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**Three dimensional internal  
imaging of materials  
structure: a feasibility study**

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Three dimensional internal imaging of materials structure:  
a feasibility study

Materials Tools Project  
DTI Performance Programme (Apr 2004-Mar 2007)

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**ABSTRACT**

A review of the current status of true 3D imaging techniques has been carried out. The findings have been reported in two master tables. The study has focused on understanding industrial drivers, barriers and scope of 3D imaging methods and on identifying new opportunities, applications and new developments in 3D techniques with inputs from key players in the UK. A brief experimental assessment has been undertaken to report results of relative performance of three 3D imaging techniques on a series of artefacts (including bio- and nano-materials) at the macro, micro and nano size scales. A list of recommendations is proposed that could influence formulation of the NMS programmes to include 3D imaging techniques. A series of dissemination activities has been planned, which include an article in a newsletter, a dedicated NPL website and a future presentation at a focus group.

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## 1 INTRODUCTION

Industrial needs for understanding and optimising behaviour of complex materials are increasing rapidly. 3D imaging techniques are needed to characterise internal structure of a wide range of materials from traditional to the emerging nano- and bio-technology areas. Specific examples are in the manufacture of metal and polymer foams, evaluating tissue scaffolds and bone cements, in developing dental restorative and soft materials and in characterising nano-composites. Beyond its conventional use, the 3D imaging information has the potential to be correlated to materials properties such as density, hardness and modulus so as to provide not just a 3D imaging model, but also the 3D distribution of anisotropic material properties. A classic example of this is bone, where bone density and Young's modulus can be correlated to the signal strength of CT (computed tomography) scans. The performance of materials at the macro level can thus be determined from 3D imaging studies of complex internal structures or imperfections in materials.

The importance of internal materials structure in dictating final properties of materials is well documented. Understanding 3D morphology, distribution, interconnectivity and crystallography of non-biological solids containing two or more phases and cellular solids is critical in characterising material properties precisely. Until recently three-dimensional structures were derived from 2D observations from optical, SEM and TEM studies. Over the last 20 years a series of novel and innovative true 3D internal imaging techniques have emerged, which are currently widely used in the medical science, but are gradually, finding their suitable applications in a much wider materials field [Refs. 1-2].

This project aims to review current status, undertake brief experimental evaluation of selected techniques and address key issues that relate to future development of true 3D internal imaging techniques (surface / topological measurements fall outside the scope of this study) for a wide range of materials.

## 2 REVIEW OF CURRENT STATUS

One of the key purposes of this feasibility study is to assess strengths and weaknesses of currently available true 3D imaging tools in understanding complex inhomogeneous structures and distribution in a range of artefacts including bio- and nano-materials. The review attempts to focus on identifying the scope, limitations and availability of 3D measurements that are generally available and are gradually gaining popularity in the materials area in the UK and elsewhere. The most promising techniques, based on their novelty, speed, accuracy, ease of operation and cost-effectiveness have been identified in this exercise and the collated data are presented in Tables 1 and 2 [Refs: 3 - 45].

Table 1 lists several criteria, which should influence the initial decision of the user in selecting the correct 3D imaging technique(s) based on the scale of operation (resolution), type of test materials, cost and the existing capability within the NMS (e.g. in NPL). Additional factors, which might also influence the choice of techniques, are also listed in the table.

**Table 1 Factors affecting selection of 3D imaging techniques for materials**

Scale of Operation	Test Materials	3D Imaging Methods (Generic)	Relative Cost of Test	NPL Capability
Macro (1 mm upwards)	Metallic, Polymeric, Ceramic, Concrete and Bio-materials. Foams (polymeric & metallic), Wood, Composite & other Cellular materials	Acoustic (Ultrasound C-scan)	Low	Yes
		Optical (Stereo microscopy)	Low	Yes
		Optical (Laser Profilometry)	Low	Yes
		Optical (SOM)	Med	Yes
		Optical (OCT)	Low	Yes
		Thermal (with CT)	Med	Yes CT (no)
		X-ray CT	Med	No
		Gamma Ray (with CT)	Med	No
		Electron (SEM, STEM, EBCT)	High	Yes EBCT (no)
		Magnetic (MRI)	High	No
Micro (sub 1 mm)	Metallic, Polymeric, Ceramic, Dental, Electronic, Composites and other Cellular materials. Bone; other Bio- and Organic materials	Nuclear (PET with CT)	High	No
		Optical Sectioning (DVI, Cryo)	High	No
		Optical (OCT)	Low	Yes
		Acoustic (SAM)	Med	Yes
		Acoustic (Ultrasound CT)	Med	No
		X-Ray CT	Med	No
		Gamma Ray (with CT)	Med	No
		Magnetic (MRI)	High	No
Nano (sub Micrometer)	Nano-tube/fibre, Polymer Nano-Composites ; Electronic, Bio- and Organic materials	Nuclear (NMR, PET with CT)	High	No
		Electron (SEM / FIB & STEM)	Med	SEM (yes); FIB (no)
		Optical (SPM / AFM)	Med	Yes
		Electron (TEM, STEM)	Med	Yes
		Electron (SEM / FIB)	Med	SEM (yes); FIB (no)

**Key:**

CT = Computed Tomography SAM= Scanning Acoustic Microscopy SPM/ AFM= Scanning Probe Microscopy / Atomic Force Microscopy

TEM = Transmission Electron Microscopy STEM= Scanning Transmission Electron Microscopy FIB= Focussed Ion Beam SEM= Scanning Electron Microscopy

OCT = Optical Coherence Tomography SOM= Scanning Optical Microscopy MRI = Magnetic Resonance Imaging PET = Positron Emission Tomography

EBCT = Electron Beam Computed Tomography DVI = Digital Volumetric Imaging NMR = Nuclear Magnetic Resonance

**Other factors that might influence the choice of techniques are:** Metallic or Non-metallic, Conductivity (magnetic or electrical); Moisture content ; Depth profile required; true or pseudo 3D image

**Estimated cost / test:** : Low = <£500 ; Medium = £500- £1000 ; High: >£1000

Eight generic 3D imaging sources have been identified in the review. Table 2 lists a brief evaluation of increasingly popular 3D internal imaging techniques by referring to their strengths, limitations, applications and other key parameters, which have been collated from literature and from discussions with the scientific community, experts & peers both at NPL and outside. The 3D techniques are classified in this report in **Generic Groups** based on their original energy source and are listed as follows:

**Magnetic**

**Nuclear**

**X-ray**

**Acoustics**

**Optical**

**Electron**

**Thermal**

**Gamma-ray**

Table 2 further expands these original imaging routes (hyperlinked from the first page) and provides technological, commercial & user details of at least 3 widely used strands that fall under each imaging category (i.e. main generic technique). It is anticipated that these additional information would provide readers relevant information to focus on their specific requirements and enable them to consider key wider issues such as, **Industrial Drivers** (covered under applications/ uses/ accuracy and resolution), **Barriers** (cost/ limitations/ resolutions/ accuracy) and **Scope** (availability / present users / key contacts/ comments) of the 3D imaging techniques.

**Table 2: Details of Internal 3D Imaging Techniques (Most techniques are non-portable)**

<i>Generic Techniques</i>	
Magnetic	5
Nuclear	6
X-Ray	8
Acoustics	9
Optical	10
Electron	14
Thermal	16
Gamma Ray	17

Generic Technique	Imaging Methods	Applications/ Uses	Cost	Resolution/ Accuracy	Limitations	Availability (commercial/ R&D)	Useful Links and contacts	Comments
<b>Magnetic</b>	Diffusion MRI (MRI)	Used mainly to measure complete tissue diffusion profiles	High	High resolution (< 1mm) High Quality Directional imaging (tensor) 6 gradient directions Fractional Anisotropy (FA), can be computed Test time is commonly 60 minutes	<ul style="list-style-type: none"> <li>• Cost</li> <li>• Large size</li> <li>• High power requirement</li> <li>• Lack of portability</li> </ul>	Commercially available	<a href="http://www-sop.inria.fr/odyssee/team/Christophe.Lenglet/pages/gallery.html">http://www-sop.inria.fr/odyssee/team/Christophe.Lenglet/pages/gallery.html</a>	<ul style="list-style-type: none"> <li>• Diffusion MRI is a specific Magnetic Resonance Imaging (MRI) modality that produces in vivo images of biological tissues weighted with the local microstructural characteristics of water diffusion.</li> </ul>
	Functional MRI	This magnetic resonance imaging technique can be used to map changes in brain hemodynamics in order to map the human brain.	Medium/ High	Very high resolution (10 micron)	<ul style="list-style-type: none"> <li>• Cost</li> <li>• Large size</li> <li>• Limited to brain mapping</li> <li>• Lack of portability</li> </ul>	Commercially available	<a href="http://www.fmri.org/fmri.htm">http://www.fmri.org/fmri.htm</a> <a href="http://www.fmrib.ox.ac.uk/~stuart/thesis/">http://www.fmrib.ox.ac.uk/~stuart/thesis/</a>	<ul style="list-style-type: none"> <li>• Main advantages to fMRI as a technique to image brain activity related to a specific task or sensory process.</li> <li>• Due to the ability to image the entire 3-dimensional volume of brain, fMRI is capable of isolating many simultaneous and coordinated brain events. This "multi-level" view of brain activity can include "executive" functions and high level cognitive tasks simultaneously with the primary and secondary input such as vision and audition as well as cerebellar contributions</li> </ul>
	Interventional MRI	Interventional magnetic resonance imaging (IMRI) defines the intra-operative application of magnetic resonance imaging technology. Used in operative procedures in specialist applications	Very High	Excellent resolution and real time operation (down to 10 micron)	<ul style="list-style-type: none"> <li>• High Cost</li> <li>• Large size</li> <li>• High power requirement</li> <li>• Limited to research applications</li> <li>• Lack of portability.</li> </ul>	R and D Some commercial units (very rare and specialised)	<a href="http://neurosurgery.umc.edu/interventionalmri.html">http://neurosurgery.umc.edu/interventionalmri.html</a> <a href="http://ccir.uhrad.com/mri/imri/devices/">http://ccir.uhrad.com/mri/imri/devices/</a>	<ul style="list-style-type: none"> <li>• The virtual real-time feedback provided by the IMRI allows the neurosurgeon to precisely localize brain and spinal lesions at the time of surgery thus facilitating more accurate surgical exposure.</li> <li>• Diagnostics of tumours in the brain (during surgery) and spine.</li> </ul>

Generic Technique	Imaging Methods	Applications/ Uses	Cost	Resolution/ Accuracy	Limitations	Availability (commercial/ R&D)	Useful Links and contacts	Comments
<b>Nuclear (and Magnetic)</b>	NMR spectroscopy	Modern NMR spectroscopy is frequently divided into several categories.	Medium/ High	High resolution (typically ~1mm)	<ul style="list-style-type: none"> <li>• Test sample should be pure, non-paramagnetic and should not be a mixture of many compounds</li> <li>• Lack of portability</li> </ul>	Widely available	<a href="http://www.ch.ic.ac.uk/local/organic/nmr_principles.html">http://www.ch.ic.ac.uk/local/organic/nmr_principles.html</a>  <a href="http://saif.iitm.ac.in/facilities/4_nmr.html">http://saif.iitm.ac.in/facilities/4_nmr.html</a>	<ul style="list-style-type: none"> <li>• High-resolution mode on homogenous solutions.</li> <li>• High power mode on highly relaxing nuclei, which exhibit very broad lines, or polymers etc.</li> <li>• NMR 3D imaging to resolutions of ~1 mm.</li> </ul>
	Solid state NMR	Used for calculating solid state structures of polymers <a href="#">Membrane proteins</a> and <a href="#">amyloid</a> fibrils. <a href="#">Alzheimer's disease</a> and <a href="#">Parkinson's disease</a> , are two examples of application where solid-state NMR spectroscopy complements <a href="#">solution-state NMR spectroscopy</a> and beam diffraction methods (e.g. X-ray crystallography, electron microscopy).	Medium	High (typically 100 microns)	<ul style="list-style-type: none"> <li>• Cost</li> <li>• Size</li> <li>• Specialized operation</li> </ul>	Research/ limited commercial	<a href="http://www.process-nmr.com/solid.htm">http://www.process-nmr.com/solid.htm</a>  <a href="http://www.elsevier.com/wps/find/journaldescription.cws_home/622947/description#description">http://www.elsevier.com/wps/find/journaldescription.cws_home/622947/description#description</a>  <a href="http://www.projects.ex.ac.uk/nmr/">http://www.projects.ex.ac.uk/nmr/</a>	Alzheimer's disease and Parkinson's disease, are two examples of application where solid-state NMR spectroscopy complements solution-state NMR spectroscopy and beam diffraction methods (e.g. X-ray crystallography, electron microscopy).

