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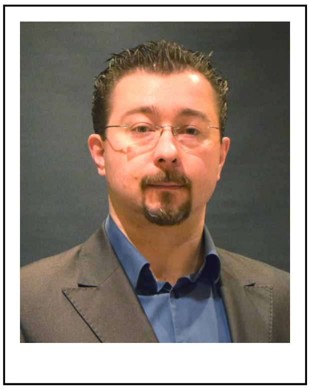

CORRELATIVE AND MULTI-SCALE ANALYSES OF ADVANCED MATERIALS

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1. ABSTRACT

Biological materials exhibit intricate three-dimensional (3D) hierarchical microstructures, resulting in unique combinations of anisotropic mechanical properties that often surpass those of synthetic materials. To develop synthetic alternatives for damaged or diseased organs, as well as for non-biological applications that leverage the exceptional properties of these materials, researchers must understand the structure of the materials they fabricate across various length scales, ranging from millimetres to Ångstroms. Advanced materials, including fibre-reinforced composites, ceramic or metal alloys, and additively manufactured components, also can possess hierarchical microstructures and complex anisotropic mechanical properties. Consequently, comprehensive investigations across different length scales are necessary for these materials as well.

This contribution presents a multi-modal and multi-scale workflow that employs micro X-ray computed tomography (μCT), focussed ion beam/scanning electron microscopy (FIB/SEM), and analytical techniques like electron backscattered diffraction (EBSD), energy-dispersive X-ray spectroscopy (EDS), and transmission electron microscopy (TEM). By combining the spatially correlated results obtained from these techniques, the workflow enables crucial 2D/3D characterisation that is vital for understanding the structures of these materials and enhancing the associated manufacturing processes. This article specifically focusses on (*i*) correlative and multiscale analyses of a fatigued and fractured Ti-6Al-4V titanium alloy medical cellular cubic scaffold manufactured using selective laser melting, and (*ii*) directly laser deposited Inconel IN718 alloy for aeroengines.

2. INTRODUCTION

Correlative and multi-scale analyses (CMA) encompass a wide range of imaging and analytical techniques, including optical microscopy (OM), micro X-ray computed tomography (μCT), focussed ion beam/scanning electron microscopy (FIB/SEM), electron backscattered diffraction (EBSD), energy-/wavelength-dispersive X-ray spectrometry (EDS/WDS), X-ray photoelectron spectroscopy (XPS), Raman spectroscopy, transmission electron microscopy (TEM), atom probe tomography (APT), and more. Figure 1 illustrates the various experimental techniques and the corresponding length scales they can explore [1].

A specific subset of CMA is correlative X-ray computed tomography and electron microscopy (C-XCT-EM) [2], which is an innovative workflow that integrates advanced imaging and analytical techniques to provide valuable insights for scientist and engineers involved in product design, material development, and manufacturing processes. This workflow combines data from various techniques such as OM, μCT [3], EBSD, EDS, serial sectioning tomography (SST) with plasma-focussed ion beam/scanning electron microscopy (PFIB/SEM) [3], and scanning/transmission electron microscopy (S/TEM). By integrating these techniques,

