Rich multi-dimensional correlative imaging

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Abstract. The use of microstructural design to tailor materials properties has increased sharply in recent years. In parallel the number and the capability of techniques able to characterise materials microstructures has increased sharply too providing structural, chemical and crystallographic information. Here we examine how correlated 3D, 4D (3D+time) and multi-dimensional imaging enable a much richer picture to be built up of a materials microstructure. We look at how a data-centric approach can support the use of materials informatics, digital twinning and machine learning to accelerate the design of new materials systems and to optimise the manufacturing of established ones. However for this to happen we need to develop ways to digitally fingerprint the microstructural images and maps we collect such that they can be incorporated into machine learning schemes. Through the use of case studies (multimodal imaging) we look at correlative imaging across scales, across time (the dilation of electrode materials in lithium batteries during discharging and fast corrosion of magnesium), as well as across multiple modalities (butterfly defects in bearings steels and the sintering and recrystallization of powders). These demonstrate how different techniques can come together to provide complementary aspects of the bigger picture.

1. Introduction
The number of techniques available to interrogate materials microstructures has increased sharply over recent years. In particular we have seen measurements extended from two dimensions to three by X-ray and transmission electron computed tomography (CT), atom probe and serial section scanning electron tomography. By exploiting the non-destructive nature of X-rays and visible light this has been extended to four dimensions through temporal studies. With the ability to map chemical and crystallographic information the number of dimensions has been extended yet further. These can be combined to obtain a rich, multifaceted picture of the structure and microstructure of materials and devices.

At the same time manufacturing methods are now able to rapidly build up and assemble materials and devices engineered across multiple hierarchical scales. This is alongside a concurrent development of advanced multiscale models for their design and simulation. In order to exploit this ability to rapidly manufacture an almost infinite variety of materials and structures, a step jump in materials imaging and characterization will be required.

This article examines the trajectory of this development which has led to the recent advancements of correlative microscopy and looks forward towards a wider, and more integrated, approach to characterization through correlative frameworks where it is possible to exploit the capability of individual pieces of equipment whilst keeping them contextually connected to other analyses. We consider how we can connect information efficiently and effectively across scales and modalities and the benefits this brings for characterization and modelling. We assess the current status of correlative
strategies and look at opportunities for application in materials science looking first at manufacturing and then at materials degradation.

2. Multiscale 2/3D imaging
As illustrated in figure 1, almost all materials are hierarchical in some sense. In each case it is important to identify the key length scales that control behaviour and to develop the tools needed to probe these scales.

Figure 1. Row 1: Bone at different hierarchical levels; a) an optical micrograph showing the cortical and trabecular bone, b) SEM of the trabecular network, c) micro CT of the Haversian system, d) Lamellar structure in polarized light, e) TEM of collagen fibrils [1], Row 2: Shale rock at a) reservoir scale, b) fractures (MacroCT), c) laminations and microstructures (MicroCT), d) Organics & clay (FIB-SEM), e) nanopores (TEM tomography) [2], Row 3: a) Ni superalloy single crystal turbine blade, b) dendrites, c) \( \gamma' / \gamma '' \) arrangement, d) EDX map of Al segregation to \( \gamma ' \), e) atomic structure across boundary (courtesy of [3]), Row 4: C fibre composite a) composite wing, b) CT image of impact damage, c) CT of woven tow structure, d) SEM image of C fibre, e) TEM image of c-fibre, Row 5: Microchip at increasing levels of magnification, a) overview, b) SEM of integrated circuit, c) FIB-SEM tomograph of 3D circuit architecture, d) 3D serial section PFIB-SEM of a through Si via, e) TEM of 14nm chip (images courtesy of TESCAN).
Of course, linking together microscopes to examine different length scales in 2D is not new. However, linking them together in 3D is more challenging because locating a potentially sub-merged region (volume) of interest identified at one scale for examination in greater detail at another is difficult. This has been done for a number of applications in metallurgy, particularly with respect to studies of degradation [4, 5]. To date, this has mostly been through the registration of sample features with manual transfer of the sample coordinates from one machine to another. Going forwards, routes for automatically transferring the sample coordinates, with very high accuracy, from one machine to another are required if multiscale 3D imaging is to become routine. A number of instrument manufacturers are aiming to do this predominantly through the use of standard stages and fiducials [6].