Generic Technique	Imaging Methods	Applications/ Uses	Cost	Resolution/ Accuracy	Limitations	Availability (commercial/ R&D)	Useful Links and contacts	Comments
	Protein nuclear magnetic resonance spectroscopy	Used to analyse structure and dynamics of proteins	Medium/ High	Very high (nanometer resolution)	<ul style="list-style-type: none"> <li>• Cost</li> <li>• Large size</li> <li>• Limited to mapping protein structures</li> </ul>	Mostly research	<a href="http://www-nmr.cabm.rutgers.edu/photogallery/structures/index.html">http://www-nmr.cabm.rutgers.edu/photogallery/structures/index.html</a> <a href="http://www.palmer.hs.columbia.edu/protein_nmr_spectroscopy/index.html">http://www.palmer.hs.columbia.edu/protein_nmr_spectroscopy/index.html</a>	<ul style="list-style-type: none"> <li>• Can be used to calculate protein bond lengths and angles.</li> <li>• Protein NMR Spectroscopy combines a comprehensive theoretical treatment of high resolution NMR spectroscopy with an extensive exposition of the experimental techniques applicable to proteins and other biological macromolecules in solution. Beginning with simple theoretical models and experimental techniques, Protein NMR</li> </ul>
	Positron Emission Tomography (PET) with CT	Mainly used in the medical field to analyse tumours or metabolic abnormalities	Medium	High (Less than 100 microns)	<ul style="list-style-type: none"> <li>• Cost</li> <li>• Large size</li> <li>• Limited to brain mapping</li> <li>• Highly specialised and non-portable.</li> <li>• Limited time of operation</li> </ul>	Hospitals/ research	<a href="http://en.wikipedia.org/wiki/Positron_emission_tomography">http://en.wikipedia.org/wiki/Positron_emission_tomography</a> <a href="http://www.radiologyinfo.org/en/info.cfm?pg=pet&amp;bhcp=1">http://www.radiologyinfo.org/en/info.cfm?pg=pet&amp;bhcp=1</a>	Positron emission tomography, also called PET imaging or a PET scan, is a diagnostic examination that involves the acquisition of physiologic images based on the detection of radiation from the emission of positrons. Positrons are tiny particles emitted from a radioactive substance administered to the patient. The subsequent images of the human body developed with this technique are used to evaluate a variety of diseases.

Generic Technique	Imaging Methods	Applications/ Uses	Cost	Resolution/ Accuracy	Limitations	Availability (commercial/ R&D)	Useful Links and contacts	Comments
<b>X-Ray</b>	Computed Tomography/ CAT Cranial/ Chest/ Cardiac/ Abdominal and Pelvic.	used to generate a <a href="#">three-dimensional image</a> of the internals of an object from a large series of two-dimensional <a href="#">X-ray</a> images taken around a single axis of rotation	High (equipment cost £500K upwards)	1000 image study in under 30 seconds (very high resolution in 4 <sup>th</sup> Generation scanners)	<ul style="list-style-type: none"> <li>• Cost</li> <li>• Large size</li> <li>• Highly specialised</li> <li>• Lack of portability</li> </ul>	Widely Available/ Commercial/ Hospital/ Research	<a href="http://en.wikipedia.org/wiki/Computed_axial_tomography">http://en.wikipedia.org/wiki/Computed_axial_tomography</a>  <a href="http://www.radiologyinfo.org/sitemap/modal-alias.cfm?modal=CT">http://www.radiologyinfo.org/sitemap/modal-alias.cfm?modal=CT</a>	X-ray slice data is generated using an X-ray source that rotates around the object; X-ray sensors are positioned on the opposite side of the circle from the X-ray source. Many data scans are progressively taken as the object is gradually passed through the gantry. They are combined together by the mathematical procedure known as <a href="#">tomographic reconstruction</a> .
	3 –D Florescence microscopy WDS/ EDX/ EDS	X-ray Florescence (XRF) Spectroscopy involves measuring the intensity of x-rays emitted from a specimen as a function of energy or wavelength	Medium/ High	Used for calculating amounts of a particular atomic type in materials, resolution –atomic. Resolution dependant on emitance.	<ul style="list-style-type: none"> <li>• Limited to identifying atomic type</li> <li>• Size</li> <li>• Specialized operation</li> </ul>	Widely available in R & D and commercial uses	<a href="http://www.amptek.com/xrf.html">http://www.amptek.com/xrf.html</a>  <a href="http://www.src.le.ac.uk/instrumentation/mcp/papers/spectroscopy/">http://www.src.le.ac.uk/instrumentation/mcp/papers/spectroscopy/</a>	X-ray florescence (XRF) is a widely used non-destructive technique for chemical analysis. In the simplest geometry, the sample is uniformly irradiated and an averaged florescence spectrum obtained from its entire area. One determines which elements are present and in what proportions but obtains no information on their spatial distribution
	Micro X-ray 3-D Microscopy	Spectrometer from <a href="#">EDAX</a> utilizes the sensitivity and range of energy dispersive X-ray detection to produce fast, simultaneous, multi-element detection for elements sodium to uranium for 3 D mapping (internal)	Medium	Up to 40 micron	<ul style="list-style-type: none"> <li>• Cost</li> <li>• Highly specialized</li> <li>• Non-portable</li> </ul>	Specialized tool for image use	<a href="http://www.mwrn.com/microscope/spectroscopy/micro_xrf.aspx">http://www.mwrn.com/microscope/spectroscopy/micro_xrf.aspx</a>  <a href="http://en.wikipedia.org/wiki/Microscopy#Fluorescence_microscopy">http://en.wikipedia.org/wiki/Microscopy#Fluorescence_microscopy</a>	<ul style="list-style-type: none"> <li>• Non-destructive sample analysis</li> <li>• Little or no sample preparation for most materials (solids, liquids, powders, films and coatings, non-conductive, polymers...)</li> <li>• Large sample chamber accommodates irregular sample sizes and shapes</li> <li>• Fast, simultaneous, multi-element detection for elements sodium to uranium</li> <li>• Sensitivity from PPM to 100% concentration levels</li> </ul>

Generic Technique	Imaging Methods	Applications/ Uses	Cost	Resolution/ Accuracy	Limitations	Availability (commercial/ R&D)	Useful Links and contacts	Comments
<b>Acoustics</b>	3-D Sonar	Operates in a similar manner to a conventional mechanically scanned sonar system with the unique addition of height information for every point scanned	Medium to High	Can resolve to 0.1 mm within the 60 receive beam-width to generate the target height	<ul style="list-style-type: none"> <li>• Limited to marine applications</li> <li>• Non-portable.</li> </ul>	Military/ Salvage and Research	<a href="http://www.marine-electronics.co.uk/3D_5150.html">http://www.marine-electronics.co.uk/3D_5150.html</a>  <a href="http://www.boatersland.com/947c3d.html">http://www.boatersland.com/947c3d.html</a>	<ul style="list-style-type: none"> <li>• Enhanced 3D sonar uses 11 interlaced sonar beams for higher resolution across 531 of bottom, and offers up to 20 times more volume information.</li> <li>• Dual function sonar provides 3D capability to 240 feet and traditional 2D capability to 1000 Feet.</li> </ul>
	Scanning Acoustic Microscopy (SAM)	A scanning acoustic microscope works on the principle of propagation and reflection of acoustic waves at interfaces where a change of acoustic impedance ( $AI = \text{density} \times \text{velocity}$ ) occurs, commonly used to view the structure of materials in research/ acoustic compliant materials	Medium	High frequency" Scanning Acoustic Microscopy (SAM) from 0.1 -- 2 GHz Low frequency" Acoustic Sounding (AS) from 25 - 100 MHz	<ul style="list-style-type: none"> <li>• Limited to materials that propagate sound</li> </ul>	Widely available	<a href="http://pangea.stanford.edu/~manika/sam1.html">http://pangea.stanford.edu/~manika/sam1.html</a>  <a href="http://www.soest.hawaii.edu/~zinin/Zi-3D.html">http://www.soest.hawaii.edu/~zinin/Zi-3D.html</a>  <a href="http://www.asylumresearch.com/Products/Mfp3DCF/Mfp3DCF.shtml">http://www.asylumresearch.com/Products/Mfp3DCF/Mfp3DCF.shtml</a>	The angular spectrum approach can be used to develop the theory of subsurface imaging in acoustic microscopy. It takes into account reflection and transmission of the sound beam at the liquid-solid interface. The theory is of importance for understanding acoustical images of the internal microstructure of the non-transparent solids. This ensures that the internal 3-D structure can be viewed.
	Real-time radio frequency (RF) 3D ultrasound	Ultrasounds method using conventional 2-d probes with specialized software (real/time) utilising a series of images obtained in 2-d array scanners but using specialised software to splice 2-D images to produce a 3-d image/ mesh	Low	Same as conventional Ultrasound however resolution is entirely dependant on type of probe utilised.	<ul style="list-style-type: none"> <li>• Limited to the driving power of the transducers.</li> <li>• Non-portable.</li> </ul>	Widely available/ Hospitals	<a href="http://www.diagnosticimaging.com/AdvancedUS/3d.jhtml">http://www.diagnosticimaging.com/AdvancedUS/3d.jhtml</a>  <a href="http://www.csse.uwa.edu.au/~bernard/us3d.html">http://www.csse.uwa.edu.au/~bernard/us3d.html</a>	Traditionally known as ultrasound holography and developed in the 1950's forward this technique has been vastly improved by using more powerful computers which render the image at a higher resolution combined with greater speed to provide the user with real time information from 2-d liner probe geometries which although provide scans of 2-d geometry can be adjoined using software to produce real-time 3-D images.

