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Imaging of 316H SENT creep sample subjected to oxidation at high temperature

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At elevated temperature (550°C) in CO₂ environment, the 316H stainless steel is known to be sensitive to intergranular creep cracking [1]. In view of assessing creep crack growth from surfacic Digital Image Correlation measurements, the surface pattern stability of the creep specimen was assessed in this preliminary study. Monitoring the creep crack initiation and growth is conducted at the grain scale using scanning electron microscopy. Small Single Edge Notched Tensile specimens (SENT) have been machined using electric discharge machining (see Figure 1). In the tests, we plan to use a/w ratios close to 0.15.

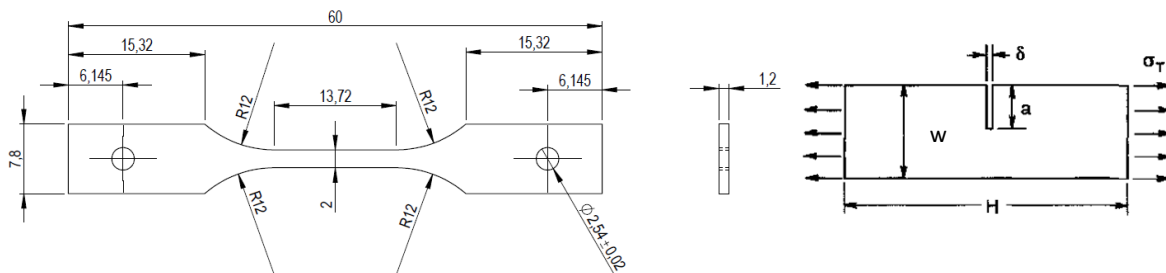


Figure 1: Single Edge Notched Tensile creep specimen drawing (metric unity: mm, crack length a , gage width w).

In order to get an adequate signal for Digital Image Correlation, a cathodic deposition of a tungsten (W) pattern obtained by microlithography (thickness 20nm) is carried out in the vicinity of the crack tip prior to the mechanical testing. Then, interrupted creep tests are conducted at 550°C in air. For each interruption, images are acquired using a SEM FEI Quanta 600 in BSED/Z contrast.

The oxidant atmosphere and the elevated temperature are likely to affect the surface pattern. Therefore a preliminar study aims at determining the optimal sample preparation to get a stable pattern. After a fine polishing (until OPS), several specimens have been subjected at 550°C in a furnace with air flow. After five weeks, ion beam sectioning and SEM observations have lead to quantify the thickness of the formed oxide (about 1 μm). The brittle oxide is detrimental to the pattern quality. Etching was then conducted to retrieve the oxide layer without damaging the

chromium-rich layer which confers to the material its good corrosion resistance. Etching conditions were HCl and HNO₃ solutions at room temperature with ultrasonication. The W pattern is then deposited on the etched surface and it is submitted to high temperature oxidation. Figure 3 shows the pattern after two weeks.

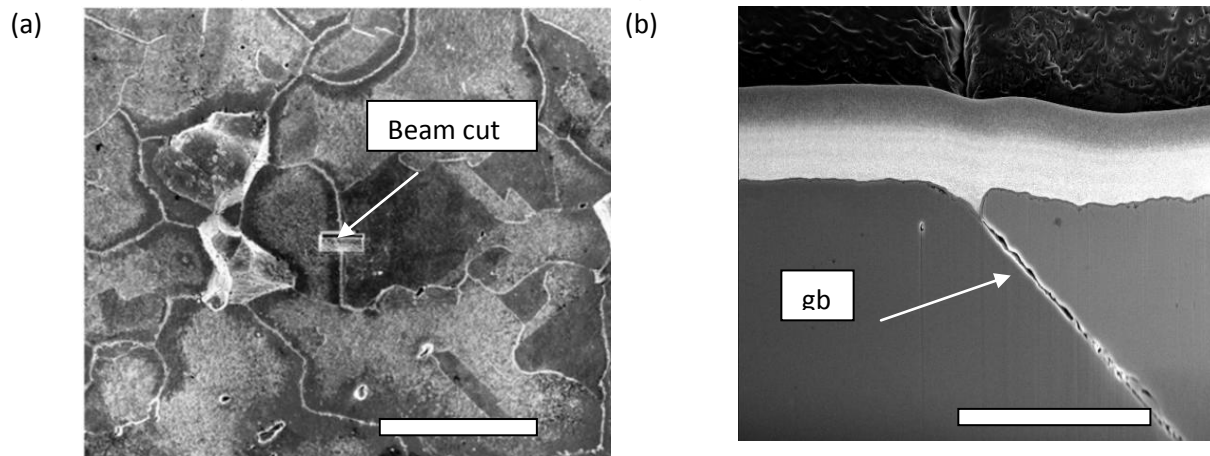


Figure 2: SEM image in SE mode of dual beam cut after etching (Quanta600 FEI) (a) (the scale bar is 100 µm in length) of a grain boundary (b) (the scale bar is 5 µm in length)

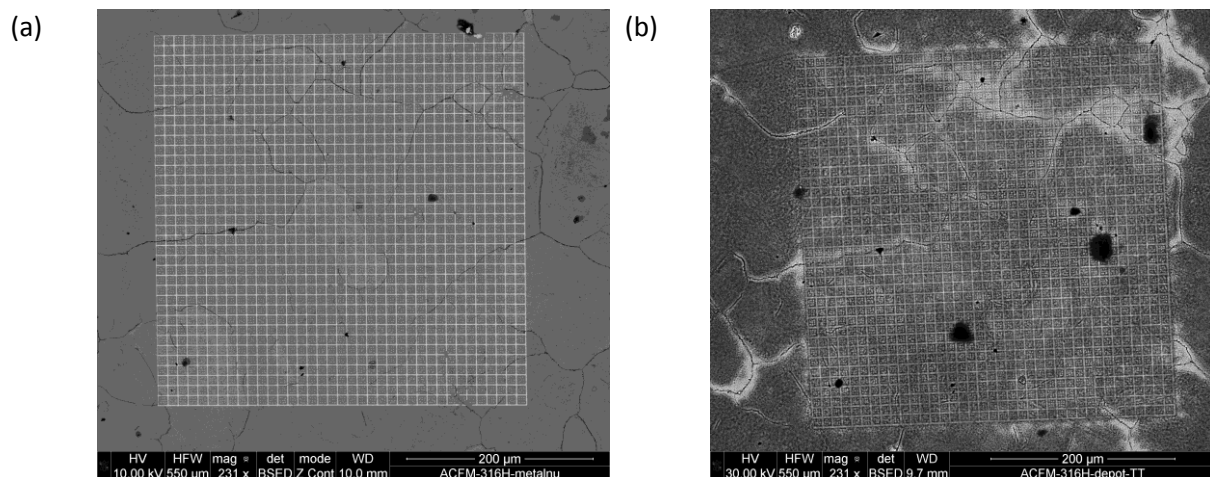


Figure 3: SEM image (BSE mode/Z contrast) of 316H patterned surface after etching and lithography (a) and after 2 weeks in air at 550°C (b).

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