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The beneficial effect of surface carbon coating on stress corrosion cracking of Type 304 austenitic stainless steels in high temperature water

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ABSTRACT

A pronounced improvement in stress corrosion cracking initiation resistance resulting from the presence of a carbonaceous surface film was investigated for specimens strained dynamically in hydrogenated high temperature water. Microstructural characterisation of the post-test samples indicated that this improvement in SCC resistance was promoted by the formation of a more uniform and thick surface oxide layer that prevented localized oxidation.

Austenitic stainless steels are widely used to manufacture internal components of the primary circuit of pressurised water reactors (PWR). Although these steels exhibit great performance and resistance to corrosion, they can exhibit primary water stress corrosion cracking (PWSCC) [1–7]. In the PWR primary water environment, PWSCC usually occurs in cold-worked austenitic stainless steels, and the deformation induced by the cold work has the effect of promoting strain localization along the grain boundaries and deformation bands [8]. The results of these material-environment interactions have been reported to promote preferential oxidation along the grain boundaries and deformation bands, and be the main contributing factor to PWSCC initiation in austenitic stainless steels [9–12].

The nature of the passive film plays an important role in the PWSCC, and specific examples can be shown in the literature. For instance, it has been shown that surface grinding can mitigate SCC for Alloy 600 [13] by the formation of a uniform external oxide as opposed to internal and preferential intergranular oxidation [13–17]. An analogous effect has been observed in austenitic stainless steel where the ultrafine-grained layer generated from machining mitigated preferential intergranular oxidation [5,18]. However, a more protective film does not necessarily imply better resistance to environmentally-assisted fracture. In fact, the enhanced oxidation that is associated with high sulphur contents in Type 304 and 316 austenitic stainless steels has been shown to retard intergranular crack propagation [7,19] and fatigue crack growth rate [20] in high temperature hydrogenated water. Similar effects were also postulated by the present authors [18] for the role of surface carbon contamination. To date, there has never been a direct observation of the effect of surface carbon on PWSCC initiation in austenitic stainless steels. Thus, this work is aimed at understanding the role of a carbonaceous film present on the material's surface on subsequent oxidation and implications on SCC initiation for forged austenitic stainless in high temperature hydrogenated water. This could be relevant to manufactured components for which a carbonaceous deposit can result from the lubricant used in machining. This suggestion is based on the observation shown by the present authors and reported in reference [18]: it was observed that SCC initiation was retarded in those regions that had carbonaceous contamination due to prolonged exposure under the electron beam during scanning electron microscope (SEM) based examination. In the present work, a very thin C layer was deposited onto a carefully polished surface of the warm-forged Type 304 austenitic stainless. The warm-worked deformation structure is similar to that generated in a weld heat-affected zone microstructure where plastic strain can be as high as 20% near the fusion line [19,21] and are regions where SCC is most likely initiate in service [14,19,21,22]. The material's SCC performance was assessed using slow strain rate tension (SSRT) tests, which have been shown to produce results that are consistent with the trends obtained with more plant-relevant tests in high temperature water conditions, such as constant load blunt notch compact tension (C(T)) tests, although in a significantly shorter time period [5,6,14,21,22]. Furthermore, SSRT tests allow greater material/environmental interactions due to the low strain rates, thus promoting more SCC.

Type 304 austenitic stainless steel used in this study had a composition (mass %) of 18.34% Cr, 7.93% Ni, C 0.029%, Mn 1.86%, Mo 0.33%, Si 0.36%, S 0.004%, P 0.023% and Fe (balance) was warm-forged (20% height reduction at 360 °C) [18]. The average grain diameter was

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approximately 25 μm. The steel contained approximately 5% δ-ferrite present as stringers that were elongated in the hot-rolling direction, and more details are available in reference [18]. Flat dog-bone tensile specimens were then extracted from the forged block so that the transverse direction of the sample was also the forging direction. Prior to testing, both sides of the gauge section of the tensile specimen used were ground using 600 grit SiC paper. One surface of the gauge section was progressively ground using finer papers and subsequently polished using diamond paste and oxide polishing suspension (OPS) to remove any deformation induced during the grinding steps. The dimension of gauge section of the specimen after grinding and polishing was 12 mm in length, 3.5 mm in width and 2 mm in thickness. Both sides of the gauge section were subsequently plasma cleaned to remove any surface C contamination. Half (longitudinal section) of the OPS-polished surface was then coated with a 20 nm thick C layer using a Quorum Q150T ES evaporator using pulsed evaporation of a 1.4 mm diameter carbon rod for 2 min with a pulse current of 54 A. To ensure that only one half of the SSRT sample was coated, one half of the SSRT specimen was masked to preserve the as-polished surface.

