS which causes a major difference in the hydrogen contents and different hydrogen assisted phase transitions. The highest activation energy value in the cathodic charged sample was ascribed to the dominant phase transformation of  $\gamma \rightarrow \gamma^*$ , whereas in the gaseous charged sample it was ascribed to the dominant formation of intermetallic compound, sigma ( $\sigma$ ). The relation between hydrogen distribution in LDS and hydrogen trapping mechanism is discussed in details.

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### 1. Introduction

Duplex stainless steels (DSS) offer an attractive combination of properties, including excellent resistance to chloride stress corrosion cracking, along with high strength and ductility. Due to these combinations of properties, the DSS family is popular in industries which use pressure vessels or underwater pipelines; services combined hydrogen and mechanical load. Nilsson [1] has divided the modern DSS into four different types according to their alloying element amount. Among them is the lower alloyed duplex grades often referred to as lean duplex stainless steel (LDS). In this work, the LDS was chosen for a number of reasons. One, its identical crystal structure as DSS - 50%/50% austenitic (FCC,  $\gamma$ ) and ferritic (BCC,  $\alpha$ ) phases. Second, its uniqueness in the different quantity of the alloying elements, which grant it better mechanical properties

with reduced costs [2,3].

One of the main problems in maximizing the service life of metals in industry is their low resistance for hydrogen. Since hydrogen is common in most manufacturing processes and services, the common used DSS are subjected to a deleterious effect known as hydrogen embrittlement. The susceptibility of DSS to the hydrogen assisted cracking (HAC) phenomenon is related to some factors, among them is the presence of the ferrite phase and the hydrogen-trap interaction [4–7]. In order to initiate a crack, a critical hydrogen concentration must be reached at potential crack sites. Therefore, factors affecting the hydrogen diffusivity into a crack site are of great importance to the analysis of DSS behavior in hydrogen environment.

In this paper, we refer and compare hydrogen trapping and desorption in LDS exposed to hydrogen by using two methods: gaseous and cathodic charging, which produce different hydrogen fugacity in LDS. previously published works were based mostly on hydrogen effect on a single phase steel [7–9], or on cathodic charged DSS [10–13]. Hydrogen different fugacity was controlled

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by the charging techniques; electrochemical charging produces high hydrogen fugacity, whereas the gaseous charging produces low hydrogen fugacity [14–18]. The hydrogen trapping characteristics were analyzed by means of thermal desorption spectrometry (TDS). The analyzed data was confirmed by LECO hydrogen determinator, X-ray diffraction (XRD) and microstructural observation with optical and electronic microscopies (OM and SEM, respectively).

## 2. Experimental procedure

### 2.1. Materials

The chemical composition of the lean duplex stainless steel, LDX 2101, which is the focus of this investigation, is presented in Table 1. The LDS was delivered as a 1.5 mm thick plate in the fully annealed condition. Samples were cut in the shape of squares with  $5 \times 10$  mm and 1 mm thickness. The samples are cathodically precharged with hydrogen or precharged at high temperature and high pressure in a hydrogen gas chamber. The cathodic charging was performed in a 0.5N H<sub>2</sub>SO<sub>4</sub> (sulfuric acid) water solution and 0.25 g l<sup>-1</sup> of NaAsO<sub>2</sub> (sodium arsenide) powder with a current density of 50 mA cm<sup>-2</sup> at 25 °C for 24 and 72 h. The sodium arsenide acts as a surface recombination inhibitor that increases the solute hydrogen concentration in the material by an order of magnitude. The gaseous charging was performed at 300 °C and 60 MPa pressure for 3 h in a gas chamber at the Federal Institute for Material Research and Testing (BAM Institute) in Berlin, Germany.

### 2.2. Characterization of LDS

#### 2.2.1. Thermal desorption spectrometry (TDS) analysis

The characteristics of hydrogen desorption were investigated by means of TDS. This technique involves accurate measurement of the desorption rate of hydrogen atoms, as solute or trapped in the material, while heating the sample at a known rate under ultra-high-vacuum (UHV) of  $\sim 10$   $\mu$ Pa. In this work, the samples were heated from 20 to 500 °C at constant heating rates of 2, 4 and 6 °C/min. The mass spectrometer was operated under the fast multiple mode detection; the measured intensity channel was set to 2 amu in order to detect hydrogen desorption. The working procedure, as described elsewhere [19], allowed for the identification of different types of traps coexisting in the specimen.

