Materials Characterization 142 (2018) 203–210

Contents lists available at ScienceDirect

Materials Characterization

journal homepage: www.elsevier.com/locate/matchar

Automated correlative segmentation of large Transmission X-ray Microscopy (TXM) tomograms using deep learning

C. Shashank Kaira[a], Xiaogang Yang[b], Vincent De Andrade[b], Francesco De Carlo[b], William Scullin[c], Doga Gursoy[b], Nikhilesh Chawla[a,*]

[a] Center for 4D Materials Science, Arizona State University, Tempe, AZ 85287-6106, USA
[b] Advanced Photon Source, Argonne National Laboratory, Building 401, 9700 S. Cass Avenue, Argonne, IL 60439, USA
[c] Argonne Leadership Computing Facility, Argonne National Laboratory, Building 401, 9700 S. Cass Avenue, Argonne, IL 60439, USA

ARTICLE INFO

Keywords:
Segmentation
Transmission X-ray Microscopy (TXM)
Deep learning
Precipitates
Aluminum alloys

ABSTRACT

A unique correlative approach for automated segmentation of large 3D nanotomography datasets obtained using Transmission X-ray Microscopy (TXM) in an Al-Cu alloy has been introduced. Automated segmentation using a Convolutional Neural Network (CNN) architecture based on a deep learning approach was employed. This extremely versatile technique is capable of emulating the manual segmentation process effectively. Coupling this technique with post-scanning SEM imaging ensured precise estimation of 3D morphological parameters from nanotomography. The segmentation process as well as subsequent analysis was expedited by several orders of magnitude. Quantitative comparison between segmentation performed manually and using the CNN architecture established the accuracy of this automated technique. Its ability to robustly process ultra-large volumes of data in relatively small time frames can exponentially accelerate tomographic data analysis, possibly opening up novel avenues for performing 4D characterization experiments with finer time steps.

1. Introduction

X-ray computed tomography has become an increasingly popular technique owing to its non-destructive nature and ability to probe large volumes of material at unprecedented spatial and temporal resolutions. With the rapid pace of advancement in its use for quantitative 3D imaging [1], both at synchrotron sources [2–4] and in lab-scale systems [5–7], there is an ever increasing need to simplify analysis of the large volumes of acquired data. This need has become even more critical with the recent advent of 4D characterization (the fourth dimension being time), which has been instrumental in investigating several fundamental phenomena such as initiation, and propagation of failure at high temperatures [8], dendritic solidification [9] and more recently, microstructural evolution of nanoscale precipitates at high temperatures in aluminum alloys [10]. The datasets generated are often quite voluminous and their analysis is non-trivial and cumbersome, as it entails several steps that aim to reduce noise in these image stacks as well as improve the quality of the desired features present in them. Depending on whether absorption contrast or phase contrast imaging is implemented, subsequent analysis of the image stacks can vary significantly. The latter results in an increased edge contrast and is primarily used for imaging features with comparable attenuation [11]. For accurate quantification and 3D visualization of the microstructure, the different features present in the scanned volume need to be classified/segmented accordingly. Post-processing the acquired 3D image stacks is widely implemented to ease the process of segmentation. Depending on the variety of features present and their homogeneity, the wide distribution of grayscale values in these images need to discretized accordingly. Presence of artifacts generated during X-ray tomography can also significantly complicate analysis of such data [12]. In most cases, these grayscale images from 3D stacks cannot be segmented using simple thresholding strategies, rendering this task quite challenging as complexity of the features can require manual intervention, making it extremely time intensive. Although, to circumvent this issue, a few studies in the recent past [13–18] have implemented automated quantitative routines to aid in identification of features based on their morphological parameters and presented quantitative analyses of 3D data captured using micro-computed X-ray tomography. More recently, introduction of semi-automated techniques [19] has rendered segmentation a relatively less laborious process. However, it still remains impractical to manually segment complete datasets, especially with the increasing use of 4D characterization and continually improving temporal resolution of data acquisition. As a result of this, segmentation and analysis is often restricted to small sub-volumes which can result in

* Corresponding author.
E-mail address: nchawla@asu.edu (N. Chawla).

https://doi.org/10.1016/j.matchar.2018.05.053
Received 17 January 2018; Received in revised form 21 May 2018; Accepted 28 May 2018
Available online 28 May 2018
1044-5803/

statistically insufficient results. This parochial analysis also hindered the ability to explore different heterogeneities present and obtain a thorough understanding of the material system.

With advances in X-ray optics, Transmission X-ray Microscopy (TXM) has made nanotomography possible at unprecedented spatial resolutions (20 and 60 nm) [20]. Using unique Fresnel zone plate optics capable of magnifying radiographs, it is capable of probing materials non-destructively at the nanoscale [21,22]. This technique was recently employed by the authors in revealing novel phase transformation reactions occurring in aluminum alloys [10] as well in establishing structure-property relationships between their 3D microstructure and micromechanical properties [23]. Transmission X-ray Microscopy is quite promising as it extends the non-destructive capability of X-rays down to the nanoscale, allowing their utility to span a wide range of length scales. As features of interest approach spatial resolution limits of the technique, substantial noise can populate in these images. Complexity of these nanoscale features as well as presence of multiple phases in such images demands careful segmentation of these image stacks. Fortunately, with the recent advent of deep learning [24] and its use in image classification [25], its application in tomography data analysis can be quite promising. Its implementation in this field can make segmentation almost an entirely automated process and its implications can be revolutionary.