A critical aspect in multiscale imaging is managing the volume of the acquired dataset. If the whole region is collected at increasingly high spatial resolutions (termed the ‘Googlemaps’ approach in [7]) then the data requirements soon become the limiting factor (see figure 2). Alternatively targeted trajectories [7] have to be identified that track certain features at increasingly high spatial resolutions, but over concomitantly smaller volumes.

![Figure 2. The relationship between spatial resolution, sample size and typical data storage requirements (non-destructive techniques denoted by dashed lines).](image)

### 3. Temporal 4D (3D+time) imaging

The non-destructive nature of X-ray imaging opens the way for acquiring a sequence of images over time. For phenomena that occur quickly relative to the acquisition times needed to collect sufficient X-ray projections (radiographs) to reconstruct a tomograph (typically 200-3000 projections), such as ductile fracture [8], then continuous streaming of the data during the process is common [9]. For longer timespans, e.g. corrosion [10], or cases where the sample must be incrementally withdrawn from a particular environment for X-ray scanning, such as during sintering [11], time-lapse intervals are often chosen [9]. Image sequences can be correlated over time using digital image, or volume, correlation to extract deformation fields, for example to map the dilatation of lithium batteries during charging [12] as illustrated in figure 3. In addition to conventional CT, 3D XRD has been developed to obtain very high spatial resolution images of grains in 3D over time [13, 14].
4. Multidimensional (>4D) imaging

Structural or microstructural images are useful, but often only provide part of the picture. Greater understanding can be gleaned by combining structural images with maps of chemistry or crystallography for example. This is now routine in 2D where scanning electron microscopy is combined with energy dispersive X-ray (EDX) mapping and electron backscattered diffraction (EBSD) to provide insights into the elemental distribution and the grain textures respectively. This technique has been extended to 3D using serial sectioning. The volume that can be mapped extends up to $(50 \, \mu m)^3$ using traditional Ga$^+$ focused ion beam, to $(300 \, \mu m)^3$ using a plasma FIB [15] and beyond with ultramicrotomy for relevant materials [16], broad ion beam sectioning [17] or laser sectioning [18].

Non-destructive multimodal imaging can be undertaken to provide a richer picture of behaviour that achievable with single modalities. For example with regard to the time-lapse sintering of metallic powders, diffraction contrast tomography (DCT) has provided detailed maps of the 3D grain structure while attenuation CT provided complementary information about the elimination of porosity during consolidation [11]. Similarly in medical imaging (positron emission tomography (PET) and CT scanning are combined – the former identifies specific metabolic functions using radiopharmaceuticals while the CT provides anatomical information and can also be used to help reconstruct the PET scans too.

Given that we have many destructive and non-destructive tools with which to probe a rich multidimensional space there are inevitably many choices to be made in terms of the trajectory by which we explore it (See figure 4a).
Figure 4. a) Correlative frameworks can illuminate a multidimensional space via many potential trajectories. Consequently the user needs to consider data gathered to date in determining the most useful pathways b) an illustrative workflow aimed at understanding the microstructure of a superalloy turbine blade [19-23].

Such workflows demand significant planning, both to take into account information gathered at one stage when considering the next step, but also because experimental considerations restrict the order of certain pathways (e.g. selecting when to excise certain regions or to employ certain destructive methods) etc. Figure 4b outlines a study aimed at understanding the manufactured microstructure in a single crystal nickel superalloy jet engine turbine blade. Here lab. X-ray CT reveals the overall 3D architecture of the turbine blade while a finer scale time-lapse synchrotron X-ray CT is able to capture the morphology and formation of the dendrites during the single crystal solidification process. Subsequently serial section FIB-SEM is needed to characterize the finer γ matrix/γ' precipitate microstructure and map their chemistry by EDX. To examine the atomic structure and local chemistry transmission electron microscopy (TEM) and atom probe tomography (APT) are applied respectively.

