MRS Advances

In Situ Testing Using Synchrotron Radiation Computed Tomography in Materials Research

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ABSTRACT

High resolution (< 1 μm) computed tomography is an attractive tool in materials research due to its ability to non-destructively visualize the three-dimensional internal microstructures of the material. Recently, this technique has been further empowered by adding a fourth (temporal) dimension to study the time-lapse material response under load. Such studies are referred to as four-dimensional or in situ testing. In this snapshot review, we highlight three representative examples of in situ testing using synchrotron radiation computed tomography (SRCT) for composites failure analysis, measurement of local corrosion rate in alloys, and visualization and quantification of electrochemical reactions in lithium-ion batteries, as well as forward-looking integration of machine learning with in situ CT. Lastly, the future opportunities and challenges of in situ SRCT testing are discussed.

INTRODUCTION

In the last few decades, high resolution (<1 μm) X-ray computed tomography (CT) has gained huge popularity in the field of materials science owing to its ability to non-destructively image the three-dimensional (3D) internal microstructures of virtually any class of materials, including metals, polymers, ceramics, and composites. Very recently, the development of high brilliance photon sources has made it possible to introduce a time-component directly into the already powerful X-ray CT, allowing researchers to conduct four-dimensional (4D) or in situ CT tests [1]–[3]. In situ CT tests provide valuable information regarding microstructural changes within the material (e.g., crack propagation [4], intergranular corrosion [5], and metal solidification [6]) as a function of time in response to an external stimulus (e.g., mechanical, environmental, and electrochemical), which can be used to guide the design and development of future materials with enhanced performance.

X-ray CT can be generally categorized as lab CT and synchrotron radiation CT (SRCT). Lab CT, sometimes referred to as µCT, usually grants users relatively unlimited access, allowing for the design and execution of complex and new experiments. However, the relatively low photon flux of lab CT means that it would require hours of scan time (>15 hours per scan) to achieve sufficient resolution (micrometer or sub-micrometer scale) for many materials, oftentimes excluding the possibility of in situ tests. On the other hand, the extremely high photon flux of SRCT enables the same level of resolution with a much shorter scan time (typically on the order of minutes per scan), opening up the potential for in situ tests. However, one of the main disadvantages of SRCT is that the beamline is usually a shared facility at a national or supra-national level, with access to the beam time being highly competitive and therefore constrained. In our experience at all 3 Tier 1 beamlines, the average allotted beam time is only 2 – 3 days per visit, and oftentimes only once per year, leaving little room for learning and iteration. Therefore, these two X-ray CT tools are not mutually exclusive and in fact are complementary to each other. See Figure 1 for a high-level comparison between the two. This snapshot review focuses on the use of SRCT to conduct in situ tests. It is worthwhile to note that despite the relatively fast scanning time of SRCT compared to lab-based CT, scanning time remains an outstanding key challenge. First, there are processes, *e.g.*, high strain-rate loading, in which the microstructure change of the material occurs much faster than the imaging rate. In this case, in situ SRCT is unable to capture the important morphology change information. Second, in situ SRCT is a very data-intensive technique, which generates very large datasets (on the order of terabytes) in a short period of time (24 hours), and it takes considerable time to analyse the data post-acquisition. In our experience, 2 – 3 days of SRCT data generates a year's worth of post-processing, analysis, and writeup for the team. The latter of these time challenges are currently being addressed by the application of artificial intelligence (AI), especially machine learning (ML), which will be discussed in the last section of this review.

In this snapshot review, we first briefly introduce some basic principles of SRCT and different types of in situ tests. Second, we highlight three representative examples in three different materials science fields to illustrate in situ SRCT as a powerful means to obtain insights on material behavior in practical operation. After those examples, we then present the most recent advances of applying ML techniques to in situ testing before concluding with future perspectives. The purpose of this paper is not to provide a comprehensive decadal review, but rather a snapshot of the fast changing field of in situ SRCT testing, via a few successful efforts across a spectrum of materials science, in hopes of inspiring ever more creative research on this topic in the decades to follow.

