INTERNATIONAL JOURNAL OF HYDROGEN ENERGY 40 (2015) 10697–10703

Available online at www.sciencedirect.com

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journal homepage: www.elsevier.com/locate/he

Critical grain size to limit the hydrogen-induced ductility drop in a metastable austenitic steel

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ARTICLE INFO

Article history:
Received 22 May 2015
Received in revised form
19 June 2015
Accepted 22 June 2015
Available online 11 July 2015

Keywords:
Austenite
Grain size
Hydrogen embrittlement
Ultra-fine grains

ABSTRACT

The metastable austenitic stainless steel is Fe-16Cr-10Ni, of which the grain size can be controlled between 1 and 21 μm. Hydrogen precharging causes a critical drop in ductility during tensile tests for the largest grain size (21 μm). In order to understand how efficient grain refinement is against hydrogen-induced ductility reduction, by varying the heat treatment conditions, it was possible to manufacture six different grain sizes and pinpoint the grain size at which the drop of ductility is critical. This change in ductility was associated with a transition from fully ductile fracture surface to a fracture surface composed of dimples, quasi-cleavages and intergranular fracture.

Introduction

One important issue in the establishment of a society using hydrogen as energy-carrier for vehicles and local electricity production is the cost. At the moment, stable austenitic stainless steels are used in hydrogen components because of their resistance to the so-called hydrogen embrittlement. However their lower strength levels and high nickel contents mean also bulky and therefore more expensive components [1]. One possibility to obtain higher yield stress in austenitic stainless steels is strain hardening [2]. Furthermore in order to reduce costs, metastable austenitic steels, less expensive with lower nickel content, are being investigated [3]. However, in the case of metastable alloys, the presence of a BCC phase may be an issue, whether it is introduced prior to testing or during testing [4] as it may serve as a highway for the hydrogen into the austenite, or as phase that will affect the level of embrittlement [5]. Furthermore hydrogen has been known to cause ductility drops even in austenitic steels [6–9].

Reducing the grain size contributes to increasing the strength. Already, several researchers have tried to investigate the effect of reduced grain size on the steel resistance to hydrogen embrittlement [10–17]. Martensitic steels, duplex stainless steels and austenitic stainless steels have been studied, with either cathodic hydrogen charging or hydrogen gas charging, to introduce hydrogen into the materials. An overall trend was that reduced grain size helped reduced the hydrogen effect, but early report could not use grain sizes below 10 μm. More recently, Mine et al. [11,12] have used high-

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http://dx.doi.org/10.1016/j.ijhydene.2015.06.111

pressure torsion to produce grain sizes below 1 μm in types 310S and 304 austenitic stainless steels. However type 310S has a high nickel content, making it an even more expensive alternative to type 316, while type 304 retained martensite even after annealing. Also, the high-pressure torsion produced disks less than 20 mm in diameter, raising the question of the applicability of such a process to obtain ultra-fine grains on an industrial scale.

In the present study, a metastable austenitic stainless steel, developed in the laboratory, was used [18]. Its three main particularities are that the grain size can be controlled down to 1 μm, that it does not require any special equipment to do so, and that the final microstructure is fully austenitic. Indeed regular cold-rolling and annealing are sufficient, for this particular alloy composition, to control the grain size. This in turn makes this alloy an interesting candidate for the manufacture of higher strength austenitic steels leading to decreased size and cost of components. The objective here is to investigate how much the grain size needs to be reduced in order to produce relatively high strength along with reduced susceptibility to hydrogen embrittlement. Six different grain sizes were used. Scanning electron microscope (SEM) observation showed the evolution of fracture surfaces for decreasing grain sizes for a specific hydrogen content.