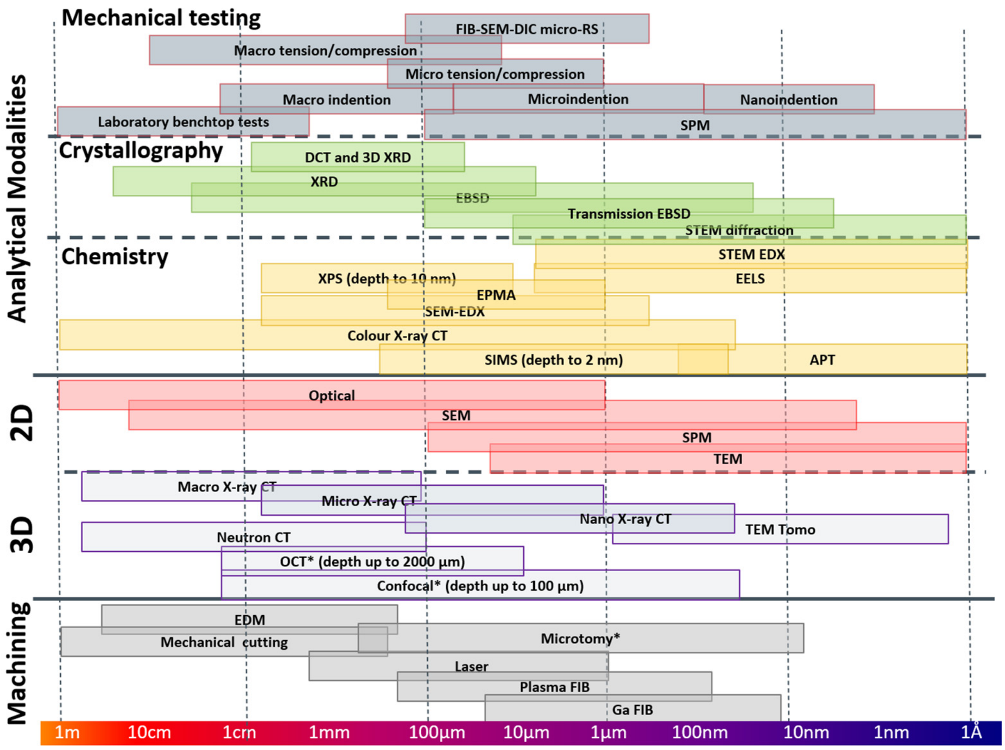

Figure 1. Graphics illustrates the various experimental techniques and the corresponding length scales they can explore. After [1].

C-XCT-EM enables the analysis of three-dimensional characteristics across length scales ranging from millimetres to Ångstroms. These characteristics include surface roughness, porosity, composition, residual stress state [4], and more. Figure 2 illustrates the length scales and resolutions that can be accessed for correlative and multimodal analyses using C-XCT-EM [5].

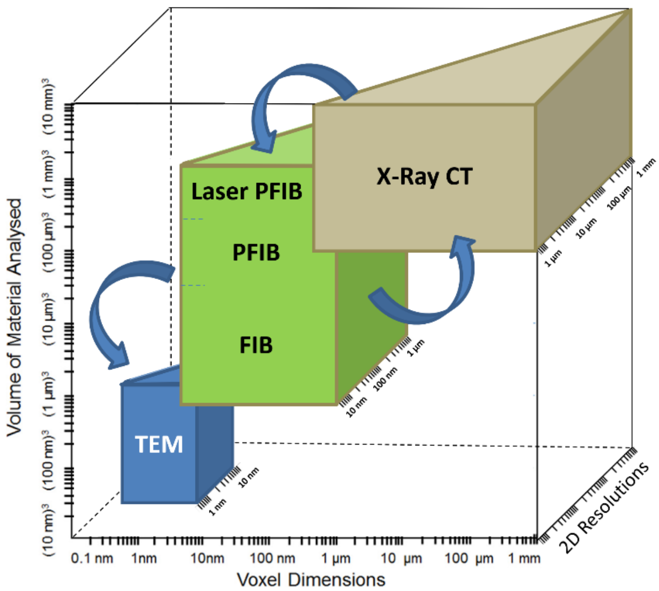

Figure 2. The graphic illustrates commonly used multi-scale and multi-modal imaging methods in materials science. The DualBeam (FIB, PFIB) and TriBeam (Laser PFIB) platforms are utilised for both sample preparation and data collection. The arrows indicate potential sample transfers and the co-registration of system stage coordinates. After [5].

To showcase the capabilities of this workflow, this article focuses on correlative and multiscale analyses of a fatigued and fractured Ti-6Al-4V titanium alloy medical cellular cubic scaffold manufactured using selective laser melting, and directly laser deposited Inconel IN718 alloy for aeroengines.

3. *Ti-6Al-4V TITANIUM ALLOY MEDICAL CELLULAR SCAFFOLD*

Open-porous Ti64 scaffolds or foams [6] are highly desirable in the field of biomedical implants due to their ability to reduce stress-shielding and enhance osseo-integration [7]. To ensure compatibility with a patient's bone, the mechanical properties and geometric characteristics of implant scaffolds are customised using computer-aided design (CAD) and finite-element analysis (FEA). Subsequently, CAD-FEA designed implants are manufactured using various additive manufacturing (AM) techniques, such as selective laser melting of Ti64 (SLM) [8]. However, certain challenges still need to be addressed, including detrimental residual stress states, discrepancies between the mechanical properties and FEA predictions, and inconsistencies between the CAD-designed and manufactured geometries. Ti-alloys, in general, exhibit high notch sensitivity [9]. The presence of surface roughness, notches, and material defects are inherent to any AM process and can act as stress concentrators and initiators of cracks, significantly impacting the fatigue resistance of an AM build [10].