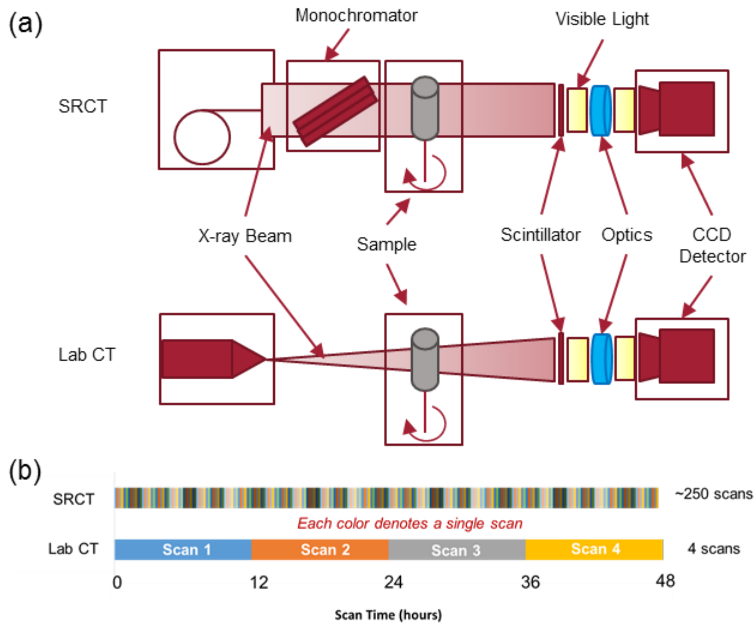

Figure 1. Synchrotron radiation computed tomography (SRCT) vs. lab-based computed tomography (lab CT). (a) The different beam characteristics between SRCT and lab CT. (b) Comparison of representative scan speed for composite materials between SRCT and lab CT.

IN SITU SYNCHROTRON RADIATION COMPUTED TOMOGRAPHY

In this section, we present some of the basics of in situ SRCT testing.

Synchrotron radiation computed tomography

When an object is exposed to X-ray, it leaves a two-dimensional (2D) shadow called a "radiograph", which represents the different material interactions with the X-ray photons passing through the material. If one acquires multiple 2D radiographs from various angles, those radiographs can then be reconstructed by computer algorithms to form a 3D representation of the object [7]. In fact, the word "tomography" derives from the ancient Greek, which means "slice". Synchrotron X-ray is a type of extremely powerful source of X-rays, produced by changing the direction of fast moving electrons in a large (km-scale) ring. The main characteristics of a synchrotron X-ray are: high coherence, high brilliance, and parallel beam geometry, which enables very fast imaging acquisition at a high resolution, unachievable by lab-based X-ray CT equipment, making in situ testing possible [8]. Example beamlines for in situ SRCT that we have been fortunate to utilize are the European Synchrotron Radiation Facility (ESRF) in France, the Advanced Photon Source (APS) in the United States, and the Super Photon ring-8 (Spring-8) in Japan.

In situ testing techniques

Because SRCT can achieve high spatial and temporal resolution concurrently, a number of in situ SRCT-based testing techniques have been developed and are receiving increasing interest in the materials science community. The central idea of in situ SRCT testing is to replicate material behaviors in real-world situations by applying an external stimulus (e.g., mechanical stress, heat, pH, moisture, etc.) and following its response as a function of time [9]. To date, the main areas of in situ SRCT investigations include: (i) mechanical testing, which refers to the application of a mechanical load, commonly in the form of uniaxial tension [10] or compression [11] to a material and imaging its resultant response such as bending, buckling, densification and cracking; (ii) environmental testing, which is usually used to study the microstructural changes within metallic materials when they are subjected to heat [12], [13], moisture [14] or corrosive environments [15], [16]. Sometimes both are combined [3]; (iii) electrochemical testing, which is often utilized to study the time-lapse behavior of functional materials, such as catalysts [17] and batteries [18], when they are in reaction or operation. Regardless of the type of test, all in situ tests require some sort of special 'loading' stages that should be stable, X-ray transparent at the region of the sample, and able to rotate 360° to collect radiographs.

IN SITU SYNCHROTRON RADIATION COMPUTED TOMOGRAPHY TEST EXAMPLES

In this section, we highlight three representative examples of in situ SRCT testing from three different fields: failure in advanced fiber composites, corrosion in aluminium alloys, and battery cycling. Together, these examples demonstrate how in situ SRCT can be utilized as a powerful tool to obtain insights of material behaviors in real-world situations and guide the development of the design of next generation materials.

