Recent advances in X-ray microtomography applied to materials

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This review highlights recent advances in X-ray microcomputed tomography (microCT) as applied to materials, specifically advances made since the first materials microCT review appeared in International Materials Reviews. Improvements in instrumentation are covered, and one focus is microCT using phase (as opposed to absorption) contrast. Instead of grouping studies by disciplines, the reviewed reports are organised by type of application, specifically the study of the spatial distribution of phases, of cellular solids (including static and temporally evolving, fibrous network solids, mineralised tissues and biomedical applications), of channel structures, of deformation, fatigue and fracture, of processing and of corrosion and environmental interactions. Metrology applications are covered briefly, and several applications where microCT is combined with position resolved X-ray scattering are described in more detail. The accuracy of microCT reconstructions is discussed before data handling challenges are outlined. The review closes with speculations on the future directions of materials microCT.

Keywords: Microtomography, X-ray imaging, Synchrotron radiation

Introduction

Less than a decade ago, microcomputed tomography or microtomography (microCT) of materials was reviewed in this journal by this author. A reader might ask why a second review is needed so soon after the first. One finds an answer to such a reasonable question by considering the very rapid development of instrumentation and the concomitant increase in accessibility (and in publication rate) that have occurred since the mid 1990s.

Dedicated microCT instruments at the third generation synchrotron X-radiation sources (e.g. APS, ESRF and SPring-8) and at other storage rings have multiplied opportunities for three-dimensional (3D) imaging at the highest spatial resolution and contrast sensitivity, but daily access is not an option. Multiple manufacturers now offer affordable, turnkey microCT systems for routine, day to day laboratory characterisation by scanning electron microscopy (SEM). Recently, commercial nanoCT systems (claimed spatial resolutions substantially below 1 μm) and in vivo microCT systems (for small animals) began to appear in research laboratories.

The increase in microCT papers since the first International Materials Reviews (IMR) review amounts to an explosion. Quantifying the rate of increase in publication of microCT papers is problematic because of artificial issues such as the division between microCT and conventional tomography (here the author arbitrarily takes the same definition as in the first review, namely that microCT describes tomographic imaging with ~50 μm voxels, which is volume elements).

Nonetheless, a feel for the increase can be gained by considering the SPIE conference series Developments in X-ray Tomography (1997, 1999, 2001, 2004 and 2006); it spans biology to engineering and its proceedings has grown from 266 to 340 to 374 to 802 to 682 pages respectively, which represents a saturation of this particular forum (limited to the papers that can be presented in a 3 day symposium). The slight decrease in pages from 2004 to 2006 probably represents a decrease in frequency (3 year gap preceding 2004 and 2 year gap preceding 2006).

Some authors of microCT studies use synonyms (including microCT, X-ray tomographic microscopy, computerised microtomography, the recent nanoCT and even just tomography) in describing their studies, and this complicates the search for relevant papers. Further, the same class of structure, requiring similar analysis tools, can occur in disciplines spanning the life sciences to art conservation to the physical sciences and engineering, and reports appear in a wide dispersion of journals and conference proceedings. One example is cellular solids with trabecular or spongy bone and bone growth scaffolds found in the biomedical literature and with metal foams in engineering publications. These two factors combine to hinder newcomers finding previous paradigms on which to base their analyses and to produce examples of unneeded (except perhaps in an existential sense) sweat expenditure via wheel reinvention.

Considering the following experiment in locating microCT papers relating to foams, a class of cellular
solids described in more detail below. Over the past several years, the author desultorily collected nine papers on microCT of cellular solids (excluding those on trabecular bone) without any particular purpose beyond the possibility of writing this review. A literature search in Compendex, a database for engineering papers, on ‘microCT and foam’, revealed one paper (one of the nine); on ‘microtomography and foam’ produced 30 hits (three more of the nine) and on ‘tomography and foam’ resulted in 139 hits (six of the nine). Separate searches on ‘cellular solid and tomography’ or ‘wood and tomography’ or ‘scaffolds and tomography’ would be required to reveal the other three papers of the nine; note that the middle search yields 204 hits most of which are irrelevant to microCT.

This review examines developments since the first review appeared in this journal1 and except where needed for understanding the more recent developments, this earlier material will not be described again. To a great extent, organisation of the papers in different topical areas is arbitrary. Further, many papers could be discussed in more than one subsection. The reader familiar with some of the literature, might, therefore, be surprised at where a given paper appears. One hopes that this reader will not be surprised by significant omissions in the literature reviewed.

Developments in instrumentation will be covered first because these dictate what has been possible in the applications that follow. The subsections of the instrumentation section are listed at the start of that section.

MicroCT of the distribution of phases is the first subsection of the materials applications section. Cellular solids such as foams and trabecular bone are covered second because the analysis techniques extend those illustrated in the previous section. Channel structures, essentially the reverse structure of foams (minority phase being open space instead of solid like in foams), are considered after cellular solids. Cracks are often an important feature in fatigue, deformation and fracture applications, and, because measurement of quantities such as the 3D spatial distribution of crack openings is not dissimilar to what is required with channel structures, these applications are summarised next. Deformation and crack opening/closing studies often require repeated imaging of the same specimen, and this thread is continued in subsections on processing and on corrosion and other environmental interactions.

MicroCT’s use in metrology is covered in a separate section following the materials applications section. Multimode studies (microCT plus another modality such as X-ray microbeam diffraction mapping) are covered in the next section. Accuracies of microCT reconstructions and of various measured quantities and data handling challenges are discussed in separate sections. A prognosis for the future of microCT imaging of materials concludes the review.

**Instrumentation**

This section covers laboratory (absorption) microCT first with the primary emphasis on available commercial systems and on promising new developments that have, for whatever reason, not appeared on the market. The second subsection reviews synchrotron (absorption) microCT. Spatial resolutions substantially below 1 μm are the province of nanoCT, and, as the instrumentation needed differs from that in the first two subsections, nanoCT is discussed in a separate, somewhat shorter subsection. Phase microCT, mainly performed with synchrotron radiation, is the subject of the rather lengthy (fourth) subsection. Fluorescence microCT and microCT employing X-ray scattering (small angle X-ray scattering, SAXS, and wide angle X-ray scattering, WAXS, or diffraction) are the subjects of the fifth subsection; these two modalities are treated separately because they require translate-rotate data collection and different detector configurations. Alternative approaches to tomography are discussed next, and the section ends with a discussion of reconstruction improvements.

Before developments embodied in the current generation of laboratory and synchrotron systems are discussed, it is useful to reiterate the interplay between field of view (FOV), the number of detector elements and the minimum corresponding voxel (volume element) size (Fig. 1). Exact reconstruction requires the specimen to remain in the FOV for all rotations (i.e. FOV equals or exceeds the specimen diameter as in Fig. 1e); otherwise, difficult to predict errors will result from parts of the specimen rotating into and out of the FOV (i.e. the situation in Fig. 1a and b for points A and B). If the specimen diameter is fov and the detector has N elements, then the minimum voxel size containing physical information is $v_{ox} = \frac{fov}{N}$ (note that one can always reconstruct with smaller voxels, but this is strictly a mathematical exercise). For a 5 mm diameter specimen and a 1 K detector, reconstruction can be with 5 μm voxels; recent literature reports increasing use of 2 K detectors which would result in 2-5 μm voxels of the same specimen. At least one manufacturer of a commercial, tube based system offers a 4 K × 2 K detector and capability to produce (8 K)$^2$ voxel reconstructions. As a very rough estimate, spatial resolution is somewhat worse than twice the voxel size, but metrics such as the modulation transfer function are required for more precise discussion of resolution. Effects like penumbral blurring (finite X-ray source size) or optical and mechanical imperfections may degrade spatial resolution beyond what is expected from the voxel size.

Angular sampling is another important variable. The Nyquist limit defines the rotation increment consistent with a given spatial sampling, i.e. voxel size. In (1 K)$^2$ synchrotron microCT reconstructions, the author employs 0-25° angular steps over 180° (somewhat more than 700 projections), and this appears adequate to produce sharp reconstructions. When the size of the reconstruction is doubled to (2 K)$^2$, the angular increment is decreased to 0-125°. Sample geometry has an important effect on the acceptable level of angular undersampling. For example, long, straight edges will cause worse streaking in the case of angular undersampling than they would under conditions of adequate sampling.

The dynamic range within the projections is extremely important in dictating the levels of contrast that can be retrieved reliably from reconstructions. The X-ray intensity flux through the specimen and the exposure time determine the total number of X-ray photons incident per image pixel and hence dictate the levels of contrast that will exist in a reconstructed slice provided the detector is not saturated by light photons; the condition for optimum contrast was discussed in the first
IMR review\(^1\) and is not repeated here. Not only do specimen characteristics affect contrast sensitivity, but several instrument characteristics also have a role. The number of light photons produced per transmitted X-ray photon (i.e., the fraction of X-ray photons absorbed by the scintillator and the output of light photons per absorbed X-ray photon) is an important variable, especially given that detectors have limits to the maximum number of photons that can be collected before saturation. Dynamic range in the projections (range of intensities from positions where the beam misses the sample to where the beam is most attenuated by the specimen) also is constrained by the bit depth of the area detector used (a 12 bit detector allows differentiation of 4096 levels; a 14 bit detector increase this four times). Noise in reconstructions decreases (more precisely, the signal to noise ratio increases) with increasing average counts in the projections, so use of the entire dynamic range of a 14 bit detector will improve contrast (roughly) by a factor of 2 compared to a 12 bit detector.\(^2\) Decreased noise in reconstructions can be achieved by frame averaging if the incident beam is reasonably stable: one expects four frame averages with a 12 bit detector to provide comparable signal to noise ratios as are obtained with a 14 bit detector. Because reconstructions are invalid if the beam saturates the detector at any position, incident beam inhomogeneities produce decreased contrast in slices from the dimmer areas of the beam. It should be emphasised that noise is X-ray count limited and for the very inefficient optical coupling, noise is at best limited by the detector’s full well capacity (which in turn has fewer contrast levels than one thinks because of the largely unexamined interplay between number of light photons produced per absorbed X-ray photon and the small fraction of light photons collected by the optics).

There are some tricks that can be used to preserve the required voxel size for larger than ideal sample diameters. A rotation axis placed to one side of the FOV and 360° specimen rotation doubles the specimen diameter possible for a given voxel size (Fig. 1c and d); the author has used this successfully with synchrotron microCT, and some manufacturers use this in their fan beam instruments. Local or region of interest (ROI) tomography is another approach (Fig. 1f), but discussion of these (to a greater or lesser extent) approximate reconstruction methods is postponed until the subsection on alternative tomographic methods.

**Laboratory (absorption) microCT systems**

Turnkey systems with presets for voxel size can be obtained from a number of vendors. Table 1 is a recent...
## Table 1 Commercial laboratory (absorption) microCT systems with manufacturer’s listed voxel and reconstruction sizes
as well as notes on specimen sizes: adapted from table compiled and copyrighted by Steven Cool, Radiation Monitoring Devices (used with permission) and supplemented by additional entries

<table>
<thead>
<tr>
<th>Manufacturer</th>
<th>Model (application)</th>
<th>Reconstruction size, voxel</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>BIR (Biologging Research)</td>
<td>MicroCT (specimens)</td>
<td>&lt;50 μm; 1024^2</td>
<td>[a]</td>
</tr>
<tr>
<td>Bioscan</td>
<td>NanoSPECT/CT (in vivo animal)</td>
<td>&lt;200 μm</td>
<td>[b]</td>
</tr>
<tr>
<td>Biospace</td>
<td>μ IMAGER-CT (small animal)</td>
<td>250 μm</td>
<td>[c]</td>
</tr>
<tr>
<td>Gamma Medica-Ideas</td>
<td>X-O (small animal)</td>
<td>To 43 μm; 512^2–2048^3</td>
<td>[d]</td>
</tr>
<tr>
<td>GE [e]</td>
<td>Expore Vista PET/CT (small animal)</td>
<td>...</td>
<td>[f]</td>
</tr>
<tr>
<td>GE [e]</td>
<td>Expore Locus MicroCT (in vivo)</td>
<td>27, 45 or 90 μm isotropic</td>
<td>[g]</td>
</tr>
<tr>
<td>GE [e]</td>
<td>Expore Locus SP MicroCT (specimen)</td>
<td>To 8 μm isotropic</td>
<td>[h]</td>
</tr>
<tr>
<td>GE [e]</td>
<td>Expore Locus Ultra CT</td>
<td>...</td>
<td></td>
</tr>
<tr>
<td>Nittetsu Elektronics</td>
<td>Ele Scan (specimen)</td>
<td>...</td>
<td>[i]</td>
</tr>
<tr>
<td>Phoenix X-ray</td>
<td>Nanotom</td>
<td>To 0.5 μm</td>
<td>[j]</td>
</tr>
<tr>
<td>Philips</td>
<td>vtom X-ray 240</td>
<td>To 4 μm</td>
<td></td>
</tr>
<tr>
<td>Siemens [q]</td>
<td>Inveon Multimodality</td>
<td>To 15 μm; to 4096^3</td>
<td>[k]</td>
</tr>
<tr>
<td>Skyscan</td>
<td>MicroCAT</td>
<td>To 15 μm; to 4096^3</td>
<td>[l]</td>
</tr>
<tr>
<td>Scanco Medical</td>
<td>XtremeCT (human peripheral in vivo)</td>
<td>41–256 μm; 512^3–3072^3</td>
<td>[m]</td>
</tr>
<tr>
<td>Scanco Medical</td>
<td>vivaCT 40 (in vivo animal)</td>
<td>10–72 μm isotropic; to 2048^2</td>
<td>[n]</td>
</tr>
<tr>
<td>Scanco Medical</td>
<td>MicroCT 80 (specimens)</td>
<td>10–74 μm isotropic; to 2048^2</td>
<td>[o]</td>
</tr>
<tr>
<td>Scanco Medical</td>
<td>MicroCT 40 (specimens)</td>
<td>6–72 μm isotropic; to 2048^2</td>
<td>[p]</td>
</tr>
<tr>
<td>Scanco Medical</td>
<td>MicroCT 20 (specimens)</td>
<td>8–34 μm isotropic; to 1024^2</td>
<td>[q]</td>
</tr>
<tr>
<td>Shimadzu</td>
<td>SMX-225CT-SV3 (specimens)</td>
<td>To 4096^2</td>
<td>[r]</td>
</tr>
<tr>
<td>Siemens [s]</td>
<td>Inveon Multimodality</td>
<td>To 15 μm</td>
<td>[s]</td>
</tr>
<tr>
<td>Skyscan</td>
<td>MicroCAT</td>
<td>To 15 μm</td>
<td></td>
</tr>
<tr>
<td>VAMP</td>
<td>Tornado 30 s (rapid examination)</td>
<td>80 μm</td>
<td>[aa]</td>
</tr>
<tr>
<td>Xradia</td>
<td>MicroXCT</td>
<td>1–6 μm; 1024^2</td>
<td>[bb]</td>
</tr>
<tr>
<td>Xradia</td>
<td>NanoXCT</td>
<td>50–70 μm; 1024^2</td>
<td>[cc]</td>
</tr>
<tr>
<td>Xradia</td>
<td>NanoXPI</td>
<td>&lt; 8 mm</td>
<td>[dd]</td>
</tr>
<tr>
<td>XRT</td>
<td>X-AMIN PCX</td>
<td>...</td>
<td>[ee]</td>
</tr>
<tr>
<td>X-tek</td>
<td>Benchtop CT</td>
<td>&lt;100 mm</td>
<td>[ff]</td>
</tr>
<tr>
<td>X-tek</td>
<td>BMX(HT) CT</td>
<td>5 μm</td>
<td>[gg]</td>
</tr>
<tr>
<td>X-tek</td>
<td>Venlo CT</td>
<td>Feature detection to 1 μm</td>
<td>[hh]</td>
</tr>
<tr>
<td>X-tek</td>
<td>...</td>
<td>...</td>
<td>[ii]</td>
</tr>
</tbody>
</table>

[a] Specimen diameter up to 25 mm, length up to 55 mm.
[b] One, two or four SPECT detectors.
[c] Maximum object size: 100 mm length, 90 mm diameter.
[d] Maximum object size: 97 mm length, 93 mm diameter.
[e] General Electric, previously enhanced vision systems.
[f] Specimen diameter up to 85 mm.
[g] Specimen diameter up to 40 mm. Cone beam system.
[h] Diameter up to 140 mm, long axis up to 100 mm/rotation.
[i] Examples of operating parameters given in user reports.340
[j] Diameter up to 45 mm, length to 50 mm.
[k] Diameter up to 125 mm, length to 150 mm.
[l] Diameter up to 500 mm, length to 600 mm. Other variants of this industrial system are available.
[m] Diameter up to 125 mm, scan length up to 150 mm.
[n] Diameters from 20 to 38 mm, scan length up to 145 mm.
[o] Diameter up to 75–8 mm, scan length up to 120 mm. Cone beam system.
[p] Diameter up to 36–9 mm, scan length up to 80 mm. Stacked (40) fan beam system.
[q] Diameter up to 17–4 mm, scan length up to 50 mm. Fan beam system.
[r] Diameter up to 140 mm.
[s] Previously CTI and Imtek.
[t] PET, SPECT, CT. Diameter to 100 mm.
[u] Cone beam. SPECT option.
[v] Diameter up to 68 mm, scan length up to 200 mm. Cone beam.
[w] Diameter up to 16 mm. Cone beam.
[x] Diameter up to 48 mm, scan length up to 140 mm. Cone beam.
[y] Diameters 20/37 mm or 35/68 depending on version.
[z] Diameter up to 82 mm, scan length up to 210 mm.
[bb] 0.5–1 mm for maximum resolution, 11 mm maximum diameter (9 μm voxels). Cone beam.
[cc] Diameter up to 40 mm, axial length up to 37 mm. Cone beam.
[dd] Diameters 5–12 mm; 16 slices.
[ee] Phase imaging.
[ff] SEM based instrument, phase and absorption imaging.
[gg] Field of view (20 μm)^2.
[hh] Diameter up to 50 mm.
[ii] Few details available online.
compilation of vendors and their systems, a listing that is almost certainly incomplete. Instruments range from single fan beam to stacked fan beam to cone beam systems, and manufacturers’ websites should be consulted for specifics of their systems. Commercial lab microCT systems designed for studying specimens with diameters \( \sim 10 \text{ mm} \) can be expected to produce highest resolutions with voxels between 1 and 10 \( \mu \text{m} \) in size. It is not uncommon for specimen diameters greater than 20 mm to be accommodated, although a moment’s reflection should make it clear that not all 20 mm diameter specimens can be studied (e.g. beam penetration is a problem for Ti samples). It is very difficult to discuss data collection rates without going into considerable detail as data collection rates and reconstruction times can vary widely for a single instrument depending on chosen operating conditions. Collection of data for 40 slices in 25 min is a reasonable order of magnitude figure for the highest sensitivity sampling (i.e. smallest voxel size with the largest integration time). Generally, reconstruction times for the highest resolution/highest sensitivity settings are longer than the time required for data collection, but this depends on variables such as computer architecture and number of processors, and the resulting lag builds up considerably for large numbers of slices. Intermediate resolutions and sensitivities, therefore, are often used for large datasets in order to increase throughput and also to decrease the hard drive space required to store the data.

In vivo microCT systems for imaging small animals have been available commercially for several years, and the radiation doses received by the tissue being imaged are surprisingly low. Other descriptions of in vivo systems have also appeared. Somewhat lower resolution clinical systems designed for imaging the human periphery (appendages), and hence constrained by required dose limitation, are also to be found and are termed peripheral quantitative CT (pQCT) systems.

The interplay of the different components of microCT systems has been carefully considered by Davis and Elliott. Cone beam systems are susceptible to certain classes of artefacts, and Davis suggested that improve reconstructions would result by combining data (for the same specimen) collected with short and long source specimen separations. Other systems’ development has also been described. A dedicated cryo microCT system has been reported, and at least one manufacturer’s system has been used in a subzero (C) environment. Because tube based microCT systems operate under conditions of photon starvation, there is considerable impetus to use X-ray optics to increase X-ray intensity. One lab microCT system has been developed that employs polycapillary X-ray focusing optics to increase the flux passing through the specimen without increasing penumbral blurring. Gurker et al. describe a system using bent multilayer optics for focusing; although the reported system is a pinhole design (single channel detector in the translate rotate geometry), the authors indicate that the system can also be used in a fan beam geometry.

Before an experiment using lab microCT is designed, it is important to appreciate the interplay between the different possible instrument settings and the microstructure of interest. Turnkey systems might have presets for voxel sizes based on the specimen holder diameters and for different sampling resolutions based on the number of projections collected. One instrument with which the author is familiar offers five diameter holders and three ‘resolution’ settings. An example of thorough characterisation of the dependence of morphometric indices (see cellular materials subsection below) on scan parameters in one commercial system appears elsewhere.

Because lab microCT employs polychromatic radiation, one expects it to provide lower contrast than synchrotron microCT with monochromatic radiation, and a simple example suffices to illustrate the magnitude of the effect. Suppose X-rays with photon energies between 15 and 20 keV comprise the beam used to study a specimen containing multiple (spatially resolvable) phases including Al. Because the mass attenuation coefficients \( \mu/\rho \) of Al for these ranges of energies spans 7.96–3.44 cm\(^2\) g\(^{-1}\) respectively, this significant smearing of absorptivity makes it very difficult to distinguish different phases, particularly in the presence of partial volumes, i.e. voxels partially occupied by two or more phases. Some quantitative comparisons are possible if one computes the effective energy of beam and compares the experimental linear attenuation coefficient(s) with the value(s) calculated for the phase(s) at the effective X-ray energy (see Ref. 27 for an illustration). Another example is calibration for calcified tissue, described in Ref. 28.

Low contrast may be improved by infiltrating contrast agents into specimens (e.g. brominated silane in wood, iodine based solutions in microcracks in a polymeric matrix composite, and lead chromate based polymer in blood vessels). Dual energy microCT has been applied to analysis of several ZrO\(_2\)/Al MMC\(_p\) specimens, and this approach enhanced the detectability of the particles enough that quantitative analyses of void clustering, of particle clustering and of particle-void association in fractured specimens could be performed.

For someone new to microCT, it is a daunting task to decide which microCT system to purchase (the other option of build it yourself is inadvisable unless manufacturers do not provide the required capabilities off the shelf and the builder has considerable experience with X-ray imaging and with instrumentation and software development). The recent paper by Schena et al. discusses design considerations for a custom built system, and Davis and Elliott discuss a new, custom built low noise, high definition system. As Table 1 demonstrates, there are quite a number of choices, and it is unreasonable to expect any one instrument to be able to do everything imaginable. The author would ask (and answer) the following questions: what are the ranges of sample diameters that it is essential to scan? What are the elemental compositions of the specimens of interest, and will the X-ray beam penetrate the specimen? What are the minimum dimensions of the features that are to be studied and what is their contrast? What specimen throughput is required, and what analysis software is needed? What computer platform is used? How does data archiving work? Once possible solutions are identified from websites and literature, the best answers to the above questions will be found by contacting the manufacturer expressing interest in a specific system and arranging for them to scan several specimens embodying the applications for which the system is intended. A
The second important step is to use a candidate system in person, either by visiting the manufacturer or the lab of someone with the instrument being considered. This may be the only way to verify considerations such as whether there is adequate space within the apparatus for in situ stages of various sorts. Friendliness of the software is particularly important if multiple users will use the apparatus.