Generic Technique	Imaging Methods	Applications/ Uses	Cost	Resolution/ Accuracy	Limitations	Availability (commercial/ R&D)	Useful Links and contacts	Comments
Optical	Near Field Scanning Optical Microscopy (SOM)	Near-field scanning optical microscopy provides a technique for examining specimens with ultra-high spatial optical resolution combined with software to produce 3D images	Low	The wavelength of the visible light used in scanning optical microscopes is between 400 and 700 nanometres (nm). The resolving powers of high-quality light microscopes are limited by the wavelength of imaging light to about 200 nanometres (0.2 microns, 0.2 $\mu$ m).	<ul style="list-style-type: none"> <li>Limited to real depth area unless the sample is destroyed then viewed using a cutting probe/ion technique</li> <li>Size</li> </ul>	Widely available	<a href="http://www.pku.edu.cn/academic/xb/97/97e318r.html">http://www.pku.edu.cn/academic/xb/97/97e318r.html</a>  <a href="http://www.life.umd.edu/CBMG/faculty/wolniak/wolniak_micro.html">http://www.life.umd.edu/CBMG/faculty/wolniak/wolniak_micro.html</a>	Also known as Confocal Scanning Optical Microscopy however there are some issues regarding this technique being truly an internal 3-D imaging technique
	Fluorescence Microscopy	In biological applications fluorescence microscopy can be used to view particular molecules in complex mixtures or in cells. Fluorescence has the advantage of providing a very high signal-to-noise ratio, which enables us to distinguish spatial distributions of rare molecules	Low/ Medium	The limit of resolution is $\sim 0.2 \mu\text{m}$ with the best available objective lenses and a good specimen	<ul style="list-style-type: none"> <li>Limited to cells, tissues and gel substances</li> <li>Non-portable.</li> <li>Size.</li> </ul>	Widely available	<a href="http://nobelprize.org/physics/educational/microscopes/fluorescence/index.html">http://nobelprize.org/physics/educational/microscopes/fluorescence/index.html</a>  <a href="http://www.nature.com/nmeth/journal/v2/n12/abs/nmeth817.html">http://www.nature.com/nmeth/journal/v2/n12/abs/nmeth817.html</a>  <a href="http://www.ptb.de/index_en.html">http://www.ptb.de/index_en.html</a>	To utilize fluorescence, we need to label the specimen (a cell, a tissue, or a gel) with a suitable molecule (a fluorochrome) whose distribution will become evident after illumination. The fluorescence microscope is ideally suited for the detection of particular fluorochromes in cells and tissues Very useful tool to solve imaging problems in the biological field,

Generic Technique	Imaging Methods	Applications/ Uses	Cost	Resolution/ Accuracy	Limitations	Availability (commercial/ R&D)	Useful Links and contacts	Comments
	Polarization light microscopy (Birefringence technique)	Birefringence microscopy (LC-PolScope™) is used to investigate the domain structure of polycrystalline thin films. In general, this imaging system allows to quickly and easily measure stress and structure in transparent materials	Low/ Medium	Resolution is similar to light microscopy	<ul style="list-style-type: none"> <li>Mainly limited to viewing applied stresses</li> <li>Size</li> </ul>	Widely Available	<a href="http://www.uni-ulm.de/ac2/Volkmer_Group/html/tools.html">http://www.uni-ulm.de/ac2/Volkmer_Group/html/tools.html</a>	This technology enables fine birefringent structures to be imaged and measured within living cells or other materials non-invasively – without harmful dyes or UV light. It enables direct observation of microtubules, actin filaments, meiotic spindles, and neural growth cones at sensitivities two hundred-times greater than conventional polarized light methods
	SPM / AFM (Laser based)	Scanning probe microscopy is mainly used in characterization of surfaces in nano-meter resolutions. Atomic Force microscopy (AFM) is a form of SPM. This technique can be applied to various types of materials.	Medium	Nano-meter Resolution	Mainly used to obtain true 3-D maps of surfaces and resolve surfaces down to nano-meter resolutions. Preparation of the sample is a limitation due to the degree of flatness required	Research/ University	<a href="http://www.mobot.org/jwcross/spm/">http://www.mobot.org/jwcross/spm/</a> <a href="http://spm.phy.bris.ac.uk/techniques/">http://spm.phy.bris.ac.uk/techniques/</a>	<ul style="list-style-type: none"> <li>Scanning probe microscopes (SPMs) pass a needle-like probe over the surface of a molecule and record an image of that surface. Different SPMs can not only map the topography but also determine the type of atoms and their thermal and magnetic properties</li> <li>An image of the sample is obtained by mechanically moving the probe with respect to the sample so that the sample is scanned line by line</li> </ul>

Generic Technique	Imaging Methods	Applications/ Uses	Cost	Resolution/ Accuracy	Limitations	Availability (commercial/ R&D)	Useful Links and contacts	Comments
	OCT	Optical coherence tomography can be used to probe the structure of any accessible tissue structure within the medical field. OCT is mainly used as a real time method to view tissue structures at micron scale.	Medium	Depths of 1–2 mm can be imaged in turbid tissues such as skin or arteries; greater depths are possible in transparent tissues such as the eye	<ul style="list-style-type: none"> <li>• Limited to biological media</li> <li>• Portability</li> <li>• Size</li> </ul>	Medical/ Research/	<p><a href="http://www.iop.org/EJ/abstract/0034-4885/66/2/204">http://www.iop.org/EJ/abstract/0034-4885/66/2/204</a></p> <p><a href="http://www.risoe.dk/ofd/oct/">http://www.risoe.dk/ofd/oct/</a></p>	Optical coherence tomography (OCT) is a recently developed, non-invasive technique for imaging subsurface tissue structure with micrometer-scale resolution. The principles of time gating, optical sectioning, and optical heterodyning are combined to allow cross-sectional imaging. Depths of 1–2 mm can be imaged in turbid tissues such as skin or arteries; greater depths are possible in transparent tissues such as the eye. Optical coherence tomography complements other imaging modalities commonly used to image subsurface tissue structure, including ultrasound and Confocal microscopy.
	Laser Profilometry	Laser profilometry is used commonly to map the surfaces of many different types of materials ranging from electronic circuits to biological media	Medium/ Low	Laser spot size of 2 micron. Sub-micron resolution in all axes, thus enabling accurate determination of 3D surface features without limitation of point density	<ul style="list-style-type: none"> <li>• Optically translucent media or mirrored surfaces cannot be scanned</li> <li>• Size</li> </ul>	Widely available, R & D through to production environments.	<p><a href="http://www.solarius-inc.com/html/laser.html">http://www.solarius-inc.com/html/laser.html</a></p> <p><a href="http://www.ndt.net/article/0798/roberts1/roberts1.htm">http://www.ndt.net/article/0798/roberts1/roberts1.htm</a></p>	<ul style="list-style-type: none"> <li>• Non-destructive profilometry for a variety of surfaces</li> <li>• Laser profilometry uses point sensors along with highly precise stages to create profiles and 3D topographies. The sample, which is placed under the sensor, is moved by the stages while the sensor transmits the height data to the measurement control unit.</li> </ul>

Generic Technique	Imaging Methods	Applications/ Uses	Cost	Resolution/ Accuracy	Limitations	Availability (commercial/ R&D)	Useful Links and contacts	Comments
	Stereo Microscopy	Used mainly in macro (100 microns and above) environments to observe/ map the structure of a material. Applications of this technique range from R&D through to industry.	Low	High Micron (100um) and mm range. Optical resolutions of lower sizes are available however image clarity is affected.	<ul style="list-style-type: none"> <li>• Commonly used for macro sampling, micro sampling is not possible at high magnification.</li> </ul>	Widely available	<a href="http://www.stereomicroscopy.com/">http://www.stereomicroscopy.com/</a>	Conventional stereomicroscopes have binocular eyepieces, both of which are arranged to view the object through a single objective lens. The optical axes of the light paths for the two eyepieces are laterally displaced from each other at the point where they pass through the objective lens. This produces viewing parallax for the two eyes, and the brain as a stereo or three-dimensional image interprets this.
	Serial Sectioning (including Cryogenics)	Used predominantly in the materials research or in medical industry to view human tissue in 3-D.	Medium to High	Materials: Optical microscopy & image processing In medical: as the technique involves both MRI and CT for image purposes the resolving power is similar to those techniques.	<ul style="list-style-type: none"> <li>• In cryogenics, mostly used in water containing biological tissue.</li> <li>• In materials work, time consuming</li> <li>• Destructive technique</li> <li>• Cost</li> </ul>	R&D very specialised.  Potential usage in materials research	<a href="http://www.arts.arizona.edu/buildingbetterhumans/vh_1.html">http://www.arts.arizona.edu/buildingbetterhumans/vh_1.html</a>	Use of this novel technique is in materials research with advanced optical microscope & computer image processing algorithms. Also widely documented in medical research, when a section of a complete human body is being profiled. The technique involves rapid freezing (to avoid ice formation) and subsequent slicing of sections using a Macrotome, which are individually scanned by MRI - CT and then manipulated by computer image processing to obtain complete internal image of the body in 3D.

Generic Technique	Imaging Methods	Applications/ Uses	Cost	Resolution/ Accuracy	Limitations	Availability (commercial/ R&D)	Useful Links and contacts	Comments
<b>Electron</b>	Focussed ion Beam (FIB) Scanning Electron Microscopy	The Focused Ion Beam (FIB) system uses a Ga <sup>+</sup> ion beam to raster over the surface of a sample in a similar way as the electron beam in a scanning electron microscope. The generated secondary electrons (or ions) are collected to form an image of the surface of the sample.	High	Similar to conventional SEM systems (down to 10 nm). Tunnelling of the beam can extend to a few microns	<ul style="list-style-type: none"> <li>• Cost</li> <li>• Size</li> <li>• Specialized operation</li> <li>• Damages sample</li> </ul>	Mostly research and development	<a href="http://www.imec.be/efug/EFUG13.html">http://www.imec.be/efug/EFUG13.html</a>  <a href="http://www.almaden.ibm.com/st/nanoscale_science/nano-fabrication/fib/">http://www.almaden.ibm.com/st/nanoscale_science/nano-fabrication/fib/</a>	<p>The <a href="#">applications</a> of FIB include:</p> <p>Cross-sectional imaging through semiconductor devices (or any layered structure), modification of the electrical routing on semiconductor devices, failure analysis, preparation for physico-chemical analysis, preparation of specimens for transmission electron microscopy (TEM) micro-machining, mask repair, on-semiconductor applications</p>
	Transmission Electron Microscopy (TEM) OR (STEM)	Transmission electron microscopy (TEM) is an imaging technique whereby a beam of <a href="#">electrons</a> is focused onto a specimen causing an enlarged version to appear on a <a href="#">fluorescent</a> screen or layer of <a href="#">photographic film</a> (see <a href="#">electron microscope</a> ), or to be detected by a camera system. The TEM is used heavily in both <a href="#">material science/metallurgy</a> and the <a href="#">biological sciences</a> . In both cases the specimens must be very thin and able to withstand the high vacuum present inside the instrument	High	Down to nanometre scale.	<ul style="list-style-type: none"> <li>• Cost</li> <li>• Size</li> <li>• Specialized operation</li> <li>• Thin film samples only</li> </ul>	Mostly R & D and some commercial.	<a href="http://en.wikipedia.org/wiki/Transmission_electron_microscope">http://en.wikipedia.org/wiki/Transmission_electron_microscope</a>  <a href="http://www.microscopy.ethz.ch/">http://www.microscopy.ethz.ch/</a>	<p>In the most powerful diffraction contrast TEM instruments, crystal structure can also be investigated by <a href="#">High Resolution Transmission Electron Microscopy</a> (HRTEM), also known as phase contrast imaging as the images are formed due to differences in phase of electron waves scattered through a thin specimen.</p>

