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Correlative Microscopy-assisted Investigation of AM-build Healable Al-Mg Alloy

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INTRODUCTION

Aluminium alloys are frequently used in aerospace and defence vehicles for building load bearing engineering structures, manufactured with complex 3D shape allowing for weight minimization [1] and lower fuel consumption. Laser Powder bed fusion (L-PBF) allows building such complex 3D parts. However, common Al alloys processed by L-PBF, like 4xxx series Al-Si alloys, have too low strength compared to the best wrought alloys used in these industries (e.g. 2xxx and 7xxx Al alloys). Recently, 3D printable Al-Mg-Sc alloys (low Mg content, ~4 wt%) have been developed to reach minimum required high strength (~500MPa) [2,3]. However, these new high strength L-PBF Al alloys still exhibit low ductility and fatigue resistance, compared to wrought Al alloys, due to excessive porosity and cracks [4,5]. A new paradigm of self-healable materials [6,7] has the potential to significantly improve the durability of parts. Healing cracks in the range of millimetres, would be particularly advantageous in applications where part replacement is difficult or impossible, for parts subjected to interrupted service or requiring high reliability or for fast manufacturing using less optimized parameters (i.e. with even more porosities) [8].

Self-healing metallic alloys require a stimulus, e.g., high temperature and pressure conditions, to trigger the atomic diffusion (nano-scale) and/or micro-scale local melting and flow of a healing agent (eutectic phase), which can thus migrate, fill the damage sites and solidify (Figure 1).

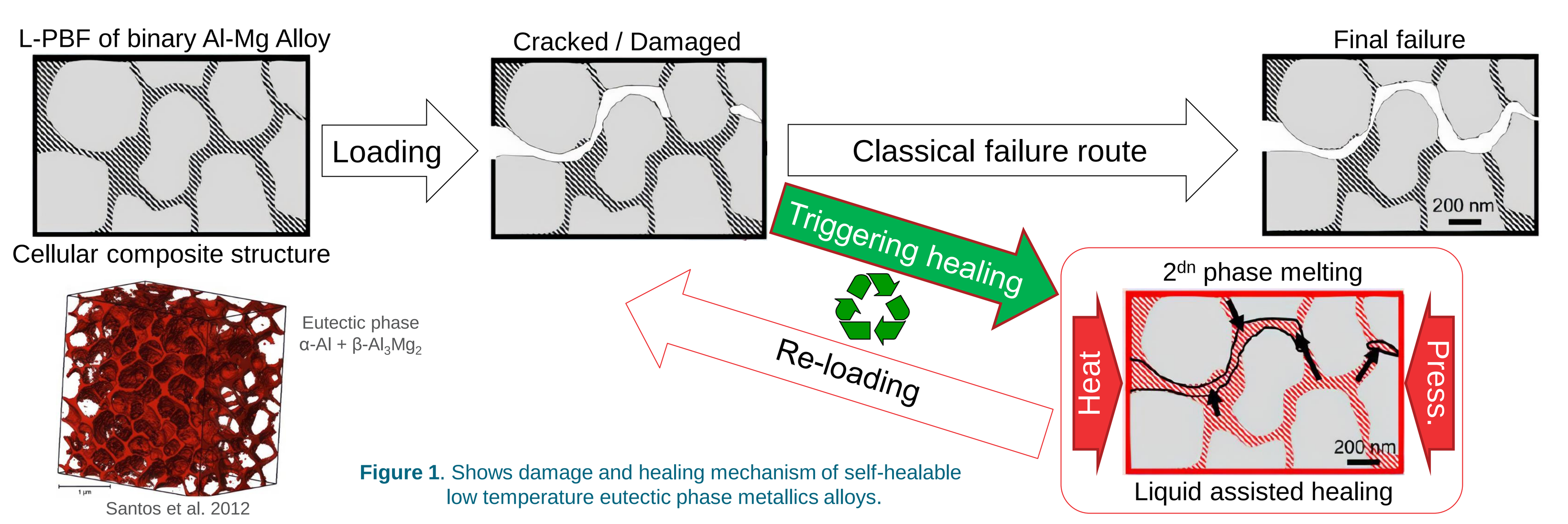

Figure 1. Shows damage and healing mechanism of self-healable low temperature eutectic phase metallics alloys.

The healing treatments optimization and ultimate understanding of healing mechanisms depends on accurate multi-scale and multi-modal microstructural analyses performed at the exact location of damage [6]. Therefore, a correlative tomography and electron microscopy imaging [9, 10] are required to evidence the microstructure healing efficiency.

In this investigation, a multi-scale correlative tomography and multi-modal electron microscopy were applied to investigate healing mechanism within Al-Mg alloy (Patent pending).