**Synchrotron (absorption) microCT**

Reviews of microCT at a given synchrotron radiation source appear periodically and typically update new capabilities. Because the components required to perform microCT are readily available, many experimental stations occasionally perform microCT in response to their users’ requests. Results in the literature increasingly come from dedicated imaging/microCT beamlines, not just because they award many more shifts for microCT but also because the production facilities tailored to a small range of activities are much more efficient.

Synchrotron microCT (without lenses) is typically performed with voxel sizes between 1 and 10 μm, although routine operation with voxels sizes below 0.5 μm is possible at certain facilities and larger voxel sizes are used upon occasion at most facilities when larger specimen diameters dictate it. Design of microCT systems is driven by the portfolio of specimen types that are expected and the features within that need to be resolved. Available resources inevitably play a role in system characteristics, and constant upgrade of capabilities is the rule at active synchrotron microCT facilities. The systems of which the author is aware are highly modular, and this allows incremental instrumental improvements.

The typical synchrotron (absorption) microCT system (that uses the parallel beam directly without focusing optics) is pretty much the same as was described in the first IMR review. The essentials are: specimen rotator, X-ray phosphor (single crystal or manufactured), optical lens and CCD detector. The available components have improved in capability and affordability, and it is certainly worth a brief discussion of what has been accomplished.

Consider first the mechanical components and the physical stability that is required for high quality reconstructions. Voxel sizes down to 1–2 μm can be achieved with (relatively) affordable positioning and optical components. Reconstructions with voxel sizes down to 0.5 μm are not uncommon (and capabilities apparently exist for voxel sizes down to 0.3 μm), but required stability increases system cost considerably. The specimen rotator is the single mechanical motion during data collection with the typical rotate only synchrotron microCT system. A rotator without wobble (unintended in plane and out of plane translations from perfect circular paths) would be ideal, but measuring the rotator’s imperfections and correcting for them improves reconstruction quality considerably (see the section on ‘Reconstruction improvements’).

X-ray detector systems in most synchrotron microCT instruments consist of commercially available modules: thin single crystal phosphors, microscope objective lenses and CCD or other area detectors for optical wavelengths. Cadmium tungstate single crystal phosphors precut and polished to the desired thickness are widely used and are relatively inexpensive. These crystals provide light wavelengths with acceptable efficiencies for CCD detectors; phosphor development continues including materials formed through thin film processing routes. Radiation damage dictates periodic replacement of phosphor crystals (and optical lenses if they are in line with the direct beam or prisms if the optical lenses are placed off the beam axis). Most instruments can switch between several optical lenses for different FOV and voxel sizes (Station 2BM of APS routinely uses ×1:25, ×2:5, ×4 and ×5 objectives providing FOV of 5-4, 2-7, 1-7 and 1-36 mm respectively, when used with a 1 K detector and twice these values when used with a 2 K camera); switches require adjustment of lens focus and can be completed in a few minutes. Most area detectors are (1 K)² or (2 K)² scientific grade CCDs with depths of 12 bits; the specialised FReLoN detector developed at ESRF provides (2 K)² elements with 14 bit depth.

Significant advances in beam delivery optics include wide band pass monochromator systems based on multilayers; these produce surprisingly uniform beams and increase throughput dramatically compared to single crystal optics. The system with which the author is familiar is based on a pair of multilayer optics with areas of different layer spacings; tuning to different energies is carried out by simple translation to the appropriate positions on both optical elements. At ID 19 of ESRF, for example, multilayer optics provide ΔE/E ~10⁻² and corresponding increases in intensity compared to ΔE/E ~10⁻⁴ for an Si(111) double crystal monochromator.

Decreased voxel sizes in synchrotron microCT are typically achieved by increasing the magnification of the optical lens coupling phosphor to area detector, but there is a limit to what optical magnifications can be used. If the beam passing through the specimen is spread before the phosphor, much smaller voxel sizes can result (at the cost of decreased FOV and increased data collection times for a given brightness incident beam). Placing a perfect crystal in the beam transmitted through the specimen, orienting the crystal to diffract from a Bragg plane inclined with respect to the surface (angle of incidence less than the Bragg angle and exit angle greater than the Bragg angle) and using this diffracted beam for the reconstruction allows smaller voxel sizes for a given area detector combination. This magnification is only along one direction, and use of a second orthogonally oriented crystal is required to magnify along the second direction. Asymmetric Bragg magnifiers have long been used in X-ray diffraction topography (imaging of nearly perfect crystals using diffraction contrast), and Bragg magnifiers have been used between specimen and phosphor in microCT. There has recently been renewed interest in this approach at the third generation synchrotron radiation sources and some of these results are described below in the section on nanoCT.

Various synchrotron microCT facilities emphasise different scientific missions, time domains or spatial domains. The author’s impressions of some of these differences follow (with apologies for its incomplete, subjective nature). At DESY, the emphasis appears to be on high energy microCT and interferometer based phase imaging. The various facilities at ESRF appear to emphasise high spatial resolution, high temporal...
resolution and phase imaging with the propagation method: for example, polychromatic radiation from a wiggler source can be used from near real time microCT, down to 10 s per set of projections for one reconstruction.\textsuperscript{77} At SLS grating based phase imaging has received considerable emphasis. Reports from SPring-8 that have come to the author’s attention are centred around high spatial resolution and on phase imaging with interferometry. At APS, GSE-CARS focuses on geological applications including measurements at high pressure;\textsuperscript{78} station 2BM at APS emphasises rapid throughput (rapid reconstruction via a large dedicated computer cluster, robotic sample changer, facilities for remote access); time resolved microCT of evolution of fuel spray (5-1 μs temporal and 130 μm spatial resolution) has been achieved using the pulsed nature of the storage ring.\textsuperscript{79}

Absorption edge difference imaging can increase sensitivity to small concentrations of the element of interest and is absolutely straightforward at most synchrotron imaging beamlines. Applications include transport in low porosity materials\textsuperscript{80} and in sands,\textsuperscript{81} mapping of flame retardants (Br and Sb) in polymers,\textsuperscript{82} mapping Cs adsorption on iron oxide hydroxide particles,\textsuperscript{83} mapping new bone formation through administration of Pb or Sr labels.\textsuperscript{84} Tetrachloroethylene with 8 vol.% iodobenzene was used in model studies of organic, water immiscible phase distribution in porous water filled materials.\textsuperscript{85,86} Multienergy data collection and reconstruction algorithms have also received attention for materials where there are no convenient absorption edges.\textsuperscript{87}

Sensitivity limits to contrast agents have been investigated. Sensitivity to a fixed concentration of KI in water was clearly much better in a coarse sand (mean particle diameter $d_{50} = 0.58$ mm in a 6 mm diameter sample) than in a fine sand ($d_{50} = 0.17$ mm in a 1.5 mm diameter sample) because the larger photon flux in the former produces a much higher signal to noise ratio;\textsuperscript{88} this example is particularly compelling because the specimens are self-similar, that is, the relative sizes of pore and particle do not vary.

Synchrotron microCT data from sources such as APS, ESRF, SLS and SPring-8 seem to invariably have a strong component of phase contrast in the reconstructions. Sometimes this can lead to anomalously large values of the linear attenuation coefficient in positions within a specimen that are not easily recognisable as being near surfaces. In one study,\textsuperscript{89} such unexpected contrast (Fig. 2) was not recognised until later.\textsuperscript{89} This effect may or may not be responsible for contrast interpreted as solute segregation (e.g. high local Zn concentration in an Al engineering alloy).\textsuperscript{90} Considerable care must be taken, therefore, in the interpretation of voxel values in synchrotron microCT. In situations like those mentioned above, viewing a movie paging through a stack of slices can be very helpful.

**NanoCT**

Commercial nanoCT systems are listed in Table 1; commercial desktop systems have been described in literature.\textsuperscript{91,92} Manufacturers report voxel sizes to 100 nm and perhaps smaller; concomitantly smaller specimen diameters than in microCT are required. Scanning electron microscopes (SEMs) possesses many of the attributes required for nanoCT of small specimens, and several groups have modified SEMs for this purpose.\textsuperscript{93–95} They produce very small diameter electron beams, i.e. a very tiny X-ray source, essential for minimising penumbral blurring and for high spatial coherence for phase imaging.

Synchrotron nanoCT reconstructions have been reported using optics to provide submicrometre resolution (parabolic X-ray focusing lenses, asymmetric crystal magnifiers and Fresnel zone plates). The nanoCT system produced by X-radia employs Fresnel zone plates, for example, as well as image registration software to correct for wobble and displacement during specimen rotation. A recent study by Bay\textsuperscript{96} illustrates use of Fresnel zone plate optics to approach 100 nm spatial resolution. Nanoplates of $\gamma$-Ag$_2$Al with different [111] habit planes were clearly resolved in the age hardening Al–Ag alloy. The reader interested in more details is directed elsewhere.\textsuperscript{37,72,74,75,97–107}

**Phase contrast microCT**

The first IMR review\textsuperscript{1} mentioned microCT using phase contrast as a future prospect, and considerable progress has resulted during the intervening years. As the emphasis of this review is materials applications, neither the fundamentals of phase contrast nor the esoterica of the different phase imaging approaches are covered except as needed to illustrate the applications. Technical developments have centred in Europe (Cloetens and co-workers at ESRF ID-19, workers at the Swiss Light
Recent advances in X-ray microtomography applied to materials

X-rays are ever so slightly refracted when passing through solids (indices of refraction differ from one by a few parts per million), enough so that X-ray wavefronts distort when passing through regions of different electron density (see Ref. 108 for an introduction). With a suitable X-ray source, i.e. one with adequate spatial coherence, it is possible to detect changes in phase related contrast resulting from X-rays traversing volumes with different electron densities. Most frequently, phase imaging is performed at a synchrotron radiation source such as the Advanced Photon Source (APS); imaging can also be performed with X-ray tube sources.109

Figure 3 illustrates four methods where phase effects are used to produce contrast in X-ray images. In the propagation method (Fig. 3a), the detector is placed much farther away from the sample than is normal for X-ray imaging (~1 m vs. ~1 cm); refracted X-rays ‘r’ diverge and interfere with other X-rays at the detector plane producing detectable fringes in the image at external and internal boundaries between materials with different electron densities. Here contrast is provided by differences in the second derivative of the X-ray phase.111–117 Images acquired at four or more specimen detector separations (typically from 5 mm to 1 m or more) are required to extract the phase information,118 and this method can be described as an analogue of the focus variation method in transmission electron microscopy.114 In diffraction enhanced imaging (DEI, Fig. 3b), an analyser crystal is placed in the X-ray beam after the sample; images recorded with different settings of the analyser isolate changes in the phase angle and this method produces image contrast based on changes in the first derivative of the X-ray phase.119,120 Essentially, the analyser selects only a small angular fraction of the refracted radiation. The grating enhanced imaging method (Fig. 3c) is analogous to DEI except that contrast from changes in the first derivative of phase is provided by translation of one analyser grating relative to a second instead of by rotation of the analyser crystal and its periodic array (the crystal lattice).53,121–125

Interferometry for phase imaging is illustrated in Fig. 3d (and in the first IMR review10). In the Bonse–Hart geometry, a beam splitter ‘S’ produces a reference beam and an imaging beam, the mirror ‘M’ redirects the beams together, the object is placed in one of the beams exiting the mirror and the analyser ‘A’ recombines the reference and object modified beams.62,126–129 An alternative is the shearing interferometer.130,131 Recently, the limited FOV of interferometers from monolithic blocks of Si has recently been improved.132–134 Interferometers allow changes in the X-ray phase to be measured directly, not merely its derivatives.

At this point in the development of X-ray phase imaging, relatively little comparison of the different modalities has appeared in literature. Kiss and co-workers135 discuss image contrast numerically for absorption v. diffraction enhanced radiography. Pagot et al.136 compared radiography for phase propagation and diffraction enhanced imaging, but it is not clear how their conclusions translate to microCT. Wernick et al.137–139 and Paganin et al.140 discuss different representations of phase imaging data, but not microCT data; and Mayo et al.141 examine these representations in microCT reconstructions.

Grating based phase imaging provides an illuminating illustration of how phase microCT techniques work. Consider first the situation where no specimen is present and the spatially coherent X-ray beam passes through phase grating G1, the lines of which show negligible absorption but substantial phase shift (Fig. 3e). Note that grating G0 is typically present only with imaging with an X-ray tube with large source size. Grating G1 acts as a beam splitter, producing the two diffracted beams used for image formation. Because the wavelength of the illuminating X-rays (~10^{-10} m) is much smaller than the grating period (~10^{-4} m), the angle between the two beams is so small that the beams...
overlap almost completely as they propagate away from G1 and interfere. The interference pattern generated could be imaged directly with an X-ray detector placed at an appropriate distance $d$ from G1 (see Refs. 121 and 123 for the relationship of $d$ to X-ray wavelength $\lambda$, periodicity and other characteristics of the grating), but lack of spatial resolution of the detector systems has led to an alternative solution, use of an absorption grating G2 positioned $d_g$ away from G1. The analyser grating G2 acts as a transmission mask for the detector placed immediately behind it and transforms the local interference fringe position into signal intensity variation. Note that the gratings must be parallel.

Placing a specimen upstream of G1 produces local wavefront distortions $W(x, y)$ and alters the interference pattern. Phase imaging is performed by translating the analyser grating G2 by small increments $x_g$ of the fringe periodicity $g$ and recording a radiograph at each position. Figure 4 shows images of polystyrene spheres for different $x_g$.123 The signal intensity $I(x, y)$ at each pixel $(x, y)$ in the detector plane oscillates as a function of $x_g$, and the phases $\phi(x, y)$ of the intensity oscillations in each pixel are related to $\Phi(x, y)$ via

$$\phi = (i d_g / g_2) \Phi / \partial x$$

where $g_2$ is the period of the absorption grating.123 The phase profile of the object can be retrieved from $\phi(x, y)$ by simple one-dimensional integration (Fig. 4g). Radiographs at as few as three positions $x_g$ are needed to extract $\phi$ if one knows a priori that the intensity oscillation is sinusoidal, but the reconstructions123 were obtained using eight phase steps per projection. Once the set of phase radiographs are obtained at different viewing angles, a pure phase reconstruction can be computed using the normal methods.

Synchrotron radiation is not essential for phase microCT.108 The X-ray source size provided by the electron beam in an SEM provides adequate spatial coherence for phase microCT,94 and modifying an SEM can be an effective way of studying small specimens. The fringe formation underlying the grating method described in the previous paragraph is independent of X-ray wavelength, and, provided a grating G0 is used before the specimen (Fig. 4c) to provide a small virtual source size (more precisely, a set of independent small sources), a relatively high power X-ray tube and gratings can be used for phase microCT.109

In specimens such as foams where the majority of volume is air, the total phase shift across the specimen varies relatively little, and holotomographic
reconstruction can utilise the absolute values of the phase. In solid cylindrical Al–Si specimens such as that used by Cloetens et al. (~1.5 mm diameter), phase shifts will vary over 200 radians at 18 keV, and this dictates that the reconstructions employ the phase variations with respect to the phase introduced by the homogeneous matrix (i.e. the X-ray phase relative to that of the matrix).

Cloetens et al. provide a clear illustration of differences in absorption and phase enhanced tomography reconstructions produced with the propagation method. Figure 5 compares the same slice from an Al-Si specimen (grains of Al embedded in a matrix of very fine Al-Si eutectic) obtained under three different imaging conditions. The radiographs for the first reconstruction were absorption dominated (i.e. they were recorded with a very small specimen detector separation DS); the radiographs for the second with a single, large DS (edge enhanced interface contrast) and the radiographs at four DS were combined via the holotomography algorithm (see above) for the third. In Fig. 5a, absorption contrast does not allow one to distinguish the Al grains and Al-Si eutectic matrix. Edge enhancement allows the two phases to be seen clearly (Fig. 5b), but because the Fresnel fringe intensity varies from position to position, segmentation of the grain and eutectic phases is challenging. The reconstruction with variation in refractive index decrement (Fig. 5c) clearly shows the different metallurgical phases whose difference in density is on the order of 0.05 g cm$^{-3}$, and segmentation is quite straightforward.

In interferometer based phase microCT, the spatial distribution of polystyrene (PS) and poly(methyl methacrylate) (PMMA) in a ~50 vol.-% mixture were imaged. The polymers are immiscible (although analyses of the values of the refraction indices of both phases suggest that immiscibility is not total) and form a phase separated system. The achieved contrast resolution was, in terms of density resolution, <4 mg cm$^{-3}$, clearly beyond what is obtainable with absorption based microCT.

Phase based microCT has been used in a wide variety of studies, descriptions of which are folded into the various subsections of materials applications. A few examples are mentioned here in closing the subsection, including damage in composites and structures in biological specimens. Real time phase radiography of insect respiration produced interesting new insights, and phase microCT, providing the third dimension, will undoubtedly prove very valuable.

**X-ray microCT using signals other than absorption or phase**

Signal variations other than changes in transmitted intensity can be used as the basis for microCT. The primary and secondary topics of this section are fluorescence microCT and SAXS microCT, and, as the signals utilised might be unfamiliar to some readers, a brief introduction is provided before experimental geometries are discussed and examples of studies are applied.

Several interactions can occur between a beam of X-rays and the atoms in the specimen through which the beam passes. If the X-ray photon energy is high enough, atoms can fluoresce, emitting photons with energies characteristic of the electronic shell transition that produced the emitted photons. These characteristic X-rays have well defined energies, an energy sensitive detector can measure the intensity of each characteristic peak and this intensity can be converted to the concentration of these atoms within the irradiated volume. Detection limits (atomic concentration) for X-ray fluorescence are much, much lower than for X-ray absorption, and the elemental specificity is much, much higher for the former; these advantages continue to drive development of fluorescence microCT for applications where small concentrations of elements need to be mapped.

X-rays can also be scattered by structures with electron densities differing from their surroundings. Scattering from periodic arrays of atoms reinforces intensity along certain directions in the wide angle X-ray scattering regime, and scattering from fibrils, particles, etc., can produce peaks in scattered intensity or other characteristic scattering profiles in the small angle regime. The larger the size or spacing of the scatterers, the smaller angle of the scattered intensity.

X-ray fluorescence occurs in all directions, and the typical experimental set-up positions an energy sensitive X-ray detector to one side of the specimen in order to count the photons emitted from the irradiated volume (in the direction of the detector). Neither an area nor a
ribbon like X-ray beam appears to be practical for use in fluorescence microCT due to the confounding cross-fire from different ray paths through the specimen. One consequence of sampling along only a single ray (i.e. of using a pencil beam) is that data collection rates are quite low. Quantification requires correction for absorption of the emitted characteristic X-rays along the path to the detector, and the reader is directed elsewhere for more details. 146,147

An interesting option is to use a polycapillary focusing optic to localise fluorescence from a single position along the beam path. This appears to be a viable option for 3D mapping, an option that does not require sample rotation to provide a complete map of elemental distribution in a slice. 148

Reports combining fluorescence and phase microCT in biomedical applications (organs labelled with iodine containing contrast agents) have appeared. 149–151 Other reports of element distributions in organs include Ref. 152. Elemental maps in slices of specimens of roots have been published: K, Fe, Rb and Cl in mahogany, 153,154 and K, Fe and Zn in tomato. 35 Mapping in small particles has been of interest, both for those of terrestrial origin (fly ash particles, 153 sediment particles, 155 and diatoms 156) and extraterrestrial origin (Si, Ca, Fe and Cr maps in a microfragment of the Tahitouane meteorite, 153,155 S, Ca, Cr, Mn, Fe, Ni, Cu and Zn maps in a cosmic dust particle, 158 Fe and Ni maps in micrometeorites 159,160). Other studies include Fe nanocatalyst spatial distribution, 161 trace elements in a SiC shell of a nuclear fuel particle, 162 metal elemental maps within inclusions in diamond and quartz 148 and light elements in biological specimen. 148 Spectroscopy related to absorption edges has been used in chemical tomographic mapping. 163,166

Small angle X-ray scattering (SAXS) microCT is an ideal approach for studying polymer texture: absorption microCT shows no contrast but differences in SAXS with position can be pronounced. 166,167 The complete SAXS pattern must be recorded for a single ray through the specimen; there would be too much overlap between patterns of adjacent rays if, for example, a ribbon beam were used. In a 5mm rod of warm drawn polyethylene (PE), different layers could be resolved with SAXS microCT (see the processing subsection for more details). Tomographic reconstructions of idealised specimens using diffracted intensity from different hkl (and different phases) have also been reported. 59,168

**Alternative tomographic methods**

Region of interest (ROI) or local tomography is an approach where portions of the specimen pass out of the FOV during rotation (Fig. 1f). The effect of the missing mass can be corrected by stitching together lower resolution data for the missing areas of the project or using known sample composition and geometry and calculating corrected views. 169,170 Uncorrected local tomography reconstructions are necessarily approximate, but the extent to which their fidelity is degraded (geometry, linear attenuation coefficient values) depends on many factors. Errors will become more important as more mass remains longer out of the FOV, and one expects a priori that specimens with anisotropic cross-sections will provide the greatest problems. In general terms, the internal geometries in local reconstructions will be reproduced with good fidelity, but if there is significant mass outside of the FOV, dynamic range may be suppressed and/or linear attenuation coefficients affected. For specimens with complex, highly anisotropic cross-sections or with high frequency, anisotropic internal structure, it is essential to ascertain the extent of artefacts. 171

A number of groups/facilities routinely use local tomography. In a custom built lab microCT system, local tomographic reconstruction compared well with reconstruction with the complete FOV. 9 Local tomography is routinely used at ESRF, so much so that it is sometimes only mentioned in passing. 172 Local tomography is particularly effective in specimens with relatively low absorption such as foams; it has been applied to good effect to study deformation of an Al foam. 90

Partial view reconstruction, where an angular range of projections is unavailable, is related to local tomography in the sense that information is missing. Interpolation of the missing views from the existing data seems to produce tolerable reconstructions, 173 but this sort of approximation should be avoided if at all possible. If only one or two adjacent projections are interpolated within an otherwise complete set of views spaced 0.25° apart, one will not normally be able to see an effect in the reconstruction.