Generic Technique	Imaging Methods	Applications/ Uses	Cost	Resolution/ Accuracy	Limitations	Availability (commercial/ R&D)	Useful Links and contacts	Comments
	Electron Beam Computed Tomography (EBCT)	This technique is very specialised as only 120 machines exist worldwide however it is an ultrafast version of a conventional CT scan. It is mainly used in cardiovascular imaging of the heart whilst beating to perform hyper accurate maps of the organ and its function.	Very high	Sub-mm level.	<ul style="list-style-type: none"> <li>• Cost</li> <li>• Size</li> <li>• Highly Specialized</li> </ul>	Very limited to the medical community.	Useful links: <a href="http://www.siggraph.org/education/materials/HyperVis/applicat/medical/electron.htm">http://www.siggraph.org/education/materials/HyperVis/applicat/medical/electron.htm</a>  <a href="http://www.healthsystem.virginia.edu/uvahealth/peds_cardiac/uebctscn.cfm">http://www.healthsystem.virginia.edu/uvahealth/peds_cardiac/uebctscn.cfm</a>	<ul style="list-style-type: none"> <li>• Electron beam tomography is a specific form of computed axial tomography (CAT or CT) in which the X-Ray tube is not mechanically spun in order to rotate the source of X-Ray photons. This technique is a very powerful diagnostic tool due to the speed at which scans can be taken and therefore images produced.</li> <li>• The principal application advantage of EB tomographic CT machines and the reason for the invention, is that the X-Ray source is swept electronically, not mechanically, and can thus be swept with far greater speed than with conventional CT machines based on mechanically spun X-Ray tubes</li> </ul>

Generic Technique	Imaging Methods	Applications/ Uses	Cost	Resolution/ Accuracy	Limitations	Availability (commercial/ R&D)	Useful Links and contacts	Comments
<b>Thermal</b>	3D Thermography	This technique is widely used to measure and detect heat within objects. 3-D thermography is primarily used as a tool to view theradiative energy distribution on the surface and infer heat distribution within object. . This can be mapped to produce a radiative energy distribution or covered to a temperature scale image knowing the emissivity of the surface image. Used widely in condition imaging,. Medical imaging, process control and NDT.	Low/ Medium	Sub-mm level dependant on detector. Thermography is sensitive to approximately -50 °C to over 2,000 °C	<ul style="list-style-type: none"> <li>Limited to materials that have a temperature differential.</li> </ul>	Widely Available	<a href="http://www.flirthermography.co.uk/">http://www.flirthermography.co.uk/</a> <a href="http://en.wikipedia.org/wiki/Thermography">http://en.wikipedia.org/wiki/Thermography</a>	<ul style="list-style-type: none"> <li>Thermography, or thermal imaging, is a type of infrared imaging. Thermographic cameras detect radiation in the infrared range of the electromagnetic spectrum (roughly 900–14,000 nanometers or 0.9–14 μm) and produce images of that radiation</li> <li>Thermal infrared imagers convert the energy in the infrared wavelength into a visible light video display. All objects above 0 degrees Kelvin emit thermal infrared energy so thermal imagers can passively see all objects regardless of ambient light. However most thermal imagers only see objects warmer than -50C</li> </ul>

Generic Technique	Imaging Methods	Applications/ Uses	Cost	Resolution/ Accuracy	Limitations	Availability (commercial/ R&D)	Useful Links and contacts	Comments
<b>Gamma Ray</b>	3D visualisation	This technique uses a radioisotope, commonly Cs-137 radioisotope to irradiate the specimen and produce a photograph of the internal structure through a detector. The 3-D image is created when slices of 2-D scans are paired.	Medium	Commonly above 100 microns to large macro samples.	<ul style="list-style-type: none"> <li>• Specialized</li> <li>• Cost can be high.</li> </ul>	R&D, some industrial (security).	<a href="http://www.ndt.net/apcndt2001/papers/1038/1038.htm">http://www.ndt.net/apcndt2001/papers/1038/1038.htm</a>  <a href="http://www.scielo.br/pdf/bjp/v35n3b/a18v353b.pdf">http://www.scielo.br/pdf/bjp/v35n3b/a18v353b.pdf</a>	This technique allows for non-destructive techniques for visualizing interiors of objects to obtain digital information on there internal 3-Dgeometry and topology.
	Also in PET under Nuclear	<b>See Nuclear</b>						

**Exclusion:** 3D Holography (in above generic groups) in order to display spatial images

A selection of 3D internal images from each of the above **generic groups** is presented below from the published literature to demonstrate their wide ranging and specific applications, particular strengths and potential benefits in the modern science at present time.

## **Magnetic [ Refs. 3-4 ] :**

### **MRI Images, Figs 1-3**



**Figure 1: Sagittal image of the head , MRI image. (Source: Lodestone Patient care, Ref. 3)**



Figure 2. MRI of ankle joint: source Lodestone Patient care [ Ref 3 ]

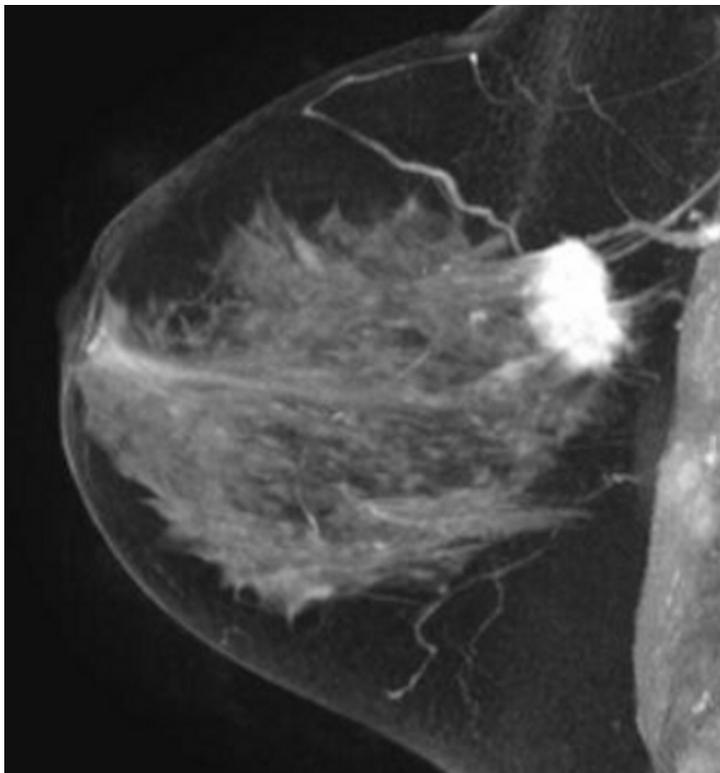


Figure 3: Sagittal image through the breast using a pathology sensitive MRI technique: source: Lodestone patient care. [ Ref 3]

## Nuclear : [ Refs. 5-6 ]

### NMR Images (Figs. 4-5)

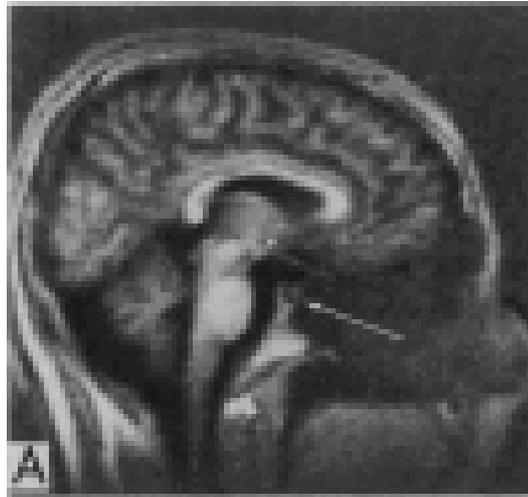


Figure 4: NMR image of human head. source: R G Henderson JRSM Journal 1983. [ Ref. 5 ]

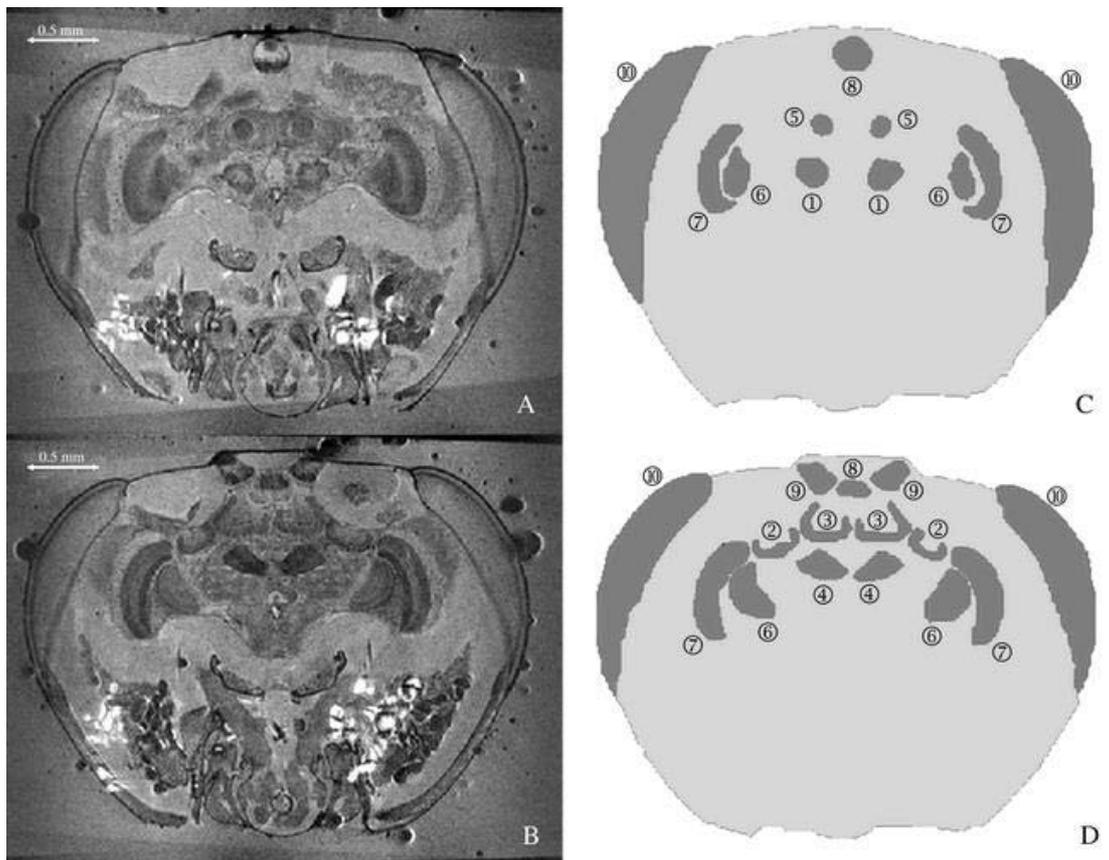
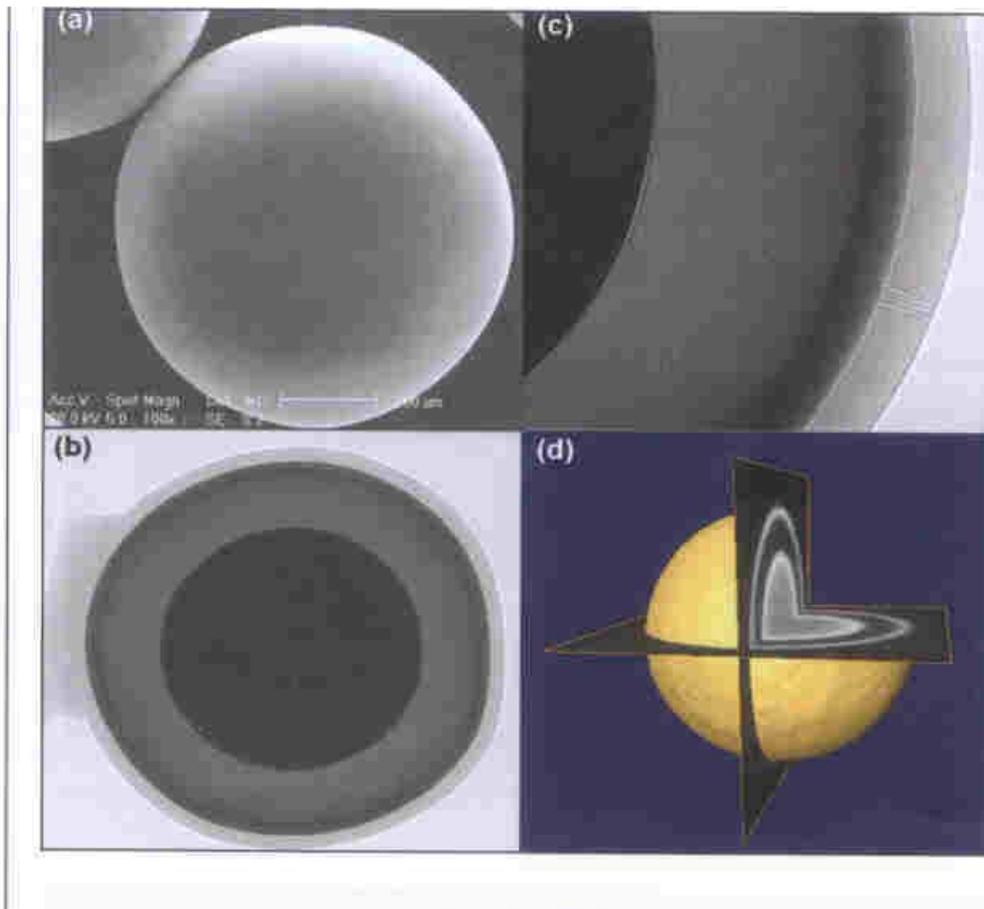