The SSRT test was performed at 300 °C in deoxygenated (O_2 < 2 ppb), hydrogenated (H_2 = 3 ppm) and lithiated (2 ppm of Li added as LiOH) water using a refreshed flow loop previously employed to achieve similar water chemistry exposure [23,24]. During the test, the specimen was initially strained at $1 \times 10^{-5}\ s^{-1}$ to 80% of the yield stress at 300 °C (575 MPa). Thereafter, the strain rate was reduced to $2 \times 10^{-8}\ s^{-1}$ until the sample was plastically strained to ~5%, which are conditions known to promote SCC in this material [18]. The higher strain rate was used to reduce the time required to reach 80% of the yield strength, whilst the slower strain rate was chosen to promote greater material/environmental interactions and therefore more SCC. In addition, three oxidation coupons were prepared for electrochemical corrosion potential measurements tests. One was prepared in exactly the same way as the SSRT sample whilst the other one had all surfaces coated with a 20 nm-thick layer of C after OPS polishing and plasma cleaning. The third coupon was not coated with C after OPS polishing and plasma cleaning. The electrochemical corrosion potentials of the oxidation coupons, as well as the one of a platinum sample, were periodically measured throughout the experiment using an Ivium

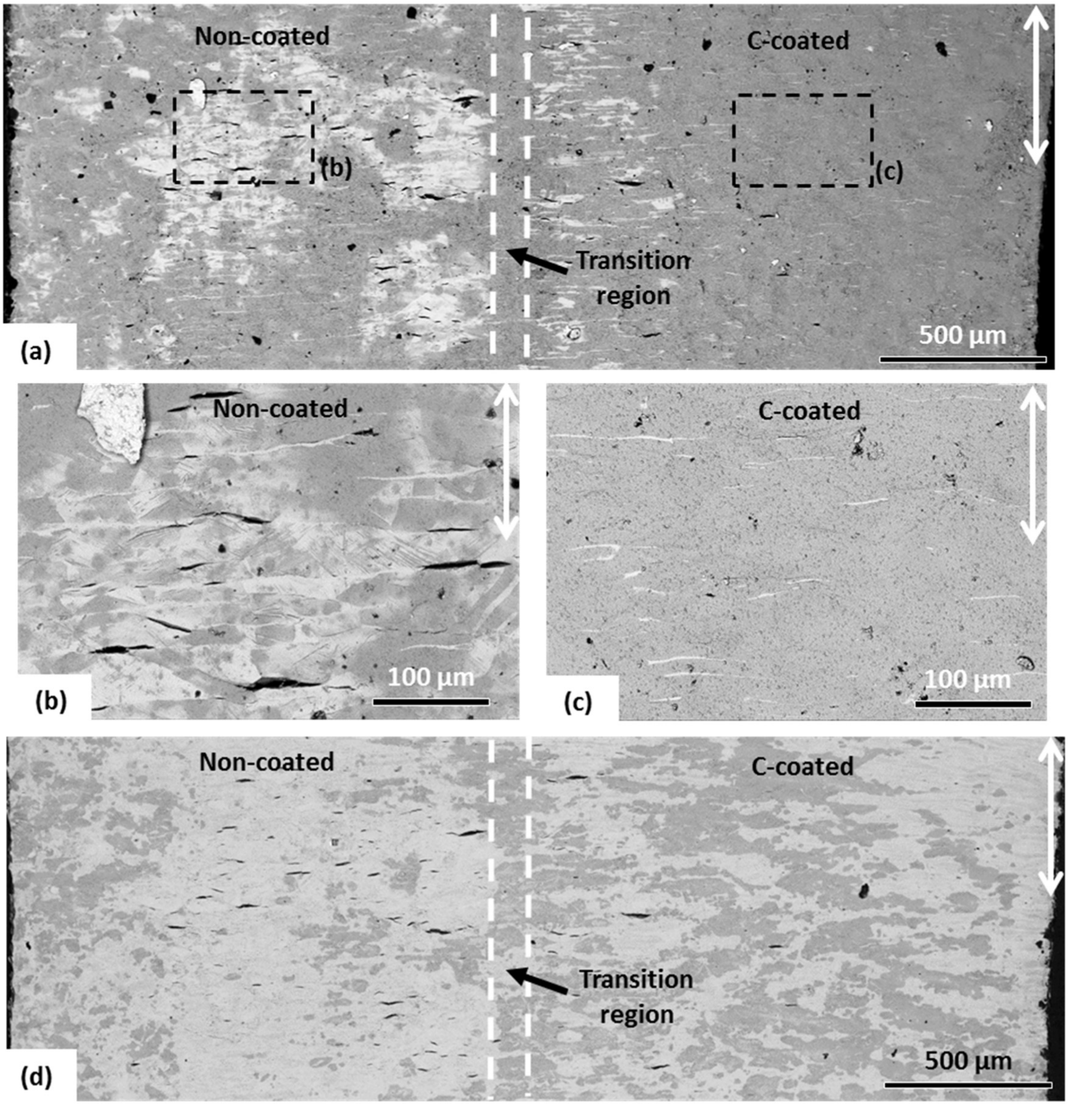

Fig. 1. Plan-view BSE images of the sample that was OPS-polished prior to SSRT test. (a) Low magnification image showing the non-coated surface (left) and C-coated surface (right); the transition between these two regions (*i.e.* region within the two dashed line) was related to the C deposition process. (b)–(c) BSE images of the regions within the dashed rectangles in (a). (d) BSE image of region shown in (a) after the oxide was removed showing a higher proportion of SCC in the non-coated region. The dark patches in (d) are regions were the inner oxide layer was not completely removed. The loading axis is identified by vertical white arrows.