In Situ Mechanical Test Example: Tensile Failure in Polymeric Composites

Advanced fiber-reinforced composites with their unique combination of lightweight and mechanical strength are used in a wide range of applications [19], notably aerospace structures. However, they are also known to display complex failure modes with multiple interacting microscale damage mechanisms. These mechanisms are often 3D in nature, and thus it is difficult for one to use conventional 2D inspection methods such as optical and scanning electron microscopes to obtain a deep understanding of the underlying damage mechanisms and progression [4]. Recently, great progress has been made in the application of in situ SRCT to study internal damage initiation and evolution of composite materials. For example, Scott et al. [20] used a simple screw driven mechanical loading frame to strain carbon fiber-epoxy notched laminates in tension at incremental load steps. At each load step, the load was maintained and 2D radiographs were recorded at 1500 angular positions over 180° of rotation. Figure 2 shows different types of matrix damage in 3D at eight load steps up to 80% of final failure. From the time-resolved damage mapping, they concluded that transverse cracks first appear in the notch region at low loads and reach saturation at about 60% of the ultimate (final failure) load, whereas 0 degree ply splits occur at 40% of the failure load and propagate along the loading direction through final failure. The work of Scott et al. can be classified in the "interrupted in situ testing" category because the load was on hold during each scan. However, such maintained loads may introduce changes in the mechanical behavior, particular in the formation of fiber break clusters [21]. Most recently, Garcea et al. [22] has combined continuous monotonic tensile loading with fast SRCT acquisition. To achieve an ultrafast imaging rate, different from most synchrotron experiments which use a monochromatic beam, they chose to use a "pink beam": a polychromatic beam with low and high energy photons removed from the white spectrum. As a result, the exposure time was set to only 2 ms, and 500 projections were collected for each tomography (resulting in 1 tomography per second) while maintaining sufficiently high resolution (~1 μm voxel size). With fast in situ SRCT, they were able to capture the damage state at 99.9% of the ultimate failure, revealing that fewer than 8% of the fibers in the 0 degree plies have fractured even at 99.9% of the failure load. See Figure 3 for the damage states very close to final failure.

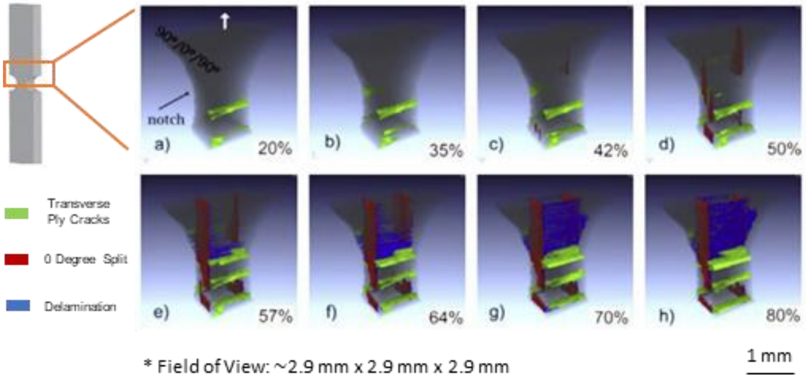

Figure 2. Progressive 3D damage of a cross-ply double edge-notched polymeric composites subject to incremental tensile load (from 20% to 80% of ultimate tensile load). Different damage modes are colored (green: transverse ply cracks, red: 0 degree split and blue: delamination). Reproduced with permission from Reference 20. © 2011 Elsevier.

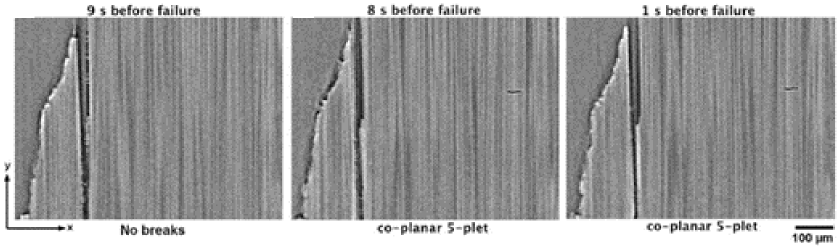

Figure 3. Cross-section along the loading direction (0 degree ply direction) for the same position of a cross-ply double edge-notched polymeric composite subject to monotonically increasing tensile load that was imaged continuously using fast SRCT immediately before ultimate failure. Reproduced from Reference 22 under the Creative Commons Attribution License (CC BY).