Laminography, also termed tomosynthesis, is an alternative approach mentioned briefly in the first IMR review, a method that is particularly valuable for specimens whose aspect ratios are impractical for conventional microCT (e.g. plate like specimens). Recent digital methods have been reviewed, although from a clinical and not a microimaging perspective, and Fig. 6 illustrates one method of determining 3D positions from a series of views limited to one side of the specimen. There is a cost in terms of degraded contrast by methods such as the shift and add algorithm illustrated in Fig. 6. Tomosynthesis has been applied to microscopic imaging in recent studies of perfusion, 175 of integrated circuits 4,76 and of non-destructive evaluation (NDE) of long objects. 177 In situations where displacement of well defined features can be followed versus rotation, the relative translations of each resolvable point can be converted in depth from one of the specimen surfaces. In this approach termed stereometry, use of 8–10 views allows a feature’s depth to be determined to higher precision than in simple two view triangulation; the 3D fatigue crack surface positions determined with stereometry were in excellent agreement with conventional microCT. 178,179

**Reconstruction improvements**

Reconstruction software must cope with various non-idealities intrinsic in the experimental apparatus and in the X-ray sources and return the highest fidelity reconstructions practical. An ideal microCT apparatus would have positioning component errors that are always much smaller than the smallest information containing voxel size specified in the system design. This ideal system would employ a bright, highly stable X-ray source amenable to flat field correction. It is best to collect the highest quality data consistent with the goals of the imaging experiment and any practical constraints during the problem at hand. Software sometimes can ameliorate the effects of instrument non-idealities and
from less than optimum sampling dictated by experimental requirements.

In current generations of microCT systems (employing ribbon or area beams), the only required specimen motion is rotation (translation along the rotation axis in order to enlarge the scanned volume does not affect the quality of reconstructions). Specimen wobble, described in the following paragraph, and rotation axis misalignment, can be significant sources of error in reconstructions. Reconstruction software typically uses each row of detector pixels to reconstruct a single slice (note that this is not true of cone beam reconstruction). Tilt of the rotation axis from perpendicular to detector rows brings material from adjacent slices into and out of the beam for specific ranges of angles; this degrades the fidelity of the reconstruction. Such tilts are best avoided by very careful alignment (note tilts of even 0° over 2 K pixels can shift projected data to an adjacent row), but this can be corrected by post-collection rotation of the projection (and resampling of the pixels) to align the rows precisely perpendicular to the actual rotation axis. Automatic routines for this geometric correction are described elsewhere.180

Accurate reconstruction requires that the centre of rotation be known very precisely, to within a fraction of a voxel of that in the intended reconstruction. Brunetti and De Carlo181 reported on a recentring algorithm that seems to work quite well. The approach is based on the form of artefacts from centring errors: tails of (apparent) mass extending from features like the corners of the specimen. These tails increase the numbers of voxels with non-zero values, and iterating through different trial centres for a representative slice of the volume allows one to select the ‘best’ centre for reconstructing the rest of the volume, even in the presence of beam fluctuations, noise and low contrast. Donath et al.182 developed metrics for optimisation of centre of rotation corrections. The centre of rotation can also be refined by eye, but this is impractical when more than a few specimens are being imaged.

Ring artefacts, existing even when careful flat field renormalisation has been performed, often pose problems for accurate segmentation. Correction with a median filter often does not work particularly well, and use of a purpose built filter taking advantage of the concentric nature of the rings seems to work well to minimise the rings.183 Ring reduction using 21 pt smooth to the average of all rows of the sinogram (2D plot showing transmitted intensity in the radiograph, for one slice, i.e. one row of the radiograph, as a function of rotation angle) offers substantial improvement over uncorrected reconstructions;61 the actual high frequency content of the slices do not appear to be affected, only the rings. Sinogram correction algorithms for ring reduction have also been investigated for lab microCT data.184 Ring artefacts can also be reduced by small displacements of the detector during data collection. These and other artefacts in lab microCT reconstructions have been recently discussed by Davis and Elliott185 and in synchrotron microCT by Vidal et al.186

Sasov187 used data from narrow cone beam system and compared slices reconstructed with a fan beam algorithm, a cone beam algorithm and a spiral scan algorithm. The quality of slices at the ends of the stack was compared to that at the centre, and this study should be considered by those interested in different strategies for rapid data collection.

Materials applications

Materials applications continue to be quite varied, and many of the studies reported below could be organized into a different collection of subsections. Most of the results presented below were obtained with absorption microCT, but results of phase microCT or other modalities will be incorporated where they fit into the coverage of different materials applications. Several materials microCT reviews and perspectives have appeared elsewhere,49,188–191 and these tend to have specific geographic foci, by design, in addition to covering a range of examples. Reviews of more interest to the biologist/bioengineer include Refs. 192–196.

The first subsection below is concerned with the spatial distribution of phases within specimens. Cellular solids comprise the second topic reviewed; this rather long subsection includes engineering as well as biological
cellular materials and provides some background into analysis techniques. Channel structures are the third area reviewed; these structures are complements of cellular materials. In this and the second subsection, the materials scientist/engineer can learn much from the biomedical microCT literature. In all fairness; biologists also have a lot to learn from the rigorous (quantitative) microstructural characterisation practiced by many materials scientists and engineers. Deformation, fracture and fatigue studies are covered in the fourth subsection. Materials processing is reviewed in the fifth subsection, and environmental interaction of materials is the subject of the final subsection.

**Distribution of phases**

Note that this use of the word ‘phase’ does not describe X-ray phases employed in imaging but rather refers to homogenous regions of matter bounded by a surface mechanically separating it from other phases. A relatively new application of lab microCT is evaluation of the spatial distribution of phases in pharmaceutical manufactured materials, that is, in solid dosage forms (tablets and soft gelatin capsules). Thicknesses and interface character in multilayer tablets, microstructure of rapidly dissolving tablets produced by lyophilisation (ice crystallisation followed by drying) and particle sizes within controlled release osmotic tablets are important characteristics directly measurable via microCT. Non-destructive microCT comparison of genuine and counterfeit tablets has the additional advantage of preserving the evidence in patent litigation or other legal proceedings. Pores in pharmaceutical granules have also been studied with lab microCT. Magnetic particles are being investigated for controlled drug delivery, for example, to tumours, and microCT has been used to measure these particles’ distribution in tissue of animal models.

In one synchrotron microCT study, quartz, magnetite and sanidine size distributions were measured in pumice clasts (isolated crystals surrounded by a low density matrix). This investigation was undertaken to address possible limitations of earlier work on the same material. Previous characterisation used a crushing + sieving + winnowing procedure to quantify the size distributions, avoiding stereology’s well known limitations in transforming 2D data into true measures of the 3D arrangements in the solid. Such processing, however, tends to cause significant loss of small crystals and frequently fragments larger crystals. This latter artefact is especially significant in that it obscures characterisation of fragment generation in magmatic processes. As the steps involved in the analysis of the microCT data are characteristic of those often encountered in microCT studies, they are described here in some detail.

In phase quantification studies, the first image analysis step (after reconstruction) is image classification, that is, assigning each voxel in the 3D volume to a given phase. The second step is identifying individual grains, i.e. clusters of voxels belonging to a single phase particle. In the study cited in the previous paragraph, contrast sensitivity (256 grey levels) was adequate to quantify the volumes and size distributions for particles greater than 5 voxels diameter in the Bishop Tuff pumice clast; noise limited the investigators’ ability to reliably identify smaller particles. Nonetheless, Gualda and Rivers found that the combination of contrast and spatial information allowed distinction between quartz and sanidine, despite the fact that the distribution of linear attenuation coefficients of the minority phase (sanidine) formed an indistinct shoulder of the quartz peak. The microCT results agreed with earlier results from destructive analysis, namely, the distribution of quartz particle sizes indicated action of magmatic fragmentation processes but that of magnetite was largely unaffected by the fragmentation process recorded by quartz. The trade-off between spatial resolution and contrast sensitivity is clearly explained in Ref. 200 as it affects these results, but the authors do not provide details of the number of X-ray counts recorded in the 2 × 2 binned detector pixels, and one is unable to assess the numerical extent to which contrast for a given phase is the spread because of counting statistics. It would have been interesting if the investigators had investigated frame averaging for improving contrast sensitivity and increasing the small particle detection limit. Of further interest in this carefully done study is the documentation of exceptional volumes that diverge quite markedly from the rest of the sample: this simple result should serve as a caution to all investigators using microCT of small sections derived from larger objects.

Bubble (vesicle) characteristics in basalts provide insight into various processes in magma before, during and after eruption; a distribution of bubble sizes, for example, can be due to multiple or continuous nucleation processes or differences in growth rates. Synchrotron microCT of five basalts from different locations showed bubbles were spheroidal, comprised 45 vol.-% lavas to 80 vol.-% scoria and were at least 90 vol.-% interconnected. The distribution of Cr particles in alumina, determined via synchrotron microCT, was used as the input for a finite element calculation of residual stresses arising during cooling from the processing temperature (1450°C). Hydrostatic stresses were found in the alumina near the Cr particles. In a separate paper, the authors compared calculations with actual residual stress measurements. Such residual stress measurements are readily performed with high energy synchrotron X-radiation. Similar input was used for finite element modelling (FEM) of dynamic response of porous (and epoxy infiltrated) shape memory alloy specimens.

Calculations of macroscopic properties have been performed using actual particle spatial and orientation distributions were measured in an Al–20 vol.-%Al₂O₃ composite. Finite element modelling (FEM) and mean field and multiscale modelling were used to compute various elastic properties, good agreement with experimental moduli measurements were obtained and somewhat larger fraction of particles fractured in the interior of the specimen compared to a zone 2–3 particle diameters from the surface. Sanchez et al. quantified graphite volume fraction in Al matrix composites and used the microCT determined spatial distribution of graphite to calculate flow of Al into the graphite preform and to simulate the spatial distribution of strains from deformation of the solid composite. As Heggi et al. discuss in conjunction with their microCT data on a graphite/Al composite, accurate models depend on employing representative volume elements that are sufficiently large to be representative.
of the material on a macroscopic scale (but are small enough to be tractable numerically); and these authors concluded that reasonably accurate predictions of resistivity could be obtained using ensemble averaging over a sufficient number of small models.

Radio-opaque polymers are desirable for dental applications; Anderson and co-workers employed lab microCT to show that barium methacrylate monomer did not blend well when diluted in methacrylate, whereas inhomogeneities could not be detected when tin methacrylate was used. Microdiamond content and size distribution in kimberlite are important indicators of the likelihood of finding coarser valuable diamonds; microCT has been applied to the problem of quantifying diamond content of drill hole cores, as discussed in a report of tomography research at De Beers. Void distribution determination in HY-100 steel is one of the few synchrotron microCT studies of very highly attenuating material. Catalytic conversion of natural gas to (clean) liquid fuels via Fe nanoparticles is of interest for lessening internal combustion related pollution, and the spatial distribution of these nanoparticles has been studied with fluorescence microCT.

The distribution of different carbon based phases in a composite was studied with phase microCT. The refractive index decrements of resin, carbon fibres and deposited carbon were clearly different and allowed clear segmentation of the different phases. Absorption contrast would have revealed only porosity.

Different biological tissue types can be regarded as phases in the materials sense, and the number of studies employing microCT of tissue specimens, scaffolds for implants, etc., dwarfs those of engineering materials; needless to say, the budgets are correspondingly asymmetric. Mineralised tissues such as bone (apatite, calcium phosphate, plus collagen) or echinoderm ossicles (calcite and calcium carbonate) can take the form of a cellular solid (i.e. plates and struts surrounded by soft tissue). These studies, therefore, are reviewed in the subsection of cellular solids. Likewise, studies of blood vessel and airway networks are covered in the channel structure subsection. MicroCT studies of dense mineralised tissues (cortical bone and sea urchin teeth) are distributed within other subsections, but pathological calcifications have also been studied. Quantitative comparisons based on microCT data have shown statistically significant differences between control and disease affected tissue.

MicroCT of soft tissue is mostly carried out in order to differentiate between different soft tissue types, although as recent data on brain tissue and on fixed lung sections showed, this is not always necessary for certain tissue types. Tumour tissue has been well differentiated from healthy tissue, and different normal tissue structures (e.g. mammary ducts) can also be made out. An example where in vivo absorption microCT was used to study soft tissue features (lung tumours in a mouse model) is typical of such studies: a priori information was necessary to differentiate tumours from other features (large blood vessels). Respiratory gating was used in this study of the accuracy of microCT characterisation, and further details are covered in the section on accuracy. Injection of contrast agents that concentrate in the soft tissue type of interest has been used to good effect to image murine liver tumours, but success depends on adequate contrast enhancement, a process that may be difficult to control.

Not all medical X-ray applications require full 3D information, and considerations of dose limitation and/or specimen geometry often indicate that simple radiography is required. It is worth a brief mention of the few phase radiographic imaging studies, then, because this modality may become more important clinically in the future. Digital phase mammography has received attention because sensitivity per unit dose to tumour cells or tumour precursors such as microcalcifications is much greater than with conventional mammography. Diffraction enhanced radiography of cartilage in disarticulated as well as in intact joints is quite promising, although the technical challenges of covering the FOV for human joints such as the ankle are considerable.

Cellular solids

Following the definition of Gibson and Ashby, a cellular solid consists of an interconnected network of solid struts (rods) or plates which form the edges and faces of cells respectively. In other words, faces separate two cells, and edges, are common to three or more subvolumes (cells) of the larger structure. The cells in the present context are, of course, different from the cells one normally encounters in microbiology. In 3D, the cells are polyhedra filling space and such materials are often termed foams. If the cells connect through open faces (the only material being struts at the cells’ edges), the material is termed an open cell foam; if the faces are solid, sealing off adjacent cells, it is a closed cell foam. Many cellular materials are produced by plants and animals, including wood, cork and bone. Engineered (by humans) cellular solids employ all classes of materials including composites and are used for: thermal insulation, packaging (energy absorption), structures (for high specific strengths), buoyancy (marine) and scaffolding for cell growth. The microstructural characteristics of cellular solids are difficult to quantify except with non-invasive, 3D methods such as microCT. In what follows, for purposes of simplicity, the solid phase will be discussed as if it occupies a relatively small fraction of the total volume.

As noted by Maire et al., cellular solids are often very challenging to analyse with tomographic techniques, particularly with respect to the different levels within the hierarchy of structural scales influencing the materials performance, specifically the scale of the constitutive material and the scale of cellular microstructure. The interplay among voxel size, contrast sensitivity and FOV is particularly prominent in materials such as foams. If, on the one hand, the distribution of cell sizes is important, then large voxel sizes may be required for the FOV to span the half dozen or more cells required to represent the structure adequately, and an instrument optimised for these parameters (perhaps an industrial or medical peripheral CT system) might prove more efficient than a microCT system. If, on the other hand, features within the cell walls are of central importance, then microCT or even nanoCT on small sections of the material is required. Both scales sometimes can be studied productively on the same instrument, and several investigations have employed either two extreme resolution/FOV...
combinations on a single instrument, two or more systems or local tomography techniques.

A structure like a cellular solid, i.e. a complex mixture of empty space and solid, requires several parameters in order to describe its microstructure, specifically, how much material is present and how the material is distributed spatially. These microstructural characteristics have largely been defined in studies of cancellous bone, and unbiased methods of measuring these quantities are products of these studies. The amount of material is given by the volume fraction of solid \( V_s \); in bone quantification software bundled with commercial software systems and in some literature, this is written BV/TV (the ratio of bone volume to total volume). In certain applications, the mean cell size and the distribution of cell sizes of a foam must be specified in order for macroscopic properties to be predicted. Also important is the 3D distribution of solid material, the components of which can be in the form of plates and struts (rods); these are characterised by quantities such as the surface area per unit volume \( S_V \) and mean thickness \( <T_h> \) of the structural elements. Accurate values of \( S_V \) and \( <T_h> \) cannot be derived from isolated slices unless one can assume the individual structural elements are all plates or all rods: this is one circumstance where a dataset of contiguous slices is essential. As discussed in detail elsewhere, one cannot simply make measurements of apparent thickness in the individual slices of a volume without risk of introducing significant, unpredictable bias into the data.

An approach called the distance transform method is a powerful method for determining an accurate mean 'trabecular' thickness or distribution of thicknesses. Analysis proceeds by calculating the metric distance of each solid voxel to the nearest solid (empty)space surface, i.e. this distance is the radius of a sphere centred on this voxel and fitting inside the structure. Redundant (smaller) spheres are eliminated producing a set of centres of maximal spheres filling the structure completely (Fig. 7). Thicknesses Th for each portion of the structure are twice the radii, and this allows maps of local thickness value to be produced in 3D renderings as well as \( <T_h> \) or distribution of thicknesses. Mean spacing \( <S_p> \) between structural elements is calculated with the same method by simply switching the background and object voxels. Iterative opening and closing in 3D (n erosions followed by n dilations with increasing n until no volume remains in the image: note that n erosions remove all structures smaller than 2n voxels) is the basis of another method (granulometry) of rapidly computing the distribution of wall thicknesses in a cellular material; the derivative of the remaining volume with respect to structural element size \( n \) gives the (size) distribution of thicknesses.

Additional quantities of importance include the connectivity density Conn.D, the structural anisotropy and the structure model index (SMI). Connectivity reports the number of redundant trabeculae in the structure, that is, trabeculae that can be cut without increasing the number of separate parts of the structure, and Conn.D is calculated by dividing the connectivity by the examination volume. A more complete discussion of Euler numbers, connectivity and edge effects is beyond the scope of this review as these topics are not emphasised in the examples below. Anisotropy, following Odgaard, can be defined by main directions (perpendiculars to symmetry planes in the structure) and by numbers quantifying the concentration of directions around the main direction. The fabric tensor compactly describes orthotropic architectural anisotropy via a \( 3 \times 3 \) matrix of eigenvectors giving main directions and eigenvalues the degree of concentration around main directions. Alternatively, the degree of anisotropy (DA) can be computed using the mean intercept length (MIL) method. The structure model index (SMI) relates the convexity of the structure to a model type and allows one to determine, for example, whether a given structure is more rod like or more plate like. An array of ideal (flat) plates has SMI=0, a set of ideal cylindrical rods has SMI=3 and a set of spheres has SMI=4. If sufficient 'air bubbles' are present within the structure, SMI<0.

Generally, the quantification techniques described above depend on, or at least, are implemented with,
Recent advances in X-ray microtomography applied to materials

Stock

Recent advances in X-ray microtomography applied to materials

One frequent segmentation approach is assignment by inspection: the operator examines a typical slice or slices and selects the threshold which best (to his or her eye) preserves the important fine scale features of both the solid and the surrounding empty space. Given that this is a highly subjective process, considerable effort has been devoted to assessing the robustness of conclusions derived from small shifts in threshold: the general consensus is that absolute numbers ($V_Y$, $<\Theta_h>$, etc.) will change somewhat with changing threshold, but so long as the features being quantified have minimum dimensions greater than perhaps 4 voxels, comparisons between specimens will be valid (see the subsection on microCT accuracy toward the end of this paper for specifics). The author uses this trial and error method but with the following precaution: the threshold is chosen based on evaluation of a preliminary subset of specimens, a subset explicitly excluded from the actual statistical comparison of different treatment groups.

Before other thresholding approaches are described, it is useful to examine an example of determination of quantities such as $<\Theta_h>$, $<Sp>$ and SMI for a cellular solid, the mineralised tissue of the sea urchin. The skeletal elements of sea urchins are termed ossicles and consist of calcite, i.e. calcium carbonate. In all but a few specialised cases, all of the ossicles are single crystals, despite having complex external and internal geometries reflecting their different, highly specialised functions. The design motif of sea urchin calcite (except in teeth) is a highly porous structure ($\sim 50$ vol.-% occupied by soft tissue and fluid) that is termed stereom. Stereom has its local minimum at the boundary between cell faces and edges and for Ti particle

One alternative thresholding method is use of the gradient in greyscale value to define the boundary between phases. Adaptive or dynamic thresholding, which explicitly accounts for varying background, is another approach. In structures where the solid phase is particularly thin and difficult to resolve reliably, the boundary between cells can be determined by a distance transformation plus watershed algorithm, as, for example, in the study of evolution of a liquid foam described below. In specimen systems where partial volumes and low contrast dominate, selective iterative thresholding may be useful: here an initial threshold is guessed and the results of this binarisation are numerically compared to those of different iterations, but it is not clear exactly how this had been implemented. Segmentation by ‘snake’ or active contour models for boundary detection can also be effective: an initial contour is deformed towards the boundary to be detected so as to minimise a functional designed to have its local minimum at the boundary. Spowage et al. described the steps required to identify surface porosity in foams. Lindquist explicitly compares three quite different thresholding methods, and the interested reader can take this paper as a starting point for learning more about thresholding.

Quite a number of investigators independently developed analysis tools for cellular or highly porous materials that are not too different from the algorithms described above. Considerable effort could have been avoided by checking engineering (e.g. Compendex) AND medical (e.g. Medline) databases for various synonyms for microCT and for the type of structure of interest. When the results of a study are evaluated using independently developed methods, it is important to establish whether the algorithms evaluate quantities only slice by slice or whether via a true 3D analysis; 3D analysis algorithms may not be important for quantities such as volume fraction, but are most certainly essential for thickness measurements.

Static cellular structures

Before covering evolving cellular structures and biological/biomedical structures in detail, it is useful to illustrate the wide variety of cellular materials to which microCT has been applied. Porosity and grain structure in wood have been studied. Bubble size distributions in simulated or actual volcanic silicate foams have been quantified with synchrotron microCT, and 2D and 3D measures of the distributions were compared and related to models of bubble growth. Open cell aluminium foams have been used as substrates/materials sources for zeolite catalyst growth, and lab microCT was used to characterise the starting cell diameters and strut thicknesses as well as to show that the zeolite film had homogeneous thickness. Pd–Ag/SiO$_2$ xerogel catalysts supported on Al$_2$O$_3$ foams were also characterised by microCT. Pore initiation by blowing agents in metal foams has been studied by synchrotron microCT, and pores in prealloyed Al powders nucleated around the blowing agent particles, whereas they tended to nucleate around Si particles in Al–Si powder blends. In a more well developed, closed cell foam produced by the blowing agent route, a large number of 3D parameters were quantified for pores (volume fraction, equivalent radius, surface area and sphericity), for the distribution of cell wall material (via granulometry) between cell faces and edges and for Ti particle geometric parameters and size distribution. Brunke et al. noted their intention to quantify the 3D spatial
distribution of Ti particles and to relate this to foam morphology and produced a simple representation, projection of the mass within each slice along the length of the cylindrical sample, which sufficed to show the principal foaming direction. Different foaming times also altered the distribution of cell wall material.236

8 Synchrotron microCT data for interambulacral plate of a Lytechinus variegatus and b–d demipyramid of Astenosoma varium. a Slice with plate exterior at top: inset box shows magnified section of ossicle indicated by arrows and horizontal FOV is 550 voxels (2.75 mm). b Slice with enlarged area inset. Black box defines ROI for numerical microstructure evaluation; pixels are binarised to either calcite (white) or void (black). Horizontal FOV is 687 voxels (3.44 mm). c Grey scale and d thresholded 3D renderings from within ROI defined in b: volumes are both 37 voxels (0.185 mm) high. In a–c, lighter pixel, higher linear attenuation coefficient. In d, higher absorption voxels are shown solid and lower values are rendered transparent. Reprinted from Ref. 242 with permission
X-ray tomography (using a fairly large voxel size, 90 μm, dictated by the required FOV for a material with cells up to 5 mm diameter) of different structural metal foam types revealed differences in cell anisotropy (distribution of cell volumes and of aspect ratios quantified as equivalent ellipsoids) stereographic projections showing distribution of cell axes versus orientation) that correlated with altered mechanical properties. Defects in the cell walls (corrugations, holes and cracks), however, substantially reduced Young’s modulus and strength from theoretical values. Another lab microCT report of a metal foam was followed by detailed FEM of foam deformation (see below).

