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In-situ observation of hydrogen induced crack initiation in a nickel-based superalloy

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ABSTRACT

Hydrogen induced crack initiation in a nickel-based superalloy was studied via in-situ Scanning Electron Microscopy observation under slow strain rate loading. Direct evidence has been obtained of crack initiation and propagation along dislocation slip bands leading to sample fracture. Post-mortem microstructural analysis showed that hydrogen induced cracking and separation along dislocation slip bands, which are along {111}γ crystallographic planes with the highest Schmid factor, leads to the formation of the typical slip-trace-like features on the quasi-cleavage surface.

Nickel-based superalloys are promising structural materials for the oil and gas deep well applications due to their high strength at high temperatures and excellent corrosion resistance [1]. However, when they are exposed to hydrogenating environments, nickel-based superalloys can suffer from hydrogen induced embrittlement [2,3]. The current lack of understanding in the mechanism of hydrogen induced embrittlement (HE) of nickel-based superalloys hampers tailoring the alloy chemistry and microstructure in order to optimise resistance to hydrogen embrittlement [3,4].

Among the various mechanisms accountable for HE of metallic materials, the hydrogen enhanced decohesion (HEDE) [5,6] and hydrogen enhanced localized plasticity (HELP) [7,8] are the mostly invoked mechanisms for materials that do not form hydrides. HEDE mechanism suggests that hydrogen can reduce the atomic cohesive strength and thus brittle fracture occurs where hydrogen concentration reaches the threshold [5,6]. In contrast, HELP mechanism proposes that hydrogen can reduce the activation stress of dislocation mobility and enhances dislocation slip localisation [9,10], which leads to premature material failure. The essential difference between the two mechanisms is the involvement of microscale plasticity, i.e. dislocation motion in the case of HELP and truly brittle failure without dislocation-mediated plasticity for HEDE. For nickel-based superalloys, due to large volume fraction of precipitates (γ′, γ″, δ and carbides), the precipitates/matrix interfaces, which might be potential hydrogen trapping locations, are likely to be the vulnerable sites for crack initiation according to the HEDE mechanism. Extensive research has been conducted to study the effect of precipitates on the hydrogen embrittlement susceptibility of nickel-based superalloys [3,11–15]. The general trend is that hydrogen induced cracking along the precipitates/matrix interface is only observed when they are deliberately coarsened and/or segregated along grain boundaries [3,13,16,17]. Direct evidence of cracking along precipitates/matrix interface is seldom seen in conventionally treated material, which suggests that the HEDE mechanism might be of limited relevance. On the other hand, substantial evidence of plasticity on the microscale has been reported for hydrogen-embrittled superalloys. Recent studies on the HE behaviour of superalloy UNS N07718 (Alloy 718) demonstrated that hydrogen-dislocation interaction under the framework of HELP mechanism might play a key role [18]. However, these observations were only based on post-mortem analysis and there is a great desire to obtain in-situ observation on hydrogen induced cracking to provide further evidence of the prevalence of the HELP mechanism. Therefore, in this study, in-situ SEM observation was conducted on hydrogen-charged Alloy 718 to reveal the interplay between slip localisation, crack initiation and propagation process in the presence of hydrogen. Additional post-mortem analysis on the in-situ tested samples was carried out in order to provide additional information about the crack initiation region and the fracture surface affected by the HE.

Alloy 718 was heat-treated to achieve a microstructure considered as typical for oil and gas applications, including solution treatment at 1040 °C for 1 h followed by aging at 774 °C for 6 h. The microstructure obtained by the heat treatment can be found in [18]. Flat tensile specimens with a gauge length of 6 mm, a width of 3 mm and a thickness

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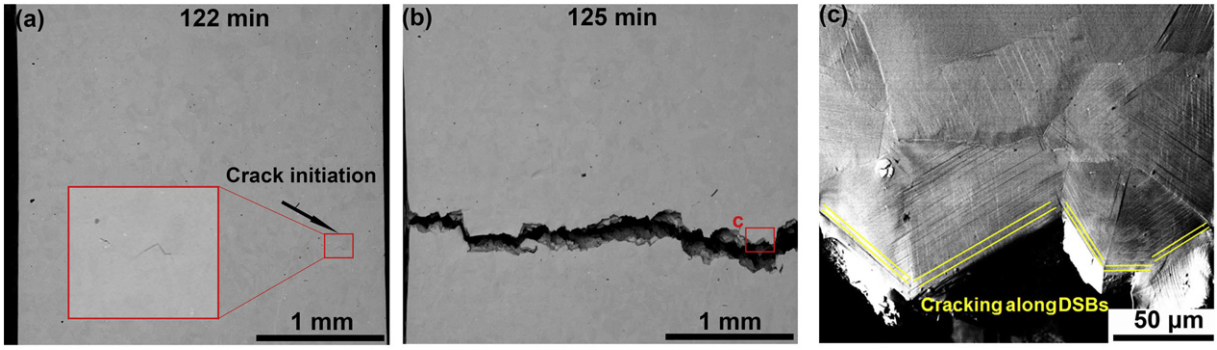

