

## Understanding Nanoscale 4D Microstructural Evolution in Aluminum Alloys using Transmission X-Ray Microscopy (TXM)

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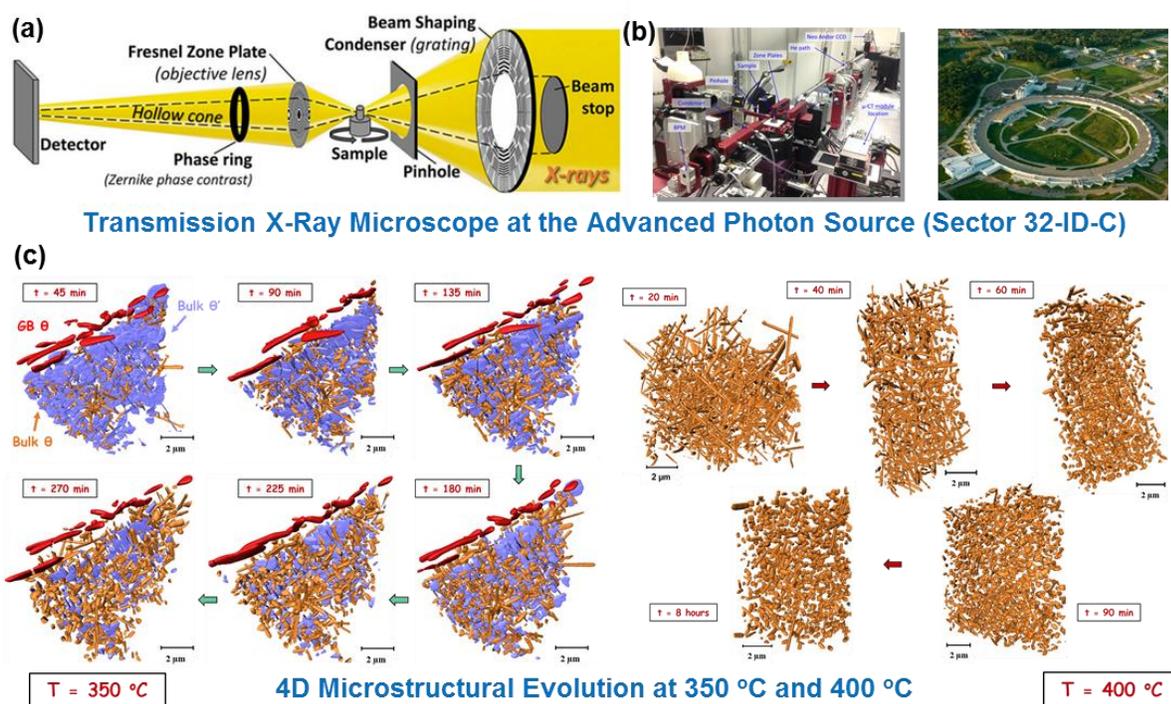
Precipitation-strengthened alloys have found uses in several structural applications and for us to better their performance and reliability, it is essential to have a thorough understanding of the microstructure and its effect on properties. Most of our understanding of these alloys date back to the mid-20th century and majorly rely on analyses carried out using transmission electron microscopes (TEMs), which are generally associated with shortcomings such as laborious destructive sample preparation, two-dimensional field of view and limited penetrability in case of metallic specimens. High temperature evolution studies in electron microscopes are also enormously influenced by surface diffusion and projected view of the specimen. We have been able to use Full-field Transmission X-Ray Microscopy (TXM) [1, 2] to overcome almost all of these shortcomings and have been successful in providing novel insights into nanoscale evolution occurring in the bulk of these alloys. Using miniaturized mechanical testing to complement this technique has enabled us to better predict the alloy's mechanical behavior. By coupling this with segmentation using Machine learning, it has also been possible to analyze ultra-large volumes of material at a very high spatial resolution.

Absorption full-field hard X-ray nano-computed tomography was performed on Al-4%Cu alloys using the new Transmission X-ray Microscope (TXM) of the Advanced Photon Source (APS) at sector 32-ID-C. Micropillars having a 30  $\mu\text{m}$  diameter and 50  $\mu\text{m}$  in height were fabricated at the tips of sharpened wires using a dual-beam focused ion beam (FIB) with a scanning electron microscope (Nova 200 NanoLab FEGSEM-FIB, FEI Co, Oregon). Imaging was performed using a monochromatic beam at 9.1 keV, just above the Cu K-edge to maximize contrast between the  $\text{Al}_2\text{Cu}$  ( $\theta'$  &  $\theta$ ) and Al phases. Fresnel zone plate lens with 60 nm outer most zone width was used as a microscope objective lens to magnify radiographs of the sample placed on a high accuracy air-bearing rotary stage. Tomopy, an open source Python based toolbox [3], was used to analyze synchrotron tomography data and perform 3D reconstructions. Subsequent 3D quantification as well as 3D visualization was also carried out in Avizo® Fire.

The morphology, distribution as well as the evolution behavior of  $\theta'$  and  $\theta$  precipitates in Al-Cu alloys was analyzed, revealing complex transformation reactions that were occurring in the alloy during aging at 350 °C. Most of these are nearly impossible to capture using other characterization techniques. 3D measurements of precipitate dimensions rendered a thorough understanding of the coarsening behavior of  $\theta$  at 400 °C, showing a transition from interface-controlled to volume diffusion controlled growth in thickness of  $\theta$ , as captured from the scaled precipitate size distribution curves. Correlation of 3D tomography data with EBSD allowed experimental estimation of the Orowan strengthening as well as quantification of the preferred 3D orientation of the  $\theta$  phase. Use of machine learning assisted segmentation enabled analysis of unprecedented volume of material, opening up several new possibilities for analysis of tomography data [4].

References:

- [1] C.S. Kaira, et al. Nanoscale Three-Dimensional Microstructural Characterization of an Sn-Rich Solder Alloy Using High-Resolution Transmission X-Ray Microscopy (TXM). *Microscopy and Microanalysis*, 1-6, 2016.
- [2] De Andrade, V. et al. Nanoscale 3D imaging at the Advanced Photon Source. *SPIE Newsroom*, 2016.
- [3] Gürsoy, D., De Carlo, F., Xiao, X. & Jacobsen, C, TomoPy: A framework for the analysis of synchrotron tomographic data. *J. Synchrotron Radiation*, 21, 1188–93, 2014.
- [4] The authors are grateful for financial support from the Army Research Office (ARO) under Contract No. W911NF1410550 (Dr. David Stepp, Program Manager).



**Figure 1.** (a) Schematic showing principle of Transmission X-ray Microscopy (TXM). (b) Instrument setup at sector 32-ID-C of the Advanced Photon Source (APS). (c) 4D Microstructural Evolution of Al-4%Cu at 350 °C and 400 °C.