Synchrotron microCT of polyvinylchloride foam (an example where cell diameters were smaller than 0.2 mm) showed that a higher processing temperature produced higher volume fraction of porosity, larger equivalent cell diameters, decreased wall thicknesses and decreased degree of anisotropy; further, selection of small sub-volumes produced the same results as for larger volumes of interest. Glass foams can be produced from silicate wastes (power generation waste plus bottle fragments plus a SiC foaming agent) and used for thermal or acoustic insulation, etc., a process of considerable interest given the amount of this waste that has been accumulating for years. Lab microCT has been used to quantify as equivalent ellipsoids; stereographic projections showing distribution of cell axes versus orientation, etc., a process of considerable interest given the amount of this waste that has been accumulating for years. Lab microCT has been used to relate foam structure and properties with processing temperature.

**Temporally evolving, non-biological cellular structures**

The 3D evolution of liquid foams over tens of hours has been studied by synchrotron microCT and the progress of drainage and bubble coarsening posed particular problems in this study. While the minimum time between each set of projections for a single reconstruction was 8 min (10 mm FOV, 10 μm voxels, 50 ms exposure per projection and 900 radiographs per dataset), nearly instantaneous changes such as bubbles bursting or moving produced occasional reconstruction artefacts. Despite an inability to image a significant fraction of thin film area, segmentation of the image into connected pores and finite element modelling of the structure. Because the pore diameters and mean cell wall thicknesses were ~2 mm, it is difficult to know whether the results of interrupted testing differ from those obtained during continuous loading. The lab microCT studies of Nazarian et al. however, found the results of stepwise compression of several types of cellular materials agreed well with data from conventional continuous testing.

MicroCT observation of deformation of cellular solids is frequently compared to FEM of the deformation based on the initial (microCT determined) structure. In principle, this allows the step by step FEM predictions of deformation process to be compared with experiment and assumptions in the FEM to be refined. The steps involved in meshing microCT for FEM are covered elsewhere, and the reader is advised to search the bone literature for further discussion of approaches for cellular material.

Several microCT studies of elastomeric foams have appeared and include both open and closed cell materials. These studies were aimed at improving understanding of deformation processes underlying the three stages of the typical elastomeric foam stress–strain curve (linear elastic stage I at the lowest stresses transitioning into a plateau region, stage II, where little increase in stress produces large increases in strain and finishing in a densification regime, stage III, of rapidly rising stress). Kinney and co-workers studied 1 mm thick, 15 mm diameter silica reinforced (~25 wt%) polysiloxane foam pads. Synchrotron microCT of this closed cell material was performed at four deformation levels from 0 to 35% strain, and analysis concentrated on the fraction of surface connected pores and finite element modelling of the structure. Because the pore diameters and mean cell wall thicknesses were ~1/3 and ~1/10 the specimen thickness, it is difficult to know whether the results can be applied to other geometries.

Plougoven et al. studied a polypropylene foam subjected to alternating cycles of interrupted shocks followed by synchrotron microCT imaging; this simulates energy absorption behaviour during crashes. The investigators concentrated on behaviour at the mesoscopic scale of grains (1–3 mm diameter within the 10 mm diameter specimen), and the hierarchy of structural scales of pores in the foam complicated the analysis. Phase contrast effects were suppressed before filtering suppressed noise, and a distance transform operation defined the cell walls. The relative densities of the different grains were then computed as a function of deformation.

In an open celled polyurethane foam, Elliott and co-workers imaged the structure at 14 strains with the observations clustered at the critical regions of the stress–strain curve (i.e. the transition between linear...
elastic and plateau regimes, the transition between plateau and densification regions and in the final stages of densification). Their focus was on strut deformation processes, and they employed local tomography to observe the central ~7 mm of a 25 × 25 mm high specimen. The initial stages of compression (below 4% strain, in the linear elastic regime) were taken up by small amounts of bending in struts that were both longer than average and inclined to the compression axis. More severe bending and reorientation of struts within a localised deformation band accommodated strains up to ~23%. After multiple collapse bands have formed and impinged on one another, the densification regime has begun. Node and strut models of the deformation were applied, but further improvements were found to be necessary.  

The microCT study of a closed cell polyurethane foam stands in contrast with what described in the previous paragraph. Synchrotron microCT (local reconstruction) was also performed on the central portion of a larger specimen (central ~2 mm of a 6 mm diameter specimen) at four strains (maximum of 20%), and the investigators performed a thorough sensitivity analysis of FEM parameters and used FEM to fill in the gaps between microCT measurements. Direct comparison of FEM derived strain distributions in the actual foam structure showed cell wall rupture occurred at local strain concentrations.

Metal foams often fail at significant lower stresses than expected from theory, and this has motivated microCT imaging of foams after discrete increments of compression. Even simple analysis methods and modest spatial resolution can provide informative results. Deformation of two fairly dense Al ‘foams’ produced by powder metallurgy (46 and 37% of full density), for example, was quantified by measuring mean porosity as a function of slice number (compression along the tomographic rotation axis), much like what was carried out earlier for density gradients in chemical vapour infiltration or more recently for liquid foam. Displacement of local maxima/minima in mean slice porosity could be followed through the different increments of deformation as the porosity diminished by up to a factor of 2. Correlation coefficients for cross-sections between two deformation states were never lower than 0.55, and the data confirm the phenomenon that the higher the cross-section porosity, the greater the deformation of that cross-section. Similar approaches were used by others for an Al foam and for compression of a glass wool.

Medical CT (in plane voxel sizes a significant fraction of 1 mm and out of plane voxels even larger) can reveal some details of wall deformation of closed cell Al foams and can identify sites of weakness, but higher spatial resolution is required to produce data that can be incorporated usefully into numerical or analytical models. MicroCT of indentation tested, low density Al foams (between 0-06 and 0-17% of full density) revealed that the deformation zone was confined to a spherical cap immediately under the indenter with only occasional buckling of cell walls farther than one cell diameter from the zone directly under the indenter. Finite element modelling based on the reconstruction agreed with analytical expressions for the size of the deformation zone.

Significant deformation occurs during the manufacture of open cell Ni foams used as battery electrodes, and synchrotron microCT of these foams has proven very useful in characterising tension and compression damage. In tension, bending, stretching and alignment of struts were observed, and the particulars depended on the initial anisotropy of the foam. Compression led to strain localisation due to buckling of struts, and this differed from the large rotations observed under compression in polyurethane.

Lab microCT of two syntactic metal foam types (preflows of hollow ceramic spheres infiltrated by liquid metal of two different Al alloys) was used to characterise deformation modes and to interpret features in stress–strain curves. The foam with commercial purity Al showed barrelling over a large part of its area with uniform, rather modest plastic deformation of the matrix coupled with sphere fracture and a thin crush band. The foam with 7075-T6 matrix had damage concentrated in two intersecting and much thicker crush bands oriented ~45° to the compression axis; sharp stress drops after the peak stress resulted from the formation of the crush bands.

Examining the behaviour of the foam and of the foam sheet interface in Al sandwich material required microCT at two very different scales. Nonetheless, microCT allowed strains to be calculated based on measurements at four deformation states. The interface appeared to be sound, and the foam in the sandwich behaved like similar free standing foams examined by this group.

Repeated observations (after different compression increments) with synchrotron microCT revealed damage accumulation in the cell walls and plateau borders of an Al foam. Local tomography was required to provide adequate spatial resolution within the volume of interest while allowing the specimen tested to have a large enough cross-section to be representative of the foam. Displacement of thousands of micropores within the solid were tracked automatically for each increment of deformation, and a microstructure gage formalism was used to calculate maps of the 3D strain tensor components. Characteristics of pores and the micro-cracks that nucleated from them were quantified for plateau borders and cell walls, and the distribution of strain was discussed in terms of these features. The microCT derived 3D structure was imported into FEM. Similar data are required in applications in medicine, for example, displacements in CT images of lungs through image warping.

**Fibrous network solids**

Fibrous network solids are a somewhat different class of cellular materials than foams. Cellulosic fibrous networks, for example, are encountered in papers and in engineered, low density, wood based fibreboards. Synchrotron microCT of low, medium and high density fibreboards was used to determine fibre network characteristics and to generate a realistic, ABAQUS based model for calculating thermal conductivity, a property important in construction materials; a design of experiments (DOE) analysis of the model parameters revealed that fibre density is responsible for 60% of local
conductivity of the network and fibre orientation and tortuosity represent 25% of the influence of network conductivity.\textsuperscript{296}

Walther \textit{et al.}\textsuperscript{297} examined various medium density fibreboards with synchrotron microCT and provide a particularly clear account of the challenging image analysis needed to differentiate the fibre volume from the surrounding air. With a voxel size of \(2.3 \, \mu m\), the lignocellulose fibre walls and the hollow lumens are clearly resolved; both need to be included in the fibre volume but real and apparent (noise related) breaks in the walls were a complication that was overcome. The maximum fibre thickness was \(14 \, \mu m\) (6 voxels), so the analysis (quantification of fibre diameters and related quantities) should be regarded as quite robust. Quantities such as volume fraction (fibre walls, lumen volume and interfibre air) and total surface area per unit volume (fibre lumen and fibre outer air) were quantified as a function of specimen density. Individual fibres and fibre fragments and their orientations were quantified, and fibre bundles (groups of fibres each with contact areas>10\(^4\) \(\mu m^2\)) were mapped. Lux \textit{et al.}\textsuperscript{298} provide similar analysis (but with different image analysis tools) of synchrotron microCT data for a very low density fibreboard and found that the resulting density agreed with that measured macroscopically. Synchrotron microCT studies of paper have also appeared,\textsuperscript{299,300} in which paper’s extreme sensitivity to changes in humidity was during foaming.\textsuperscript{307} phenolic foam incorporating short glass fibres revealed short fibre reinforced foams, and lab microCT of a distributions are also important to the behaviour of this data could certainly be imported into numerical models. Contaminant classification in cotton fabrics is another area where microCT was applied.\textsuperscript{304}

The relatively good environmental stability of metals makes metal fibrous networks attractive for heat transfer, filtration and catalyst support applications. Lab microCT and skeletonisation analysis were used to study two bonded stainless steel fibre assemblies.\textsuperscript{305} The distribution of fibre segment lengths between the two specimens differed as did the distribution of fibre orientations (shown on stereographic projections), and this data could certainly be imported into numerical models of stiffness (or other quantities) as in Ref. 306. Determination of fibre orientation and fibre length distributions are also important to the behaviour of short fibre reinforced foams, and lab microCT of a phenolic foam incorporating short glass fibres revealed fibre preferred orientation attributed to shear generated during foaming.\textsuperscript{307}

Plant roots can be regarded as a fibrous network and can be analysed with approaches similar to those described above. For example, Kaestner \textit{et al.}\textsuperscript{308} studied root networks in two older specimens.

\textbf{Mineralised tissue and biomedical applications}

Mineralised tissue is an important naturally occurring biomaterial that has been studied extensively with microCT. Indeed, a number of commercial lab microCT systems were optimized for studying the mineralised tissue of the widest clinical interest, bone. In this subsection of cellular materials, the focus is on cancellous bone, that is, spongy or trabecular bone, and on analogous skeletal tissues such as the stereom of echinoderm ossicles. Cortical bone and tooth contain much smaller fractions of open volume, and microCT studies of these materials are described elsewhere in the review.

Aging populations are characteristic of countries in the developed world and have motivated, at least in part, microCT studies of bone. Osteoporosis is a disease of increased bone fragility seen in both elderly females and males, and the classical view is that decreased bone mineral density (BMD) explains fracture susceptibility. This paradigm fails in a significant fraction of patients, and the first refinement to this model is the proviso that decreased bone competence may also be the product of inferior bone microarchitecture, that is, impaired networks of struts and plates of the trabecular bone, a major constituent of vertebral and femoral head, both major risk sites for osteoporosis related fractures. Quite some time ago, it was realised that microCT was an ideal method for characterising bone microarchitecture, and microCT studies of bone occupied a significant fraction of the first IMR review.\textsuperscript{1} Biopsies of human tissue or whole limbs of small animal models continue to be a major subject in microCT materials characterisation. A few examples of such studies follow.

MicroCT has examined trabecular bone loss accompanying steroid treatment (for inflammation).\textsuperscript{309} Ovariectomy (OVX, mimicking estrogen loss at menopause)\textsuperscript{310,311} or sciatic neurectomy\textsuperscript{312} and gastrectomy\textsuperscript{312}. Post-menopausal osteoporosis is often treated with bisphosphonates, and several studies of bisphosphonates’ effects on bone loss have been reported for animal models.\textsuperscript{313-316} Parathyroid hormone (PTH)\textsuperscript{317-320} or fibroblast growth factor (FGF)\textsuperscript{320,321} treatment of osteoporosis models has also been investigated. Patterns of bone resorption and the effect of dose were examined for calvarial culture and IL-1 and PTH treatment.\textsuperscript{247}

Effects of disease processes on bone (infections, tumours and arthritis) have also been examined with microCT.\textsuperscript{322-328}

Synchrotron microCT and synchrotron X-ray diffraction were used to study the microarchitectural and physical changes of human vertebrae during fetal growth.\textsuperscript{329} Interestingly, the trabecular bone network was denser than in adult vertebrae, perhaps in compensation for the immature cortical shell in the fetal bone. An important maturation stage in the long bones of many animals is the fusion of the growth plate and until microCT was applied to the problem, it was unclear whether epiphyseal growth plates remained open throughout life in rats, a popular skeletal (trabecular) model for aging humans. Rats are not good models for bone remodelling (replacement of impaired bone by the
action of osteoclasts and osteoblasts does not occur except under unusual conditions, and, if their bones never ceased to extend however slowly, this would impact the usefulness of this model. The microCT data of Martin et al. suggest that growth plate fusion does take place in rats and the fraction closed follows a sigmoidal pattern.

Changes in snail shells during growth were studied through in vivo microCT. MicroCT has also been used to study an extreme case of growth, the regeneration of limbs in adult newts.

Bone structure is expected to change in response to in vivo loading. Craniofacial joints, for example, are expected to change in response to different forces encountered in eating (i.e. to exhibit adaptive plasticity) and to attachment of different sized muscles. MicroCT was used to examine such changes in rabbits (animals eating a hard diet versus those on a soft diet), in myostatin deficient mice versus normal mice (the larger than normal muscle mass of the former has led to informal label ‘muscle mice’) and in subfossils of the extinct primate Archaeoeluridae. One expects differences in bone microarchitecture for animals that are phylogenetically close relatives but which have different modes of locomotion. MicroCT analyses of trabecular bone in the femoral head and neck for two primate genera showed no difference in cancellous bone volume density but did show differences in trabecular orientation. Changes in bone loading history are easily imposed in rat and mouse models and form the basis for a number of microCT studies.

The material bone is a discontinuously reinforced composite of apatite (mineral) nanocrystallites dispersed in a matrix primarily of collagen, and it is important to realise that in many genera, the mineral content varies considerably from older, mature areas (highest mineral content) to newly remodelled osteons (lower mineral content). The sensitivity limits of microCT make it ill suited for quantifying small differences in composition, but a number of studies have shown that the mineralisation levels in bone can be mapped. Synchrotron microCT of low mineralised versus high mineralised volumes in trabecular as a function of bisphosphonate mineralisation levels in bone can be mapped. Synchrotron microCT of low mineralised versus high mineralised volumes in trabecular as a function of bisphosphonate treatment was the subject of one study. Microradiography of thin sections of bone has long been used to show remodelling, and determination of the degree of bone mineralisation via this method has been compared to that with synchrotron microCT. Synchrotron microCT has also been used to study the degree of mineralisation in human normal vertebra, osteoblastic metastases and sites with degenerative osteosclerosis and in different mouse genetic strains.

Strength in bisphosphonate treated bone has been characterised along with bone microarchitecture in OVX rats. Elastic moduli from indentation measurements have been determined for PTH and FGF treated bones and structures compared with microCT. Bone modelling and simulation was the subject of another study. MicroCT derived bone microstructures are routinely imported into finite element models and the response to mechanical loading investigated numerically. Imaging of entire vertebrae, for example, allowed, via importation into FEM, assessment of the contribution of cortical and trabecular structure to strength; this study also compared actual and numerical estimates of yield stress for OVX versus control versus OVX plus alfalcacidol treated rats. Importation into numerical models is particularly valuable in highly variable biological systems because the same starting structure can be perturbed virtually and the response determined numerically.

Several in vivo and in situ microCT characterisation studies have appeared. Multiple observations of rat bone by synchrotron microCT is one example, and this approach has been extended to mice recently. The reader should consult the latter study for a nice discussion of the relationship between X-ray dose and signal to noise ratio in the reconstructions. Observation of changes in trabeculae in the murine hindlimb unloading model is another example. Damage accumulation in cancellous bone specimens has been followed during in situ loading. In situ straining of bone was the subject of another study.

 Determination of longitudinal changes in trabeculae by direct comparison of the reconstructed volumes was a rather more ambitious approach employed in an in vivo lab microCT comparison of OVX and control rats. In this study, image registration (translation and rotation between volumetric datasets) was performed to maximise mutual information. The registration algorithm worked well for the controls, for which changes were very small, but the large changes in the OVX animals required a modified approach based registration of a relatively small number of large invariant structures. Clear patterns of bone resorption and apposition were documented for the OVX rats, and new bone formation was shown to correspond with areas of calcein labelling. These results highlight the difficulties inherent in direct registration approaches and suggest that there continues to be real value in comparison of overall 3D specimen maps [of a particular quantity such as crack opening instead of point by point matching (subtraction) of features]. It is also possible to administer Pb and Sr to living animals in order to label the bone in much the same way as fluorochromes such as tetracycline or calcein as used; imaging to either side of the absorption edge increase sensitivity in synchrotron microCT studies.

A prominent feature of clinical radiographs is the texture produced by the (partially) overlapping trabeculae at the ends of long bones and within vertebrae. An obvious question is whether analysis of the texture can provide useful information about the trabecular microarchitecture, and this has, in fact, received attention. Luo et al. used synchrotron microCT to show that plain radiographs contain architectural information directly related to the underlying 3D structure; they suggested that a well controlled sequence of radiographs might allow monitoring of trabecular changes in vivo and identifying individuals at increased risk of osteoporotic fracture. Later synchrotron microCT based analysis by others examined which 2D texture parameters correlated best with the underlying 3D structure. This approach may prove useful for analysis of other cellular solids, for example, metal foams undergoing high strain rate deformation (with complete tomographic reconstructions only possible of the initial and final states), but much more detailed descriptions of the radiograph’s texture need to be developed (instead of...
one or a few global measures of texture, a dozen or more parameters might be appropriate) and a priori knowledge could be utilised (for this example, the known initial structure and corresponding radiograph, the final structure and radiograph and the fact that the \(i\)th radiograph resulted from the \((i-1)\)th structure and evolved into the \((i+1)\)th structure).

Bone formation around implants and the resulting structural integrity (or lack thereof) is a biomedical engineering topic of increasing importance as the number of hip (and knee) replacements increases. Examples of microCT studies include the 3D analysis of bone formation around Ti implants,\(^{363}\) characterisation of Mg bone implant degradation,\(^{364}\) study of bone around dental implants\(^{365}\) and investigation of human tooth alveolar bone complex.\(^{366}\) One complication in many such imaging studies is that the high absorptivity of many implants such as stainless steel or Ti washes out contrast in bone. In the author's experience, this can cripple analysis if contrast is confined to linear, 256 level greyscale; such an 8 bit approach seems to be favoured in many different analyses, so this is not an academic concern. Use of non-linear contrast scales or high end clipping can be effective; contrast enhancements for bone structures beside implants are described elsewhere.\(^{367,368}\) In synchrotron microCT, phase contrast at edges of different low absorption tissue types may also aid segmentation in the presence of a very absorbing material.

Not all mineralised tissues are based on the collagen–apatite composite system that comprises bone: echinoderms, for example, utilise calcite and a trabecular structure reminiscent of that in bone. The illustration of \(<\text{Th}>\) determination above was for the stereom in a demipyramid of \textit{Asthenosoma varium}. Sea urchin spines fill a rather different function (they protect the animal's body from predators and high energy, rough surf, environments), and the spines of different phylogenetic families have different characteristic architectures as well as a wide range of lengths and diameters. In addition to stereom, spines can contain radial wedges or a dense cortical shell, and reports of structural analyses have appeared recently.\(^{369,370}\) Numerical analysis of the structures revealed by microCT may shed light onto why certain characteristic structures have persisted over millions of years, providing insight into functional advantages conferred by certain structures. Figure 9 shows the complex 3D spine structure of one type of sea urchin.
urchin: the radial wedges and hollow centre concentrate mass where it will be the most effective in resisting bending, and the bridges between wedges and the perforated central cylinder link the wedges. This structure is a single crystal, and the exquisite biological control of mineralisation geometry and crystallography at ocean temperatures is even more remarkable because the calcite’s high Mg content grows at equilibrium only about several hundred degrees Celsius. Several studies suggest that spatial constraint of the mineralisation space controls single crystal calcite growth in sea urchin ossicles. Materials processing employing this strategy and suitable macromolecular crystallisation adjuncts appears to be a very attractive route to improved cellular solids.

MicroCT imaging of cellular mineralised tissues is useful even when the microstructure cannot be resolved. The finest stereom found in echinoderm ossicles can have submicrometre dimensions, for example, and nanoCT would be required. Often the ossicle dimensions are up to a centimetre, and there is a strong desire to avoid destroying the specimen. In studies of functionality of ossicles and of phylogenetic changes in stereom microarchitecture, the 3D distribution of stereom fabrics is of great interest and is largely unexplored for ossicles other than test plates. Stock and co-workers379 used lab microCT to image a demipyramid of the sea urchin *Lytechinus variegatus* at a resolution too coarse for the trabecular structure of stereom to be resolved. Because this ossicle consisted of only two phases (high Mg calcite and air), the 3D distribution of linear attenuation coefficient values was interpreted in terms of partial volumes of calcite. The complex structure revealed by microCT probably reflects the conflicting mechanical requirements (increased mass for rigidity versus decreased mass for metabolic savings) and the need for transport to keep cells viable in the stroma (interstitices of the stereom). The structure revealed by microCT certainly could be imported into a finite element model and the role of the different features probed numerically, but this remains to be carried out.

Scaffolds for bone replacement via osteointegration have been actively researched, and microCT is a natural tool for studying these structures that generally mimic trabecular bone. As this is a very active field, only a few examples are cited here. Biocompatible materials including scaffolds were discussed in Ref. 375, and other reports of scaffold structures and function include Refs. 44 and 376–379. Quantification of bone on hydroxyapatite scaffolds380 and 3D study of bone ingrowth into calcium phosphate biomaterials381 may be of interest to the reader. Bone formation in polymeric scaffolds has been evaluated by proton magnetic resonance microscopy and microCT.382 The fluid dynamic microenvironment in tissue construct has been considered based on microCT data.383 Other studies include pore interconnectivity in bioactive glass foams, structure and properties of clinical coralline implants,385 scaffolds of carbonated apatite-collagen sponges386 and polymer composite scaffolds.387

**Channel structures and cortical bone**

Channel structures are simply structures complementary to those of cellular solids: once again, the volume of interest occupies the smaller fraction of the total, but here the empty space (or other phase filling the channels) is of interest. Many analyses used for cellular solids can be and are used for channel structures with the solid and empty space subvolumes inverted.

