Characterization of Quasi-continuous Reinforcement Network in the Selective Laser Melted Titanium Matrix Nanocomposite using Correlative FIB-SEM Tomography and STEM

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Microscopy and Microanalysis, **29** (Suppl 1), 2023, 1432–1433
https://doi.org/10.1093/micmic/ozad067.737

Microscopy AND Microanalysis

Proceedings

Characterization of Quasi-continuous Reinforcement Network in the Selective Laser Melted Titanium Matrix Nanocomposite using Correlative FIB-SEM Tomography and STEM

Dian Li[1], Sydney Fields[1], Xing Zhang[2], Rongpei Shi[3], Yiliang Liao[2], and Yufeng Zheng[1,*]

[1]*Department of Chemical and Materials Engineering, University of Nevada Reno, Reno, Nevada, United States*
[2]*Department of Industrial and Manufacturing Systems Engineering, Iowa State University, Ames, Iowa, United States*
[3]*School of Materials Science and Engineering, Harbin Institute of Technology, Shenzhen, Guangdong, China*

*Corresponding author: yufengz@unr.edu

Due to high specific strength, great chemical resistance, good wear resistance and excellent high-temperature durability, titanium matrix nanocomposites (TMNCs) reinforced by Ti_5Si_3 nanoparticles have shown great potential in the field of aerospace and automotive industries [1]. Recently, TMNCs with so-called quasi-continuous network distribution of reinforcement have been developed via casting or sintering, showing a good combination of strength and ductility [2]. With the development of advanced characterization techniques, e.g., tomographic focused ion beam-scanning electron microscopy (FIB-SEM), the microstructure in three-dimension (3D) can be investigated in detail. For example, we have used the FIB-SEM to study the 3D morphology of grain boundary precipitates in the titanium alloy [3] and evaluate the 3D dual-phase cellular structure and defects in the additively manufactured Ti-48Al-2Cr-2Nb alloy [4]. In our recent work, we have reported, for *the first time*, a nanoparticle-mediated approach to in-situ fabricate nano-Ti_5Si_3 reinforced TMNC coating through selective laser melting (SLM) of Ti powders and a minor amount of SiC nanoparticles, showing ultrahigh surface microhardness of 706 VHN [5]. This presentation will introduce our latest study of 3D distribution of quasi-continuous reinforcement network formed in the SLMed Ti+ SiC using correlative FIB-SEM tomography and scanning transmission electron microscopy (STEM).

In the first part of this work, the 2D and 3D microstructure in the SLMed Ti+SiC were investigated using SEM backscattered electron (BSE) imaging, electron backscatter diffraction (EBSD) and FIB-SEM tomography using ThermoFisher Scientific Scios 2 FIB/SEM. Bimodal globular α and plate-shape α precipitates, carbon-rich particles, and network structure around bimodal α precipitates were characterized in the SEM BSE imaging (Fig. 1(a)). FIB serial sectioning was performed in the volume of 9 μm × 4.5 μm × 9 μm with 10 nm step size along the marked z direction, shown in Fig. 1(b). Feature-by-feature 3D microstructure was reconstructed using MIPAR™ image analysis software, showing the disconnected nature of the network structure in Fig. 1(c).

The second part of the present work focuses on identifying the composition of the quasi-continuous network structure using FEI Talos F200s scanning transmission electron microscope. STEM-high angle annular dark field (STEM-HAADF) image in Fig. 2(a) reveals a similar microstructure observed in SEM BSE imaging, including the bimodal α microstructure, carbon-rich precipitate and network structure surrounding the bimodal α precipitates. STEM X-ray energy dispersive spectroscopy (XEDS) mappings show clearly the Ti depletion and Si enrichment in the network structure in Fig. 2(b-c), indicating that the quasi-continuous network structure belongs to Ti_5Si_3 reinforcement [6].

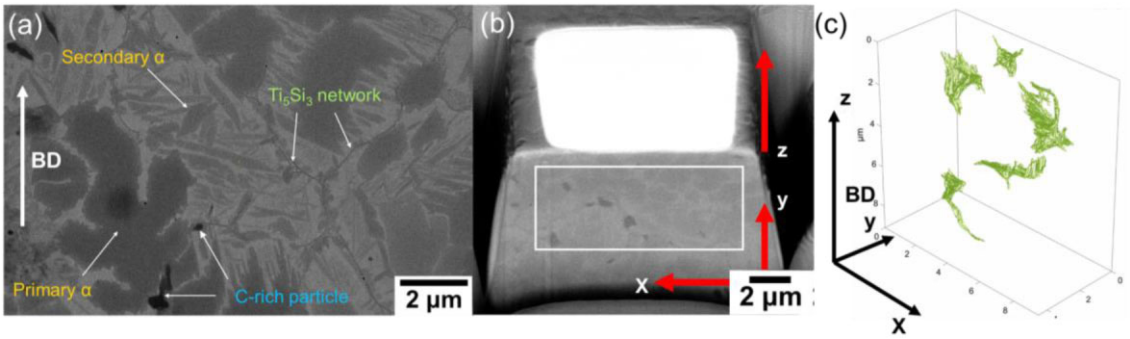

Fig. 1. 2D and 3D characterization of microstructure in SLMed Ti+SiC: (a) SEM BSE image showing bimodal α microstructure, carbon-rich particles, and network structure; (b) SEM image showing the volume for FIB-SEM tomography analysis; (c) 3D reconstruction of quasi-continuous network structure.

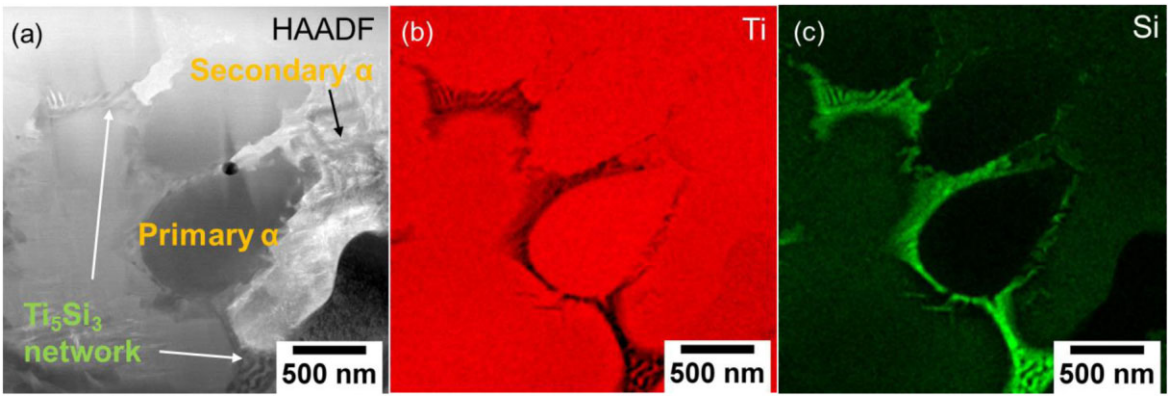

Fig. 2. STEM-HAADF imaging and XEDS mappings showing the quasi-continuous Ti_5Si_3 network structure in the SLMed Ti+ SiC: (a) HAADF image showing bimodal α microstructure, carbon-rich particle and quasi-continuous Ti_5Si_3 network; (b-c) XEDS mapping showing the distribution of Ti, and Si in the quasi-continuous Ti_5Si_3 network.

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6. DL and YZ acknowledge the funding support from the National Science Foundation, grant CMMI-2122272.