A deep neural network approach is utilized in this study to learn the mapping between the original images and manually segmented image (s). The trained network is then used to perform automated segmentation on large datasets. This technique can emulate the manual segmentation approach to segment X-ray images with reliable quality and it can speed up the process by several orders of magnitude. Convolutional Neural Networks (CNN) is the main branch of deep learning that was originally developed for pattern recognition [26]. Recently, it has also been used in X-ray image analysis [27]. The CNN configuration used in this study is similar to that used by Yang et al. in calibrating the rotation axis for X-ray CT [28]. However, the objective here is to implement the supervised learning approach [28] to the segmentation process by using an acquired 2D TXM slice and a corresponding manually segmented (single) image, as training input for the CNN model. Different hierarchical levels of the trained network are used to identify features of varying complexity. The trained network is then used to segment the entire 3D image stack. A schematic of this workflow has been depicted in Fig. 1.

2. Experimental Procedure

Al-4wt.%Cu wires of 5N purity having a 0.5 mm diameter (Princeton Scientific Corp., Easton, PA, USA) were solution treated at 535 °C for long times to obtain large grain sizes. This was followed by immediate quenching in ice water and subsequently aged at 350 °C for 45 min. These wires were mechanically sharpened to fine tips and micropillars were fabricated at their tips using a dual-beam Zeiss® Auriga focused ion beam (FIB) workstation (20 μm in diameter and 40 μm height). Absorption full-field Transmission X-ray Microscopy (TXM) was performed at sector 32-ID-C of the Advanced Photon Source (APS), using a monochromatic beam at 9.1 keV, just above the Cu *K*-edge to maximize the contrast between the Al_2Cu and Al phases. Using an ultra-stable stage design, the amplitude of mechanical vibrations was reduced to about 4 nm (RMS) and it was possible to extract a sub-60 nm spatial resolution from the TXM (with a voxel width of 16 nm). A more detailed description of the stage design [20] and scan details [10] have been addressed elsewhere. 3D reconstructions were performed using Tomopy, an open source Python based toolbox used to analyze synchrotron tomography data [29,30]. Subsequent 3D segmentation, quantification as well as visualization was carried out in Avizo® Fire. A Python toolbox named Xlearn (https://github.com/tomography/xlearn) was used to implement the aforementioned CNN model. The toolbox is based on the Keras and the Theano packages.

For post-scanning imaging using Scanning Electron Microscopy (SEM), the tip of the sample was cross-sectioned flat using a focused ion beam at an accelerating voltage of 30 keV and a current of 1 nA.

3. Results and Discussion

The Al-4%Cu alloy's microstructure mainly consists of orthogonal plate-like θ′ precipitates with a tetragonal crystal structure (Al_2Cu) [31], needle-like bulk θ precipitates (Al_2Cu with a different lattice structure) and coarse grain boundary θ precipitates suspended in the α Al matrix. On aging, the metastable θ′ phase eventually transforms into the equilibrium θ phase [10,31]. The relative proportion of each phase can play a significant role in controlling the alloy's mechanical properties [32]. The interfacial properties of these precipitates which play an essential role in their shape determination also vary significantly [33]. These phases can be easily distinguished owing to their differing attenuating properties, which aid in their segmentation. Further detailed information on the microstructure of these precipitates can be found in refs. [34, 35]. To improve the quality of the acquired data, various image filters were utilized. The stack of images was post-processed using a combination of Mean 3D, Bandpass and Non-local means denoise filters in ImageJ [36] as shown in Fig. 2. These filters were used cautiously and precipitate dimensions were carefully tracked to ensure that the edges don't broaden or deplete and that they are not over/under-estimated. Use of a 3D filter was seen to improve the quality of the image stacks owing to the three-dimensional nature of the nanoscale particles. The Bandpass filter was used to normalize the background and enhance contrast between various phases. The most important of these is the Non-local means filter [37], which is an edge-preserving filter that improves the quality of individual features present in images (improvement in signal to noise ratio) and enhances their contrast, without distorting their edges. Although these filters aided in significantly improving data quality, segmentation of different features in these images would still require manual intervention. From Fig. 3, it is clearly evident that conventional grayscale thresholding is completely insufficient as it either leads to over-thresholding (Fig. 3b) or under-thresholding (Fig. 3c) due to the diffuse edges of particles. Manual segmentation was performed using a semi-automatic 3D region growth based technique in Avizo® Fire, as shown in Fig. 3d. It makes use of the local contrast gradient to select a feature in three dimensions. These gradients need to be carefully chosen to ensure the particle volume is appropriately selected in 3D and not over/under estimated. It is important to note that this process is quite crucial as it could have a pronounced effect on subsequent quantification of segmented data. It is also important to note here that this approach is not limited by the computational resources utilized and only requires significant manual intervention. To ensure accurate segmentation as well as analysis of 3D TXM data, a unique correlative approach has been utilized in this study. As features imaged using Transmission X-ray Microscopy approach the resolution of the technique itself, imaging and segmenting edges of such features can be challenging and hence, introduce significant uncertainties. To overcome this, Scanning Electron Microscopy was implemented to image the same feature and aid in its segmentation. Following scanning using Transmission X-ray Microscopy, the sample was cross-sectioned normal to the rotation axis using the FIB. SEM Images of the sectioned surface were then carefully compared to the corresponding slice from the 3D TXM Image stack, as shown in Fig. 4 to facilitate a 1:1 comparison of the exact same plane. A distinct large feature of interest was chosen in both the images for comparison purposes. Grayscale intensity line profiles were constructed across the same interface for both the images. It is quite clearly evident that interfaces are more broad and diffuse in TXM slices when compared to SEM images, owing to the latter's finer spatial resolution (of about an order of magnitude). The sharp nature of the interface clearly delineates particle dimensions as seen from the line profile in Fig. 4(b). This is quite beneficial as it aided in calibrating manual segmentation of