In Situ Environmental Test Example: Measuring Local Corrosion Rate in Aluminium Alloys

Aluminium alloys are common structural materials due to their high specific strength and stiffness, but they are susceptible to corrosion [23]. For a safety-critical structural component, it is important to be able to measure the corrosion rate of such materials. It is well-known that corrosion is a localized process; however, most conventional techniques such as weight loss measurements, electrochemical impedance spectroscopy, and optical measurements are either 2D in nature or provide an averaged bulk measurement. Singh et al. [24] provides an example of using in situ SRCT to measure the localized corrosion rate of aluminium alloys by following the hydrogen bubbles produced from the corrosion of inclusion particles (i.e., Mg_2Si particles in this case) as a function of time. To perform their experiment in a liquid environment, they modified an existing mechanical loading stage by adding a Kapton tube containing deionized water and replacing the steel grip with a PEEK cylindrical grip. A single edge-notched fatigue-cracked 7075 aluminium specimen was then immersed in the water. The evolution of hydrogen bubbles occurs very fast (on the order of minutes). In order to capture this process, they also used the pink beam as described in the previous section and achieved a voxel size of ~2 μm with scanning time on the order of seconds. By doing so, both the initiation and the following time-resolved volume change of the hydrogen bubble was captured (see Figure 4). To calculate the localized corrosion rate, they converted the rate of volume change of the hydrogen bubble to the weight loss due to magnesium dissolution, as one mole of magnesium produces one mole of hydrogen gas. They found the local corrosion rate of the Mg_2Si to be ~3×10^2 g/m^{2}d. This novel method could be extended to other types of metals and their associated corrosion mechanisms.

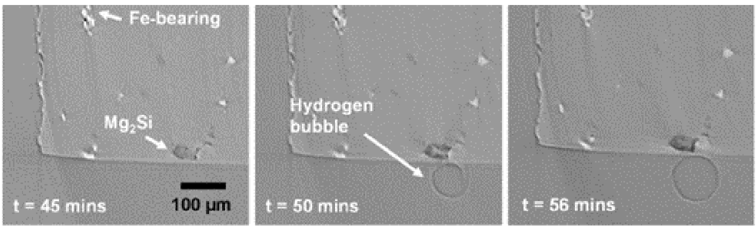

Figure 4. Evolution of corrosion of Mg_2Si particles in 7075 aluminium alloys in deionized water. The initiation and the subsequent volume change of the hydrogen bubble was captured as a function of time and used to calculate the local corrosion rate. Reproduced with permission from Reference 24. © 2016 Elsevier.

In Situ Electrochemical Test: Lithium-ion battery cycling

Lithium-ion batteries (LIBs) are the most commonly used energy storage devices in portable electronics and electric vehicles and are growing in popularity in military and aerospace applications [25]. However, the understanding of the complex interplay between the electrochemistry and mechanics during LIB operation is still lacking. In situ SRCT can provide valuable time-resolved information of the chemical composition and morphology change of the active particles during realistic battery operating conditions, which helps the design of future high-performance and safe battery materials. A good example of an in situ SRCT-based battery study is Ebner et al.'s work [26]. In their study, they used in situ SRCT to visualize and quantify the electrochemical reactions and mechanical degradation in LiBs, particularly the volume change of the electrodes resulting from the conversion and alloying reactions. Specifically, they fabricated a porous electrode comprised of SnO particles, carbon black, and polymeric binders, and they inserted the electrode into a SRCT-compatible electrochemical cell with Li_2O acting as the matrix. The electrochemical cell underwent reduction (lithiation) for 12 hours and oxidation (delithiation) for 5 hours, simulating a typical charge-discharge cycle of battery operation. In the meantime, tomograms of the entire electrode were collected every 15 minutes with a 0.65 μm voxel size, capturing the time-resolved 3D microstructural data of individual particles going through phase transitions and the resultant cracking. Qualitatively, they were able to observe two consecutive processes (formation of nanosized Sn clusters and alloying reaction of the Sn clusters with lithium to form $Li_{4.4}Sn$), which is consistent with the conversion reaction of SnO and its associated volume changes, crack formation, and crack propagations. Quantitatively, they were able to identify and measure the different chemical compositions and phase transformations of the active particles within the electrodes at different stages of the chemical reactions by analysing the distribution of the X-ray attenuations coefficients and the size of the 3D reconstructed particles (see Figure 5). This technique can be applied to a number of anode and cathode materials.