Figure 5: NMR images of insect brain. source: D Haddad, J of Insect science [ Ref. 6 ]

**X-ray :** [ Refs. 7 - 18 ]

**X-ray-CT images (Figs. 6-13)**



**Figure 6: SEM and XuM (X-ray ultra-Microscope) imaging of an intact microsphere**  
a) SEM image; b) & c) XuM X-ray image; d) 3D tomographic reconstruction image  
[ source : L Brownlow, XRT Ltd. Ref. 14 ]

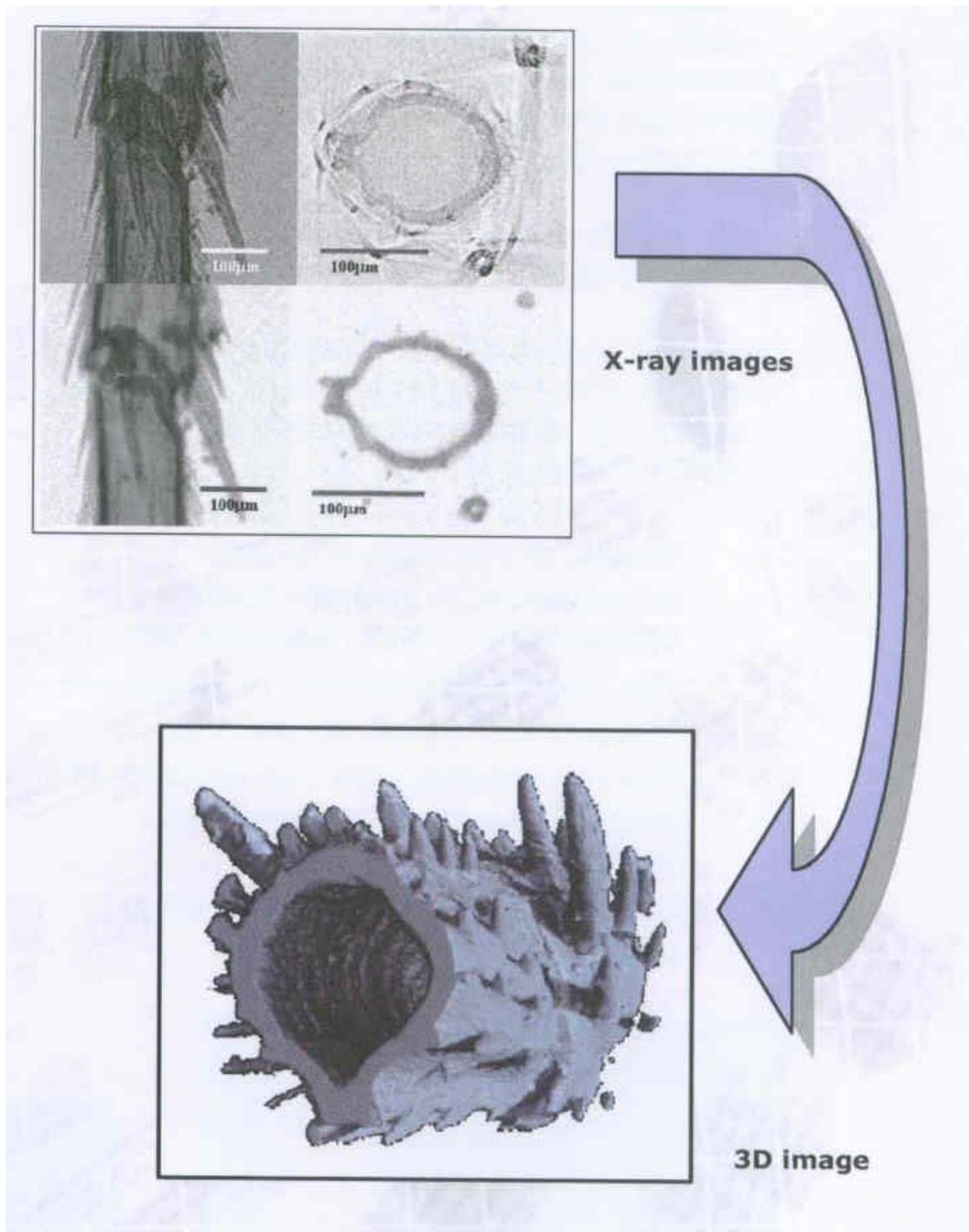


Figure 7: XuM (SEM hosted X-ray Ultra-Microscope) Image of an insect leg in 3D [ Ref . 10 ]

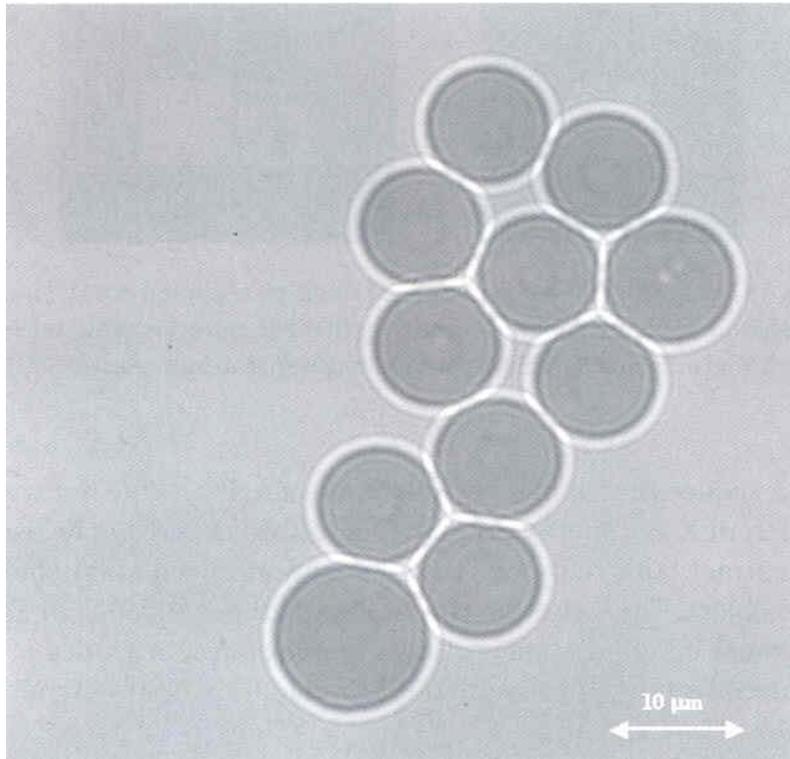
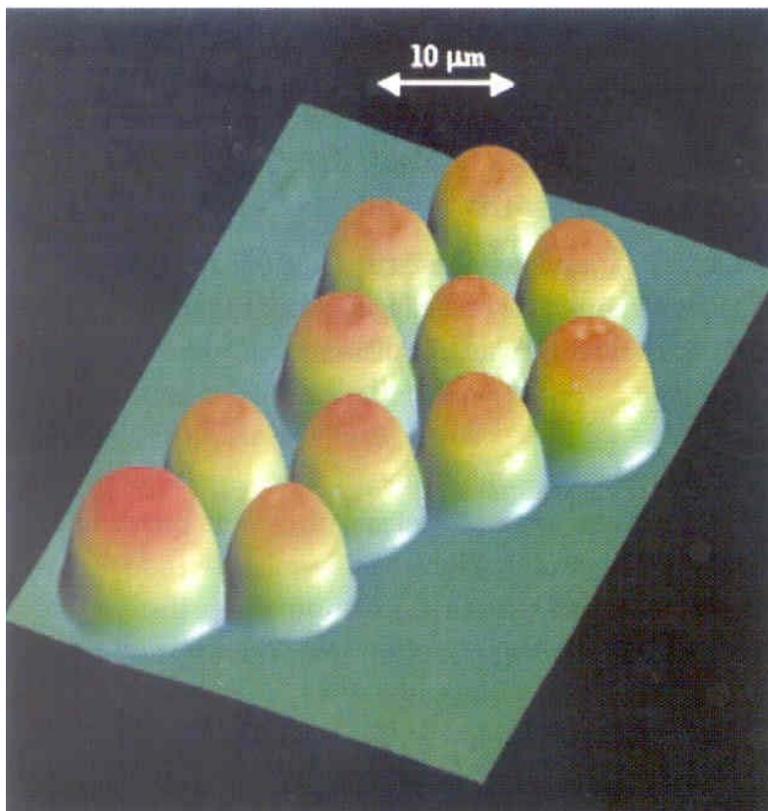