Fluid content and drainage were studied in a wide range of porous specimen sizes using an industrial CT system [76 mm diameter with (368 µm)³ voxels], a medical CT scanner [76 mm diameter with (150 µm)² x 2 mm voxels] and synchrotron microCT [27 mm diameter with (76 µm)³ voxels down to 1.5 mm diameter with (6.7 µm)³ voxels]. Note that similar comparisons were performed for trabecular bone337 and for bone containing Ti implants.388 The industrial and medical systems could detect heterogeneous drainage patterns but not the pore spaces. In coarse sand, synchrotron microCT could partition the specimen volume into KI doped water, air and solid and reliably detect different interfaces; changes in fluid distribution after drainage could also be imaged clearly.341 Contrast sensitivity for water in pores (undoped and doped with tracers such as KI) was established in other work30 and is discussed below in the section on microCT accuracy. Other synchrotron microCT studies of interfaces and water saturation include Refs. 389 and 390.

Sheppard et al.355 characterised a number of materials with porosities between 18.5 and 54% using microCT and either compared these determinations to results from other techniques (see section on microCT accuracy below) or used the data as input to fluid transport or other models. In addition to quantification of grain size, these authors looked at grain coordination number in different types of specimens, pore connectivity and local flow paths. Similar analyses were performed by Knackstedt et al.391 on eight different industrial foams (microcellular polyurethane formed by four different processes), including thermal conductivity, permeability and elastic properties (Young’s modulus). The same group describes a vertically integrated centre for the analysis of transport (and mechanical) properties in a wide class of solids.392

Polymer foams can be used to absorb oil from spills, and the ingestion of fluids into cellular materials such as polyurethane foams has, therefore, considerable practical importance but has received relatively little attention. Oil uptake (two densities of oil) of polyurethane foams (three densities) were examined for two temperatures (simulating winter and summer seawater temperatures) using lab microCT.393 Weight uptake as a function of time was the principle measure of polyurethane foam performance, and a few reconstructions and 3D renderings of the as received foam were provided. Unfortunately, the foam structure was not analysed, structure was not correlated with performance nor were reconstructions presented of the foam with absorbed oil.

Blood vessel structure in organs is an important topic in medicine and biomedical engineering, and microCT based analysis of these systems of channels is an efficient quantification method. The approaches developed can be directly translated to materials problems. Ritman and co-workers9,394–397 described blood vessel quantification for different organ systems including coronary arteries, hindlimb vascular trees, kidney glomerular microvasculature and biliary trees. The hundreds of branches present in a typical organ are typically filled with a contrast agent, but phase imaging can also be used to
image blood vessels without contrast agents.\textsuperscript{398,399} Automated analysis routines are required, however, if an adequate number of replicates are to be analysed. This last requirement is particularly important in biological studies where interindividual variability is very large. In common with analyses of cellular structures, the first step in the analysis is extraction of the vessels from the image and suppression of noise. It is important to note the tree like nature of the vessel system and branches because this differs from the situation pertaining in cellular materials and conditions analysis algorithms. After extraction of the vessel tree, quantitative data can be computed, and numerical analysis of the tree characteristics can be performed (e.g. partition into mother/child sibling relationships for the different branches that focuses analysis of functionality and dimensional changes onto equivalent portions of the network).

Wan et al.\textsuperscript{31} studied the coronary arterial tree of a rat and focused quantification on arterial lumen cross-sectional area, interbranch segment length, branch surface area at equivalent generation, interbranch and intrabranch levels. In coronary circulation, arterial blood inflow increases during diastole and venous outflow increases during systole; direct comparison of microvessels in the myocardium for diastolic arrested and systolic arrested hearts allowed investigators to identify the characteristics of the capacitance vessels.\textsuperscript{400} Pulmonary arterial wall distensibility, decreases of which are important in various lung pathologies, was examined with a liquid contrast agent injected into pulmonary arteries and imaged with lab microCT at different arterial pressures spanning the physiological range.\textsuperscript{401,402} main arterial trunk diameter versus distance from the inlet was determined for each of four pressures, and a linear relationship between diameter and pressure was found for a single point on one vessel. This same group used the self-similarity of the arterial trees to improve analysis efficiency\textsuperscript{403} and employed these tools to compare vascular remodeling for hypoxia treated rats with rats living under normal oxygen partial pressures.\textsuperscript{404} Bentley et al.\textsuperscript{405} reviewed the use of microCT to study alterations in renal microvasculature caused by development of cirrhosis in a rat model; they found changes in microvascular volume fractions in different portions of the kidney that may contribute to changes in salt and water retention that accompanies cirrhosis. Sled and co-workers\textsuperscript{406} applied semiautomatic analysis to microvasculature in mouse kidneys (simple threshold, distance transformation, special routines for local contact between vessels); Toyota et al.\textsuperscript{407} studied heterogeneity of glomerular volume distribution in a rat model of early diabetic nephropathy and found statistically significant (greater) coefficient of variation within individuals of the model compared to controls. Quantitative microCT analysis of collateral vessel development after ischemic injury in a mouse model revealed that the vascular volume was reconstituted as a series of highly connected, small diameter, closely spaced and isotropically oriented vessels as soon as 3 days after surgical ligation of the femoral artery;\textsuperscript{408} simple thresholding and vessel diameter measurement based on distance transformation were used. Corrosion casting of the vasculature system in the brain (perfusion of a polymer followed by maceration of the soft tissue and decalcification of the bone) and a two resolution approach (lab microCT, 16 \textmu m voxels, to identify volumes of interest for subsequent local tomography reconstruction with synchrotron microCT, 1.4 \textmu m voxels) have been used to compare an Alzheimer’s disease model mouse with the wild type using metrics described above.\textsuperscript{409}

Corrosion casts of a canine lung and a mouse lung were imaged with conventional CT and microCT respectively, and, due to effects like bubbles in the polymer, a small amount of manual segmentation was required.\textsuperscript{410} Analysis of the airway tree structure in terms of generations, etc., was similar to that reported in Ref. 31; interestingly, the approach was accurate and efficient for up to six generations for the canine cast and ten generations for the murine cast, presumably because of instrumentation differences. \textit{In vivo} quantification of regional lung gas volumes in rabbits was recently reported using synchrotron microCT, mechanical ventilation with Xe–O gas and K edge subtraction imaging.\textsuperscript{411} \textit{In vivo} imaging with respiratory gating of laboratory rodents has been used to examine lung damage from tumours and from chemotherapy.\textsuperscript{412}

Channel networks have also been studied in cortical and trabecular bone. In all bones above a certain thickness, nutrients must be transported actively via canals of various sorts. In rabbits and smaller mammals such as rats and mice, the author (and many others) observed these canals to be quite narrow; in larger mammals such as dogs, horses and humans, canals are larger and serve for an additional purpose, as described below. The character of the channel systems in cortical bone has been quantified using methods similar to those described earlier in this section.\textsuperscript{413,414} Internal channels in trabeculae have also been described.\textsuperscript{415}

The nutrient canals, termed Haversian if they run longitudinally in bone and Volkmann if transversely oriented, serve as sites for bone remodeling, that is, the replacement of old, damaged bone with new bone. The process involves the concerted action of osteoclasts gouging out old bone followed by osteoblasts depositing bone matrix and mineral. The cylinders of remodelled bone are termed osteons and for some time after they are formed contain lower mineral density than mature bone. The decreased mineral levels in newly formed osteons have long been imaged with microradiographs of thin sections of bone, and synchrotron microCT more recently has been used to good effect to reveal the qualitative spatial distribution of mineral density.\textsuperscript{315,316,328,329,414–417} Dyck et al.\textsuperscript{38} discuss calibration of mineral levels in lab microCT for various calcified tissues, but it is normally quite difficult to see remodelled osteons in lab microCT. It should be possible to quantify differences in mineral density between osteons of varying ages within a bone specimen, but this appears still to be carried out. Standards measurements can be used to remove systematic biases in values of linear attenuation coefficients resulting from beam hardening;\textsuperscript{418} although this is important for correctly interpreting mean mineral level, it does not help with detection of differences between recently formed and mature osteons.

Reports have been published on determination of Ca/P ratios for bone from synchrotron microCT scans: linear attenuation coefficients from bone were compared
to those of two phantoms of calcium phosphate salts with different Ca/P ratios. Although differences in bone mineral density certainly appear to be supported by this group’s data, interpretation of differences in terms of Ca/P ratios appears problematic at best. In bone, the relative amounts of collagen and mineral are expected to vary, the density of osteocyte lacunae can alter the apparent linear attenuation coefficients and other variations will be present (see the discussion of Dowker and co-workers and that of Kozul et al.).

Thin bands of high absorptivity were found in synchrotron micro-CT data of bone. In animals dosed with SrCl₂, these were interpreted as zones of bone with high Sr replacement of Ca in the mineral. Similar bands of high absorptivity were observed in specimens of human femoral neck bone in extraosteoferal areas near to and parallel to the external surface of the cortex; these features in aged bone were interpreted as regions of high mineral content. The author has observed similar high absorptivity bands within cortical bone of animals including newts and mice, bands that examination of adjacent slices showed could not be due to out of plane geometrical features producing unexpected phase contrast. The magnitude of the difference is quite striking: linear attenuation coefficients in the mouse cortical bone were 40% or more greater within the very small volume of hypermineralised tissue than in the ‘normal’ bone, the difference being over twice the standard deviation seen for areas of ‘normal’ bone. It is difficult to imagine that these features, observed by several investigators working as different synchrotron radiation facilities, are an artefact of some sort, but their origin remains obscure.

The current model of bone remodelling holds that Haversian systems are the most active where bone undergoes the most extreme loading, i.e. accumulates the greatest amount of damage such as microcracking (see Ref. 103 for examples of synchrotron micro-CT imaging of microcracking associated with bone porosity including osteocyte lacunae). Clustering of osteons might be a result, particularly in bone of aged individuals, and the presence of such a significant stress concentrator could lead to unexpected fracture. Several studies of channels in cortical bone have focused on establishing the equivalence of micro-CT based measures of porosity and osteon dimensions with long standing methods such as microradiography or histology. Pore content and mineral levels determined from micro-CT correlated with axial ultrasound velocity; the data showed the structure in the outer 1 mm of cortex (about 1/2 cortex thickness) affected velocity and suggest clinical usefulness of this non-invasive monitoring method. A rat ulnar loading model was studied with pQCT, with FEM and with attached strain gages (to assess load sharing between ulna and radius), and the greatest bone formation in response to fatigue loading was found in regions of high compressive strain.

Deformation, fatigue and fracture

There have been quite a number of micro-CT studies of deformation, damage, cracks and fracture since 1999, but not all are covered in this section. Deformation of cellular materials was covered above. Deformation studied as part of materials processing is postponed until the processing subsection, and the subsection on environmental interactions and corrosion covers micro-CT characterisation of these aspects of cracking. Increasingly, investigators using micro-CT observe a given sample four or more times during its evolution, and this will be a continuing theme in the future. Accounts of micro-CT studies of monolithic materials open this subsection, and reports on composite materials, including cortical bone, a natural composite, close the subsection.

Strain localisation, the concentration of deformation into narrow bands of intense shear, occurs not only in cellular solids but also in most geomaterials. Viggiani et al. observed a stiff soil at several strains with synchrotron micro-CT; a single shear band was formed at an axial strain of 2.7%. In situ loading plus micro-CT of concrete has shown that the fracture processes must be treated not as a 2D crack but as a system of smaller 3D cracks. The non-recoverable work of loading, calculated using a linear elastic fracture mechanics approach, in compression of mortar, was determined by measuring the incremental changes in crack surface area revealed by micro-CT as load was increased; the incremental fracture energy rose with increasing damage indicating secondary toughening mechanisms such as friction made up a greater fraction of the measured energy.

Several micro-CT studies have focused on the role of porosity in failure of metallic samples. Model copper specimens have been manufactured with a regular array of interior pores, and their coalescence in the final stages preceding fracture was studied with synchrotron micro-CT. Micro-CT revealed considerable void growth but not nucleation of new voids (i.e. no strain localisation between the pre-existing manufactured voids). Evolution of pore size distribution during creep was studied by micro-CT and X-ray diffraction in a three phase copper alloy; details of this study are discussed later in the section on multimode studies. High velocity impacts generate refraction waves within the target, waves that superimpose and generate zones of high density of pores; synchrotron micro-CT has been applied to small specimens cut from impact tested Ta disks. These data show that pore volume distribution obeys a power law, at least for the larger pores, and that results from 2D methods, even with correction, could not be extrapolated to the actual 3D distribution. Lab micro-CT of specimens from five positions in a high pressure, die casting of Mg alloy AM60B revealed considerable variability in the amount and distribution of microporosity; location of the fracture plane, the fracture strain and the fracture strain agreed with predictions of a critical strain model based on the initial pore distribution. Void growth near a notch was followed at several loads with synchrotron micro-CT (~0.5 μm isotropic voxels) of a cast A356 Al specimen (Al grains surrounded by a Si rich eutectic phase), and this study noted constraint effects on the specimen faces (compared to the middle of the specimen) as well as considerable void-Si particle spatial correlation.

Fatigued commercial bone cement (PMMA beads in a BaSO₄ filled PMMA matrix) was studied with synchrotron micro-CT, macroscopic failure was found to be linked to the presence of large voids, crack deflection was observed at matrix beads and crack arrest was found within beads. In an Al engineering alloy, the
distribution of pore sizes determined from individual lab microCT slices and from the 3D stack of slices were compared: the 2D measurement showed a significantly lower mean pore diameter, even after Saltikov-type correction for sampling biases, than the 3D measurement.\(^{440}\) The interrupted fatigue test specimens showed the crack tended to deviate toward pores that were near the transverse plane (normal to the load axis) that the crack was following across the specimen.\(^{440}\)

Lab microCT with rather large voxels (0-08–0-12 mm) was used to investigate the mechanisms responsible for rising crack growth resistance with increased crack length (R curve behaviour) in a fairly large specimen of polygranular graphite.\(^{441}\) After crack extension, the crack was held open by inserting a wedge. Significant crack face contact was observed behind the crack tip, and the authors report a zone of discrete, low attenuation features around the crack tip and in the crack wake, features that were interpreted as microcracks hypothesised to be responsible for the R curve behaviour. It would be interesting to confirm this interpretation by performing either phase enhanced microradiography plus 3D stereometry (approach described below\(^{178,179}\)) or by performing synchrotron microCT of specimens cut from one of the samples.

The first IMR microCT review\(^{1}\) reported several studies of fatigue crack closure in AA2090. Several addition closure studies on AA2090 followed the earlier work,\(^{360,442,444}\) and correlation of fatigue crack path in these specimens with different scales of crystallographic texture is described in the section on multimode studies below. In situ loading during synchrotron microCT compared patterns of crack opening in two notched tensile specimens that exhibited different crack geometries.\(^{443}\) In small compact tension specimens of the centre of AA2090 plates, Morano et al.\(^{442,444}\) compared fatigue crack path and 3D pattern of crack closure as a function of applied stress for a specimen cracked under load ratio \(R=0.1\) and one tested under \(R=0.75\). Lab microCT was also used to compare the 3D pattern of crack closure (as a function of applied stress) in small compact tension specimens before and after crack extension; the voxel size was rather larger than ideal for crack opening studies and limited quantification of opening in the immediate vicinity of the crack tip.\(^{360}\) Others addressed this sensitivity issue by cutting a small volume of material from around the crack tip and imaging this with the highest available spatial resolution (see the following paragraphs), but Ignatiev et al.,\(^{178,179}\) studying fatigue cracks in intact miniature compact tension specimens from the same lot of AA2090 as described above, adopted a novel approach they termed stereometry (tracking features relative displacements versus specimen rotation in multiple radiographs and computing 3D positions from this data, see the section on ‘Alternative tomographic methods’ for a brief description) and were able to map fatigue crack geometry in 3D with much smaller voxels than would otherwise be possible.

Several synchrotron microCT studies have appeared of small section of Al specimens cut to contain the tip of the fatigue crack.\(^{116,445,451}\) The resulting small voxel size and the strong phase contrast in these reconstructions increase crack visibility enormously compared to reconstructions with pure absorption contrast and in intact specimens. One is never quite sure how much the change of constraint (from removed material) affects the observations, so some caution should be exercised in interpreting these results. Buffiere et al.\(^{451}\) summarise their experience in visualising cracks in these types of specimens, as well as the drawbacks and advantages of microCT in the presence of significant phase contrast, and a few comments on their studies and others’ follow.

Toda et al.\(^{445,446}\) found that the large transients in contrast from the Fresnel fringes parallel to fatigue crack surfaces indicated that robust crack opening measurements required use of features somewhat displaced from the crack plane. Therefore, near crack tip opening displacements in in situ loaded AA2024-T351 were measured using small microvoids (a small distance away from the crack faces) as fiducials,\(^{445,446}\) in much the same way that Breunig and co-workers\(^{270,452}\) worked with C cores in SiC fibres on either side of a crack in an Al/SiC monofilament composite. The high resolution closure observations of Toda et al. were in agreement with earlier work,\(^{360,442,444,453,454}\) namely, loss of surface contact occurs gradually up to the maximum load of the fatigue cycle, mixed mode surface contact is very important and near tip contact is suggested as producing crack growth resistance.

Decoration of grain boundaries in Al with Ga liquid (the melting point of gallium is 30°C) allowed grain boundary positions to be correlated with the 3D crack geometry and changes in crack path, without sectioning the specimen.\(^{66,116,447–450,455,456}\) In this approach, the authors first performed the in situ loading experiments on the material cut from larger specimens; subsequently Ga was applied. Paths of short cracks are well known to be dominated by crystallography of the few grains cut by the crack, and electron back scattering diffraction (EBSD) was used to provide the crystallographic information needed to understand which changes in grain orientation produced large deflections in the crack path.\(^{116}\) Analysis of crystallographic character of several branches of a short crack in a cast AS7G03 Al–Si alloy specimen containing artificial pores illustrated the power of this approach.\(^{116}\) In the same material, 11 observations for different crack extensions were made of a short fatigue crack that had nucleated in a narrow ligament between a pore and the specimen surface,\(^{455}\) and the evolution of the crack front shape was interpreted with respect to the surrounding grain microstructure and pore positions. Figure 10 shows a 3D view of the tip of a fatigue crack in an AA2024-T351 specimen; the solid material is rendered transparent and only the Ga labelled grain boundaries and crack are shown. Large portions of the crack (cut from the larger specimen to include only the near tip region of the long fatigue crack) were within five degrees of \{100\} or \{111\}, with steeply inclined sections following \{111\};\(^{450}\) these crack paths are those observed in AA2090 TSE41.\(^{457}\)

Ferrié et al.\(^{66}\) studied fatigue propagation in an ultrafine grained, powder metallurgy alloy (AA5091 material system with mechanical properties equivalent to the T1 condition). This alloy was selected because fatigue cracks follow highly planar paths, and an in situ fatiguing apparatus was mounted directly on the synchrotron microCT rotation stage. Radiography was used to monitor crack initiation, and, after each of nine increments of crack extension, data for reconstruction...
were collected with the specimen under maximum applied load. The crack grew more elliptical with increasing number of cycles with the major axis perpendicular to the specimen surface, and the authors attributed this to differences in closure stresses along the crack front. Local stress intensity range $\Delta K$ was calculated via FEM for the portion of the crack growing parallel to the surface and for the portion growing perpendicular to the surface. Plots of crack growth rate $\frac{da}{dN}$ v. $\Delta K$ showed comparable power law exponents, but the surface curve was displaced to lower $\Delta K$ relative to the bulk curve and the latter followed the experimental long crack growth curve. The authors concluded that, for the specimen geometry studied, a single Paris equation can predict the observed crack growth that, for the specimen geometry studied, a single Paris mental long crack growth curve. The authors concluded to the bulk curve and the latter followed the experi-
mental long crack growth curve. The authors concluded that, for the specimen geometry studied, a single Paris equation can predict the observed crack growth

Marrow and co-workers$^{458}$ performed synchrotron microCT on a very small ductile cast iron specimen and focused on characterising changing crack front geometry as the short cracks interacted with pores and graphite nodules. A gage diameter of $\sim 0.35 \text{ mm}$ was required to give adequate transmission through the iron specimen at the highest photon energy that was practical for use with the high resolution X-ray detector (30 keV). The difficulty of working with such fragile specimens is undoubtedly the reason that relatively few experiments are performed steels, copper or still more attenuating metals or composites. Even Ti poses challenges for microCT imaging.

The characteristics of a model discontinuously reinforced composite system (0, 5 and 10 vol.-%Ni particles blended with AA2124 powder and hot extruded) were studied with lab microCT.$^{459}$ Particle clustering was quantified by 2D (SEM) and 3D (microCT) methodologies, and good agreement was found. Subvolumes of the microCT reconstructed volume were meshed and incorporated into FE simulations of the three materials, and the actual and simulated stress–strain curves showed quite good agreement.$^{459}$

Buffière et al.$^{443}$ used synchrotron microCT to study a more challenging composite system than Ni–Al: 10 vol.-%SiC particles in a matrix of AA6061-T4. As the mass attenuation coefficients of SiC and Al differ by less than 3% at 23 keV, phase enhanced imaging (detector specimen separation of 830 mm instead of a few tens of millimetres) was used to provide contrast between particle and matrix and to enhance crack visibility. The same volume was compared at five strains (initial microstructure, at the yield point of the stress–strain curve and at three strains up to 13%), the fraction of broken particles was greater in the bulk than near the surface and FE calculations (normal stress, total stored elastic energy in particles) were supplied to explain the observations.$^{443}$ The spatiotemporal distribution of fractured SiC particles was mapped in a subsequent study.$^{466}$ Interest in microCT based FEM analyses of particulate reinforced composites continues.$^{461}$

The association between deformation induced porosity in several Al matrices and particulate reinforcements (ZrO$_2$) was investigated with lab microCT using a dual energy reconstruction technique.$^{32,462}$ This is an example where an energy sensitive X-ray detector (and a translate–rotate or pinhole data collection scheme$^1$) is required. Variance analysis was used to show that little if any clustering of the particles was present and to determine that voids also were not clustered. A direct relationship between volume fraction of particles and void volume fraction was demonstrated.$^{32,462}$ Synchrotron microCT and in situ loading have also been used to study damage in aluminium–zirconia composites.$^{456,463}$

Indentation damage has been studied in carbon fibre reinforced plastic composites, but this study was limited to qualitative comparisons with results of ultrasonic characterisation.$^{364}$ A more complete focus on detection limits for different types of damage in fibre reinforced polymer matrix composites was provided by a second study that examined the same cracks before and after (high X-ray absorption) dye penetrants were added.$^{365}$ In this lab microCT study, the authors reported crack detection limits without penetrant that were similar to those determined by Breunig et al.$^{270,452}$ but cracks open 0.5–1 $\mu$m in $\sim 20 \mu$m voxels (opening $<5\%$ of the voxel size) could be detected when penetrant was added. Other polymeric composite systems containing cracks and studied with microCT were the elastomeric material of autotires$^{456}$ and aged dental composites.$^{466}$

Fracture of unidirectional composites is generally thought to occur when a cluster of broken fibers reaches a critical number $N^*$, and microCT is an ideal tool for
assessing whether the critical cluster concept is valid and, if so, what \( N^* \) might be for a given composite system. Synchrotron microCT of uniaxially aligned quartz fibre, epoxy matrix composites investigated this concept with \textit{in situ} loading, and simple stochastic failure models were reported to underpredict \( N^* \) by a factor of 3–5.467

In a uniaxial monofilament Al/SiC composite, microCT of mechanically induced damage was compared with unloading modulus.468 While this study of monotonic deformation and of fatigue damage was performed some time ago,470 a more complete report (other than a thesis) appeared only recently. Macroscopic measures of damage (changes in unloading compliance and in unrecovered strain) correlated with microCT quantification of microstructural changes (fibre separation, fibre misorientation relative to the load axis, fibre carbon core fracture). More recent generations of SiC monofilaments have improved properties, so these results are not indicative of current performance. Few complete fractures of SiC fibres were observed except after specimen failure; the authors concluded SiC fibre fractures were responsible for decreased compliance but, upon unloading, residual stresses from intact fibres presumably pulled fracture surfaces back together in the damaged fibers. Because this synchrotron microCT data were obtained under imaging conditions where phase contrast was negligible (i.e. during the earlier 1990s at CHESS and SSRL), crack visibility in the SiC fibres was substantially lower than that in the studies reported in the following paragraph, and it is not surprising that tightly closed cracks might be invisible.