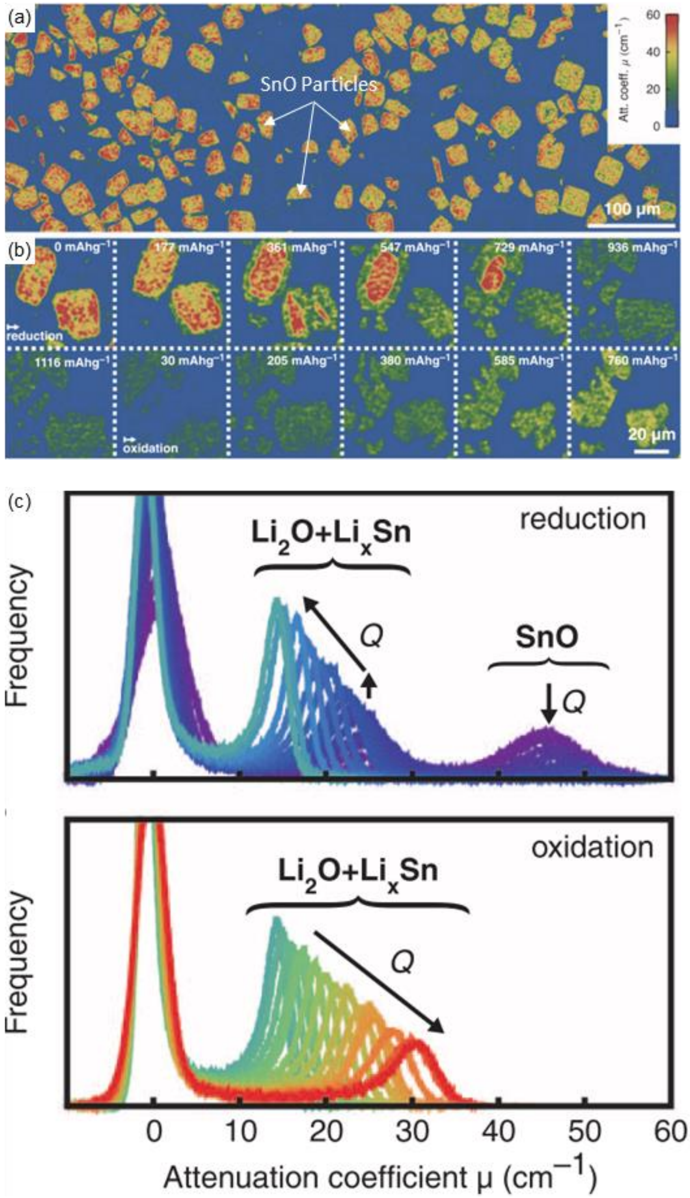

Figure 5. SRCT of the active particles (SnO) in electrodes (blue background) during lithium-ion battery operation. (a) Cross-sections of the SnO particles (yellow/red) in the electrode showing good resolution and contrast against the carbon black, binder, and electrolyte phase (blue). (b) Time-resolved cross sections of two SnO particles undergoing initial reduction and subsequent oxidation during battery operation, showing a core-shell process, volume expansion, and particle fraction. (c) X-ray attention coefficient histograms during electrochemical (top) reduction and (bottom) oxidation, revealing the evolution of the chemical composition of the active particles. Q, capacity. Reproduced with permission from Reference 26. © 2013 Science.

MACHINE LEARNING FOR COMPUTED TOMOGRAPHY DATA

As discussed in previous sections, time is a significant challenge for in situ SRCT experiments, both during acquisition and post-processing. For the acquisition step, the imaging rate should be significantly faster than the change rate of the material microstructure; otherwise, the final 3D images would be blurred. For the post-processing step, one in situ SRCT experiment typically generates a few terabytes of data over a period of a few days. Extracting useful information from such a large dataset is by no means a trivial task [9]. Researchers usually spend months or even years analysing the data acquired from just one synchrotron trip.

In recent years, artificially intelligence (AI), most notably machine learning (ML) [27], has revolutionized many fields in science and engineering. The use of AI is pervasive in our society, yet many non-computer science researchers are likely to be confused by the different terms associated with AI, especially the difference between AI, ML, and deep learning. To put it simply, AI is defined as a computer system able to perform tasks that normally require human intelligence, which has the broadest scope of all. ML is a subset of AI and is the most popular way of achieving AI. Deep learning, or more precisely deep artificial neutral networks, is a subset of ML. It is a set of computer algorithms that are especially useful for computer vision, sound recognition, and natural language translation. Here we simply focus on ML techniques for in situ SRCT testing. To date, very few users in the CT community have used AI/ML for their work. If ML can be added into the already powerful in situ SRCT testing techniques, it could greatly accelerate the time needed for materials discovery and characterization, particularly yielding faster acquisition and fast data analysis. Below we will highlight some of the most recent advances in applying ML techniques to the pipeline of in situ SRCT testing.