5. Exploiting rich multidimensional datasets: Microstructural Informatics

Computational materials science has been proposed as a means to shorten the time to design and manufacture new materials systems [24]. Initiatives such as the Materials Genome Imitative (MGI) and Integrated Computational Materials Engineering (ICME) have focused considerable effort into the use of computational materials science to discover new materials systems, as well as to optimise the manufacturing processes needed to deliver high performance components.

Perhaps the greatest successes to date have been in areas where the materials properties depend little on microstructure. In such cases the desired properties (stiffness, thermal expansion, electrical or thermal or basic coupled properties) can be predicted on the basis of the crystal unit cell. In such circumstances thousands of materials systems can be trialled in silico and the most promising ones selected and means investigated to make them. Unfortunately, the majority of performance characteristics of materials depend heavily on many aspects of microstructure and defect structure not captured by the unit cell, from the atomic defects right up to the macroscale.

Reflective of the fact that there are key length scales that can control specific phenomena, materials models have particular scales over which they are valid, from ab initio methods to continuum finite element models. Multiscale modelling sets out to address a number of challenges to effectively represent hierarchical systems. Further multiscale, multi-physics models provide an opportunity to make engineering predictions rigorously based on physical principles. In this context it should be
remembered that properties are scale-specific and that the microstructure at each scale can play a key role in determining these properties with microstructural variation being a key aspect in terms of understanding scatter in performance. Weinan [25] identifies 4 challenges to multiscale modelling:

1) We need to understand the mechanisms, model parameters, boundary conditions, microstructural variation, etc. operative at each scale.
2) Ideally, we should be able to derive coarse-grained models from more detailed ones in the appropriate limit, providing a link from scale to scale.
3) We need to understand how to couple together models at different levels together smoothly, without creating artifacts. It is common for large errors to occur at the interface where two models join.
4) We need to understand how to formulate models at intermediate levels where physics based models do not exist. Although our understanding of some scales may be mature it is common for certain domains to be poorly understood, e.g. the mesoscale gap relating discrete dislocations, dislocation networks and grain boundaries in metallic systems which mean that experiments play a critical role in filling this gap.

Rich multidimensional microstructure datasets provide an opportunity to tackle some of these challenges and thereby to improve the accuracy of such models. Ideally, the models at each length scale should be tied to the microstructures they represent (see Figure 5). In this way each model can be separately tested and validated. Furthermore in cases where materials models do not exist or where the couplings between models at different scales are poorly defined it may be possible to design experiments or workflows which deliberately address these gaps to allow the bridging of scales. In practice the link between materials models and microstructure are not so well defined and many semi-empirical models use parameters which are not simply related to the microstructures they represent (e.g. continuum damage parameters). At the present time, one of the main obstacles is the lack of well characterised, and widely available, multi-dimensional microstructural datasets.

In this respect, one of the principle issues is that at present we have no agreed way of quantifying microstructures beyond a few simple parameters (grain size, phase fractions, etc). To make progress we need to configure our microstructural models in terms of agreed microstructural parameters that can be unequivocally measured and develop machine learning methods that can relate aspects of the microstructural features to material processing or performance. As Wodo et al. point out [27] the goal of microstructural imaging is to provide detailed, high-resolution maps, and hence, imaging techniques inevitably produce high-dimensional data sets, while the goal of establishing quantitative process-microstructure-property relations (PMPRs) is to derive the smallest set of variables that explains most of the variability of the data. Consequently, microstructural data need to be reduced to a small number meaningful descriptors, a digital fingerprint (or gene sequences), to enable them to be used in developing property microstructure processing relationships. Discovering this reduced descriptor space as applied to microstructure is the basis of what has been termed microstructural informatics. At the same time such fingerprints would also enable archives of large 2D or 3D microstructural images or volumes to be stored efficiently.