#### 2.2.2. LECO hydrogen determinator analysis

In order to determine the quantity of absorbed hydrogen in the sample, vacuum extraction using a LECO RH-404 hydrogen determinator was applied after the charging process; measurement accuracy was estimated for  $\pm 0.05$  wt ppm ( $\sim 2\%$  of the result). This method includes heating the sample to the sample's melting temperature by an electrode furnace. The emitted hydrogen is detected by a thermal conductive cell, which has the ability to detect differences in thermal conductivity of different gases.

#### 2.2.3. X-ray diffraction (XRD) analysis

Hydrogen related phase transformation in the metal, which appeared as a result of hydrogen absorption/desorption was analyzed by means of XRD spectra with Cu-K $\alpha$  radiation

**Table 1**  
Chemical composition of the investigated LDS (%wt).

Sample	C	S	P	Mn	Si	Ni	Cr	Mo	N
LDX 2101	0.026	0.001	0.025	4.9	0.63	1.53	21.53	0.2	0.22

( $\lambda = 1.54$  Å). Quantitative phase analysis was performed using Rietveld refinement technique. Rietveld method is one of most popular approaches to quantitative phase analysis in XRD, for it provides information about the quantity of the crystalline by a simple Gaussian model for the different intensities [20,21]. The quantitative analysis is based on the fact that intensity of different peaks on XRD depends on concentration of this phase in the material studied.

### 2.2.4. Microstructural analysis

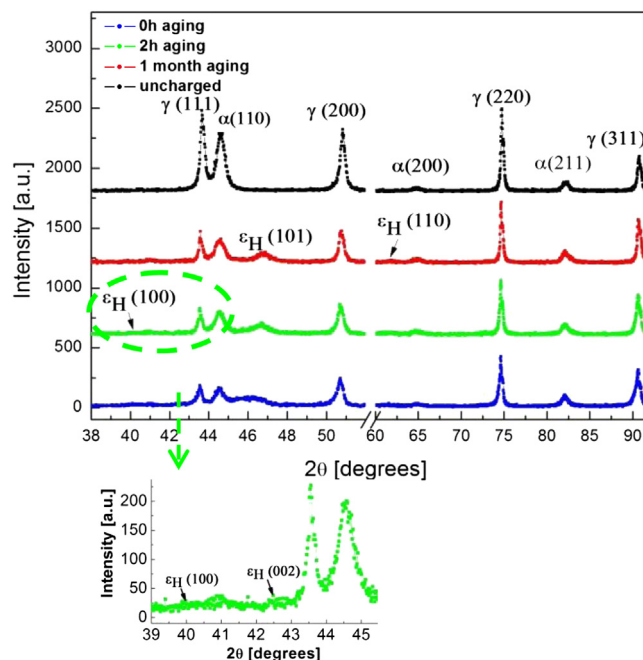
In order to examine the LDS microstructure before and after the hydrogenation process, the samples were polished to 1  $\mu$ m diamond lubricant and were etched with Glyceregia etchant (glycerine: HCl: HNO<sub>3</sub> to 3: 2: 1 parts) for 6 min. The microstructures were examined using optical microscopy (OP) and scanning electron microscopy (SEM).

## 3. Results and discussion

### 3.1. XRD and microstructural analysis

#### 3.1.1. The effect of electrochemical hydrogen charging (high fugacity hydrogen)

The XRD diffraction patterns (Fig. 1) show a comparison between LDS without hydrogen and LDS with hydrogen aged for different times at room temperature (RT). On exposure to 72 h electrochemical hydrogen environment (0 h aging), hydrogen induces strain in the LDS sample. The  $\gamma$  phase reflections exhibit a decrease in intensity and increased lattice parameter of  $\sim 2\%$ . Additional reflection observed was ascribed to the  $\epsilon_H$ -martensite, a hydrogen-containing hcp solid solution, which appears as a result of the combination between hydrogen induced in the  $\gamma$  phase and plastic deformation [3,22–25]. These observations were already seen in cathodic charged DSS alloys [3,11,25,26]. A similar result is also seen in hydrogen cathodic charged Ti based alloys, where hydrogen increases the lattice parameter and can lead to second phase's precipitation [27]. The plastic deformation is created during



**Fig. 1.** XRD pattern of the uncharged and hydrogenated LDS for 72 h and aged for different time intervals.

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