Fig. 1. SEM images of the sample during in-situ tension captured at different times (a) 122 min with ε_p of ~7.3%, when a visible crack was detected, and an enlarged image of the framed region was inset (b) 125 min with ε_p of ~7.8%, when the sample fractured, (c) BSE image shows the crack initiation site, which corresponds to the framed region in (a) and (b). The loading axis is vertical.

of 1 mm were machined by means of electro discharge machining. The machined samples were ground and polished with 40 nm colloidal silica suspension (OPS, Struers) to achieve mirror quality surface finish. Cathodic hydrogen charging was conducted at 80 °C in a NaCl (1 mol/L) solution. The samples were charged with a constant electric current density of 7.7 mA/cm^2 for 168 h. As reported in previous work [18], the H penetration depth is estimated to be ~140 μm. To remove the contamination and passive layer on the sample surface due to hydrogen charging, the sample was polished by OPS for about 5 min to retrieve mirror surface finish. Subsequently, in-situ loading was conducted on an FEI Quanta650 field emission gun SEM. To minimize H degassing, the charged sample was put into the microscope for mechanical loading within half an hour. Before loading, the sample surface was checked by SEM and no hydrogen induced defect was seen. Slow

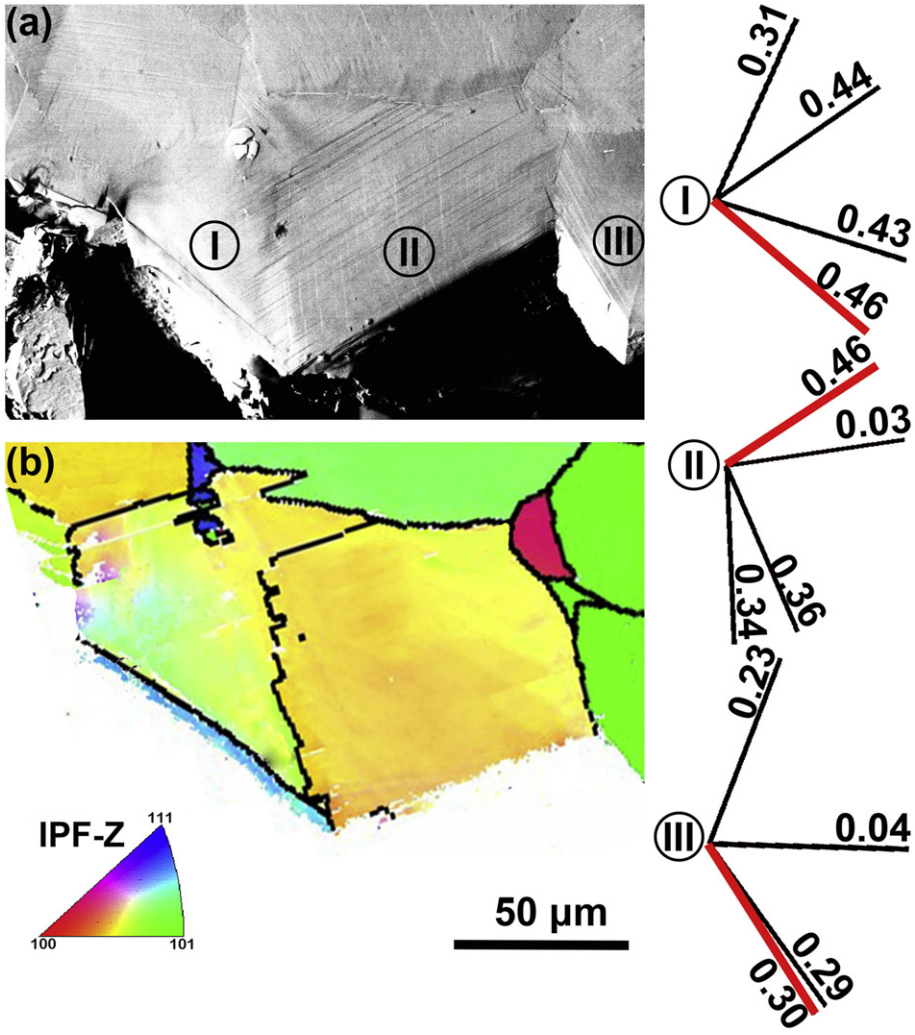

Fig. 2. (a) A BSE image from the crack initiation region, (b) an EBSD orientation map from the same region. The {111}γ plane traces from the regions I, II and III are shown on the right side with the maximum Schmid factor of each slip plane indicated. The slip planes that are parallel to the cracking path are coloured in red. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)