ICME approaches exploit a digital twin (see figure 5) that represents in silico the attributes of the manufactured component as it undergoes the manufacturing process. In essence the microstructural data and the associated microstructurally cognisant materials models are like the two strands of DNA providing the underlying characteristics that control behaviour. Gaps in one strand can be compensated for by information from the other. Further machine learning approaches can then help to identify microstructural traits (or genes sequences) that give rise to specific performance characteristics or that can be used to assess the degree to which two microstructures have similar characteristics helping to direct microstructural control for performance optimisation.
Figure 5. Multiscale experiments and multiscale models need to be developed synergistically and in parallel both to improve our understanding of materials across the key length scales, but also to enable the multilevel design of new materials. The important microstructural scales are illustrated for the deep drawing of an aluminium alloy [28-32]. Model schematics after [33] and layout after [7], FE model courtesy Dirke Raabe.

6. 4D imaging: Corrosion

Magnesium alloys are attracting a lot of interest across a broad spectrum of applications, where light weighting is a key attribute. Conventional processing to sheet involves a number of steps and for magnesium, being a hexagonally closed packed material, this are hindered by its limited formability. Twin roll casting (TRC) is a viable, cost effective alternative, which involves steep cooling rates. This affects the distribution of microstructural features across the section thickness [34], in addition to significantly influencing the near surface texture [35]. This is a critical factor because many magnesium alloys show a strong link between crystallographic texture and corrosion behaviour.

For TRC AZ31 Mg alloy (2.9 wt% Al, 0.88% Zn, 0.34 % Mn, 0.02 % Si, 0.007 % Fe, 0.009 % Cu) under saline conditions, corrosion initiates via micro-galvanic coupling between cathodic intermetallic phases including sub-micron sized, rosette-shaped Al₇Mn₃ particles [36], β-Mg₁₋ₓ(Al,Zn)₁₂ phase and the adjacent Mg matrix, once the hydroxide surface layer has been compromised [37]. Propagation of filiform-like corrosion fronts are then observed running along the dendrite arms whilst being constrained by the Al- and Zn-rich interdendritic boundaries [35]. As a result, corrosion can be highly microstructure dependent and very non uniform.

There is still a real need to better understand the fundamentals associated with corrosion of Mg and Mg-based alloys as the low corrosion resistance of Mg is its “Achilles heel” hindering uptake in many engineering applications. Previous time-lapse nano-CT imaging conducted on TRC AZ31 Mg alloys showed that the corrosion fronts were restricted within the interdendritic regions [38]. However, the exposed samples were intermittently removed from the saline environments between consecutive CT
scans, essentially rendering it an “ex-situ” characterization study. The CT images of the corroded specimens showed shrinkage cracks within the dried corrosion products; where the possibility of their influence on the corrosion propagation during the successive exposures cannot be ruled out. Secondly, only a relatively small sample surface could be accommodated within the field of view of the nano-CT scans.

In situ studies of the evolution of the interdendritic corrosion fronts in TRC AZ31 Mg alloy have avoided these issues [39]. Here X-ray imaging was able to resolve the Al- and Zn-rich phases thereby delineating the interdendritic boundaries (see figure 6). These show a range of dendrite orientations relative to the surface of the sheet with a gradation of microstructure from the surface towards the central plane of the sheet. The experiment benefited from a relatively larger field of view, and the samples were continuously exposed to the saline electrolyte, whereby the sequential corrosion events could be captured, preventing any changes upon removal and exposure to air, in particular preventing the drying and cracking of the hydrated hydroxide surface layer that forms during exposure to the saline environment. However, this adds an additional challenge as the attenuation of the metal and the water are similar using relatively hard X-rays making contrast generation more difficult. Normally sub-micron resolution scans (on a Zeiss Versa 520) would take too long to acquire given the rapid rate of corrosion. Here image quality has to some extent been sacrificed to achieve shorter acquisition times. An acquisition time of 4 h per scan was adopted, which was followed by an additional 8 h of electrolytic exposure, before the next scan was conducted. As shown in figure 6, the images are of sufficient quality to observe the advance of the corrosion fronts as a function of the dendrite orientations.