Uniaxially aligned monofilament Ti/SiC specimens were imaged with synchrotron microCT under \textit{in situ} loads, and fibre fracture geometry and spatial distribution were characterised.189,267,469–474 Single fibre, single ply and multiple ply specimens were studied; artificially fractured fibres within the one ply specimen and fibre bridging across a fatigue crack in the multiple ply specimen were studied. The SiC fibre fractures were similar to what had been reported for Al/SiC monofilament composites, namely, wedge cracks and spiral cracks. Careful consideration of synchrotron phase enhanced microCT renderings of the fractured fibres identified with wedge cracks (e.g. Figs. 5b and 6 of Ref. 472) reveals complex contrast between the wedge edges (where contrast was the strongest), and the discussion in this paper clearly identifies small fragments that give rise to the complex contrast. The fainter contrast regions suggest that the SiC material between the wedge edges contains additional (albeit more tightly closed) crack segments (small fragments of the locally shattered fiber). Although this may seem to be a minor point, the fine details of fibre fracture could provide important insight into interface bonding or into stress wave interactions during fibre fracture. The longitudinal sections through the fibre centres also revealed that the fibres curve along the length of the specimen (smaller apparent width of the fibre’s core at top and bottom of the sections). As an integral part of the study was microbeam diffraction mapping of strains in the Ti matrix and of the longitudinal fibre strains, more detailed discussion is postponed until the section on multimode studies.

Most microCT deformation studies of bone concern trabecular bone and were reviewed in the cellular materials subsection above. One bovine cortical bone study used lab microCT to examine short rod chevron notched tension specimens for fracture toughness determination.475 The V shaped notch allows steady state crack propagation in a sample diameter rather smaller than a standard compact tension specimen, an important advantage given limited dimensions available even in the long bones of large animals. In principle, fracture toughness for this specimen geometry does not require measurement of the crack length, but practically realisable geometry does not meet the assumption for the calculation and compliance tests and crack length measurements (via microCT) were used for more robust determination of the plane strain stress intensity factor.475

Nanoindentation is increasingly popular for applications such as quantification of the anisotropy of elastic moduli in bone, and such moduli have been shown to agree well with moduli from load deflection curves.476 Some assumptions are required in the analysis, and Hengsberger et al.476 used synchrotron microCT to provide some of this information (specimen mineral levels, porosity and cross-sectional dimensions).

Cortical bone exhibits good toughness, and two views of the source of toughness are: bridging by uncracked ligaments in the crack wake and microcracking ahead of the crack tip. Both absorb energy that would otherwise be used to extend the crack. Establishing the relative importance of these mechanisms would suggest treatment strategies for osteoporosis prevention. Nalla and co-workers477,478 have examined mechanistic aspects of crack growth resistance in human cortical bone by determining crack growth resistance curves (R curves) and using synchrotron microCT to image the 3D crack structure. Fracture toughness rose linearly with crack length, but there were clear differences in behaviour between bone from young mature adults (age<41 years old, described as young bone below) and that from aged individuals (age>85 year old, described as aged bone in what follows). \textit{Ex vivo} crack initiation toughness decreased 40% from young to aged bone, and crack growth toughness present in the young bone was essentially eliminated over this period. Quantification of the amount of crack bridging versus crack extension (practical only with microCT) revealed considerable initial bridging for both young and aged bone; after some extension crack, bridging for young bone remained comparable to the initial levels but for the aged bone was virtually absent. MicroCT showed that bridges were present throughout the specimen thickness (demonstrating that SEM data for bridging at specimen surfaces is valid479) and that cracks tended to follow cement lines bordering osteons. The bridging zone length was on the order of 5-5 mm long for this human cortical bone. While toughness values for bone and dentin, related collagen–apatite composites, were comparable and were thought to reflect the nanoscale structure, differences in time dependent crack blunting between the two mineralised tissues were thought to reflect the very different micrometre level structures.

Diffraction enhanced imaging (microradiography) was used along with bone’s diffraction peak widths in an attempt to identify damage in cortical bone.480
Neither method revealed damage: even with the tenfold increase in sensitivity to small cracks compared to absorption based imaging, this is not surprising because microcracks are very tiny features and the effect of overlapping depths will obscure even larger features. This conclusion should not be taken to demonstrate that X-ray diffraction cannot reveal useful information for damaged bone; as examples in the section on multimode studies demonstrate below, this is not the case.

Processing

Solidification is a profitable processing application for microCT. One application that can be studied readily is the inhomogeneous distribution of particles in a discontinuously reinforced composite. Clustering of reinforcement particles can be deleterious from a fracture resistance perspective or can be used to great effect to provide a component with a hard, wear resistant (although low toughness) outer surface and a tough internal volume.

In an *in situ* composite of Al and TiB₂, Watson and co-worker 481 used a novel sampling procedure to withdraw material from the melt and observed boride particle clustering with synchrotron microCT as a function of melt hold time. Small amounts of melt were drawn off at times up to 2.6 × 10³ s and quickly solidified. MicroCT revealed the maximum cluster size decreased from an initial value of 50 μm to 10 μm at the end of the experiment. Even though the sampling technique may bias the clustering results somewhat, use of the same method throughout means that the changes observed almost surely reflect changes occurring in the melt vessel. In an Al/SiC₃, functionally graded composite fabricated by centrifugal casting, Velhinho et al. 482 observed slight gradients in particle volume fraction away from the SiC rich surface. Because their mass attenuation coefficients are very similar, SiC and Al are difficult to distinguish based on absorption alone, and phase enhanced interface contrast in synchrotron microCT can help segmentation considerably.

Solidification with segregation of atoms with different absorptivities is another area where microCT has been applied. This segregation, and the accompanying range of solidification temperatures, can lead to undesirable excess porosity or cracks (hot tearing) appearing at the end of solidification. Ludwig et al. 47 followed *in situ* solidification of Al-4 wt-%Cu in 3D with ultrafast synchrotron microCT. A complete 512 (perpendicular to the rotation axis) × 256 scan (500 projections over 180°) was recorded every 10 s using polychromatic wiggler radiation; this was a case where contrast sensitivity was sacrificed for temporal resolution. A cooling rate of 0.1°C s⁻¹ was used for these *in situ* experiments; despite the reconstructions encompassing structures averaged over a ~1°C temperature range, the slices were quite clear, showed growth and linkage of the solid phase particles and showed increasing Cu content in the liquid phase. Evolution of the experimentally determined solid volume fraction with temperature was compared to different solidification models; shrinkage versus solid fraction was linear; SV evolved as expected and mean Cu content (wt-%) in the solid and liquid phases (determined from analysis of the linear attenuation coefficients) agreed reasonably well with that predicted by the liquidus and solidus temperatures of the equilibrium phase diagram. 47 In an Al–15.8 wt-%Cu alloy, the investigators were also able to show that rapid quenching produces a higher volume fraction of solid phase than was present at the starting temperature, 47 therefore, models based on quenching data may contain a bias that must be corrected. An earlier report of microCT materials quenched from the solid state appeared elsewhere. 483

The grain size distribution and number of faces per grain were measured in synchrotron microCT reconstructed volumes of solidified Al–Sn with 1, 2 or 3 at.-%Sn. 484,485 Sn is immiscible in Al and segregates to the Al–Al grain boundaries. Some areas of the grain boundaries appeared to be free of Sn, a possible effect of the sensitivity limit, and a special algorithm was derived to fill in the missing boundaries. The authors concluded that size distribution agreed with and the number of faces per grain differed from metallography data in literature. 485

Retained porosity in cast Al–Si was studied as a function of H₂/Ar gas ratio introduced during stirring of the melt. 460 Two populations of voids were observed. The smaller voids were associated with microshrinkage when the metal solidified, and the population characteristics (equivalent size versus sphericity) did not vary with gas composition nor did the volume fraction of microshrinkage pores. The larger voids were from artificial incorporation of gas bubbles, and the volume fraction of gas pores increased exponentially with H₂ content.

Highly non-equilibrium solidification occurs in spray deposition of thermal barrier coatings, and synchrotron microCT is invaluable for determining pore shapes and for quantifying pore volume fraction in the coating as a function of distance from the substrate. 486–489 Different processes produced different pore shapes that can be directly visualised and compared with the results of techniques such as small angle neutron scattering (SANS) and nanoindentation for elastic moduli determination. 486,489 Gradients in porosity have also been correlated with indentation derived moduli and SAXS. 487,488

MicroCT characterisation of vapour phase processed materials does not appear to have received much attention since the first IMR review. Bernard and co-workers reported some results of isobaric chemical vapour infiltration of a C/C composite. 490 Kang et al. 491 showed some synchrotron microCT images of cracks in C fibres grown from the vapour phase.

Superplastic forming (very high strains in certain alloys without rupture of the starting material) is finding application in aerospace and automotive fields. Superplastic deformation is generally limited by strain induced cavitation leading to fracture. Using synchrotron microCT, Martin et al. 492 found that the number of cavities per unit volume versus strain (~1 < ε < ~1.7) in AA5083 followed model predictions and observed developing cavity linkage. 492,493 Pore evolution from rolling of an Al–6Mg alloy was studied with lab microCT, and the authors demonstrated that the highly tortuous pores would be difficult to detect in polished sections. 494 The tortuous pores spheroidised during homogenisation, and accelerated centreline intrapores coarsening observed during initial, low reduction ratio rolling passes was attributed (through finite element modelling) to local tensile conditions, a counterintuitive but not unreasonable result. 494
Warm drawing of a 6 mm diameter PE rod down to ~5 mm diameter was studied with SAXS microCT. Circular zones of differing lamellar sizes (longitudinal and lateral) were clear in the reconstructed slice presented and undoubtedly much more can be carried out with this approach, especially in specimens that would appear featureless in absorption tomographs. It would be interesting to compare phase tomography to the SAXS derived reconstruction.

Synchrotron microCT of an Al–Mg industrial alloy (AAS182) has tracked the size distribution and spatial dispersion of intermetallic particles (iron rich, Mg2Si) and of voids from as cast + homogenised to hot rolled to cold rolled to tensile tested state. In addition to 3D views of a small portion of the structure (four thresholds with Al rendered transparent, voids as black and the intermetallics in two different greys), numerical values for mean, minimum and maximum equivalent radii of the intermetallic particles showed the progression of fragmentation expected for the different processing steps. Similar data for volume fraction, number density and mean equivalent radii were measured for pores. One limitation in the study with 0.7 μm voxels was that very small particles (those with the smallest dimension < 1 μm) could not be included in the analysis; the authors expected that this would be improved with imaging with 0.3 μm voxels.

Movement of marker particles within metals or in unconsolidated powders can be used very effectively to map displacement fields in response to deformation. Specimens of Al–1 vol.%Ti (particle diameters between 1 and 10 μm) were imaged with synchrotron microCT at the 2 μm level, and displacement gradients for deformations up to 9.5% compression were quantified. Bulk material flow has also been studied in friction stirred welds in Al using the particle technique and synchrotron microCT. Closed die compaction and sintering of powders have long been used to fabricate metal and ceramic components, but constitutive models for loose powder behaviour under intense shear deformation need to be developed. McDonald et al. used lab microCT in correlation to follow particle displacements when a cylindrical punch was pushed into a somewhat wider diameter cylindrical die; they report uncertainties in strains of ~0.05% and correlate particle displacement vectors with dilational strains calculated from the particle displacements (Fig. 11). Generally speaking, these studies found that high contrast particles (e.g. Ti, Sn or W in Al) need to have diameters of several voxels for reliable detection and automated tracking of displacement.

Particle shapes, size distributions and packing are important in processing and also in fluid transport; discussion of those studies related to transport in the open phases is postponed until the following subsection on environmental interactions. The 3D size and shape characteristics of collections of particles have been studied effectively with microCT, note that microCT can eliminate the need to disperse particles originally in a dense assembly or packing or even in a somewhat agglomerated state and avoid artefacts inevitable in any physical separation process. In Ref. 501 the analysis algorithm was developed in an dimensionless fashion, that is, in terms of voxels per unit particle diameter, so that it could be applied to different sized particles’ distributions studied with different voxel sizes. The approach was based on simultaneous solid phase and void phase burn (the algorithm moves away from the interface and assigns a value to each voxel that equals the number voxels it is away from the interface). The local maxima of burn number was used as a particle centre, and all voxels previously identified as solid were assigned to one or another particle with additional steps required to accurately partition contacting grains. Particle volume, surface area, orientation, aspect ratio and contact statistics flowed directly from the assigned particles; computation performance metrics were supplied and the results for computer generated structures and for microCT of a standard sand showed the approach works quite well. Lin and Miller characterised three collections of well defined particles with lab microCT: nearly spherical beads, an isotropic quartz sand and quite jagged rock fragments. The data showed the expected surface area versus volume behaviour, and the plot for the irregularly shaped particles was offset from that of the beads and sand, as one would expect. Fu and co-workers examined in situ compaction of powder with lab microCT;
Richard et al.504 observed granular packing resulting from vibration with synchrotron microCT and Seidler et al.505 characterised the distribution of granules packed under the influence of gravity. MicroCT derived particle characteristics from a well characterised ore have also been compared with 2D measures.506 Agglomeration of particles and the breakage of agglomerates during compression have begun to be studied with microCT combined with numerical modeling.507,508

Sintering/cementation of powders has also been studied by microCT,509–511 new areas include analysis of rapid prototyped material and evolution of materials with nanoparticulate precursors. Bernard and co-workers512,514 employed local tomography to quantify porosity elimination and neck evolution in a glass powder and in a lithium borate powder over 1-6 × 10⁴ s at 700 and 720°C respectively; growth of necks were particularly well illustrated by matched pairs of renderings, one showing the solid phase and the second the complementary void space. Sintering of copper and steel (Distaloy AE) powders were also studied with synchrotron microCT, and changes in pore geometries in copper and elimination of very thin interparticle voids but not large pores in Distaloy were observed with increasing sintering time.509,510 Final dimensional changes are strongly anisotropic in Distaloy AE Densmix (axial swelling during delubrication and axial shrinkage during sintering), and repeated in situ synchrotron microCT observations during the different sintering stages (initial structure, lubricant burn-off, sintering and cooled structure) were used to investigate these changes.511 Analysis of the orientation distribution of three populations of pores (highly elongated, near circular and intermediate geometry) was one probe; the second was an image correlation based, local strain mapping comparison for the different directions in the compact.

Corrosion and environmental interactions

Consideration of the transition of snow to firn to ice (porosities of >95%, ~40 %, <10% respectively) follows naturally from the discussion of sintering of engineering materials. The particles (snow) are at a relatively high homologous temperature (i.e. a large fraction of melting temperature) and are frequently under pressure from the weight of subsequent snowfall; thus, the process and the structures are very similar to those covered above. Properties of snow/firn/ice are important in understanding avalanches; if porosity is closed in specimen cored from ice, an air archive exists (atmospheric information) that can be compared to the ice archive (climatic information). Synchrotron microCT of a snow sample collected at the failure site of a slab avalanche, for example, showed a cohesive layer above a weak layer,515 a situation encountered in skier triggered avalanches. The metamorphosis of snow to ice depends on local 3D curvature and solid vapour surface area; under isothermal conditions, minimisation of local curvature (minimisation of surface energy) is thought to govern the densification of the structure, i.e. a structure with many sharp, flat grains transforms to a much more rounded structure. Results of synchrotron microCT of snow at different points during transformation have been compared with numerical models evolving from real (microCT derived) 3D microstructures, and the agreement between actual and modelled structures was quite encouraging.516,517 One should note that considerable technical challenges were overcome in the preparation of unaltered snow specimens for microCT examination, but these will not be discussed here.515

Understanding reactive percolation (CO₂) through porous limestone is important if, as some have proposed, rises in atmospheric CO₂ are to be combated by CO₂ sequestration in subsurface reservoirs. Synchrotron microCT has followed changes in limestone porosity during flow of CO₂ charged water over 3 × 10⁴ s and related changing microstructure to increasing permeability.514 Other studies directly compared successive structures in the same solid-fluid system over 8 × 10⁴ s.518,519 As limestone has historically been an important construction material, its attack by CO₂ and by industrial organic pollutants and repair of such damage is an important area of research.520 Vapour transport through two types of bricks has been examined with synchrotron microCT, and the relationship between permeability, diffusivity and pore size agreed with analytical expressions.183 Water transport in the proton exchange membrane fuel cell was also tracked with microCT.521

Several studies of soils have appeared.53,524 Metal contaminant absorption characterisation is a current area of activity.525 In Ref. 83, iron content was mapped, relative amounts of iron oxyhydroxides and iron bearing clays were determined semiquantitatively and the relationship of Cs adsorption with iron bearing materials was imaged using absorption edge difference techniques.

Soil microbes and plant roots microengineer their habitats by changing porosity and pore cluster characteristics. Synchrotron microCT revealed soil modified by root action over 30 days had significantly greater pore volume and pore connectivity than in soil without roots.251,522 These and other results reported indicate that the soil ecosystem exhibits self-organisation in relatively short periods of time. Some aquatic plant species sequester metal(loid) species on root surfaces, and synchrotron microCT and other techniques have been applied to show association of As to regions of enhanced Fe content.523

The location of the metal species of interest within or at the surface of particles governs the extent to which that metal may be recovered by leaching operations. In order to determine how well lab microCT might function as an assaying tool (i.e. predicting leaching performance for comparison with actual efficiency), Miller et al.525 quantified the volume fraction of copper bearing minerals in contact with the surfaces of ore particles. Standards of natural particles of copper bearing mineral phases (known to exist in the ore of interest) were used to set thresholds for segmentation and allowed 3D assessment of mineral exposure versus particle size to be determined for the ore of interest. MicroCT predicted recovery generally underestimated actual recoveries from column leaching tests, an unsurprising result given the finite resolution and sensitivity of the microCT system and the additional exposure that partial leaching might produce.525 Silver is associated with pores in certain ores, and Chen et al.526 used lab microCT to determine that porosity was insufficient to allow silver to be leached from the interior of a coarsely ground ore sample.
Disordered packing of idealised particles (e.g. spheres, equilateral cylinders) has been examined with tomographic techniques. Packing in various multiphase systems has also been studied, and properties of pore networks (relatively large open volumes connected by much narrower throats) have received attention. The influence of flowrate and porous media microstructure on macroscopic relationships such as capillary pressure versus saturation can be effectively examined with synchrotron microCT. Using one idealised packing system (alumina cylinders, 1 mm diameter, 3–4 mm length), pore scale modelling and microCT visualisation were used to study the spatiotemporal evolution of liquid phase clusters; behaviour in untreated (hydrophilic) and silanised (neutral) packings were compared. Observations of the evolution of water distribution with drying were compared with numerical simulations derived from the initial microstructure, and this sort of analysis typifies the direction many microCT investigations are taking. Numerical modelling of equilibrium distributions of water within partially saturated rock has been based on microCT derived structures and appears to be a very valuable approach.

In natural systems such as sandstone, the extent to which pores are connected indicaties not only reservoir characteristics (see Ref. 490 and the first IMR review) but also the rate of contaminant transport. Cluster labelling analysis of synchrotron microCT data of a 14% porous sandstone suggests that isolated pores comprise only 11% of the total porosity and unresolved connections between pores may contribute significantly to fluid transport. Multiscale modelling (lattice Boltzmann transport) was used to simulate the dissolution of 4 mm length, pore scale modelling and microCT packing system (alumina cylinders, 1 mm diameter, 3–4 mm length), pore scale modelling and microCT visualisation were used to study the spatiotemporal evolution of liquid phase clusters; behaviour in untreated (hydrophilic) and silanised (neutral) packings were compared. Observations of the evolution of water distribution with drying were compared with numerical simulations derived from the initial microstructure, and this sort of analysis typifies the direction many microCT investigations are taking. Numerical modelling of equilibrium distributions of water within partially saturated rock has been based on microCT derived structures and appears to be a very valuable approach.

The presence of organic, water immiscible liquids, often as blobs, is a key complicating factor in the remediation of hazardous waste sites. The pore scale morphology of such liquids residing in porous media (sand) was investigated with synchrotron microCT, and the distribution of blob sizes and morphologies were quantified. A follow-on study tracked blob dissolution after each of three column rinsing cycles; changes in blob number, volume and surface area determined with microCT correlated well with effluent concentrations and validated a first order mass transfer expression for effluent concentration. Note that these studies used a contrast agent to enhance blob visibility. Digital laminography with an X-ray tube source was used to map residual oil and water saturation in porous media. X-ray imaging in a lab microCT system visualised the dynamic adsorption of organic vapour and water vapour on activated carbon, but the report left unclear whether microCT or simple radiography was the modality used.

Transport during filtration has aspects related to flushing of porous materials, and filtration studies typically have focused on efficient elimination of water from the remaining solid. Flocculation is one method of extracting suspended solids, and characteristics of the resulting aggregates affect downstream solids recovery. MicroCT has been combined with numerical modelling to predict permeability in a model flocculated system. Drying of mechanically dewatered wastewater sludges and of filtered particulates in mineral processing (filter cake) are two examples where microCT has suggested how to improve energy efficiency of these processes. MicroCT has been used to identify microstructures resulting from drying sludges and their correlation with drying rates and energy consumption. Lin and Miller used numerical modelling to examine flow through well defined simulated filter cake, and they also simulated flow based on structures directly derived from microCT.

