

## ON THE MECHANISTIC ORIGINS OF THE INCREASED HYDROGEN ENVIRONMENT-ASSISTED CRACKING SUSCEPTIBILITY OF AM 17-4PH STEEL

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### Abstract

Literature results indicate that the hydrogen environment-assisted cracking susceptibility of additively manufactured (AM) 17-4PH steel fabricated using laser powder bed fusion is increased relative to comparable wrought 17-4PH. This study seeks to understand the mechanistic origins of this increased susceptibility through a detailed examination of near-crack deformation, alloy microstructure, and hydrogen-metal interactions. Based on these data, it is determined that sub-micrometer porosity present in the AM material provides a primary contribution to the degradation in HEAC resistance. The mechanistic basis for the influence of porosity is considered in the context of an existing model for HEAC. The implications of these findings on the broader AM community are then discussed.

### 1. Introduction

Due to its high strength and good corrosion resistance, the precipitation-hardened, martensitic stainless steel 17-4PH is widely employed in aggressive operating environments across the marine, petrochemical, nuclear, and aerospace sectors. While 17-4PH has a long history in its conventional wrought form, there is increased interest in leveraging AM to manufacture 17-4PH components. However, laboratory testing and in-service failures have established the susceptibility of 17-4PH to HEAC. Moreover, studies have demonstrated that the susceptibility of wrought 17-4PH to HEAC is heat treatment-dependent, confirming an important role of microstructure on HEAC susceptibility. However, despite the importance of the HEAC failure mode for 17-4PH components and the likelihood of differences in HEAC behavior for AM and wrought 17-4PH due to microstructural variations induced by the AM process, studies on the environment-assisted cracking behavior of AM 17-4PH are highly limited. The objective of this study is to understand the mechanistic basis for differences in the HEAC behavior of AM and wrought 17-4PH stainless steel heat-treated to obtain nominally similar yield strengths and mesoscale microstructures.

### 2. Results

The AM and wrought 17-4PH were both heat-treated to the over-aged (OA) condition. The AM material first underwent a hot isostatic press (HIP) treatment, followed by a standard solution annealing (SA) heat treatment, and then tempering to the OA condition (in lab air at 552°C for 4 h). The wrought material underwent the same SA and OA treatments; the two materials are referred to as AM (HIP+SA+OA) and wrought (SA+OA). Nominally similar microstructure and mechanical properties were observed between the wrought (SA+OA) and AM (HIP+SA+OA) 17-4PH. However, while the AM material approaches full density (~99% of wrought) after this heat treatment protocol, a large population of small, sub-micrometer pores are observed during metallographic evaluation.

Comparative slow-rising stress intensity experiments were completed on additively manufactured and wrought 17-4PH stainless steel heat-treated to the over-aged condition *via* full immersion in 0.6 M NaCl while polarized to -1.1 V<sub>SCE</sub>. Results demonstrated that the AM (HIP+SA+OA) material exhibits consistently elevated crack growth rates (~10-fold increase) relative to the wrought (SA+OA) alloy for a given K. Fractography revealed that both alloys exhibit widespread intergranular fracture morphologies, though numerous circular features were noted on the facets of the AM (HIP+SA+OA) alloy. Interestingly, these circular features were of the same size as porosity observed during the metallographic assessments.

Distinct differences in hydrogen-metal interactions were noted between the wrought (SA+OA) and AM (HIP+SA+OA) materials (Figure 2). In particular, AM (HIP+SA+OA) exhibits a higher hydrogen

diffusivity and lower diffusible hydrogen concentration (at  $-1.1 V_{SCE}$ ) than the comparable wrought material. However, correlation of these data with observed crack growth behavior suggests that microstructural features, and not hydrogen-metal interactions, are providing a primary contribution to the increased HEAC susceptibility of the AM material.