The X-ray micro-CT results show the manner in which the corrosion preferentially attacks the Mg matrix along the dendrite arms, with the Al- and Zn-rich interdendritic and grain boundaries showing increased corrosion resistance and acting as a local barrier (Figure 6b). At the same time, evidence of detachment of the corrosion products was also captured within the CT scans, as highlighted in image (b). The images show gradual increase in the local corrosion depths with respect to time. The results also confirm that the hydrated, near-surface oxide/hydroxide layer of corrosion products are relatively loosely held and render minimal protection to underlying microstructures. More importantly, after 51.6 h of exposure, the alloy microstructure showed a significant number of cracks within the corrosion products, as evident in image (c), and also observed in the previous studies [38] and the fall out of bulky chunks of corrosion products as confirmed in image (d) after 59.5 h. The formation of cracks and the falling out of corrosion products can be attributed to the weight of the loosely held interdendritic skeleton along with hydrated corrosion products. The 3D structure of Mg-dendrites in TRC AZ31 Mg alloy shows a non-continuous distribution of eutectic along the interdendritic regions.
Therefore, it can be suggested that the interdendritic regions without any eutectic are the locations where the separation/fallout of the bulkier portions of corrosion products occurs. This observation obtained from the micro-CT results is very important from a corrosion propagation perspective. Whilst we have studied the initiation at the surface elsewhere by SEM, [34, 37, 39] the time-lapse CT allows us to track the propagation of corrosion across the surface and then critically when it starts to penetrate deeper into the material at a later stage. It is this later stage that is most damaging to the structural integrity of the material.

7. Correlative multimodal 3D mapping: Butterfly defects

Under rolling contact fatigue bearings tend to produce a population of defects subsurface that initiate from inclusions at around $10^5$ cycles. These arise well before failure normally after around $10^8$ cycles [40]. The defects that grow from these inclusions typically have a ‘butterfly’ morphology and they are associated with intense plastic deformation and dissolution of the metal carbide particles. These regions appear as white etching matter (WEM) and include the butterfly wings as well as regions which decorate cracks. Traditionally these defects have been observed in 2D in metallographic sections such as that in figure 7a. Limited 3D information of butterfly defects has been obtained by coarse serial sectioning [41].

![Figure 7](image-url)
same butterfly viewed from the axial direction. [26]

In order to build up a more detailed and complete picture of the nature of butterfly defects, correlative imaging is required because no single technique is capable of distinguishing all the features:

- secondary electron (SE) imaging can delineate the cracks (see figure 7a),
- electron back scattered diffraction (EBSD) imaging can distinguish the WEM as a non-indexing feature (shown black in figure 7b),
- EPMA is needed to detect changes in the level of carbon in solid solution (figure 7c).

The EPMA maps in figure 7c clearly show that, for the alloys they have studied at least, the WEM has less C in solution than the parent alloy. This is surprising given the dissolution of the carbides in this region and runs counter to previous inferences [40-42].

Curd et al. [26] have brought these techniques together with serial sectioning by broad ion beam to build up a 3D detailed picture of the spatial distribution of these features (figure 7d and e). This shows the butterfly is extended in the circumferential direction in accordance with the sketched representation drawn by Becker [41]. Furthermore, there is cracking at the interface between the WEM and the parent as well as cracking through the WEM itself. In this case, two dominant cracks are seen either side of the inclusion giving a bi-plane appearance. The WEM tends to extend further behind the particle than the cracks.