Figure 8: 3D XuM Image (below) of a cluster of 9 micron latex spheres from 2D image above [ Ref. 17 ]



### Xray Micro-CT Images

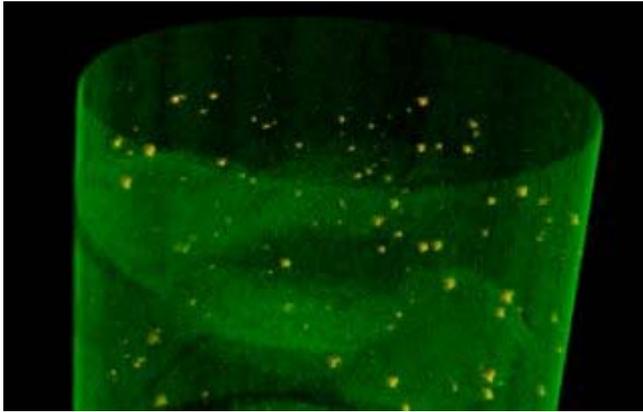


Figure 9: Powder Compact - Semi-Transparent 3D Render (source: X-Tek, Ref. 16)

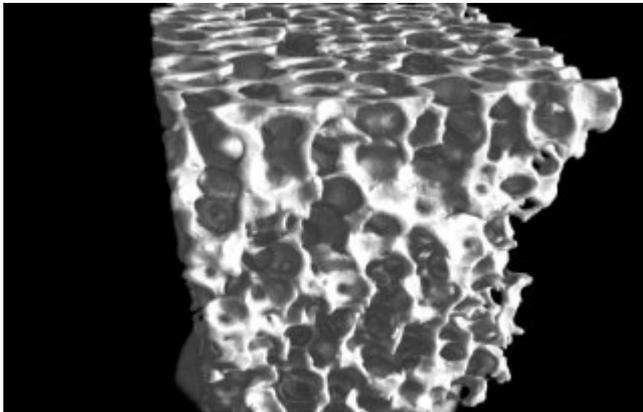


Figure 10: [source: X-Tek , Ref. 16 Ceramic Foam - 3D Reconstruction ]

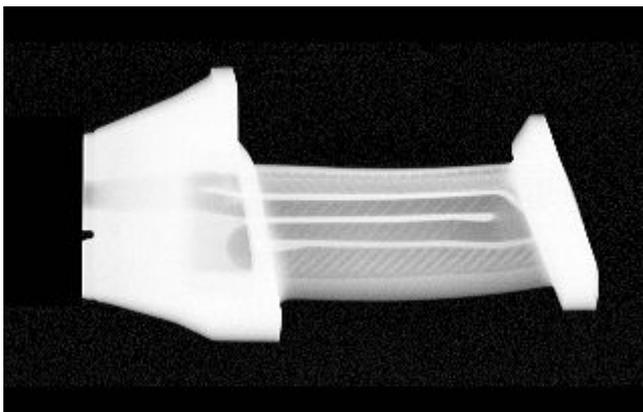


Figure 11: [ source: X-Tek , Ref. 16 3D image of Turbine Blade ]

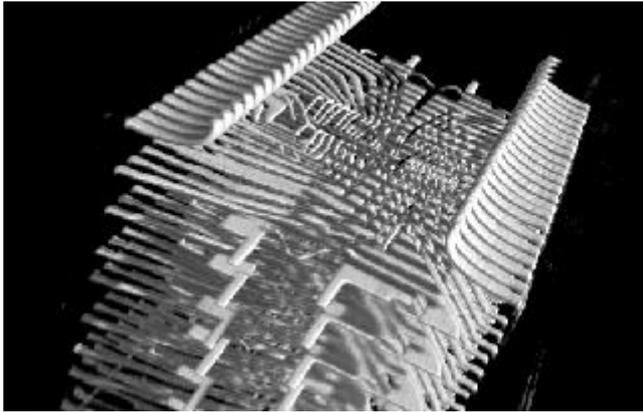


Figure 12: [ source: X-Tek, Ref. 16 Stacked IC assembly Xray CT 720 views, 120Kv, 5 micron focal spot, 16 micron voxels (volumated pixels)

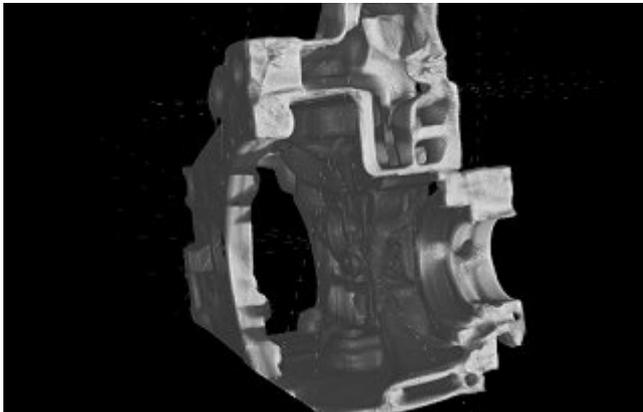


Figure 13: [ source: X-Tek, Ref. 16 Light Alloy Casting - 3D Rendering ]

## Acoustics : [Refs. 19-22 ]

### Ultrasound-CT images (Figs. 14–15)



Figure 14: 3D Ultrasonic CT image of a baby [ Ref. 21 ]

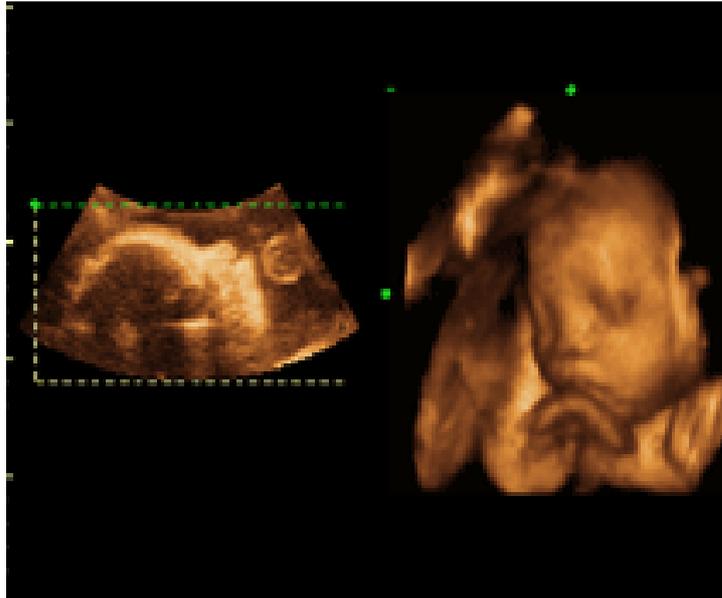


Figure 15: 4D Ultrasonic-CT images of a baby in mother's womb [ Ref. 19 ]



Optical : [ Refs. 23 – 31 ]

### Cryogenic Serial Sectioning images

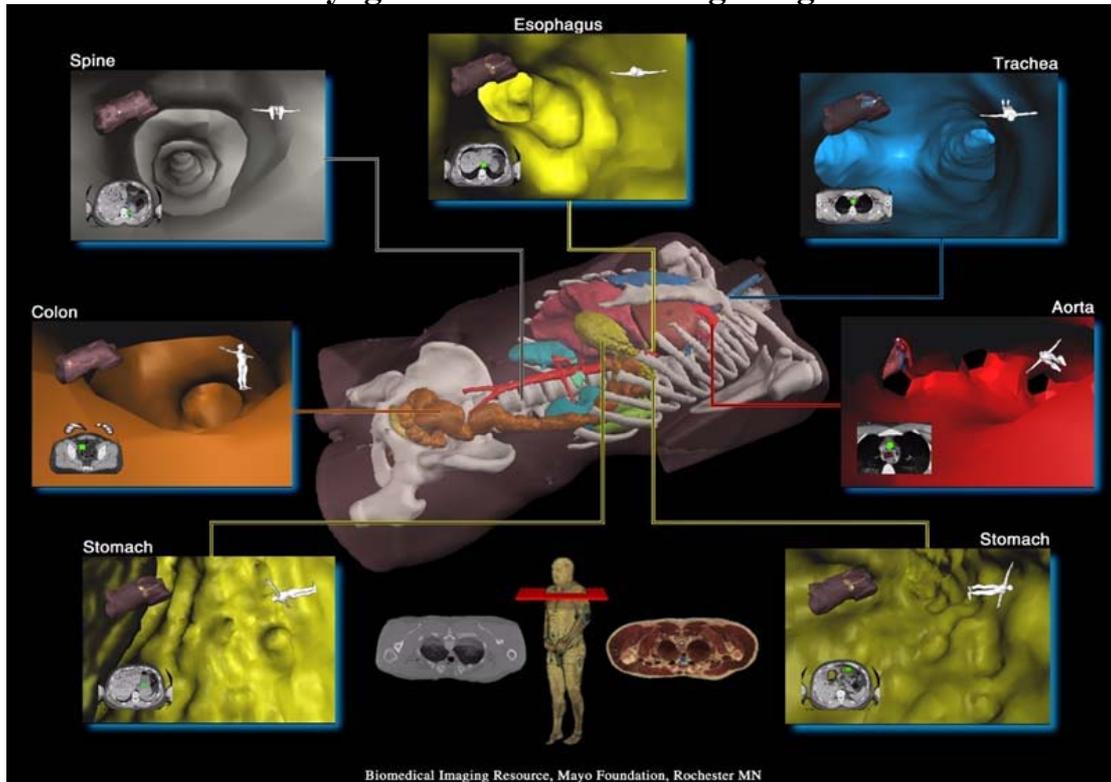
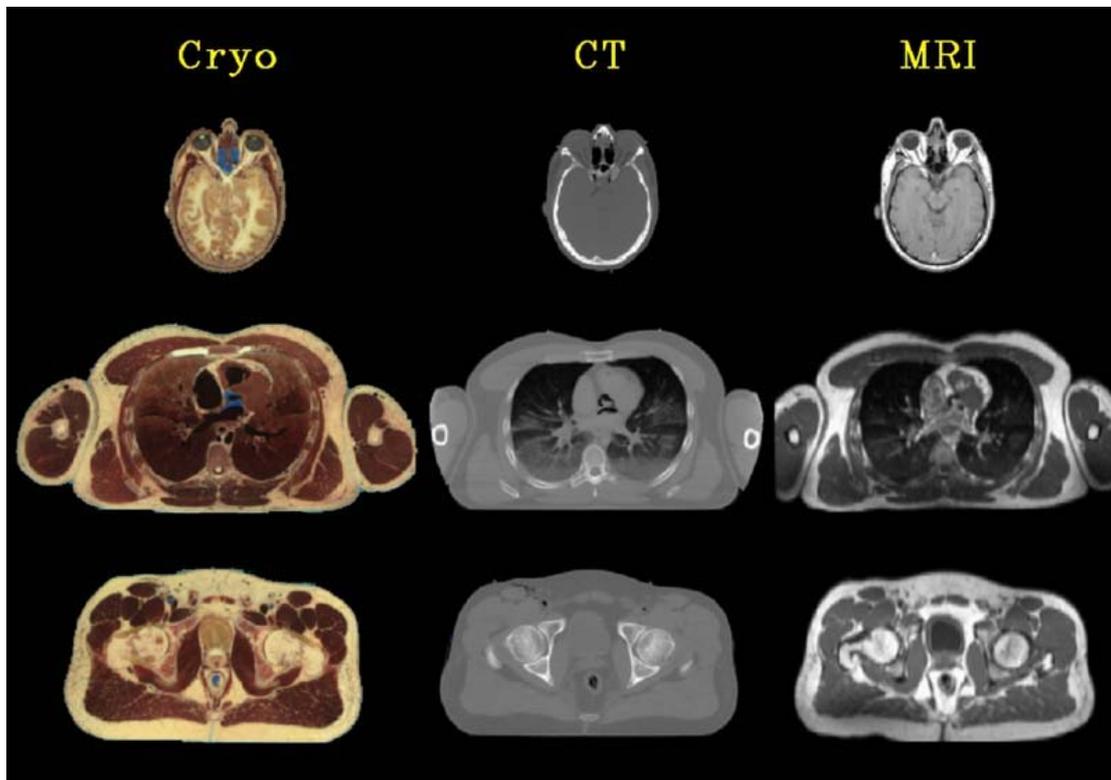
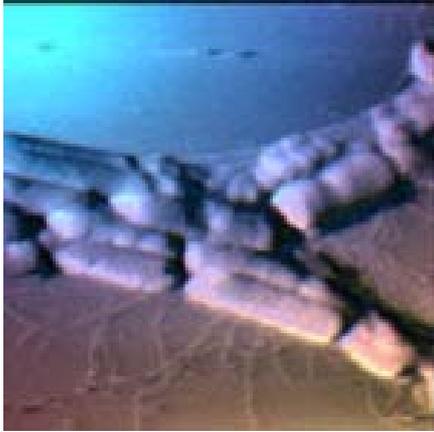


