



# Mechanisms of hydrogen trapping in austenitic, duplex, and super martensitic stainless steels



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## ABSTRACT

Hydrogen interaction with structural materials, especially stainless steels, is of great importance due to the acute effect that it can have on them. Stainless steels have become very common in many applications, and in water and high pressure environments in particular, due to their high levels of corrosion resistance and broad range of strength. Steel's durability is very much dependent on its microstructure and interaction with hydrogen. The action of hydrogen can lead to changes in mechanical properties, phase transformation and eventually to environmentally-assisted failure, which is known as hydrogen embrittlement (fracture). The susceptibility of steels to this hydrogen fracture mechanism is directly related to the interaction between traps (defects) and hydrogen. In this research, we study hydrogen fracture mechanisms through hydrogen interaction with trapping sites by thermal desorption spectrometry (TDS), and the calculation of hydrogen trapping energies states. Microstructure effects on hydrogen were investigated by exploring different stainless steels, including: austenitic stainless steel (AUSS), ferritic-austenitic (duplex) stainless steel (DSS), and super martensitic stainless steel (SMSS). The objective of this study is to determine the influences of thermal desorption analysis on the crystal structure of different stainless steels in order to better understand the trapping mechanisms of hydrogen in a variety of structure materials. It was found that the AUSS has the greatest stability of austenitic ( $\gamma$ ) phase— ~22% higher than DSS and ~45% higher than SMSS. Moreover, the AUSS presented the lowest hydrogen trapping values of ~31% compared with DSS and ~25% compared with SMSS.

Hydrogen fracture mechanism was found to be highly dependent on the hydrogen trapping states and even more on the  $\gamma$ -phase stability. The hydrogen trapping mechanisms are discussed in detail.

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## 1. Introduction

Stainless steels are the most common structural material for a variety of technologies and applications, particularly for oil, gas, pipeline and pressure applications — all of these industries combine hydrogen and mechanical load [1,2]. These materials are very attractive due to their high level of corrosion resistance and varied mechanical properties. Problems in maximizing those steels' full potential may arise from their interaction with hydrogen. In many applications these steels are exposed to hydrogen, which may lead to a deleterious effect known as hydrogen embrittlement. The base condition of this fracture mechanism is highly dependent on the dislocation process and is determined by hydrogen distribution combined with the local stress state in the material [3–5].

Understanding the hydrogen embrittlement phenomenon in the variety of stainless steels requires true insight of hydrogen solubility, diffusivity and distribution within their different microstructures, in particular inside phases and defects [6–8]. Since steel's defects (trapping sites for hydrogen) affect the steel's diffusivity, the crack initiation is highly dependent on the trapping energy states. It is known from earlier publications [9–14] that traps with activation energy  $\leq 60$  kJ/mol (also characterized as reversible traps) will have a major influence on the material's susceptibility to hydrogen embrittlement. In this work we study hydrogen trapping behavior in three different stainless steels: austenitic stainless steel (316), ferritic-austenitic (duplex) stainless steel (SAF 2205), and super martensitic stainless steel (SMSS).

The purpose of this work is to study hydrogen thermal desorption behavior in order to better understand its trapping mechanisms in a variety of structural materials.

We study the hydrogen trapping behavior with thermal desorption spectrometry (TDS), and hydrogen trapping energies

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are analyzed by the Lee and Lee model [15]. The analyzed data was confirmed by X-ray diffraction (XRD) and microstructural observations.

## 2. Experimental procedure

### 2.1. Microstructure analysis

In this research we used three different stainless steels: duplex stainless steels (DSS), SAF 2205, austenitic stainless steel (AUSS), 316L, and super martensitic stainless steel (SMSS). These steels were received in the fully annealed condition, as a 2 mm thick plate. The chemical compositions of these steels are presented in Table 1. These chemical compositions were confirmed to belong to the aforementioned structures according to the attached Scheffler diagram, Fig. 1.

The hydrogenation technique was electrochemical (cathodic) charging. The charging was performed at room temperature (RT) in a 0.5 N 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) with a constant current density of 50 mA cm<sup>-2</sup> for 72 h (h). The microstructure, phase composition, and lattice parameter of DSS were examined both before and after hydrogenation, by means of an X-ray diffractometer (XRD). XRD patterns were measured at RT using a Philips PW 1050/70 diffractometer, operating at a power of 40 kV and 30 mA, with Cu anode, and Ni-filtered generating Cu-K<sub>α</sub> radiation (1.54 Å). Data collection was performed by step scanning of the sample in a 2θ range between 30° and 100° at steps of 0.02° with 4–10 s per step depending on peak intensity, and a scan rate of ~0.3°/min.

