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Report:

Stainless steels exhibits an attractive combination of good mechanical properties and corrosion resistance, leading to their wide use in diverse applications. However, these properties may deteriorate if the manufacturing process is not carefully controlled. The brittle sigma precipitate phase is of particular concern, because it has rapid formation kinetics and can dramatically decrease the toughness and corrosion resistance of the steel. It is therefore essential to understand the formation characteristics of these precipitates such as growth kinetics, spatial distribution and connetivity.

In the original proposal two different types of experiments were planned; static experiments and high-temperature (T=900°C) in-situ annealing experiments. Unfortunately, initial technical problems with the high resolution detectors did not leave sufficent time for the more challeging high-temperature in-situ experiments. It was therefore decided to concentrate on a careful static charaterization of the samples that could serve as input to the subsequent FEM modelling efforts.

Results

Tomography of a strongly absorbing material like steel and furthermore, having a very small density difference between the austenite matrix phase and the sigma precipitate phase of interest, is not a trivial task. Thus, optimisation of the experimental settings were crucial for a successful outcome of the experiment. After testing various experimental set-ups, it was decided to work with pure absorption contrast at an x-ray energy of 21 keV – just above the molybdenum edge – maximising the contrast between the Mo rich sigma precipitates and the matrix.

From standard surface techniques it was known that the sigma precipitates in the AL6XN alloy primarily nucleate in narrow concentrations bands associated with the centerline of the steel sheet (Fig. 1a). However, three-dimensional morphology, spatial distribution and connectivity, which are all critical parameters for acurate predictive modellening of the mechanical response, were not known prior to this investigation.

Fig. 1b an Fig. 2 show the results of a preliminary analysis from one of the investigated samples. A very important, and unexpected, result that became evident from the 3D micro-tomography data was the presence of micro-voids associated with the centerline sigma precipitates. From the preliminary analysis it appears that every sigma precipitate is associated with micro-voids, and sometimes these voids form entire networks connecting the precipitates (Fig. 1b). The precense of 3D micro-voids is an excellent example of how 2D inspections sometimes fail to give a reprensentative description of the the real 3D microstructure.

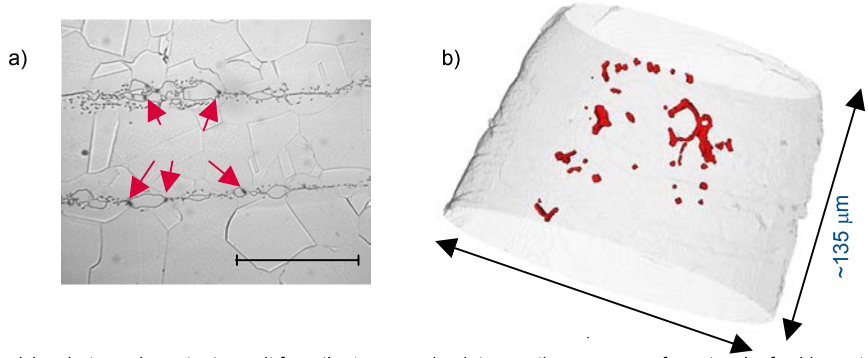

Fig. 1. A surprising, but very important, result from the tomography data was the presence of a network of voids contained in the centerline layer. In 2D inspections they appeared as dark features associated with the coarse sigma precipitates (left figure) but were thought to originate from pull outs of the brittle sigma phase during polishing. The 3D tomography study did, however, reveal that these dark features were 3D micro-voids sometimes constituting whole networks (right figure).

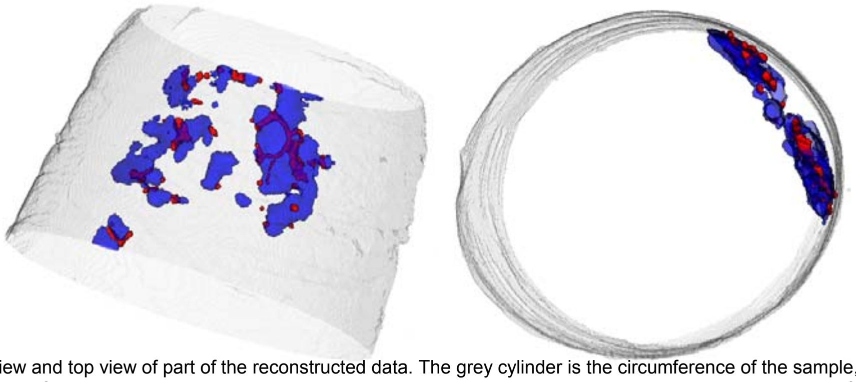

Fig. 2. Side view and top view of part of the reconstructed data. The grey cylinder is the circumference of the sample, the red part is the voids as shown in figure 1 and the blue part is the coarse sigma precipitates. The experimental set-up did not allow for detection of the very fine sigma precipitates (~1 μm). An important observation is that the sigma precipitates always seems to be associated with a void – or visa versa.

From a technological point of view these new findings are of critical improtance. Large efforts as be carried out to analyse for sigma (often non-destructively) in commercial/production scenarios, because of its anticipated detrimental affect on the properties of the alloy. However, if the voids are so associated with sigma precipitates in 3D, as the preliminary results show, then two crucial questions arises: (1) is it the voids (not the sigma per se) that cause more of a problem, and (2) do the voids form sigma or vise versa? And thus how do we develop schemes to avoid both.

These questions are currently being adressed by supplemtary investigation of selected parts of the reconstructed volumes by dual beam FIB/EBSD analysis at NRL, adding information on crystal orientation relationships between precipitates and the surrounding matrix grains, and by image-based finite element analysis of the mechanical properties of the alloy using the tomography data as input.

In summary we have fulfilled the original aim of characterizing the 3D morphology, spatial distribution and connectivity of sigma phase precipitates in an super austenitic stainless steel. In addition we have revealed the presence of a substaintial network of micro-voids which appears to be assiciated with the coarse sigma precipitates. A result which is of crucial technological importance for the application of the AL6XN stainless steel alloy.

Finally, it should be mentioned that preliminary tests on two-phase titanium alloys (not specified in the current proposal) conducted at the very last part of the beamtime showed very promising results that will be used for the planning of future research proposals.