8. Multimodal 4D imaging: Sintering of Copper

Pressureless sintering of loose or compacted granular bodies at elevated temperature occurs by a combination of particle rearrangement, rotation, local deformation, diffusion, and grain growth. In order to optimise the performance of the sintered body and eliminate defects, it is important to be able to understand the relative importance of these mechanisms as well as the extent of grain growth that occurs during densification. Conventional attenuation based X-ray CT can provide 4D information about the evolution of the general shape and volume as well as tracking voids, while X-ray diffraction can be exploited to learn about the rotations of the particles as well as information about the grain growth. Diffraction contrast tomography [43] enables the combination of attenuation and diffraction based CT to provide all this information. Originally developed for application at synchrotrons, it has recently been developed to run on laboratory X-ray sources [44].

Here the technique is used in a time-lapse manner to follow the sintering process in pure copper. It is evident from figure 8 that as the sample consolidates some grains grow and others shrink. A clear example of the recrystallized grain growth is given by the abnormally large grain (rendered purple) in the centre of the compact.

Figure 8. Attenuation CT (greyscale) alongside the grain structure mapped by lab. DCT (Courtesy S.A. McDonald, T.L. Burnett, P.J. Withers).
The densification was recorded by the attenuation CT and is shown in figure 9a showing that initially densification is quick but that this slows as 80% density is approached. It can be seen in figure 9b by combining the DCT and attenuation CT that there is a degree of correlation between the number of touching particle neighbours and the extent of particle rotation (quantified by the rotations of the constituent grains in each particle). These rotations become smaller as the sintering process develops.

Figure 9. a) Densification of the powder body across the sintering steps at 1050 °C showing the increase in density for the full sample and, inset, comparing two regions of interest (RoI) for the first 5 steps, RoI2 corresponding to an interior region and RoI1 to an exterior region b) Quantification of particle rotations and effect of particle coordination Showing the mean particle rotations relative to the original arrangement as a function of the number of contacting neighbours, for the whole sample studied [11]

9. Conclusions
While in many cases a great deal can be learnt from 2D images using a single instrument there are many other instances where a more complete picture is required. In particular the multiscale models needed to drive the computational design of new materials and manufacturing processes are highly dependent on rich, multiscale and multidimensional information for validation and to ensure the applicability across the required range of scales. X-ray imaging can provide 3D information across a range of length scales and can be combined with 3D serial sectioning techniques within the electron microscope to extend such data to finer length scales while TEM tomography and atom probe methods extend this to finer scales still.

The non-destructive nature of X-ray imaging opens up 4D studies, using either time-lapse, or fast streaming high frame rate imaging, to cover a wide range of time scales. Here an example was described where the crystallographically dependent rate of corrosion can be followed over time for twin roll cast AZ31 Mg alloy.

The ability to bring together complementary insights from multiple imaging or mapping modalities enables the superposition of data to form multi-layered (multifaceted) 3D datasets. Here we describe an example where secondary electron imaging, electron backscattered imaging, energy dispersive mapping and electron microprobe analysis are all brought together first in 2D to understand the relationship between cracking, crystallography and white etch matter, inclusion chemistry and carbide and carbon solute distributions associated with a butterfly defect. Using a coupled broad ion beam – scanning electron microscope system (BIB-SEM) the morphology of the WEM and cracks that make up the butterfly could be exposed in three dimensions. Bringing together information from different instruments is more complex in terms of the co-registration of data, as well as the transfer of samples between instruments.

In particular complementary X-ray techniques open the way for multi-modal 4D datasets. Here we presented data combining attenuation CT to record in a time-lapse manner the consolidation of copper particles during sintering alongside information about particle rotations as well as the consumption of certain grains by the growth of others. This is important if the sintering conditions are to be optimised to control grain growth during sintering yet achieve high levels of densification.
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