To investigate the potential influence of these sub-micrometer pores on the HEAC behavior of AM 17-4PH, the intact near-crack tip region of an interrupted AM (HIP+SA+OA) fracture specimen tested in 0.6 M NaCl at  $-1.1 V_{SCE}$  was examined using correlative electron microscopy and EBSD, shown in Fig. 1. Based on the correlative backscatter image (Fig. 1a), inverse pole figure map (Fig. 1b), and kernel average misorientation map (Fig. 1c), it appears that the pores are acting to concentrate deformation along the grain boundary ahead of the crack tip (which was the preferred fracture pathway for this condition). Such evidence suggests an important role of the sub-micrometer porosity with regards to the increased HEAC susceptibility of the AM (HIP+SA+OA) condition.

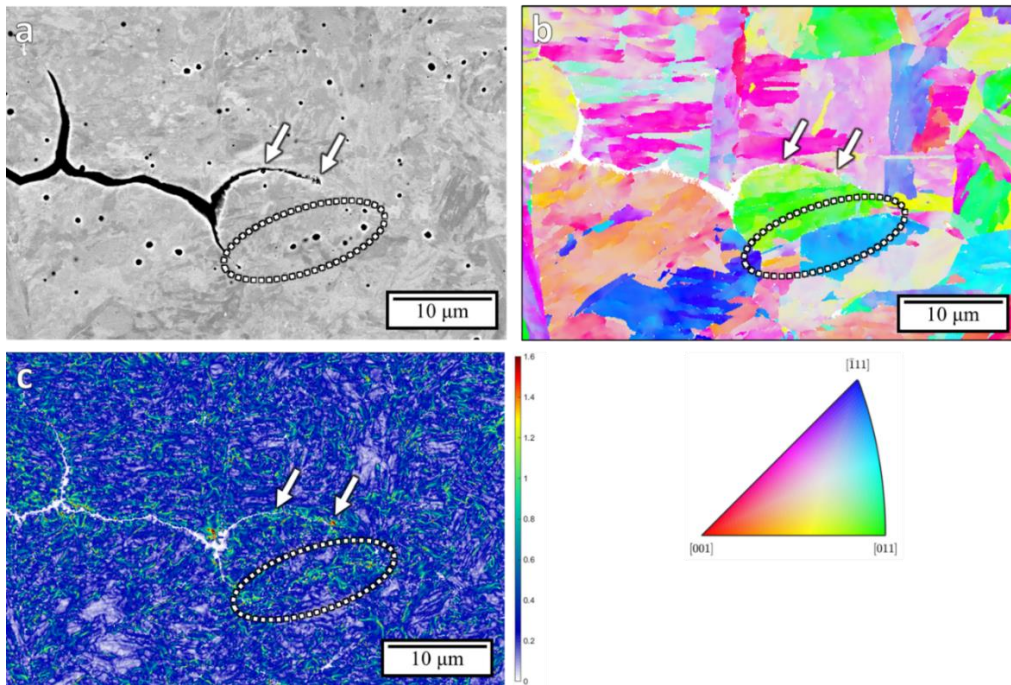


Fig.1 – Correlative (a) backscatter micrograph, (b) IPF map, and (c) KAM map from the crack tip of an interrupted AM (HIP+SA+OA) specimen tested in 0.6 M NaCl at  $-1.1 V_{SCE}$  and  $K \approx 54 \text{ MPa}\sqrt{\text{m}}$ . Crack growth is from left to right and the dashed oval represents the same location in all images.

### 3. Conclusions

Characterization of the crack tip region from an interrupted fracture experiment on AM (HIP+SA+OA) immersed in 0.6 M NaCl at  $-1.1 V_{SCE}$  suggests a casual role of the sub-micrometer porosity on the enhanced HEAC behavior of AM (HIP+SA+OA) relative to wrought (SA+OA). Specifically, coupled microscopy and EBSD analyses reveal that the sub-micrometer porosity is concentrating plastic damage along a grain boundary in front of the crack tip, likely aiding intergranular crack extension and subsequently increased HEAC susceptibility.

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