Material and experimental methods

The metastable austenitic stainless steel used here is Fe-16Cr-10Ni [18]. The chemical composition is 16.4Cr, 10.1Ni, 0.08P and 0.002C, the remainder being Fe. After solution treatment, 15 mm thick plates were cold-rolled to 1.5 mm thick sheets, the resulting sheets being more than 90% strain-induced martensite. Annealing in argon gas was then conducted to transform the martensite back to austenite, with six different conditions: 923 K for 10 min, 1023 K for 10 min, 1073 K for 10 min, 1123 K for 10 min, 1173 K for 10 min and 1173 K for 30 min. The grain sizes d obtained were 1, 5.8, 6.3, 9.6, 12.3 and 21 μm, respectively. Electron Backscattered Diffraction (EBSD) maps showing each grain size are shown in Fig. 1. The microstructure after annealing did not present any texture for any of the grain sizes. All the sheets were shown, by saturation magnetization measurements, to be fully austenitic upon completion of the reversion treatment.

Tensile specimens (gauge 18 mm in length, 3 mm in width and 1.5 mm in thickness) and chips (5 × 5 × 1.5 mm) for Thermal Desorption Spectrometry (TDS) were cut out and polished with buff. The specimens were charged in 10 MPa hydrogen gas at 543 K for 72 h. After hydrogen charging, the hydrogen content in the material was measured with the TDS chips (TDS maximum temperature 1073 K, heating rate 0.33 K s^{-1}). The tensile tests were all conducted at room temperature in laboratory air, at a crosshead speed of 0.02 mm min^{-1} (strain rate: 2×10^{-5} s^{-1}). The fracture surfaces were all analyzed by Scanning Electron Microscope (SEM).

Results

Hydrogen charging

As Fe-16Cr-10Ni is metastable and has a grain size dependency for its M_s temperature [18,19], the specimens were not stored in liquid nitrogen to be tested at an ulterior time, to avoid having the larger grained specimens transform into martensite. Instead, all specimens were tested in the same day after hydrogen charging. Due to the very low hydrogen diffusivity in FCC phase at room temperature [20], it was not expected that hydrogen would diffuse out of the tensile specimens and TDS chips. The TDS chips were polished after charging to decreasing thicknesses (1.5, 1.1, 0.7 and 0.2 mm) and the TDS measurements were conducted. The hydrogen

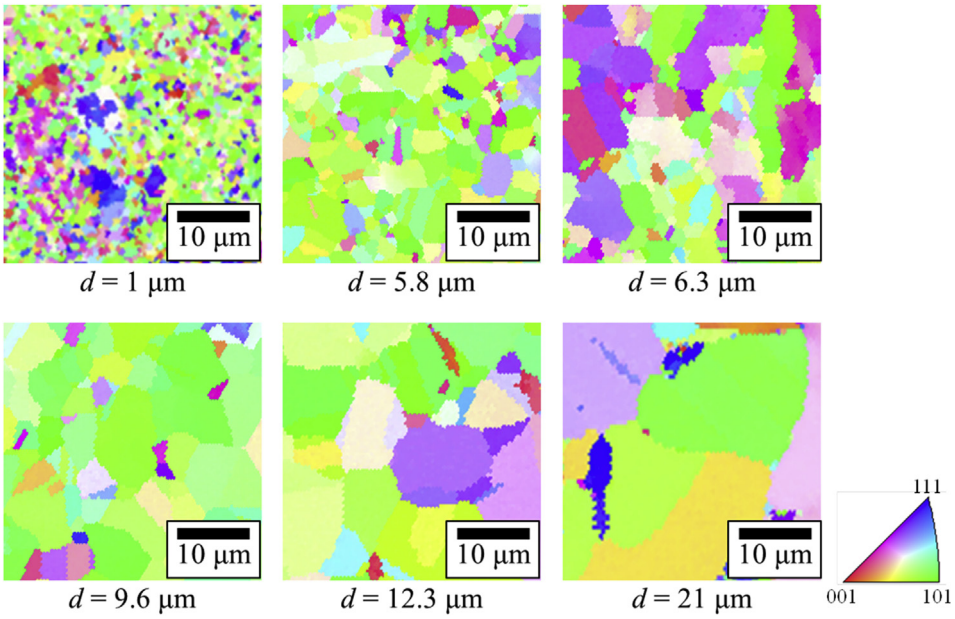

Fig. 1 – EBSD orientation maps for all grain sizes.