METHODS

In situ heat treatment of L-PBF manufactured sample was performed at ID16B beamline at the European Synchrotron Radiation Facility (ESRF), combined with the 4D X-ray nano-tomography imaging before and after the healing process [11]. Full sample volume tomograms (voxel: 200 nm) were acquired at the critical damage locations. These served for localization of the high-resolution ROI scan data (voxel: 35 nm). Healed sample, mounted on the cross-platform correlative holder containing unique fiducial markers, was finally scanned (voxel: 2 µm) with lab CT system Waygate Nanotom|M, for coordinates alignment between µCT and DualBeam imaging systems. Next, the healing microstructure was further investigated with the correlative microscopy imaging approach.

Precisely allocated, based on the nano-tomography ESRF data, sample sub-volume containing the healed damage was further investigated using Helios Hydra Plasma FIB-SEM and Talos F200X TEM both combined with the EDX elemental material composition mapping. Automated Xe PFIB serial sectioning tomography (SST) and acquisition of SEM back-scattered electron images with 60 nm voxel size, was launched in location defined by the CoSMic [10] correlative data registration tool within Avizo software. Subsequently, large 50 µm wide TEM lamella for nano-scale study of healed damage was resected and prepared to electron transparency using the Xe focused beam with the final polishing using 500 eV, and imaged using the TEM. Finally, all the data were post-processed and co-registered in Avizo 3D data processing and analyses package.

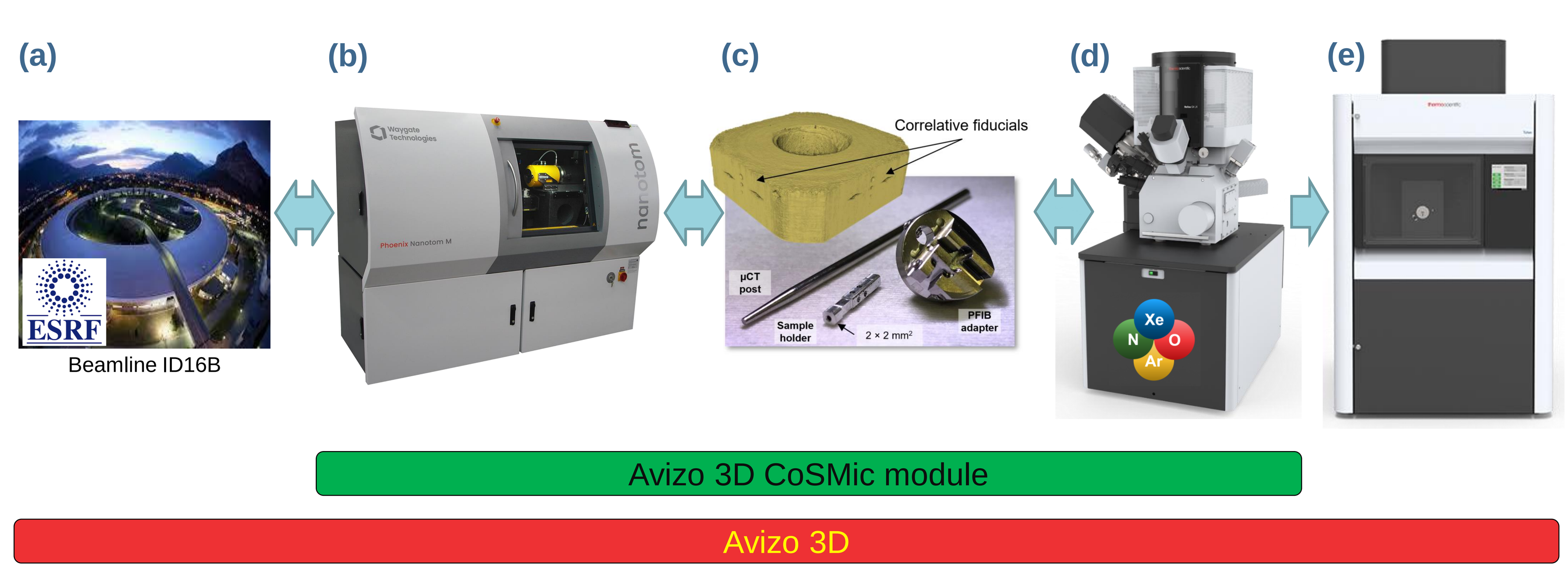

Figure 2. The Correlative Microscopy research equipment used in the current investigation. (a) Nano-analysis beamline ID16B at the ESRF, (b) Waygate Nanotom M micro-CT, (c) the cross-platform correlative holder kit, (c) Hydra PFIB-SEM, (d) Talos 200X CFEG TEM

RESULTS

The results of correlative microscopy studies are summarized in Figure 3.