Figure 16: Cryogenic sectioning images & a comparison with CT and MRI images [ Ref . 26 ]



**Optical : [ Refs. 23 – 31 ]**

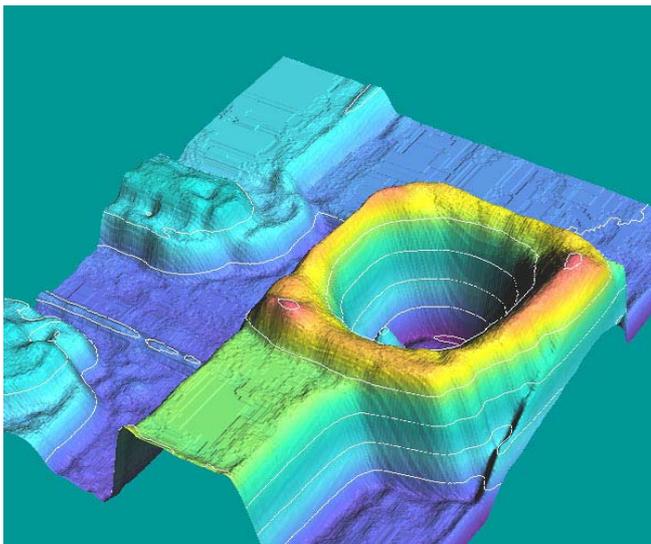
**AFM images (Laser based) Figs. 17-19**



**Figure 17: Image of Bacteria ; source: Novascan Technologie**



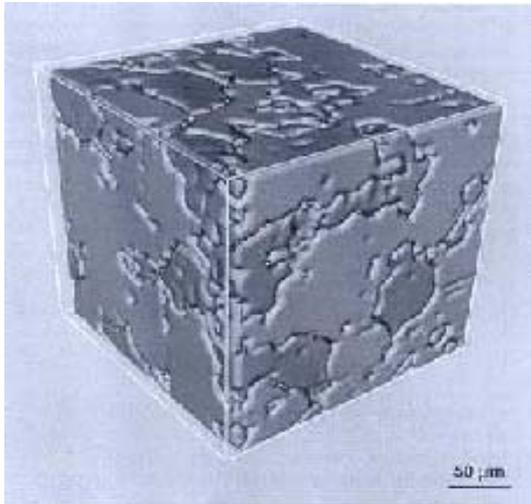
**Figure 18: Phase contrast AFM image showing AFM cantilever and epithelial cells: source: As Research**



**Figure 19: Semiconductor circuit AFM image, source: Ref. 25**

**Optical:** [ Refs. 26, 29-31 ]

**Serial Sectioning** (Figs. 20-21)



**Figure 20:** 3D microstructure of a sintered 67%Fe – 33%Cu P/M alloy by “Robo-Met”.  
Comprise 100 optical serial sections , ~1.2 micron apart. [ Ref. 26 ]



**Figure 21:** 3D image of a coarsened Pb-Sn alloy. Scale: 100 micron. [ Ref: 26 ]

## Electron: SEM, STEM, EBCT images [Refs: 1, 2, 32 – 38 ]

### SEM images

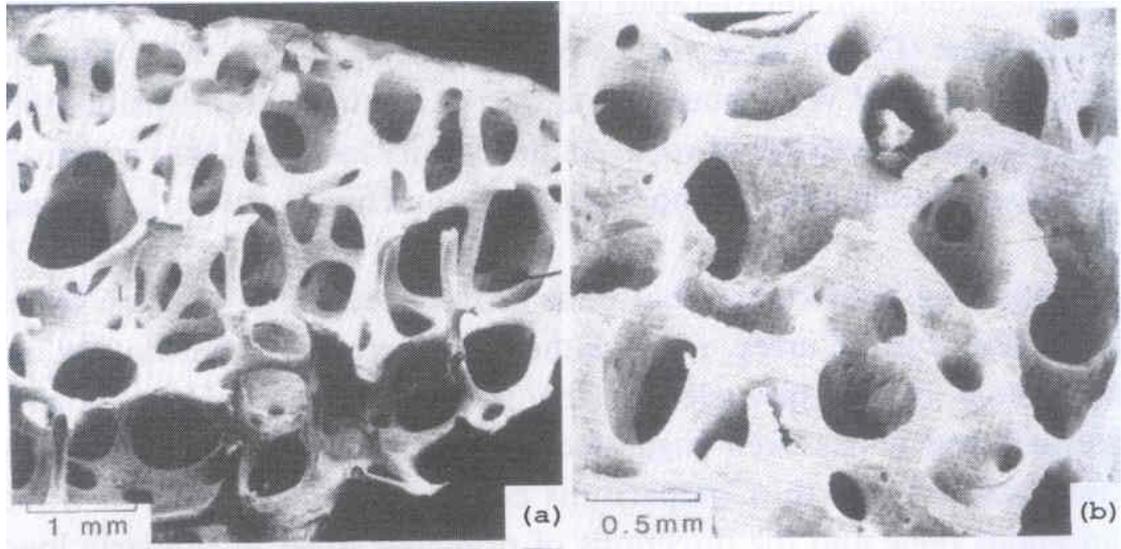


Figure 22: Cellular human bone structure in SEM; source : Gibson and Ashby. [ Ref. 1 ]

### STEM image [ Ref. 32 ]



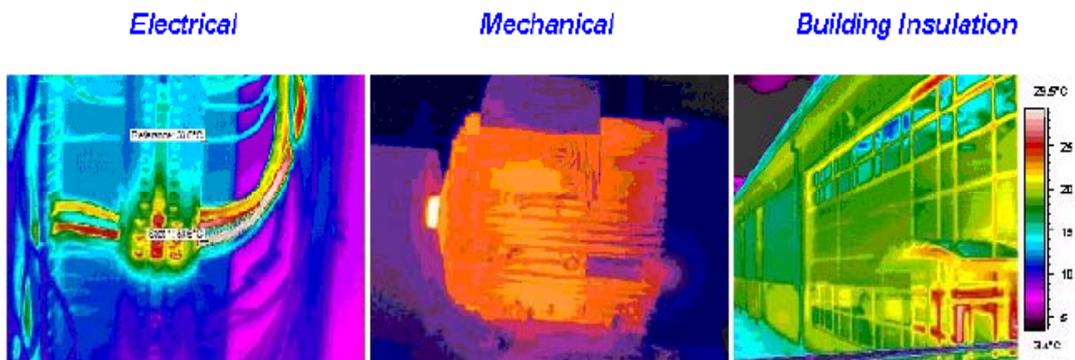
Figure 23: STEM image of an electronic circuit board in 3D, 30 keV, electron transparency 100nm.

**Electron: EBCT image [ Ref. 38 ]**



**Figure 24:** 3D non-invasive electron beam scan of abdominal & pelvic regions gives detailed information of colon and rectum problems. [ Ref. 38 ]

**Thermal : 3D Thermography images [ Refs. 39 – 40 ]**



**Figure 25:** Electrical: Thermographic image on an electrical installation  
 Mechanical: Thermographic image testing on mechanical device  
 Building Insulation: Thermography applied to buildings

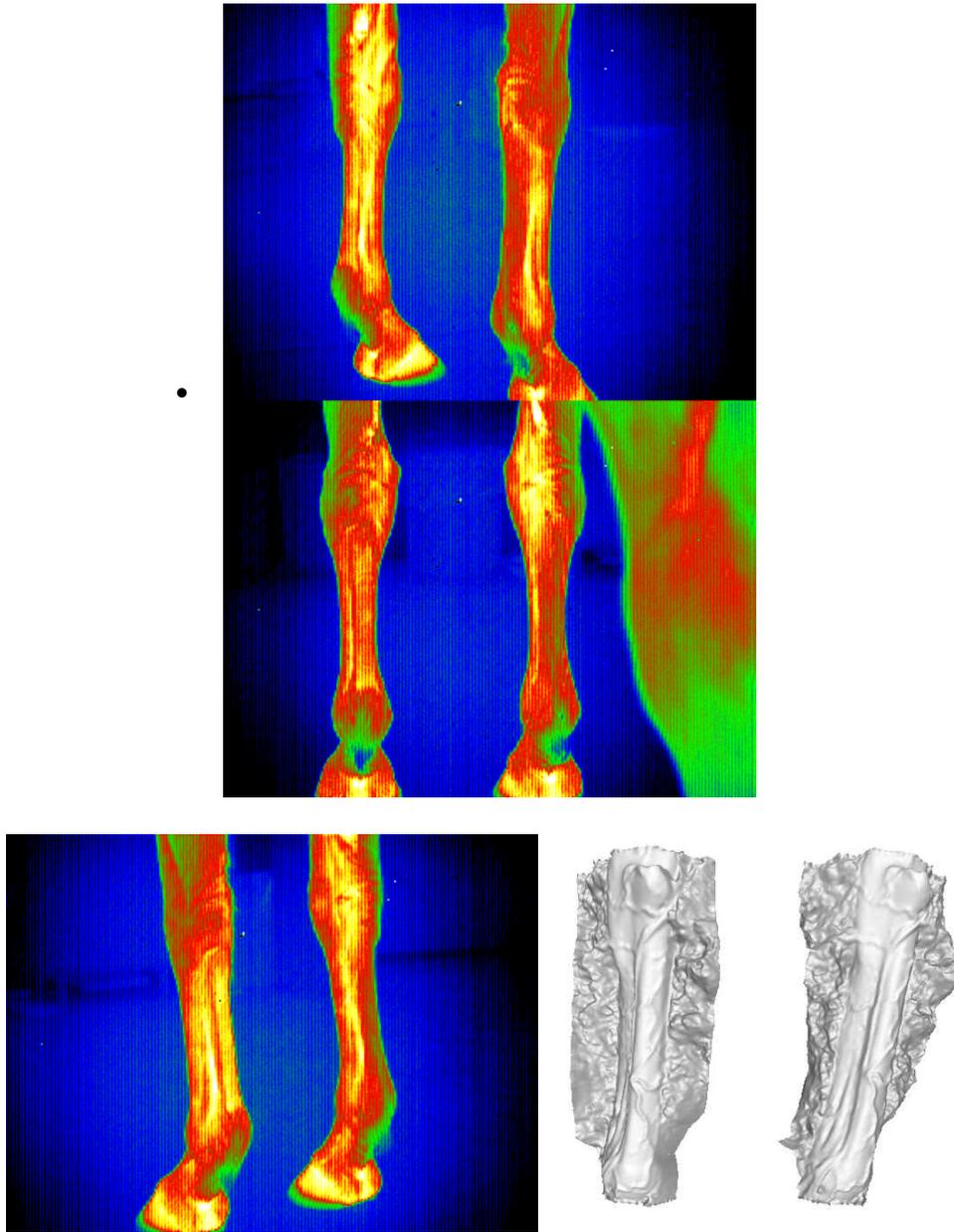


Figure 26: Thermographs of a horse with a tendon injury and 3D scan of leg model [Ref. 40]