In a previous study [8], the fatigue and quasi-static test results of a hot isostatic pressed (HIP) Ti64 scaffold were compared with finite element (FE) calculations based on two different geometries: the as-designed geometry of a regular cubic cellular scaffold and the as-built geometry reconstructed from µCT scans. It was observed that both the elastic modulus and the fatigue resistance exhibited a strong correlation with the number and severity of defects. Furthermore, the study demonstrated that predictions of the mechanical properties based solely on the as-designed geometry were found to be inaccurate [8].

The present study focuses on the investigation of a fatigued and fractured HIPed SLM Ti64 cubic cellular scaffold [8] using the C-XCT-EM framework (Fig. 1). The CMT workflow leverages the imaging, analytical, and metrological capabilities of various instruments, along with the cross-platform correlative holder kit. These instruments include helical µCT, PFIB/SEM, and laser PFIB/SEM, where three beams coincide at a single point. Additionally, other apparatus such as transmission electron microscopes are utilised. The correlative investigations are supported by MAPS-based [6] and AVIZO-based workflows [7]. Figure 3 shows diagram of the workflow used in this study [5].

Figure 4 presents a visual summary of the correlative study conducted. The fatigued and fractured scaffold undergoes µCT scanning to analyse the distribution of strut thicknesses and identify pores within the SLM build. A detailed Maps study reveals that these defects within the SLM build have a marginal effect on the fracture pattern observed

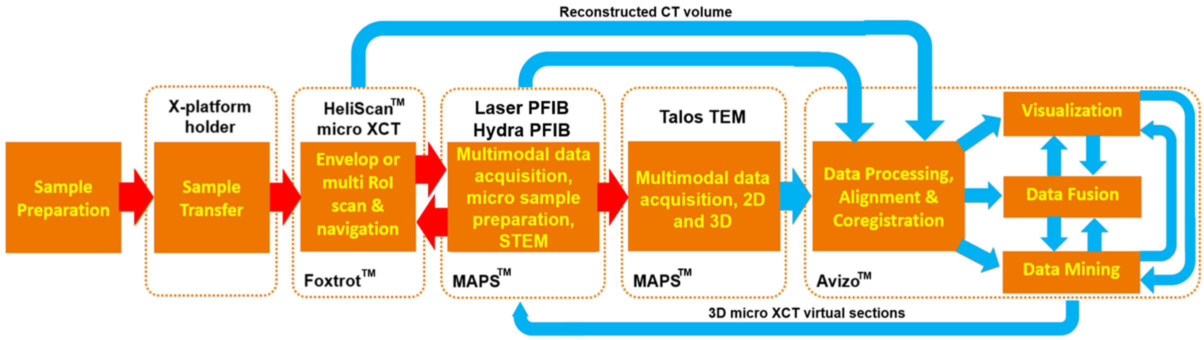

Figure 3. Diagram shows the C-XCT-EM workflow in material science. Red arrows indicate sample transfer, while blue arrows show data transfer. After [5].

(Figs. 4c and 4e). Notably, brittle, ductile, and mixed-mode fractures occur in locations resembling notches, which correspond to the smallest cross-sectional area of the struts (Figs 3d and 3e). These areas experience the highest fatigue stresses and strains. The type of fracture exhibited by each strut strongly depends on the crystallographic orientation of the α- and β-phases, with an increased presence of the brittle α-phase. These findings are further supported by a comprehensive study utilising auto slice and view serial sectioning tomography (Figs. 3g and 3h). Electron channelling contrast analysis reveals the presence of plastically deformed grains near the fractured surface. The microstructure of the bulk material exhibits residual stress and strain, as qualitatively indicated by variations in the electron channelling contrast, as previously measured [4].

Further investigations, including TEM examinations, are being conducted to provide more detailed insights into the fractured zones.

This study presents preliminary findings from a 3D C-XCT-EM investigation of a fatigued and fractured cellular scaffold commonly used in trabecular bone implants. The presented multi-scale imaging solutions, workflow, and cross-platform holder kit offer versatility for practical applications and contribute to a comprehensive understanding of additive manufacturing and related areas. These results provide valuable insights into this field and pave the way for further research and practical implementations.