The charged specimens were aged at RT and reexamined after one month, in order to observe the stability of hydrogen-induced phase transformations and lattice parameters after hydrogen desorption.

### 2.2. Thermal desorption spectrometry (TDS)

The characteristics of hydrogen desorption, and trapping states 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 treating the sample by non-isothermal heating at a known rate under UHV, ~10 μPa. In this work, the samples were heated from RT to 500 °C at constant heating rates of 2 °C/min, 4 °C/min 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 [15], allowed for the identification of different types of traps that coexist in the specimen.

## 3. Results and discussion

### 3.1. Microstructure changes in the presence of hydrogen

#### 3.1.1. Duplex stainless steel

The XRD diffraction pattern obtained from 72 h cathodic hydrogen charged DSS and aged for one month at RT is shown in

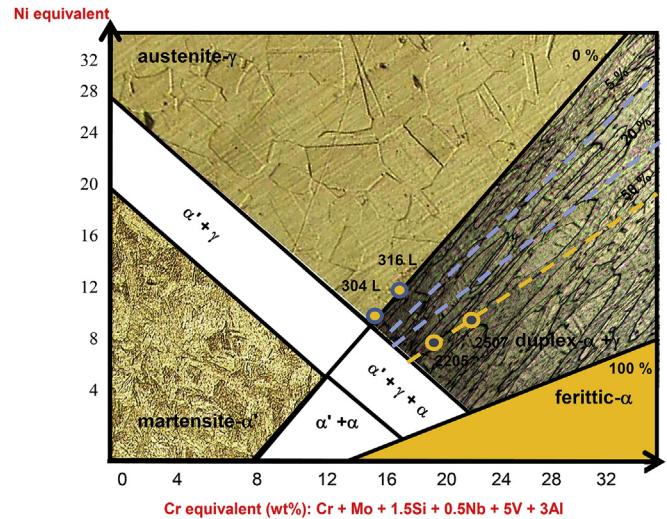


Fig. 1. Scheffler diagram for different steel compositions.

Fig. 2. This pattern is compared to an uncharged sample. The uncharged DSS (black line) displays ferritic-austenitic phases ( $\alpha/\gamma$ ) in equal amounts of about 50% for each phase.

The hydrogenation process reveals the significant changes in the  $\gamma$ -phase compared with those of the  $\alpha$ -phase. The charged samples showed a significant decrease in  $\gamma$ -phase intensity and shifts to small  $2\theta$  values; the latter change indicates a larger lattice parameter, which can be explained by hydrogen solubility in that phase. The significant changes between both phases are related to the lower diffusion coefficient and the greater solubility of hydrogen in the  $\gamma$ -phase [16,17]. These changes were seen simultaneously to the formation of an additional  $\epsilon$ -martensite phase, an hcp solid solution phase, and  $\gamma^*$ -phase, an additional fcc phase with a 2% higher

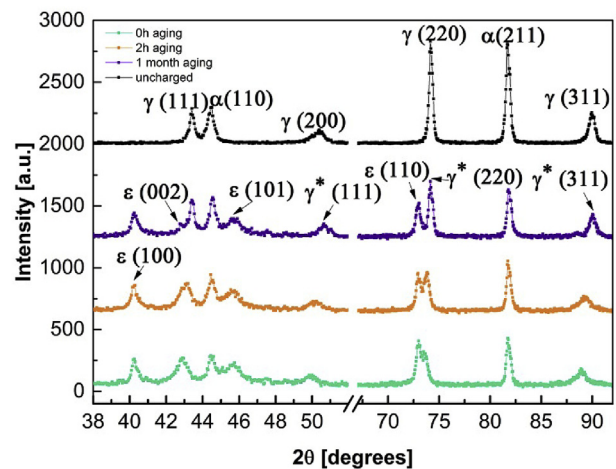


Fig. 2. Phase quantities of 72 h cathodic charged: DSS, AUSS and SMSS, after one month at RT.

Table 1  
Chemical compositions of the studied stainless steels (DSS, AUSS, SMSS) in wt%.

Sample	C	S	P	Mn	Si	Ni	Cr	Mo	N	Cu
SAF 2205	0.025	0.004	0.022	0.411	0.288	6.745	24.012	4.63	0.3	0.072
316L	0.04	0.006	0.04	1.164	0.375	10.693	15.612	2.748	0.039	0.274
SMSS	0.063	0.002	0.021	1.859	0.364	7.002	10.513	3.315	0.009	0.467

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