

The Role of an Al-induced Ferritic Microfilm in Martensitic Steels on the Hydrogen Embrittlement Mechanisms Revealed by Advanced Microscopic Characterization

M. Pinson¹, S.M. Das², H. Springer^{2,3}, K. Verbeken¹, T. Depover^{1*}

¹ Department of Materials, Textiles and Chemical Engineering, Ghent University, Ghent, Belgium.

² Department of Microstructure Physics and Alloy Design, Max-Planck-Institut für Eisenforschung, Düsseldorf, Germany.

³ Institut für Bildsame Formgebung, RWTH Aachen University, Aachen, Germany.

* Corresponding author: tom.depover@ugent.be

High strength martensitic steels are a cornerstone for a multitude of industrial applications due to their high hardness. However, their high strength levels are generally linked with an inherently brittle character which is the main limitation for these type of steels. To increase ductility, small amounts of ferrite can be introduced by ferrite stabilizing elements such as Al. This concept is validated for Fe-0.4C steels with the addition of 2 wt.% of Al which results in the formation of a ferritic microfilm along the prior austenitic grain boundaries (PAGBs). A detailed microstructural analysis of this ferritic microfilm is performed by means of scanning electron microscopy (SEM), channeling contrast imaging (ECCI), nanoindentation and transmission electron microscopy (TEM) and the results are shown in **Figure 1**. The ECCI results in **Figure 1(a-b)** show that the microfilm has a needle-shaped morphology with a clear dislocation structure. The nanoindentation experiments in **Figure 1(c)** confirm that the ferritic microfilm has a much lower nanohardness than the surrounding martensitic matrix. The bright field TEM image with corresponding EDS data in **Figure 1(d-f)** gives additional information on the formation mechanisms; the C depletion of the ferritic microfilm indicates that the ferritic phase is formed first upon cooling which causes C diffusion towards the surrounding martensitic phase which has a higher C saturation level.

To evaluate the impact of this ferritic microfilm on the hydrogen embrittlement (HE) mechanism, in-situ hydrogen bending tests are performed and combined with complementary detailed microscopy analysis of the corresponding fracture surfaces. The results show that the presence of the ferritic microfilm influences the mechanical behavior to a high extent. When tested in air, the ferritic microfilm increases the bulk ductility by over 100% when compared to samples with a similar hardness which do not contain such microfilm [1]. As shown in **Figure 2** (left), fracture in air occurs at the ferritic microfilm which results in a fracture surface with microvoids dispersed over the underlying martensitic microstructure. Moreover, the ferritic microfilm also influences the mechanical behavior in the presence of hydrogen. The HE mechanisms of an Al-free martensitic Fe-0.4C steel without grain boundary ferrite is linked to the hydrogen enhanced decohesion (HEDE) mechanism since hydrogen causes a brittle cleavage type of fracture linked to the embrittlement of the martensitic packets/blocks, as demonstrated in [2]. However, the Al-alloyed samples fail according to a different HE mechanism since an almost purely intergranular fracture is detected when tested in a hydrogen rich environment (cf. **Figure 2** (center)). The interface between the ferritic microfilm and the martensitic matrix suffers from decohesion by an interplay between the HEDE and hydrogen enhanced localized plasticity (HELP) mechanism, often termed as the hydrogen-enhanced-plasticity mediated decohesion. Nonetheless, this is paired with a delay in sample fracture since crack propagation is redirected along the ferrite/martensite interface instead of through the martensitic matrix. However, when the samples are tested at a slower deformation rate in the presence of hydrogen, the ferritic film is also embrittled and loses its capability to influence the fracture path, thus

resulting in a similar cleavage fracture surface (cf. **Figure 2** (right)) as samples without the presence of the ferritic microfilm [2].

