Probing 4D Microstructural Evolution in Aluminum alloys using Transmission X-Ray Microscopy (TXM)

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Summary: Use of high resolution Transmission X-Ray Microscopy (TXM) at the Advanced Photon Source (32-ID-C) enabled us to monitor nanoscale evolution of the microstructure of Aluminum alloys in 3D. Coarsening as well as transformation reactions were captured. The 3D tomography data was also used to derive Structure-property relationships that further our mechanical understanding of these alloys.

1. INTRODUCTION

To extract better performance and reliability from precipitation-strengthened alloys, which are extensively used in several structural applications, 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 several shortcomings such as laborious destructive sample preparation, 2D field of view and limited penetrability. High temperature evolution studies in electron microscopes are also enormously influenced by surface diffusion. Full-field Transmission X-Ray Microscopy (TXM)[1,2] overcomes almost all of these shortcomings and has been useful 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. Coupled with segmentation using Machine learning, it has also been possible to analyse ultra-large volumes at a very high spatial resolution.

2. EXPERIMENTAL METHOD

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 µm diameter and 50 µm in height will be fabricated at the tips of these 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 Al_2Cu 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.

3. RESULTS

The morphology, distribution as well as the evolution behavior of θ′ and θ precipitates in Al-Cu alloys was analyzed. Aging at 350 °C revealed complex transformation reactions that were occurring in the alloy, most of which are nearly impossible to capture using other characterization techniques. 3D measurements of precipitate dimensions rendered a thorough understanding of the coarsening behavior of θ at 400 °C. A transition from interface-controlled

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to volume diffusion controlled growth in thickness of θ was 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 orientation of the θ phase in 3D. Use of machine learning assisted segmentation enabled analysis of unprecedented volume of material, opening up several new possibilities for analysis of tomography data.

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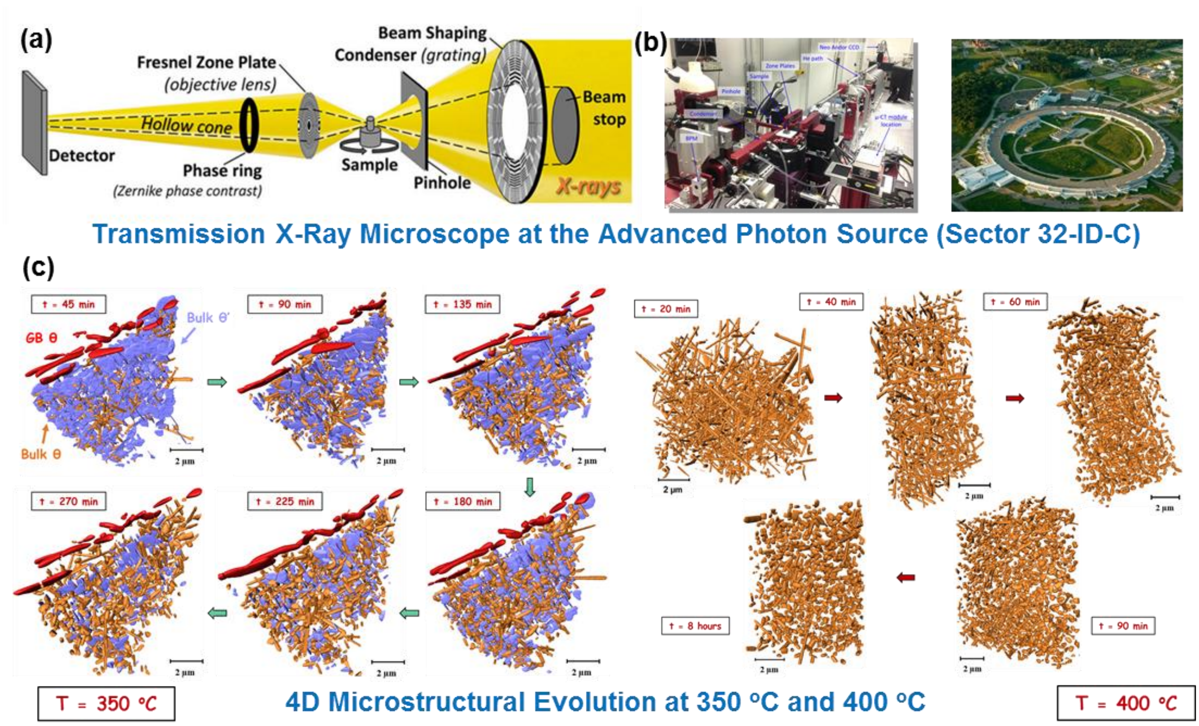

Figure 1: (a) Schematic showing principle of Transmission X-ray Microscopy (TXM). (b) Instrument setup at sector 32-ID-C of the Advanced Photon Source (APS). (c) 4D Microstructural Evolution of Al-4%Cu at 350 °C and 400 °C.