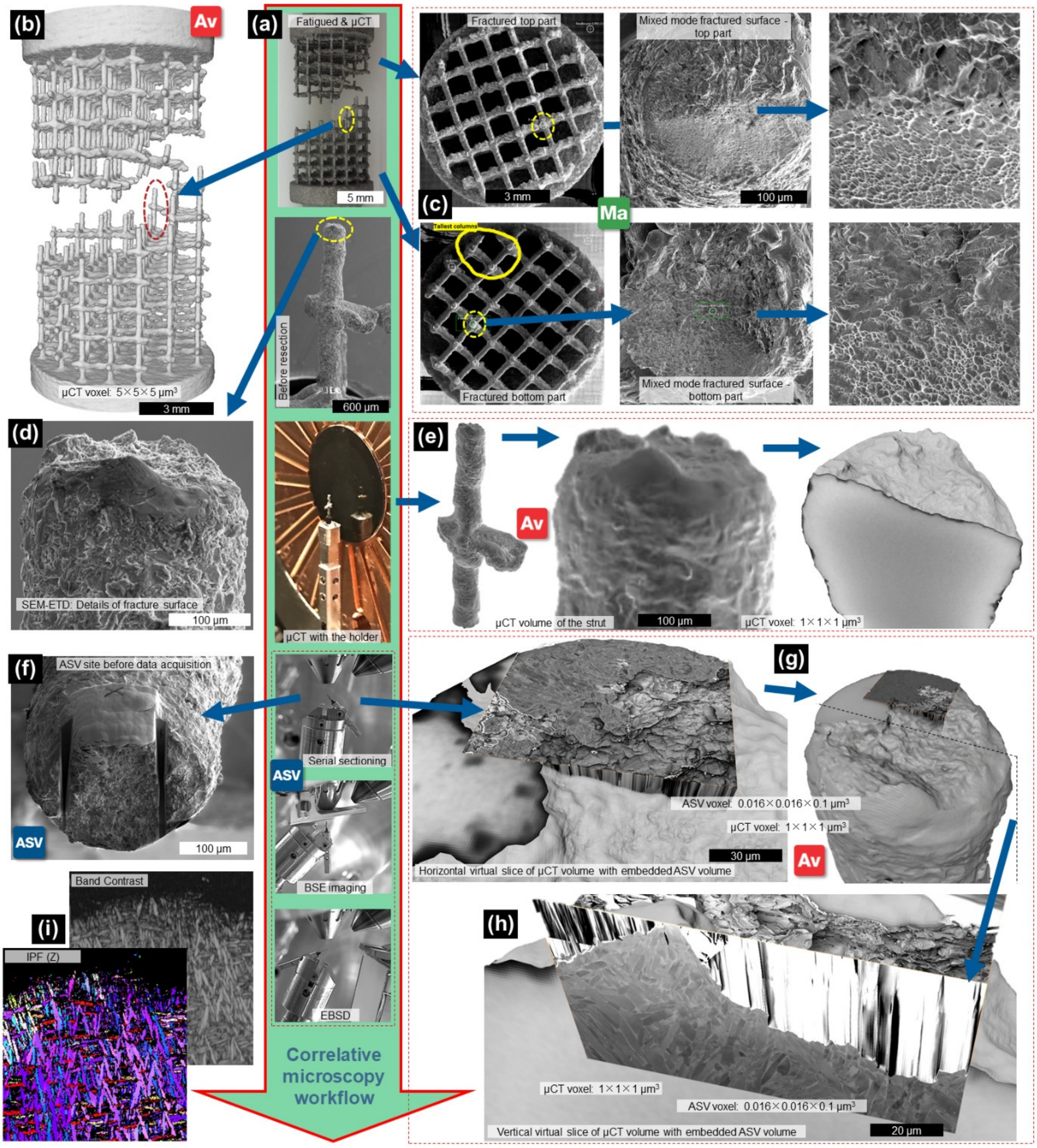

Figure 4. Shows post-fatigue investigation of Ti64 scaffolds by 3D correlative tomography using cross-platform correlative holder kit. a) Correlative microscopy workflows steps; b) µCT visualisation of fatigued and fractured scaffold; c) Fractography study using tiled high resolution SEM images in MAPS; d) SEM image of fractured strut selected for subsequent µCT scan; e) Visualisation of high resolution µCT data of PFIB resected strut; f) Site ready for SST using PFIB; g) and h) SST volume of fractured surface volume embedded in µCT volume; i) EBSD map collected after SST. After [5].

