Correlative Tomography for micro- and nano- scale defects reduction analysis in Additive Manufactured healable aluminium alloy

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Abstract

Designing self-healing metals is challenging because it requires high temperature conditions which can trigger the diffusion and/or local melting process of the healing agent. Proper understanding of the healing mechanism, followed by the healing treatment optimisation depends on a successful microstructural analysis performed at the exact location of a damage. Therefore, a multiresolution and multimodal imaging approach with a spatial resolution from micro- to nano- scale is required to evidence the microstructure healing efficiency.

Keywords: Correlative Tomography, L-PBF, Aluminium, Self-healing alloys

1 Introduction

Aluminium alloys are widely used in aerospace and aeronautic industries because of their excellent strength-to-weight ratio. In these applications, overloads can quickly lead to a damage of the functional part which requires its immediate replacement. In order to increase the part's lifetime, a solution would be to use a material able to heal its damage and restore its continuity. The most advanced man-made self-healing materials are polymers. They are composed of encapsulated healing agents which are released when a crack propagates, leading to the crack closure [1]. Designing self-healing metals is more challenging because the damage is usually healed via solid-state diffusion or by melting of the eutectic phase and its migration to the defects, which cannot be triggered at room temperature. It requires a heat treatment to trigger the migration of the healing agents and allow the healing of these cracks and/or cavities [2]. However, the damage and its healing are hard to quantify using only surface observations. Additionally, due to a multiscale distribution of the microstructure and damage size, a multiresolution and multimodal imaging approach with a spatial resolution from micro- to nano- scale is required to evidence the microstructure healing efficiency. Correlative tomography (CMT) [3] is a concept/workflow of spatial registration in two and three dimensions (2D and 3D) of many imaging modalities - light microscopy (LM), electron/ion microscopy (EM, IM), X-Ray tomography, 2D/3D EBSD [2], EDS, Raman, etc.) - that allows various types of information, and at different length-scale, to be collected for the same region of interest (ROI). Therefore, the aim of this research was to develop a precise, robust and controlled multiscale analysis protocol for evaluation of the cracks and pores dimensions within a healable AlMg alloy, using three-dimensional (3D) correlative microscopy/tomography.

2 Materials and methods

Laser powder bed fusion (L-PBF) also called Selective Laser Melting (SLM) was used to manufacture a healable aluminium alloy. After the additive manufacturing process, the parts contained defects such as porosity and cracking. Therefore, a heat treatment was applied in order to restore the metallic continuity by melting the eutectic phase and its migration to these defects.

Applied 3D data collection strategy required a non-destructive testing of a larger volume, followed by a high-resolution image and materials composition analysis using the scanning electron microscope. In this contribution, we utilized a non-destructive character of X-ray based imaging linked with high-resolution 3D volume analysis within PFIB-SEM via Correlative Spectroscopy and the Microscopy (CoSMic) module, available within Avizo software (Themo Fisher Scientific), designed for advanced correlative data acquisition and processing [4].

A whole volume of a sample with dimensions 1 mm x 1 mm x 10 mm has been scanned using µCT lab-based system (TESCAN XRE HR, Belgium) with voxel size of 2.5µm, in order to identify a sub-volume region for further investigation with higher resolution (0.6 µm voxel size) scan. Additionally, a pre-defined sample section has been investigated using TOMCAT beamline at Swiss Light Source SLS facility of Paul Scherrer Institute, providing high quality 3D data with 0.65 µm voxel size. After inspection of high resolution µCT data, a 100 μm^3 volume region of interest (ROI) has been defined at a location indicating a characteristic defect.

The specific ROI µCT data and sample surface SEM images have been co-registered in order to define the specific ROI coordinates within µCT images and to be traced further with FIB-SEM. Automated serial sectioning tomography (SST) has been launched directly from the CoSMic tool and performed at the specific sample location using the Xe Plasma FIB-SEM microscope (Themo Fisher Helios Hydra DualBeam), SST SEM back-scattered electron images with 60 nm voxel size (Figure 1).

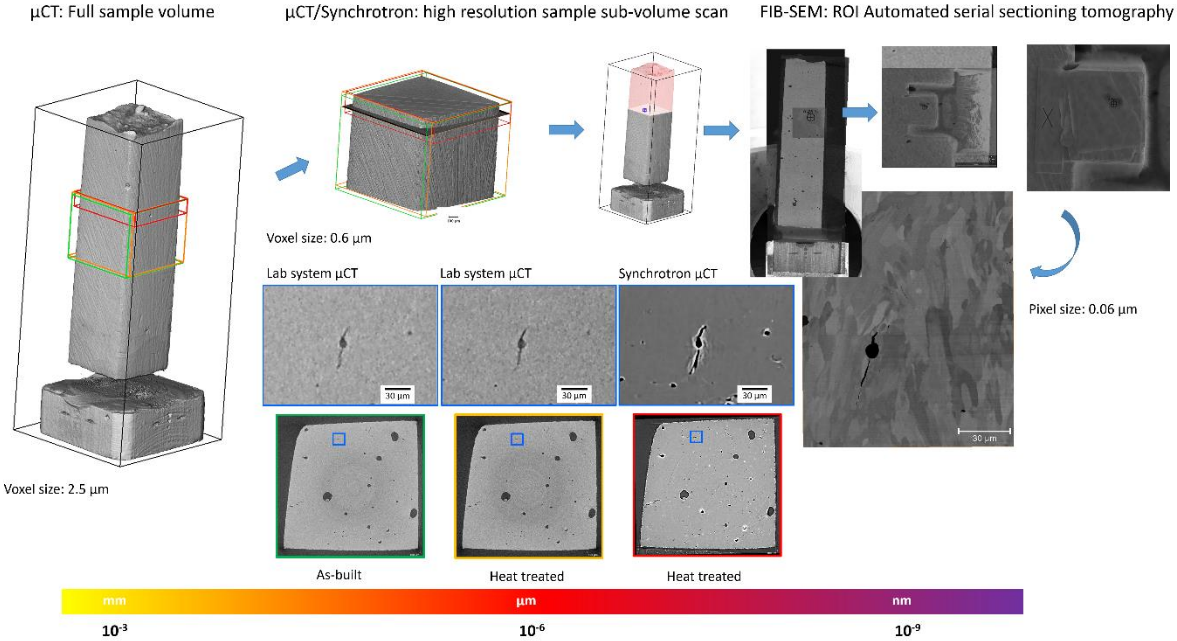

Figure 1: Correlative microscopy approach used in the current study.

3 Results and discussion

Multiscale data from FIB-SEM study were combined with µCT results using the correlative analysis within Avizo platform. A critical defect type of a crack originating from the internal porosity has been traced with multi-resolution tomography approach (Figure 1) with precision of 5 µm. High quality synchrotron tomography results revealed presence of micro-pores in surrounding of the crack origin. µCT data guided applications of PFIB-SEM system in the workflow, allowed in excess hundreds of microns size cross-sections in all dimensions, and SEM recording of nano- sized microstructural features at the identified ROI. The results of the eutectic phase migration to the defects, thus, the microstructural damage healing efficiency have been evidenced thanks to post-tomography correlation with PFIB-SEM nano-scale 3D information. These preliminary results indicate that the correlative microscopy approach for acquisition of the serial multi-resolution tomography data correlated with FIB-SEM tomography allow for precise location and characterisation of the critical defects. Furthermore, investing as built sample with the multiscale tomography approach followed by extended correlative imaging of the same location at the *ex-situ* or ideally *in-situ* heat-treated sample, can lead to better understanding and quantification of the healing mechanism.

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