Colloid transport in porous media is often treated as a filtration problem, and Li and co-workers observed (with lab microCT) the deposition of 36 µm gold coated microspheres in two porous media (glass beads and quartz sand, both ~780 µm mean particle size). In the absence of an energy barrier (native particle surfaces), the logarithm of the deposited microsphere concentration decreased linearly with increasing transport distance, a result consistent with filtration theory. In the presence of an energy barrier (treatment of the columns with polyoxyethylene laurel ether), colloid deposition was strongly influenced by local geometry (particularly grain–grain contacts) and did not vary monotonically with transport distance.

Before changing the focus of discussion from porosity and transport to environmental attack of solids, consider a recent study applying texture analysis to 3D microCT datasets of five porous specimens (mineral carbon forms from different geographical locations with similar topological structure that differed mainly in textural quality). Texture refers to the structure contained within a region, and most readers would have little difficulty visualising smooth, rough or periodic textures (see Fig. 11.19 of Ref. 252). In the textural analysis cited here, robust measures of structural texture were extracted from the gray scale images. A set of 96 texture features comprised the texture vector for a particular sample that could then be related to the texture space defined by prior measurements on known training specimens, i.e. probabilities could be computed for the likelihood that a given specimen belonged to each population. One expects that, with further development, this approach will be valuable not only for classification of complex specimens but also for rapid (automated) identification of key structural differences.

Construction materials frequently are exposed to aggressive environments over a period of years or even centuries, and, as much of the resulting damage is subsurface, a number of reports of microCT applied to degradation of building materials have appeared. One of the most important construction materials is concrete, and Portland cement used in this composite has been studied from several perspectives. A collection of useful baseline cement paste (synchrotron microCT) images have been published, and features such as unhydrated cement particles, regions of hydrated cement and ettringite needles were identified. Based on this data, cement particle shapes were analysed and were expected to improve computational models of cement hydration and to serve as a resource for those needing microstructures for modelling.

The pore structure of cement based materials seriously affects resistance to environmental attack as well as mechanical properties. Often pozzolans (fine natural materials) are added to the Portland cement to improve resistance to chemical attack and to increase long-term strength and durability. Efflorescence and corrosion are important issues for concrete buildings.

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volcanic ash, fly ash from power generation, diatomaceous earth, etc.) are added to cement to produce a fine structured composite. A study of the pore structure of Portland cement composites with pozzolan (neat cement versus 25 wt-% fly ash versus 10 wt-% metakaolin) found mean pore size and maximum pore diameters decreased for the composites compared to the simple cement. Chloride permeability is one cement durability issue, and microCT measured pore structures of a reference concrete and of fly ash, silica fume and slag modified concretes were compared with results of a rapid chloride permeability test. With 4 μm voxels, little pore connectivity over distances of 100–200 μm was documented for any of the four conditions; with 1 μm voxels the reference concrete showed deep pore penetration while the modified concretes showed clear gaps in interconnected pore spaces. The somewhat limited data showed chloride permeability correlated clearly with disconnected pore distance.

Leaching of cement in mortar (sand plus Portland cement) was studied by repeated synchrotron microCT of the same specimen over 2–2 × 10^5 s (the 4 day scheduling window at ESRF dictated the length of the experiment). Because of the time constraint, calcium efflux was increased by a factor of × 300 compared to deionised water using an ammonium nitrate solution (which produces the same mineral end products as water alone). Variation of linear attenuation coefficient μ in the cement phase was followed as a function of depth from the specimen surface for four exposure times. Within a zone near the surface, μ decreased rapidly during the first 24 h (8–6 × 10^4 s), and the authors inferred that this was due to decalcification of Portlandite crystals (calcium hydroxide, CH) with little C–S–H (calcium silicate hydrate) involvement. Between 24 and 48 h of leaching (1× 10^5 s), μ decreased more slowly, indicating that CH was completely removed from this portion of the specimen and that the less soluble (than CH) C–S–H was being removed, a process continuing to the end of the test. The variation of the thickness of the leached region agreed with diffusion controlled kinetics (i.e. square root of time dependence).

Naik and co-workers studied sulphate attack of Portland cement paste using lab microCT and repeated observations on each specimen. Two cement types, different water to cement ratios, two different cation sulphates and the presence/absence of aggregates were examined. Figure 12 shows how cracks develop in the same slice over 52 weeks sulphate exposure (pores within the cement paste are used as fiducials). Figure 13 shows 3D renderings of sulphate induced cracking (that develops into spalling) in one specimen over 32 weeks of exposure. As the interpretation of the results depended to a significant extent on use of an additional X-ray modality (position resolved energy dispersive X-ray diffraction), further discussion is postponed until the section on multimode studies.

Synchrotron microCT has proven very valuable in studying wood degradation. Fungi enzymes and metabolites degrade the structural integrity of tracheids (thick walled, tubular structures with hollow centres containing air and/or water), and significant strength can be lost early in the decay process. Over the 96 h following fungal inoculation, tracheid pore volume increased somewhat as did pore interconnectivity. Siloxanes/silanes mixtures are often applied as wood preservatives, and microCT was used to study penetration of the preservative into two types of wood using brominated silane as a contrast agent. Boundaries between treated and untreated wood were clear, and one expects that with further work improved wood preservation protocols will result.

Environmental attack of apatite in tooth enamel has been studied with lab microCT and scanning microtomography for a number of years by Elliott and co-workers. These investigators periodically examined (over 70 days, with microCT) packed powders of carbonated apatite in an acidic buffer; these data were supplemented by infrared spectroscopy and Rietveld analysis of X-ray diffraction of the dissected internal surface. This same group also carefully considered microCT derived mineral levels in sound and carious enamel, and these papers will repay careful study.

A de-remineralisation model using small coupons of bovine tooth (enamel plus dentin) has also been studied with synchrotron and lab microCT; analysis concentrated on quantifying the gradients of mineral content. The papers cited in the preceding two sentences illustrate two approaches to use of values of the linear attenuation coefficients in quantitative analyses. Elliott and co-workers took the fundamental approach of relating the linear attenuation coefficients back to mineral standards and expressing measured quantities in terms of absolute amounts of mineral present per unit volume of tissue. Similar approaches have been utilised by Peyrin and co-workers on studies of bone as well as numerous others including Ritman and co-workers. Although this approach allows for direct comparison between studies conducted under different conditions or with different techniques, extreme care must be taken to avoid systematic errors that might bias comparisons. The quantification of de-remineralisation of enamel employed an operational approach, assuming that linear attenuation coefficient values away from the surface were identical from specimen to specimen and scaling all values to this presumed reference. This might be thought to be a poor assumption because enamel mineral levels can differ by several per cent or more between the tooth surface and volumes near the dentinoenamel junction, but the profiles were normalised to values of the linear attenuation coefficient at essentially the same depth from the tooth surface, thereby rendering this consideration moot. Very little of either sort of analysis has appeared to date, although to be fair, some investigators have explicitly verified that experimental values of linear attenuation corresponded to values expected for the material being studied. Peters et al. used lab microCT to analyse root canal geometry by adapting tools developed for histomorphometry of trabecular bone. Other tooth studies include development of a library of microCT images for teaching 3D dental structures, an application recapitulating that of Dowker et al. described in the first IMR review.

The dentin/adhesive interface was studied under static loading (via synchrotron microCT) and with FEM, fatiguing in a solution of silver ions (and imaging at an energy just above the silver absorption edge) allowed interface leakage to be studied. Efficacy of endodontic...
seals was studied with synchrotron microCT. The in vivo degradation of Mg implants in bone were also studied. Fissures in enamel and changes in mineralisation were the subject of another report. Phase microCT revealed tubules in dentin, features whose diameter are only slightly larger than the voxel size in the reconstructions; it will be interesting to compare these results with higher resolution data when it becomes available.

Repeated observations of the same specimen have also been performed on stainless steel specimens undergoing localised corrosion and intergranular stress corrosion cracking. Synchrotron microCT observed localised corrosion morphology within Al specimens exposed in situ to a chloride environment (Fig. 14), and lab microCT was used to investigate the morphology and quantify the transition from localised corrosion to stress corrosion cracking in steel specimens exposed ex situ to a simulated corrosive condensate environment. A 302 stainless steel wire was heat treated to produce a stress free, fully sensitised microstructure (i.e. one with grain boundary chromium carbides) and examined with synchrotron microCT after three increments of stress in an acidic environment. Analysis centred on identifying bridging ligaments formed during the first increment of crack propagation and on following the progressive failure of the ligaments using a combination of 2D sections and 3D renderings (Fig. 15), and these authors noted the presence of unresolved cracks that could still be detected through their phase contrast. Three-dimensional (3D) finite element models were devised to investigate the development of crack bridging

Matching lab microCT slices of cement paste sample produced with water to cement ratio of 0.485 and exposed to 10 000 ppm of sulphate ions in sodium sulphate solution for a 21, b 36, c 42 and d 52 weeks. Crack C1, radial crack RC1, cracks within body of specimen BC and pores P1–P5 are labelled. Horizontal field of view is a 15.3, b 15.1, c 16.1 and d 15.2 mm; and reconstruction was with 37 μm isotropic voxels. Lighter pixel, more absorbing voxel. Reprinted from Ref. 560, Copyright (2006), with permission from Elsevier.
and its effects on crack propagation and crack coalescence in intergranular stress corrosion cracking.

Liquid metal embrittlement was studied through use of the model system of liquid Ga applied to polycrystalline Al. The Ga penetrated many but not all of the grain boundaries as discussed elsewhere. Most studies of Ga on Al grain boundaries, however, focused on its use as a decoration so that grain boundary geometry could be correlated with fatigue crack path (see the deformation subsection above).

**Metrology**

MicroCT is frequently used to measure internal or external dimensions and shapes both in manufactured and biological objects. While this topic is not central to...
this review, it is useful to list some of the applications falling under the general category of metrology. High definition inspection of fuel injector components is being investigated in the automobile industry. \textsuperscript{576} MicroCT of the auditory apparatus and associated blood vessels has received attention. \textsuperscript{577–581} Structures in small insects were imaged in an SEM based microCT system. \textsuperscript{582} MicroCT of structures in fossil fish \textsuperscript{583} and in other palaeontological specimens \textsuperscript{584} have been reported. MicroCT was used as the input for quantifying skeletal chord lengths for estimating doses received by the cells in the bone marrow during nuclear therapy. \textsuperscript{585} Comparison of trabecular structure across different mouse genetic strains \textsuperscript{345} and of femoral heads of two different primates with very different sizes and loading environments \textsuperscript{139} were other studies with a metrology component.

**Multimode studies**

A number of groups have employed X-ray microCT and another X-ray modality to gain a more complete understanding than either method could have provided separately. One example of combined position resolved X-ray scattering and absorption microCT appeared elsewhere. \textsuperscript{586} Here, the first example is microstructural characterisation of sea urchin teeth: X-ray microbeam mapping and precision lattice parameter determination was combined with microCT. The second example is sulphate attack of Portland cement paste where energy dispersive X-ray diffraction mapping of reaction products was used along with microCT. Three combined diffraction and microCT studies of mechanical responses of specimens provide the next examples: X-ray mesotexture analysis and microCT quantification of changes in fatigue crack opening; microCT plus X-ray diffraction of creep damage and diffraction based strain mapping plus microCT of load redistribution in monofilament metal matrix composites. The final examples are on the composite material bone: internal stress analysis plus microCT of loaded bone and diffraction characterisation and microCT of mineralisation versus fetal age in human vertebrae.

**Sea urchin teeth**

Sea urchin teeth differ from the urchins’ other skeletal elements in that the tooth does not consist of stereom (see the section on ‘Cellular materials’) but rather of a dense, single crystal calcite structure suited to rasping food from the surfaces of rocks. As sea urchin teeth contain their entire developmental history, they have received considerable attention as a biomineralisation model. At the tooth’s aboral end, deep within the urchin, little mineral is present, but by the adoral end, i.e. the cutting edge, mineral has filled virtually all of the tooth’s volume. Within the tooth, myriad reinforcement strategies are employed to make a functional structure from an otherwise wretched structural material (calcite). In order to understand the features’ roles, a brief digression on sea urchin tooth structure and its development must precede discussion of the microCT and microbeam diffraction results.

Sea urchin teeth have cross-sections shaped like ‘U’ or ‘T’ (sand dollars, closely related to the regular sea urchins discussed here, have teeth with diamond cross-sections), but brevity requires the discussion be limited to the ‘T’ shaped or camarodont teeth, the subject of this subsection. The leg of the ‘T’ is called the keel, and the bar across the top is termed the flange. The five teeth mounted in the five pyramids (each consisting of two demipyramids, Fig. 8) of the oral apparatus have their flanges tangent to the oral cavity and their keels running radially from the flange to the axis of the jaw structure. Each tooth functions as a T girder, a structure providing considerable resistance to bending per unit mass, with the flange loaded in compression and the keel loaded in tension.

The microstructure of the mature flange and keel seem well adapted to compression loading. The flange consists of a stack of plates paralleling the upper and lower surfaces of the bar of the ‘T’ and close to parallel to the tooth’s axis, and the interior of the flange contains thin needles running along the tooth’s axis and extending into the keel where they widen into much larger diameter prisms. The flange plates and needles are compressed end-on during eating, a geometry allowing high resistance to wear and to catastrophic failure. The keel, loaded in tension when the flange is compressed on hard surfaces, is essentially a composite structure reinforced by fibers (prisms) aligned along the tensile loading axis. In addition to the prisms within the centre of the keel, teeth of sea urchins such as *Lytechinus variegatus*, the subject of the multimode studies described below, also have carinar process plates running along the flanks of the keel. Synchrotron microCT observations of carinar process plate orientation in *L. variegatus* suggested that these plates (on the sides of the keel) serve to prevent deflection (and fracture) of the keel along a secondary bending axis, i.e. a situation that might be encountered.
when only one side of the flange is in contact with a hard substrate.89

Sea urchin teeth mineralise in two stages. The plates within the flange form first followed by needles/prisms and carinar process plates. In terms of the macroscopic structure, the flange forms first followed by the keel (which begins midway along the tooth’s length). The stacks of primary and secondary plates consist of individual single crystalline, spatially separate but highly crystallographically aligned elements. These plates, as well as the needles/prisms and carinar process plates that form after the flange plates, are termed the primary mineralised tissue and in *L. variegatus*, are non-equilibrium calcite $Ca_{1-x}Mg_xCO_3$ with $x=0.13$, as determined by synchrotron X-ray diffraction.27 At about the point where the keel develops, the primary skeletal elements begin to be linked with what are termed columns or discs. The columns are much higher Mg calcite with $x=0.33$.27 These columns, linking adjacent primary structural elements into a rigid structure, are often described as being polycrystalline, but the transmission X-ray diffraction data clearly show that the high Mg ($x=0.13$) and very high Mg ($x=0.33$) phases have their crystallographic axes identically aligned. The view of Stock and co-workers,27 therefore, is that the tooth is a compositionally modulated crystal much like multiple quantum well structures from molecular beam epitaxy.178 X-ray diffraction mapping

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15 Intergranular stress corrosion cracks in sensitised 302 stainless steel immersed in 0.15 mol K$_2$S$_2$O$_8$ acidified with dilute H$_2$SO$_4$ to pH 2. Imaging was with 30 keV synchrotron X-radiation and with detector 40 mm from specimen, and reconstruction was with 0.7 μm isotropic voxels. Labels have following meanings: CA1 and CA3: positions of crack arrest, L1–L4 uncracked ligaments; C1 and C2: crack segments. Nine rectangular images on left side of figure are parallel numerical sections (cuts) through stack of slices; vertical direction is direction along which stress was applied. This data were recorded after initial increment of cracking under applied stress of 100 MPa and with open electrical circuit. Relative separation between the cuts (in μm) is given by value of $x$. White blurred edging along cracks is from phase contrast. Renderings in right hand portion of figure show same volume of specimen after second increment of crack growth which was carried out with applied stress of 60 MPa (a, top) and after third increment under 40 MPa applied stress (d, bottom). Circled numbers identify different grains, and rectangular images at far right at oblique cuts through volume indicated on renderings (i.e. oblique cut ABCD in b shows ligament L4 in a). © 2006 The Institute of Materials Minerals and Mining. Reprinted from Ref. 574, with permission of Maney Publishing (http://www.ingentaconnect.com/content/maney/mst)
(precision lattice parameter and crystallite size/microstrain broadening determinations) provided additional structural information supplementing 3D microCT derived geometric information.

**Sulphate ion attack of Portland cement**

Study of sulphate ion attack of Portland cement via microCT was introduced briefly above (environmental attack) where it was noted that microCT showed the results of the attack but provided little information about the reaction phases producing the damage (i.e. the softening, cracking, loss of adhesion, etc., of the cement). Position resolved X-ray diffraction with high energy synchrotron X-radiation is a good method of mapping phase content as a function of depth, and using this method and microCT provided much more information than either technique by itself.\(^5\)\(^6\)\(^7\)\(^8\)\(^9\)\(^\text{560,587}\) Energy dispersive X-ray diffraction was used instead of the more normal single wavelength methods because the former allowed more precise definition of the sampling volume combined with simultaneous collection of diffraction patterns from multiple phases within this same volume. The reader is directed elsewhere for more details specific to this application.\(^5\)\(^9\)

Sulphate attack is (simplistically) described in literature by one of two classes of reaction and associated damage. The first is gypsum formation which is associated with loss of adhesion and strength. The second is ettringite formation associated with expansion and cracking. After considerable sulphate exposure, energy dispersive X-ray diffraction identified an ettringite rich, gypsum free layer outside of cylindrical cracks paralleling the outer surface of the cylindrical specimens (i.e. C1 in Fig. 12). Inside the crack, i.e. closer to the cylinder centre, a gypsum containing volume was identified.\(^4\)\(^5\)\(^8\)\(^-\)\(^6\)\(^0\)\(^\text{,586,587}\) While the same identification might have been performed by destructive specimen preparation (with considerably more effort), the results could have been criticised as affected by exposure to the atmosphere, etc.

**Fatigue crack path and mesotexture**

MicroCT of cracked AA2090 specimens revealed complex 3D patterns of crack face contact as a function of applied load (see the deformation subsection). Roughness of the crack faces produced the closure effects and is intrinsically related to the low fatigue crack propagation rate for this material. The underlying question is what drives the crack to assume this highly non-planar path: fracture mechanics indicates that the energetically favourable path would be more or less directly across the specimen (i.e. a path perpendicular to the applied tensile load). Yoder and co-workers\(^4\)\(^5\)\(^7\) related the average texture or macrotexture to the faces of asperities (large peaks) on the fracture surface; while this data explained the geometry of asperities and why they, on the average, formed, these observations did not identify the cause of the transition between an asperity and a relatively planar section of the crack. Microbeam Laue pattern mapping revealed the scale of crystallographic texture between that of individual grains (macrotexture) and the average specimen texture (macrotexture).\(^3\)\(^8\)\(^8\)\(^9\)\(^\text{,588,589}\) This particular type of mesotexture consisted of groups of 5–10 adjacent pancake shaped grains with nearly identical orientations, that is, these adjacent grains comprise near single crystal volumes. Asperities formed when the fatigue crack passed through the border between near single crystal volumes with different orientations. Further, a large fraction of the volume of the plate centres of AA2090 T8E41 consists of near single crystal domains, and this differentiates AA2090 from other Al-Li alloys with similar macrotextures\(^5\)\(^9\)\(^0\) and produces decreased fatigue crack growth rates compared to the other alloys.

**Creep damage**

Pyzalla *et al.* studied creep of a three phase copper alloy (Pb particles in a mixture of z and β brass) using synchrotron microCT and X-ray diffraction. Three detector systems were positioned so that microCT, energy dispersive X-ray diffraction and angle dispersive X-ray diffraction could be performed sequentially without realignment or recalibration. The microCT determined pore size distribution was reported for 10 time intervals and agreed with an exponential growth dependence. The decrease in diffraction peak FWHM ended when the voids started to reach appreciable size and changes in peak intensity after this point in the creep test revealed texture formation.

**Load redistribution in damaged monofilament composites**

Failure of uniaxially aligned, monofilament reinforced composites depends on many factors. MicroCT allows one to study where and at what applied stresses the reinforcements fail; repeated observations of the same specimen are particularly important because fibers such as the SCS series SiC monofilaments will often fracture several times within a 10 mm gage section; the location of each successive break is an important input for modelling. The increasing strain within the fibre, longitudinal and transverse strains within the matrix, strain relaxation to either side of fibre fractures and the fibre matrix interface strength are other important quantities that microCT alone cannot define. As demonstrated by a series of reports on Ti/SiC\(_\text{c}\) composites, combining high energy X-ray microbeam diffraction mapping with microCT has proved to be a powerful approach to measuring these quantities.\(^4\)\(^6\)\(^7\)\(^8\)\(^9\)\(^\text{,470,474,591}\)

Preuss *et al.*\(^4\)\(^\text{34}\) studied deformation of a single SiC fibre in a Ti-6Al-4V matrix: synchrotron microCT revealed the position and morphology of SiC fibre fractures and as a function of applied stress, microbeam diffraction quantified the matrix and fibre strains. At each of nineteen loading steps, mapping with transmission X-ray diffraction along the length of the fibre (100 diffraction profiles spaced by 50 \(\mu\)m steps at each load) revealed SiC longitudinal strains of at least 1-5% before the fibre cracked at the first point (equivalent to a failure stress of at least 6 GPa for \(E=400\) GPa). Above the nominal yield stress for the matrix, strains in the matrix became only slightly non-linear but the fibre longitudinal strain rose very rapidly. At 790 MPa, the load preceding first fracture, two local strain maxima were observed along the length of the monofilament; the next deformation increment produced a load drop and local strains approaching zero at the positions of the two maxima, positions that corresponded to fibre fracture revealed in microCT. Longitudinal strains in the matrix were relatively uniform at 790 MPa, but at the next deformation state, rose sharply at the positions where the fibre fractured. In other words, localised strain
concentration occurred in the matrix in the vicinity of the SiC breaks. Fitting the data to a partial sliding model allowed the authors to estimate a constant interfacial friction shear stress of ~200 MPa that was significantly higher than results from fibre push-out tests. The authors note that blind application of conventional full fragmentation post-mortem analysis of fragment lengths would suggest a significantly higher interfacial strength (~700 MPa) and suggest that fibre strength decreases after the first fast fracture event.