4. *INVESTIGATION OF CRACKING IN ADDITIVELY MANUFACTURED IN718*

The emergence of additive manufacturing (AM) methods [11], such as selective laser melting (SLM) of powder bed and blown powder laser deposition (BPLD), hold great promise in overcoming the limitations of conventional metallurgical processes, especially for small production volumes, complex part configurations, near net-shape dimensions, high-performance requirements, and part repair/cladding applications, such as those involving Inconel alloys. Inconel 718 (IN718) belongs to a family of nickel-based superalloys extensively used in aircraft turbines, jet engines, steam turbine power plants, and more, due to their exceptional resistance to high-temperature corrosion, fatigue, and creep [12]. The desired microstructure of IN718 components should comprise a γ solid supersaturated solution matrix (enriched in Ni, Cr, and Fe) predominantly strengthened by γ”-Ni_3Nb- and γ’-Ni_3(Al, Ti)-precipitates [12, 13]. However, the presence of Nb can lead to segregations and the formation of undesirable brittle phases, such as NbC, δ-Ni_3Nb, and Laves phases (Ni, Cr, Fe)(Nb, Mo, Ti), along with residual stresses that can adversely affect tensile ductility, fatigue, and creep properties [12, 13].

In this study, we employ C-XCT-EM to examine crack formation in BPLD IN718 coupons manufactured using a 5 kW laser power and feed rates of 750 and 1,250 mm/min. The C-XCT-EM analysis is facilitated by state-of-the-art equipment, including the HeliScan micro X-ray CT system, Helios Plasma FIB-SEM, and Talos STEM microscopes. Additionally, we utilise a unique instrumental environment that integrates inter-linked software, namely AUTO SLICE&VIEW 4, AVIZO 3D, and MAPS, to provide comprehensive data analysis and visualisation.

At the macroscale, we observed the presence of large millimetre-long open cracks near the base of the coupon and along the interface (Fig. 5a). These cracks tend to form along grain boundaries of the columnar/dendritic grains, as indicated by the EBSD map. Notably, the typical weld-like microstructure associated with deposition passes was not observed. Visualisation of the micro CT data reveals a network of cracks spanning across the component's thickness (Fig. 5b). Microscale analysis indicates that the cracks primarily develop in the regions enriched with Nb, which exhibit elements segregation and a network of Laves phases (Fig. 5c). Furthermore, the presence of porosity and a large 30 μm diameter pore within a grain was observed. At the nanoscale (Fig. 5d), we identified the presence of γ"-precipitates, aluminium oxide, titanium nitride, and niobium carbide. The combined capabilities of the hardware and software utilised in this study provided valuable 3D insights into the manufacturing defects that occur during BPLD.

