DOI: 10.1017/S1431927605509462
Microsc Microanal 11(Suppl 2), 2005

Three-dimensional Analysis of Creep Voids

Azmi Abdul Wahab, Milo V. Kral*

*Department of Mechanical Engineering, University of Canterbury, Private Bag 4800, Christchurch, New Zealand.

In petrochemical plants, the production of hydrogen from natural gas is carried out in hydrogen reformers where the following reactions occur in the presence of a nickel catalyst:

$$CH_4 + H_2O \rightarrow CO + 3H_2 \qquad CO + H_2O \rightarrow CO_2 + H_2$$

Reformers tubes endure operating temperatures of more than 900ºC at stresses less than 20 MPa, resulting in relatively low creep rates (8×10^{-11} s^{-1}). Reformer tubes are typically expected to sustain service for 100,000 hours (11.4 years) with diametral strain not exceeding 3%.

Several ex-service and laboratory creep-tested hydrogen reformer tubes have been studied in the present work. The material characterized in the Figures below was in service at approximately 940°C and 2 MPa internal pressure for 60,000 hours (6.8 years). Characterization of the sample using electron backscatter diffraction (EBSD) in scanning electron microscopy (SEM) and selected area diffraction (SAD) in transmission electron microscopy (TEM) revealed Cr-rich $M_{23}C_6$ and NbC precipitates at austenite matrix grain boundaries as well as intragranular $M_{23}C_6$ and TiC. Successive polishing and etching steps were performed to obtain serial section images at 0.5 µm depth increments for sixty-five layers [1]. These images were first aligned using NIH ImageJ 1.34 software. In addition, Research Systems Inc. IDL 6.1 was utilized to generate 3D reconstructions from the image stacks, as shown in Fig. 1. Characteristics of the voids such as size, location, and surrounding precipitates were determined from 3D data sets and the results are summarized in the graphs of Fig. 2. Observations in 3D show that voids are not uniform in size, shape or location distribution. Most of the voids are not spherical and voids predominantly occur at grain edges and corners. In addition, all the voids observed were in contact with the Cr-rich $M_{23}C_6$ precipitates somewhere along their perimeter.

After every tenth section, the areas of interests in the sample were subjected to EBSD scans where EBSD patterns were recorded in spot mode for specific locations of the matrix and precipitates surrounding the creep voids. The orientation relationship (OR) between two adjacent locations were calculated from the EBSD data and the results for OR between $M_{23}C_6$ and austenite are shown on a standard stereographic triangle in Fig. 3. EBSD results that showed less than 20% of the $M_{23}C_6$ precipitates adjacent to these voids show a near cube-cube orientation relationship (OR) with the austenite matrix on one side of the boundary, whereas previous work indicates that 50% should obtain a cube-cube OR. This suggests a possible preference for void nucleation at interphase boundaries with an 'irrational' matrix:precipitate OR. Further work on materials at earlier stages of creep is in progress. [3]

[1] M. V. Kral and G. Spanos, Acta Metall. 47 (1999) 711-724.
[2] A. D. King and T. Bell, Metall. Trans. 6A (1975) 1419-1429.
[3] This work was supported by grants from Methanex (NZ) Ltd. and Schmidt & Clemens (Spain).

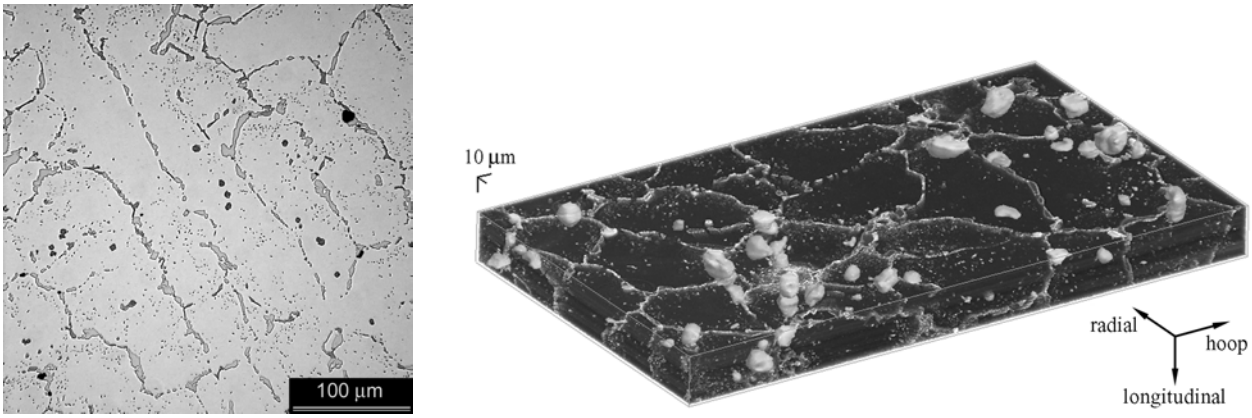

FIG. 1: A typical microstructure (left) and 3D reconstruction of a 403×204×32 µm volume (right) with austenite shown as black and creep voids and precipitates shown in shades of grey.

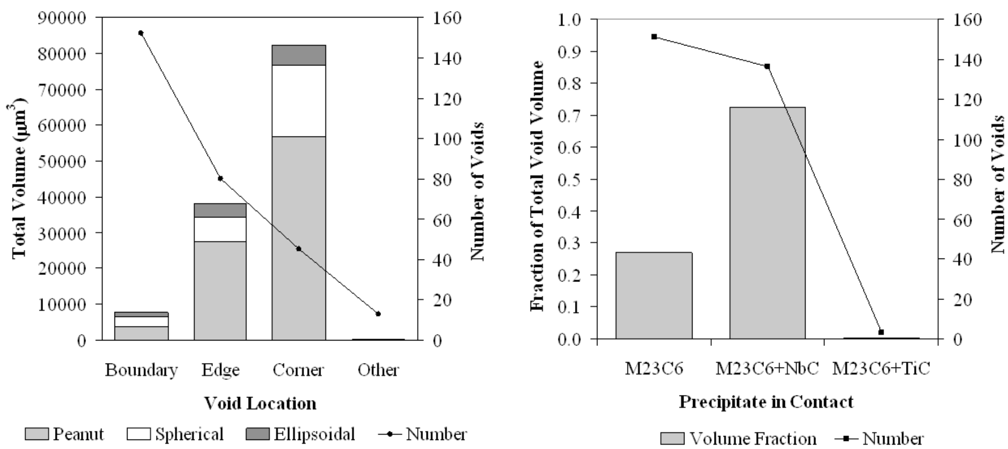

FIG. 2: A summary of 3D measurements of creep voids, (left) relationship between size, number, location, and shape and (right) correlation between volume fraction, number, and precipitate in void contact.

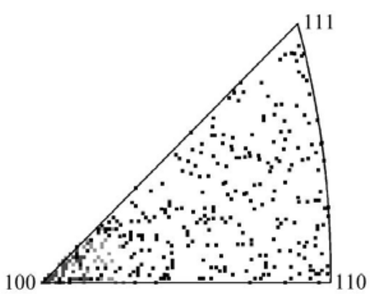

FIG. 3: Stereographic triangle showing the orientation relationship between austenite and grain boundary $M_{23}C_6$ in contact with creep voids.

https://doi.org/10.1017/S1431927605509462 Published online by Cambridge University Press