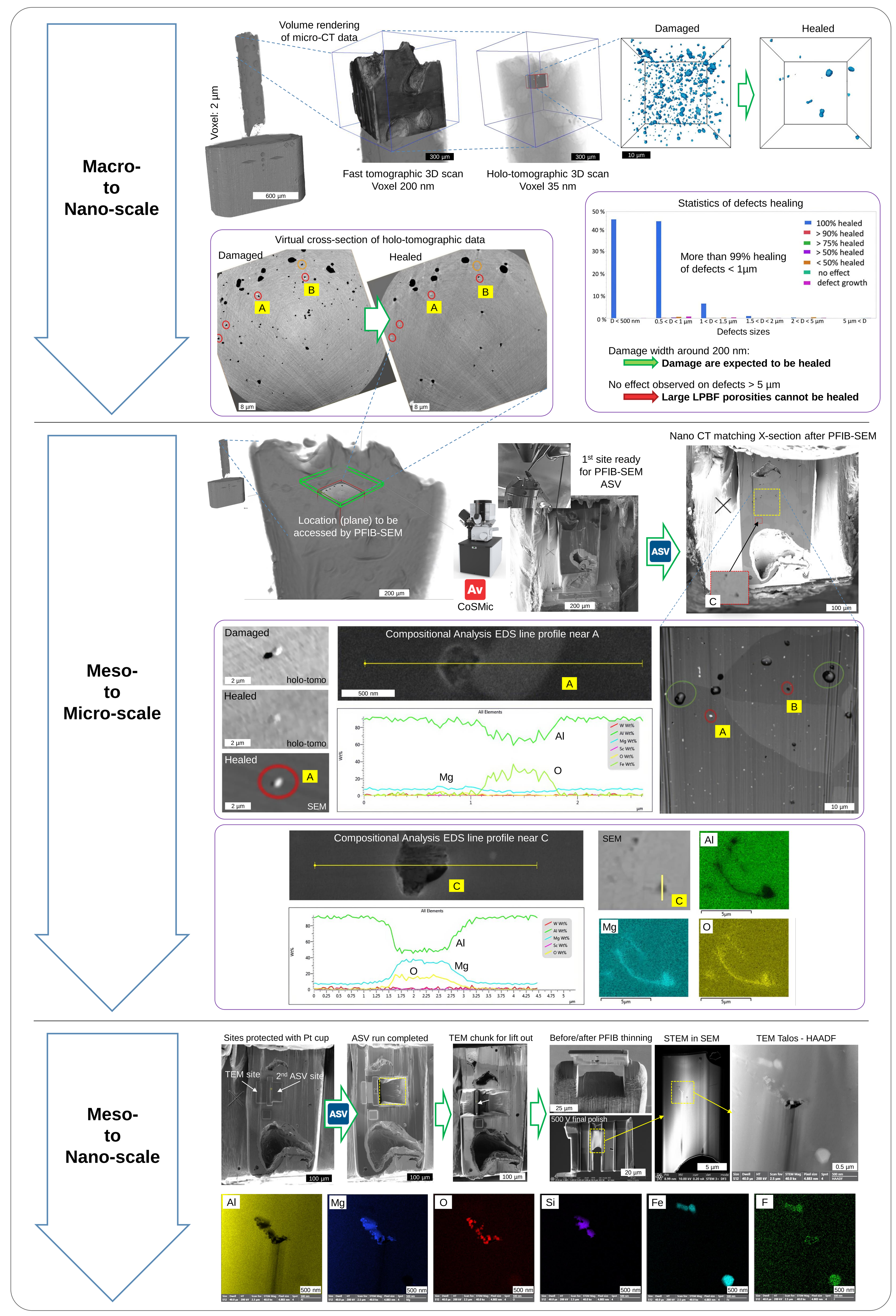

Figure 3. Results of correlative microscopy studies applied to the healable aluminium alloy

CONCLUSIONS

The heat treatments aimed to restore the metallic continuity by melting the low-melting point healing agent phase and its migration to the defects introduced by tensile loading [6]. 4D nano-tomography highlighted the filling of the damage sites at different temperatures, allowing to optimize the healing temperature. A statistical tracking analysis used to determine the fraction and size of the healed damage sites, confirmed effectiveness of applied healing process, by revealing significant reduction of micro-pores and cracks. Applications of PFIB-SEM system and TEM in the workflow, guided by multiscale data from synchrotron and lab-based µCT, allowed reaching the exact location - with precision of ~ 10 µm - and characterization of the 100 µm³ ROI sample sub-volume, containing the critical defects. These results confirmed the healing potential of this Al-Mg alloy. We showed that the correlative tomography and microscopy approach allow for precise location and characterization of the critical defects, leading to better understanding and of the healing mechanism.

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