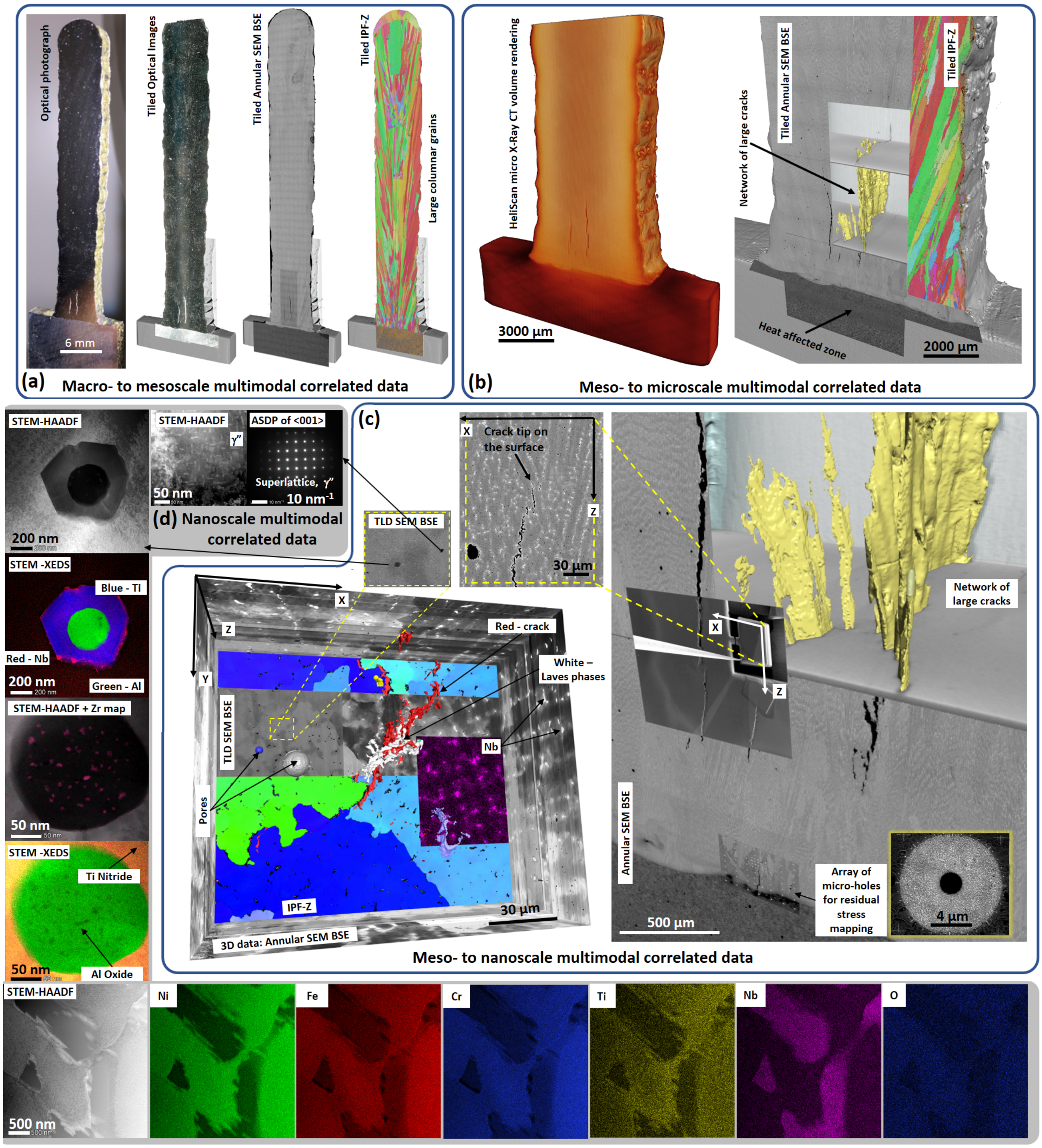

Figure 5. 3D multiscale and multimodal results (laser power 5 kW with feed 1,250 mm/min, powder feed rate 40 g/min, 84 layers with area 115×7 mm^2, 0.5 mm layer height offset). a) Investigation using tiling and stitching; OM 4.5 μm/pix, SEM 0.65 μm/pix, EBSD 10 μm/pix. b) μCT volume with co-registrated SEM and raw EBSD maps. c) On the right zoomed in region with cracks and location from where a large chunk (with a crack tip) was resected for further 3D PFIB serial sectioning study (100 nm^3 voxel); on the left visualisation of the crack tip region with co-registrated raw EBSD and EDS information. Note: SEM TLD BSE and annular BSE (CBS) signals were collected simultaneously. d) STEM study revealed γ'', aluminium oxide, titanium nitride, niobium carbide precipitates, Nb-rich Laves phases, and elements segregation. After [14].

5. CONCLUSION

In conclusion, correlative and multi-scale analyses (CMA) comprise a wide array of imaging and analytical techniques, offering versatile tools for investigating materials and structures at different length scales and imaging modalities. Within this framework, correlative X-ray computed tomography and electron microscopy (C-XCT-EM) stands out as an innovative workflow that integrates advanced imaging and analytical techniques, providing valuable insights for scientists and engineers involved in product design, material development, and manufacturing processes. By combining data from various techniques such as optical microscopy, micro X-ray computed tomography, electron backscattered diffraction, and transmission electron microscopy, C-XCT-EM facilitates the analysis of three-dimensional characteristics across a wide range of length scales, from millimetres to Ångstroms.

This paper has showcased the capabilities of the C-XCT-EM workflow through the examination of a fatigued and fractured Ti-6Al-4V titanium alloy medical cellular cubic scaffold manufactured using selective laser melting, as well as the investigation of directly laser-deposited Inconel IN718 alloy for aeroengines. These case studies demonstrate the potential of C-XCT-EM for correlative and multiscale analyses in different material systems, highlighting its relevance and applicability across various fields. The integration of multiple techniques and the ability to explore a wide range of length scales offer valuable insights into the structures and properties of materials, contributing to the development of improved manufacturing processes and materials for diverse applications.

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