Well defined defects were introduced into a single ply Ti/SiC₃ composite, and redistribution of loads from damaged fibres to neighbouring ones was investigated with microbeam diffraction mapping.473 Load redistribution around damage sites increased the load in the nearest neighbour fibres by ~25% and second nearest neighbours by ~10%. The interfacial fractional shear stress was found to be similar or slightly larger than that cited in the previous paragraph. Reverse sliding was observed during unloading and produced compressive residual stresses near the fibre ends. Wedge crack geometry was frequently observed.471,472

In the examples of a single fibre and a single ply composite described above, simple phase enhanced microradiography would have sufficed to correlate fibre fracture and maxima/minima in the fibre and matrix strain profiles. In multiple ply composites, the overlapping fibre images necessitate use of microCT, and microCT plus microbeam diffraction mapping was applied to multiple ply Ti/SiC₃ to determine the stress partition between fibre bridging a fatigue crack and broken fibres.473,474,591 Initially, strain mapping averaged over the entire thickness of the specimen,473 but subsequent experiments used a narrow receiving slit and 20° scanning to limit the gage volume to a single SiC fibre plus the surrounding matrix material.474 Strain distribution in an intact fibre in the crack wakes was compared for maximum and minimum applied loads, for example; strain distribution as a function of distance from the crack plane was analyzed using a partial debonding shear lag model.474 Measurements of crack opening displacements showed that the fatigue crack front bowed out between fibers when it emerged from a ply and advanced preferentially towards fibres when the front was between plies.591 Further, the 3D distributions of crack opening were measured for three stress intensities characteristic of a fatigue cycle (K_max, K_min and K_incl), and very little variation in crack opening was observed parallel to the crack front irrespective of the proximity to bridging fibres.591

Bone

X-ray scattering measurement of internal strains (and conversion to internal stresses) in loaded bone (or tooth dentin and enamel) is a relatively uninvestigated research area and one where a combination with microCT will provide valuable insight. For bone, the collagen D period (~67 nm) along the fibril axis produces SANS peaks, and the Angstrom level periodicities of carbonated apatite (cAp) crystallites produce diffraction peaks in the WAXS regime. While the mineral nanoparticles in long bone have a pronounced crystallographic texture, there are still enough orientations present to produce more or less complete cones of diffracted intensity for monochromatic X-rays; Debye cones from different hkl exist simultaneously and produce rings of increased intensity on area detectors. Force applied to a specimen distorts the unit cells and alters the Debye cones. Hydrostatic applied stresses (those with equal magnitude in all directions) uniformly alter the diameter of cones, whereas deviatoric stresses (those with directionality) change the shape of diffraction rings. Similarly, SAXS peak positions (from the collagen D period) alter in response to applied stress.

X-ray scattering measures quantities such as d_hkl in cAp or the D period in collagen, and changes in these quantities define the internal strain imposed during loading, i.e. strain in cAp is \( \varepsilon_{\text{cAp}} = (d_d - d_d\text{initial})/d_d\text{initial} \) and in collagen is \( \varepsilon_{\text{collagen}} = (D - D\text{initial})/D\text{initial} \). Internal stress is a quantity derived from internal strain, and stress \( \sigma_1 \) and strain \( \varepsilon_2 \) are second rank tensors related through the fourth rank elastic constants \( C_{ijkl} \) i.e. \( \sigma_1 = C_{ijkl} \varepsilon_{kl} \). Describing the conversion of deviatoric strains to deviatoric stresses is beyond the scope of this review, and the reader is directed elsewhere for details as pertains to bone internal stress measurements.

The inverse of the slope of macroscopic strain \( \varepsilon_{\text{macro}} \) (measured by an attached strain gage) as a function of \( \sigma_{\text{applied}} \) (measured by the load cell of the mechanical testing apparatus) is Young’s modulus for the specimen. Such slopes for the WAXS and SAXS data \( (\varepsilon_{\text{cAp}} \text{ and } \varepsilon_{\text{collagen}} \text{ versus } \sigma_{\text{applied}}) \) reflect Young’s modulus for the individual constituent phase of bone. While this last extension may not be strictly correct, it does provide a numerical operational probe of how the the individual phases differ from pure (inorganic) cAp or collagen. For a section of canine fibula, the resulting moduli (90% confidence limit) are: \( E_{\text{macro}} = 24\text{-}70\text{(2)} \) GPa, \( E_{\text{cAp}} = 41\text{(1)} \) GPa and \( E_{\text{collagen}} = 18\text{(1)} \) GPa.593 The value for \( E_{\text{macro}} \) is in good agreement with moduli of similar bone types reported in literature; for cAp, it is about one-third of that of inorganic apatite; for collagen, it is at least nine times higher than one would expect.593 The data demonstrate the extent to which the local environment affects the different phases’ response to applied load.

In the studies described in the preceding pair of paragraphs, microCT was used to measure cross-sectional area and to account for internal porosity. This is a rather trivial usage of microCT because of the simple geometry. In specimens containing bone in complex geometries, however, i.e. specimens containing trabecular bone or cortical plus trabecular bone, determination of the spatial distribution of bone segments and of their orientation relative to the load axis will be essential for proper interpretation of the scattering data. Obtaining such 3D maps is impractical except through microCT, especially when one considers that the different bone segments may suffer significant relative displacements during loading, displacements that may change from load to load and that may not be preserved during post-testing serial sectioning.

X-ray diffraction and synchrotron microCT were combined in a study of changing mineralization during fetal vertebrae growth.529 From microCT, trabeculae were much thicker and more widely spaced in the interior of the vertebrae than in the peripheral volume, and bone volume fraction increased linearly over gestational ages of 16 through 24 weeks. X-ray diffraction revealed a linear increase in crystallite size with age and a linear increase in lattice parameter ratio \( c/a \) (both

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over the same gestational range as above). As complete understanding of bone mechanical properties depends not only on properly incorporating microarchitecture but also inclusion of proper materials properties (in bone these include crystallite dimensions and distortions of the apatite unit cell), more studies of this sort need to be completed and the data imported into numerical models of elastic moduli, etc. In another study, diffraction with small diameter synchrotron X-ray beams was combined with synchrotron microCT to characterise coronary atherosclerosis in vitro.\footnote{594} It may be that diffraction based measures of crystallite size, microstrain or \( \text{cr} / \text{mc} \) ratio will help define the natural history of such pathological mineralisation processes or even the rate of development of these dangerous structures.

**MicroCT accuracy**

The question of how accurate microCT reconstructions are, has received considerable attention. The many direct comparisons have established beyond doubt that microCT provides accurate reconstructions of specimen volumes; further, microCT’s limitations are nearly as well established (that is not to say that microCT data cannot be, and has not been, misinterpreted or the technique misused or misapplied). Also important is understanding what can be resolved (spatial resolution, differences in values of linear attenuation coefficient) and what can be detected (sensitivity limits). Knowledge of the dependence of spatial resolution and contrast sensitivity on experimental parameters as well as the intrinsic microstructural characteristics is central to reasonable interpretation of microCT data.

Assessing the accuracy of microCT reconstructions requires comparison of reconstructions with the results of another independent technique on the same specimen. Lab microCT versus microMRI (magnetic resonance imaging) provided one comparison,\footnote{55} and synchrotron microCT versus scanning acoustic microscopy of osteonal bone furnished a second example verifying microCT’s accuracy.\footnote{395} Bone formation in polymeric scaffolds was evaluated by proton magnetic resonance microscopy and microCT.\footnote{382} Physical sectioning compared to microCT slices has been the subject of still other studies confirming validity of reconstructions: confocal optical microscopy of thin serial sections contrasted with microCT of lung specimens;\footnote{596} histology versus microCT of cortical bone;\footnote{427} and histomorphometry versus microCT of biopsies of cancellous bone.\footnote{597, 598} Calcein labelling is a standard method used to show where new bone is formed, and, in a longitudinal in vivo study of rat tibiae positions of calcein labelling matched positions where microCT showed new bone had formed.\footnote{539}

MicroCT determination of cortical porosity and of mineral levels showed good agreement with the results of axial ultrasound velocity measurements in the human radius.\footnote{416} MicroCT versus radiography of the same portions of human femora showed good agreement for measures of cortical porosity.\footnote{424} Over a significant range of strains (\( \approx 1<\text{cr}<1:7 \)), Martin et al.\footnote{492} found excellent agreement between the volume fraction of cavities measured with synchrotron microCT and macroscopic measurements of density. The distribution of particle sizes in pumice clasts showed good agreement between synchrotron microCT and the classic crushing/sieving/winnowing method.\footnote{200} Yarn dimensions and spacings in 3D textiles and their variability were identical in measurements performed with lab microCT, surface scanning and optical microscopy of cross-sections.\footnote{303}

Sheppard et al.\footnote{255} found good agreement between simulations of mercury invasion capillary pressure based on 3D pore quantification in four varied specimens (microCT based) and actual measurements performed on the same specimens, with the main differences being attributable to microporosity below the resolution limit in the reconstructions. In a specimen of packed, monodisperse beads (3-0 mm nominal diameter), the algorithms of Sheppard et al.\footnote{598} determined a mean particle diameter of 2:98 mm (full width at half maximum: \( \approx 0:06 \) mm, slightly less than one 63-4 \( \mu \)m voxel) in the reconstruction; in a second specimen of unconsolidated sandstone, the distribution of particle diameters from lab microCT agreed with results of laser light scattering.

Accuracy and precision of lung tumour volume measurements were determined from respiratory gated in vivo microCT of a mouse model.\footnote{224} Lung tumour volumes were both reproducible (2\% operator variability) and accurate (6\% average error), and tumour number assessed at necropsy correlated significantly with microCT. Relatively poor contrast between soft tissue types (tumours, blood vessels) was typical of absorption microCT, but the authors employed careful differentiation procedures. Spatial resolution was somewhat limited in both microCT (91 \( \mu \)m isotropic voxels) and in optical inspection (0:5 mm detection limit for tumours). Despite these limitations, this study is convincing, in no small part because of the thorough account provided.

Davis discussed expected image quality and accuracy in data obtained with a lab cone beam microCT system;\footnote{599} different specimen geometries were examined for different cone beam angles. Correction for beam hardening in microCT quantification and the effect of beam hardening on resolution have been discussed.\footnote{600} Determination of actual versus nominal resolving power was described elsewhere.\footnote{601}

One investigation of the reproducibility of microCT data collection found the results stable with respect to replication, and displacement of the 6 mm long ROI by up 4 mm along the axis of the trabecular cored specimen produced little change in microscopic parameters.\footnote{602} Olurin et al.\footnote{25} examined the dependence of morphometric indices for closed cell Al foam and for two thicknesses of Al foil as a function of scan parameters in a lab microCT system and found characteristics such as volume fraction and mean feature thickness did not depend appreciably on voxel size for their specimens and scanner.

Examination of potential differences in reconstructions produced by different microCT instruments has been the subject of other studies. For example, results of the second generation versus the third generation synchrotron microCT as well as absorption versus phase microCT have been compared for claws of cats (bone as well as tough cornified tissue).\footnote{603} Comparisons of lab and synchrotron microCT are particularly informative for applications such as bone or tooth around metal implants where there is a large difference in absorptivity: One such study examined bone surrounding Ti
implants\textsuperscript{388} and a second characterised bone around dental implants.\textsuperscript{365}

Repeated imaging of the same trabecular bone specimen with three different systems (voxel sizes between 14 and 2 μm) showed that the larger size provided adequate characterisation of the trabecular structure.\textsuperscript{237} A result to be expected because trabeculae are typically \(\sim 100\) μm thick, i.e. on the order of 5 voxels along the minimum dimension. The effect of different scanning and reconstruction voxel sizes on trabecular bone parameters was examined for one instrument, and, for the extreme case (voxel size of 110 μm versus mean trabecular thickness of 120 μm), differences in specific surface area (i.e. per unit volume of bone) were as large as 100\%.\textsuperscript{604} This study suggests that morphometry studies performed on low resolution pQCT systems should be evaluated very carefully before being accepted (see Ref. 605 for discussion of circumstances where pQCT is accurate).

In a study of liquid foam with synchrotron microCT, variation of segmentation threshold by \(\pm 3\) units (on a 256 level grey scale) altered the volume fraction of liquid phase by on the order of \(\pm 2\%\).\textsuperscript{255} Lab microCT data were collected on trabecular bone biopsies (6, 23 and 230 weeks old porcine vertebrae); the three data sets were investigated systematically using a range of segmentation levels that an observer might select; the segmented scans were converted into FEM and a geometric assessment and not accuracy in contrast interpretation. As the considerations affecting contrast sensitivity were briefly discussed in the instrumentation section and as examples of analyses of linear attenuation coefficient (and estimates on the validity of the data) were provided in the examples on mineralised tissue, the discussion will end here with the note that much more work needs to be carried out in this area of data analysis.

**Date handling challenges**

The large amounts of data collected with lab microCT systems (potentially running 24 h of every day) and with synchrotron microCT (see below) are very challenging to handle. While the specifics vary from system to system, considering a single example (from the author’s recent experience at the synchrotron microCT facility at station 2-BM of APS) illustrates the main points.

In November 2006, nine 8 hour shifts were assigned, and the principle goal was to image a large number of sea urchin spines for a detailed comparison of design variations within one phylogenic family. As such, maximising throughput was essential, and this dictated that the 2-BM specimen placement robot\textsuperscript{45} would be used. One shift was lost to overnight problems with the robot, but otherwise the robot performed smoothly. A total of 91 specimen volumes were imaged during the eight shifts (1-3 specimens/h); with the present hardware and operating system one reckons that the maximum is 3 specimens/h (1200 s/specimen). It is instructive, therefore, to examine the durations of various portions of the data collection cycle and their effect on throughput.

The November run was done with a 2 K \(\times\) 2 K detector coupled with an optical lens to a single crystal of CdWO\textsubscript{4}, and views were collected every 0-25° (significant angular undersampling outside the central region of 1 K voxels diameter). Two-thirds of the specimens were reconstructed with 1-4 μm isotropic voxels and the balance with \(\sim 2-8\) μm voxels. All but six specimens were imaged with 20 keV photons; otherwise 26 keV X-radiation was used. Typical acquisition times were 0-2 s/view (slightly fewer than 4 K counts collected at the most intensely exposed pixels of the reference images) or less using the double multilayer monochromator (DMM), for a total of \(\sim 150\) s of actual acquisition. The other 87\% of the time is occupied mainly by sample motions and handshaking among various hardware components. Removal and placement of specimens occupies no more than a couple of minutes per specimen. Loading the specimen holders onto the robot sample tray (up to 24 specimens per tray) requires \(\sim 10\) min (the specimens are placed on the holders while the previous tray is being collected), so this is a negligible delay when spread over multiple specimens and multiple trays.

The difference between specimens actually imaged (91 specimens) and the expected throughput (192 specimens) arises from sources other than those listed above. Some ‘wasted’ time is inevitable between finishing one tray and starting the next, but this is a very minor component. In the absence of other effects, writing to an old disk array (the late 1990s technology) was about twice as slow as writing a newer disk array (2006 parallelised and scalable array); however, data for most of the trays described above were written with the newer array. The \(\sim 50\%\) decrease in data collection rate is probably produced by non-optimum tuning of the various network and hardware components.\textsuperscript{306}

Reconstructing the data sets at the tomography facility at station 2-BM of APS is very rapid compared to other aspects of data handling, and most users can expect to leave the facility at the end of their shifts with a significant fraction of their data reconstructed but probably not in hand. Currently data is in HDF-4 format; each stack of 2 K slices from a single specimen amounts to 20–25 GByte, depending on the amount of dynamic compression possible, and writing this data to DVDs is no longer practical. During the November 2006 run, transferring 1 TByte data (40–50 specimens) from the data analysis cluster (Linux) to an external USB-2 hard drive attached to a PC running Windows required 40 h. When the drives were attached to a Linux machine and formatted as ext2, the transfer took 15 h.\textsuperscript{306}

Data collection rates will continue to increase. One hopes that the overall infrastructure (network, disks, etc.) keeps pace, or, at least, does not lag further behind. Of course the biggest bottleneck of all is the lag between...
Speculations on future trends

Several trends for future micro- and nanoCT are clear from recently published studies. First consider data presentation and what constitutes an ‘adequate’ study. Many recent microCT papers incorporate colour images (if not in the hardcopy, then in the online version of the journal). As the human eye distinguishes more levels of contrast in colour images than in greyscale images, colour is sometimes used to increase the dynamic range visible in slices, but this is used less frequently than one would expect. Colour has been used primarily to present four- or more dimensional data or to label different discrete subvolumes within 3D renderings, and these types of colour images will be increasingly important in descriptions of scientific and engineering studies. More than a few colour varieties have appeared, however, that could have been equally effective as greyscale images, but this, perhaps, is an overly pedantic observation.

Supplemental data, in particular movies, posted on journal websites, have become an increasingly important component of publications. Movies paging through a stack of slices, showing different perspectives of a rendered volume or removing outer layers of an object and exposing interior structure are popular supplements. Earlier, microCT was used to create physical models with rapid prototyping manufacture (e.g. osteoporotic and normal trabecular bone) but this is too expensive to do on a routine basis and 3D renderings (spinning or viewing perspective under user control) can be produced without particular difficulty or expense.

Some groups maintain websites where extensive microCT or CT datasets are posted. For example, the digital morphology website (http://www.digimorph.org) includes anatomical data on a wide variety of animals, renderings and slices of which can be viewed in a variety of ways. The visible cement website maintained by NIST contains synchrotron microCT datasets and serves as a standard for benchmarking new analysis programs for this class of materials.

As the number of microCT publications has increased, expectations for the quality of and depth of analysis in published studies have risen. What was a strong PhD thesis in the early to the mid 1990s, became, by the late 1990s (in this author’s opinion), only an adequate MSc thesis. The same is true of papers in archival journals. Although long explanations of analysis methods and of the principles of microCT data collection remain appropriate for theses, very little of this should appear in journal papers, given previous coverage in literature. One now expects not only interpretation of geometry defined via single thresholds of number remain appropriate for theses, very little of this methods and of the principles of microCT data collection, but also (brief but rigorous) consideration of numerical values of linear attenuation coefficients. If binary segmentation is used for numerical analysis, short but detailed examination of the effect of threshold choice should be incorporated; more complicated segmentation routines require presentation of more details.

Future studies will incorporate more elaborate loading apparatus and environmental chambers (furnaces, cooling stages, high pressure chambers) and more elaborate and better calibrated monitoring of experimental conditions. Peripherals purpose built by manufacturers for their commercial microCT systems are already appearing and will undoubtedly appear in future publications. Emphasis will surely continue on repeated observations of the same specimen: a wider range of in situ and in vivo studies with increased data acquisition rates. Approaches such as that of Bay (and many others for deformation of cellular materials, cited above), i.e. computation of local strains from incremental microCT or nanoCT derived deflections, will probably be used more frequently as computing power continues to increase. Such digital image correlation techniques are not, of course, the exclusive province of those doing mechanical testing, and one expects their increasing use in quantitative studies of temporally evolving structures.

More studies will appear on very fast phenomena using gating to freeze movement such as found in sprays; such gating is quite involved, however, so the number of such studies will remain relatively small. The author expects data acquisition rates to increase with tube based microCT systems, but the biggest changes in this area will probably occur at synchrotron radiation sources. Data in real time involving changes occurring over several minutes (time averaging over times approaching 10 s) will continue to increase. As new generations of undulator sources are commissioned, the author would not be surprised if, in the next decade, data acquisition advances to the point where (512) reconstructions with greater than 8 bit contrast can be obtained for time intervals of 1 s or less. Exactly how to store and process such amounts of data will be a continuing challenge.

Studies looking at evolution in the structure of individual specimens should emphasise incorporation of proper boundary conditions which in practice means larger volumes of material surrounding the volume of interest. Incorporation of microstructure directly into finite element or other numerical models will be an area that will continue to grow. Reconstructions using 2 K × 2 K detectors are now standard, and introduction of 4 K detector widths (in the plane of reconstruction) will be a direct approach for preserving spatial resolution while examining large diameter samples. More frequent use of local tomography also is expected at synchrotron radiation sources, although specimens with significant absorption will be limited in size by the decrease in contrast from the extra absorption of the material outside of the region of interest; sensitivity is severely affected by noise accompanying large decreases in the number of photons traversing the material of interest. The author hopes that the manufacturers of commercial microCT systems will add the option of local tomography data acquisition to their systems (if this capability is already incorporated in an off the shelf system, the author is not aware of it), but this may be of too limited an interest to be commercially supported.

MicroCT use will be applied more often as part of studies integrating it with other scales of testing and analysis or with other techniques such as X-ray microbeam diffraction mapping. The multimode studies described in a preceding section provide examples, but one should not forget that methods other than those employing X-rays can be used. One expects more studies will centre on key specimens linking microscale (samples
with optimum dimensions for contrast sensitivity) with macroscopic scale of more normal engineering specimens. Some intermediate sized specimens may also need to be studied to complete the linkage between different structural scales. Although such studies are not as novel as they were a few years ago, the earlier demonstrations may actually make it easier to organise the resources required for more detailed, multiscale research programmes.

More nanoCT and phase micro/nanoCT studies will appear in the future, especially as more commercial nanoCT systems are installed. One expects in the near future to see commercial phase microCT imaging systems using the grating method or perhaps the analyser crystal (diffraction enhanced imaging) method. The propagation method will probably not be used because of the large but precise translations needed. Stability issues may be a critical determinant of whether it proves practical to produce commercial phase microCT systems. Crystal optics of any sort can be finicky, and implementing a robust system of collecting lab based diffraction enhanced phase radiographs will probably require considerable additional hardware (e.g. feedback circuitry) to guard against optics drift. Collecting views at three or more positions of the analyser crystal will increase data collection times, therefore, by at least a factor of three compared to normal absorption microCT acquisition (and this does not include the effect of decreased flux from the optics, i.e. from wavelength rejection due to beam monochromatisation). Grating based phase microCT systems should be relatively stable, and the micrometre sized translation of the second analyser grating should not be too difficult to implement with current piezoelectric translators. All wavelengths passing through the specimen contribute to image formation. Whether the translation of gratings in a commercial system can be controlled robustly enough for day in and day out data collection by users or staff without extensive experience remains to be proven.

The range of voxel sizes one sees in literature goes down to ~100 nm, and one expects this limit to inch downward but probably at an ever decreasing rate due to the engineering challenges. Transmission electron microscopy, for example, allows one to see nanometre sized features in specimens, and the main positioning requirement is that the specimen be held still during the exposure of the analog or electronic micrograph. Tomography requires specimen rotation, however, and, even with the best components and best autoalignment software, achieving sub 100 nm voxel size with tomography will be an interesting proposition. An often overlooked practical complication is the need to handle 10 μm or smaller sized specimens.

A developing but unproved method, one that should be tracked by all those interested in the highest achievable resolution, is the diffraction imaging of very small objects such as single protein molecules in solution.607 In this approach, a small diameter X-ray beam shines through a narrow stream of liquid containing a single macromolecule at a time. After an instant’s exposure, the macromolecule drops out of the beam and is replaced by a second macromolecule. Laser light is used to align the macromolecules so that the small angle diffraction patterns from each are identically oriented. X-ray induced damage of the macromolecule is minimised while allowing good statistics to be collected in the scattering pattern. Changes in folding, etc., accompanying crystallisation, can also be avoided. Reconstruction of 3D macromolecule is then undertaken from analysis of the scattering pattern. One supposes the macromolecule orientations could also be altered over time by reorienting the laser beam, something which is quite simple to do with standard optics, and perhaps this information would help with the reconstruction.

In summary, the reviewer sees a period of consolidation in the area of materials microCT characterisation. Instruments are widespread, new applications will certainly appear but truly novel developments or applications will be few as investigators concentrate on exploiting areas pioneered in the last decade. Of course, the thing about the most profound innovations is that they seem to come out of nowhere, so it will be interesting to see whether the next few years bring surprises in the area of nano- and microCT.

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