Virtually all CT data needs some sort of segmentation, in which the users label different phases of materials within the image by grouping together voxels that have similar grey scale values. The simplest way to do so is by thresholding the greyscale histogram. However, this approach is ineffective when the image contains multiple phases with convoluted grayscale values. Perciano et al. [28] developed a ML approach termed PMRF that exploits probabilistic graphical models, specifically Markov Random Fields (MRF). PMRF takes advantage of graph partitioning and is fully parallel (for both parameter estimation and optimization), leading to high computation efficiency. They tested PMRF on real CT datasets, and the accuracy was over 95%. See Figure 6 for a comparison of CT image segmentation using different techniques. Another challenge in post processing CT datasets is for users to be able to organize and retrieve specific images fast and accurately. Ushizima et al. have developed a new Convolutional Neural Network (CNN)-based software package, pyCBIR [29] that enables fast retrieval of the most similar images and sorts them according to the selected metrics. Furthermore, researcher are actively developing methods that use neural networks (NN) to perform tomographic reconstruction, which is expected to push the limit of the time resolution at SRCT [30].

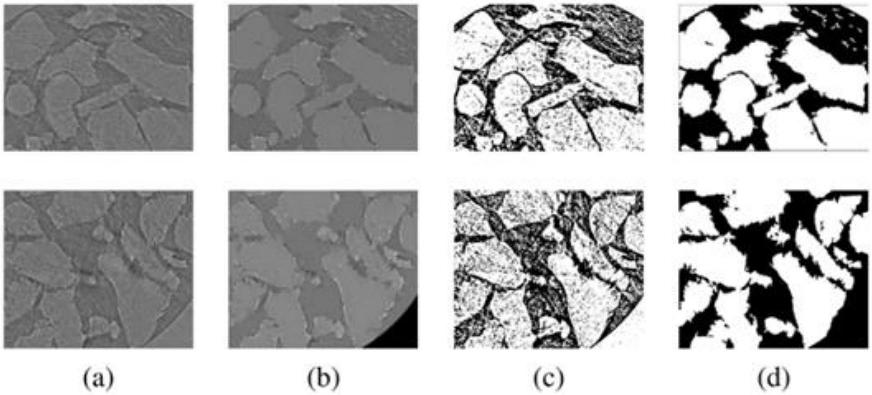

Figure 6. Comparison of CT image segmentation using different techniques. (a) Original CT slice. (b) Over segmentation using Statistical Region Merge. (c) Segmentations obtained after smoothing and manual thresholding (d) Segmentations obtained using machine learning (PMRF framework). Reproduced with permission from Reference 28. © 2016 IEEE.

FUTURE PERSPECTIVE

Although in situ (or 4D) SRCT testing has already begun to have impact on a number of areas in materials science, it is still a young and vibrant field with many exciting new opportunities outstanding. First, the temporal resolution of in situ SRCT can be further improved. Currently, the fastest SRCT can take ~10 tomography per second at microscale resolution. If the speed can be further increased while maintaining the same high resolution, we can be one step closer to realizing true 4D imaging (3D microstructural data + 1D continuous time) to capture the vast outstanding number of temporal materials topics. Second, the rich in situ SRCT data provides insights into the structure-property relations of various material classes, and yet it remains challenging to use that understanding to build a reliable model to predict material response given the microstructural data. Third, from a practical perspective, the field of view is still very small (3 – 5 orders of magnitude) compared to many material sizes when deployed in a realistic environment. Therefore, it is important to ensure that the information obtained in the small field of view is relevant to the practical service conditions. Finally, the large datasets from in situ SRCT tests have proven to be a significant challenge to analyze. Nevertheless, the preliminary application of ML has demonstrated its great potential to automate data processing. In the future, one can envision a material design framework that combines multiscale modeling, in situ CT testing, and automated data analysis to create novel materials with unprecedented properties.

ACKNOWLEDGEMENTS

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