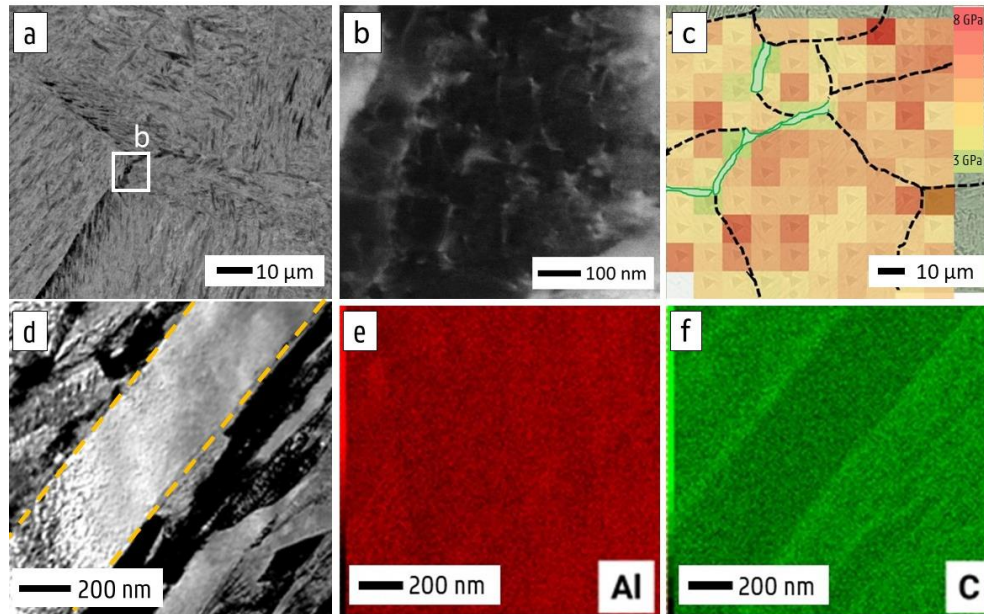


Figure 1: (a) ECCI image of the microfilm along the PAGBs, (b) detailed ECCI image indicating the dislocation structure, (c) nanoindentation results on top of the etched optical microscopy image where the PAGBs are indicated as black lines and the ferritic microfilm is indicated by green zones, (d) STEM bright field image of the grain boundary microfilm (in between the yellow dotted lines) and corresponding elemental maps of (e) Al and (f) C.

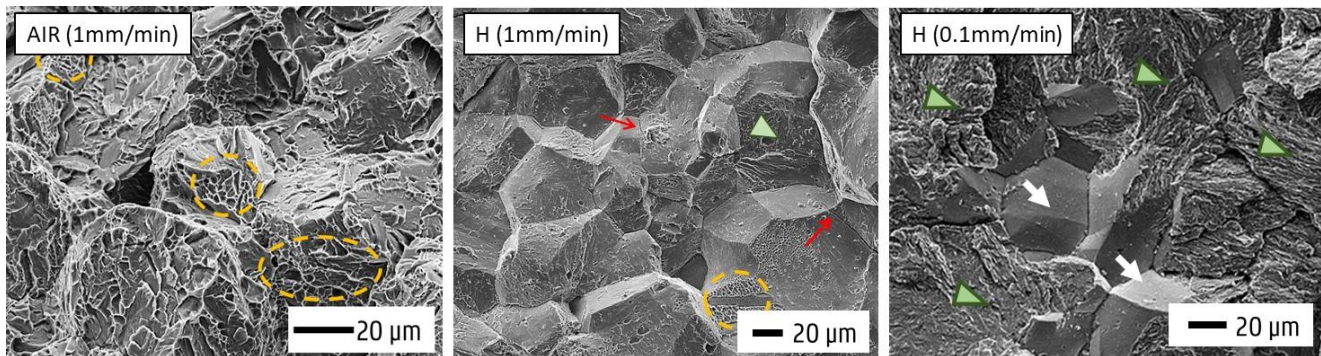


Figure 2: Scanning electron microscopy investigation of the fracture surfaces of the Fe-2Al-0.4C material tested in both air and in an hydrogen rich environment at two deformation rates, i.e. 1mm/min and 0.1mm/min. Yellow dotted circles indicate zones with microvoids, white arrows represent intergranular features and green arrowheads demonstrate cleavage features. Cracks are illustrated by fine red arrows.

References:

- [1] M. Pinson et al., *Scripta Materialia* **213** (2022), p. 114606, doi: 10.1016/j.scriptamat.2022.114606.
- [2] M. Pinson et al., *Materials Science and Engineering: A* **792** (2020), p. 139754, doi: 10.1016/j.msea.2020.139754