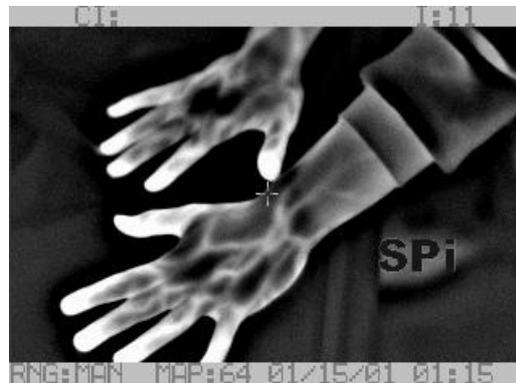
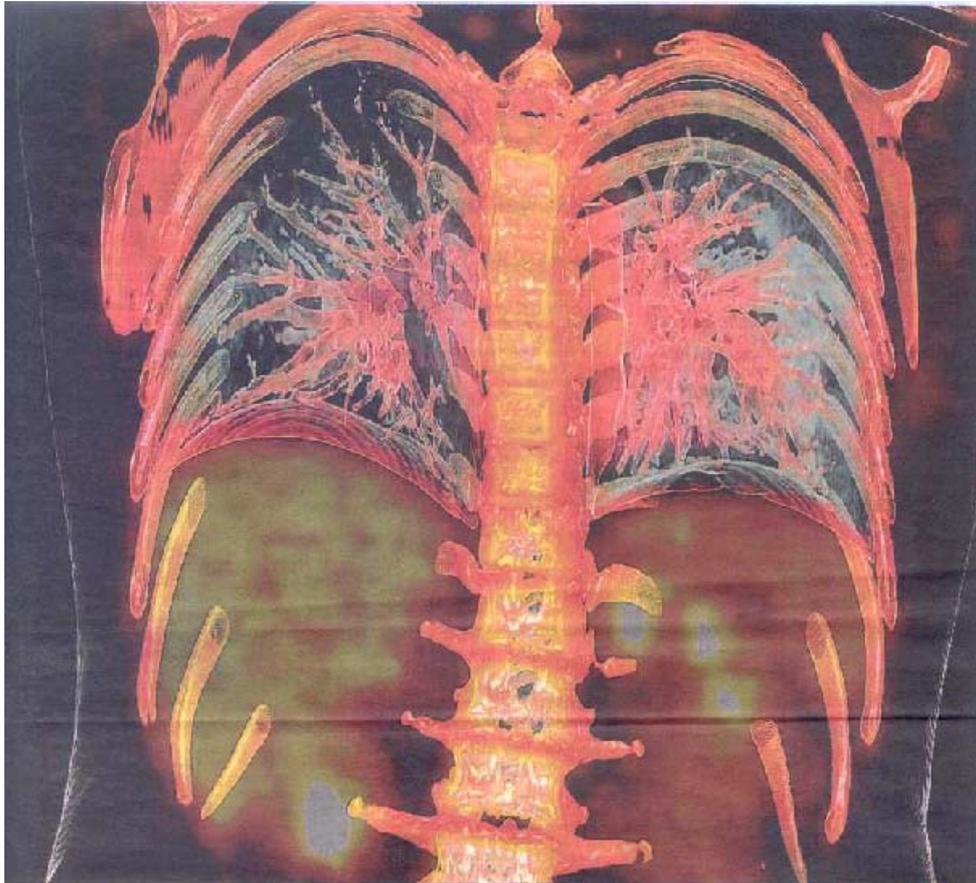
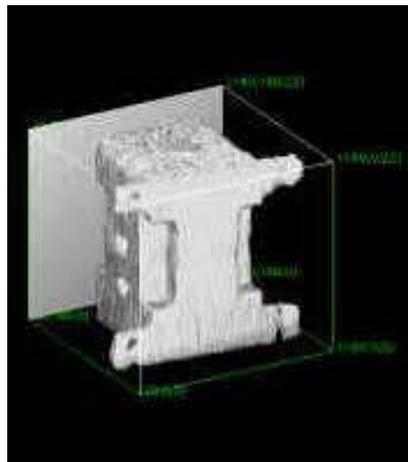


Figure 27: Vascular IR imaging [Ref. 39]

**Gamma-ray : PET- CT imaging [ Refs. 41- 45 ]**



**Figure 28: PET-CT image of chest using radioactive tracers to highlight active body parts (such as organs and tumours) and subsequent CT scan to pinpoint location. [ Ref. 42 ]**



**Figure 29: 3D Gamma ray image of a metallic artefact [ Ref. 41 ]**

### **3 EVALUATION OF 3D TECHNIQUES AND CASE STUDIES**

It is expected that Table 2 will enable the reader to identify new opportunities, applications and new developments in 3D imaging techniques for characterising complex internal materials structure. Table 1 refers to various imaging techniques one may select for a given artefact or in a specific application.

As part of this feasibility study, a brief experimental assessment (4 case studies carried out on a limited scale) has been undertaken with four establishments (University of Surrey, University of Exeter, University of Hull and Simpleware Ltd.), who are regarded to be amongst the experts in this field with facilities for 3D imaging and analysis in the UK. The focus has particularly been given in investigating different classes of artefacts (metallic and non-metallic items, including samples of bio- and nano- materials) requiring specific scales of operations at macro-, micro- and nano- levels. Results and experiences gained from these case studies are reported below and further covered in Sections 4 and 5 to report general conclusions and to produce a list of recommendations on future strategy in true 3D imaging techniques. The project summarises options, where 3D imaging tools could be developed or extended within the NMS.

The findings are also being planned to be widely promoted to at least 3000 companies in the UK through an article in a NPL newsletter and via a dedicated NPL website.

The purpose of this exercise is to measure performance of three promising 3D imaging techniques on a number of complex artefacts (metallic and non-metallic, including bio- and nano- material samples). The aim of these case studies is also to obtain at least qualitative information (where possible) to assess possible future applications of the selected 3D methods. Techniques that are considered suitable and have excellent potential for wider industrial “uptake” in materials area are:

#### **X-ray micro-CT, SEM-FIB and AFM.**

**CASE STUDY 1: SEM with up to 60° tilt (at NPL & in University of Surrey)**

**CASE STUDY 2: SEM / FIB (in collaboration with University of Surrey)**

**CASE STUDY 3: X-ray Micro CT (in collaboration with Universities of Exeter, Hull and Simpleware Ltd.)**

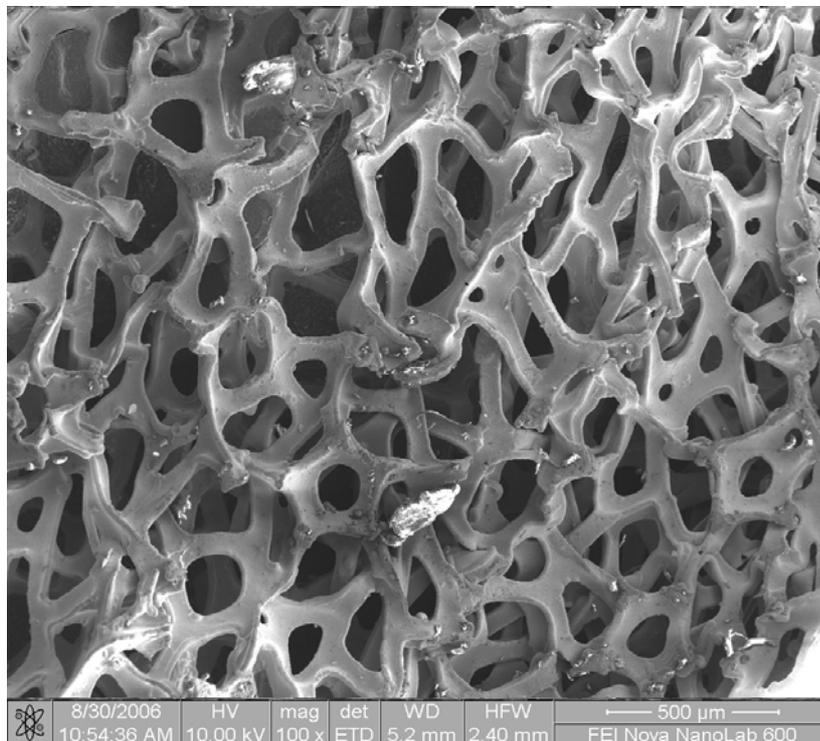
**CASE STUDY 4: AFM (at NPL)**

### CASE STUDY 1: Scanning Electron Microscopy (SEM) with up to 60° tilt at macro- and micro- resolution levels (at University of Surrey and at NPL).

The **scanning electron microscope (SEM)** is capable of producing high-resolution three-dimensional characteristic images of material artefacts. The electron beam, which typically has an energy ranging from a few hundred eV to 50 keV, is focused by one or two condenser lenses into a beam with a very fine focal spot sized 1 nm to 5 nm. The interaction volume, extends from less than 100 nm to around 5  $\mu\text{m}$ . The spatial resolution of the SEM fall somewhere between less than 1 nm and 20 nm depending on type of samples. In general, SEM images are much easier to interpret than TEM images.

In this work, the SEM instrument had been used both at NPL (Hitachi) and at the University of Surrey (FEI dual beam FIB-SEM) to examine mettalic and polymeric foams at macro- to micro- resolution levels. In some cases, SEM study was conducted with up to 60° tilt on Z-axis to assess images of internal structure close to 3D level (demonstrated on the NPL website currently under construction: <http://www.draft.npl.co.uk/materials/biomaterials/z2metal.html>). The SEM image of a fine meshed nickel metallic foam with intricate structure, is shown below. However, the work on the polymeric foam was less successful due to difficulties associated with the sample preparation and excessive charging of the polymeric sample during examination.

#### Metallic Foam (Nickel)



**Figure 30: Nickel foam sample showing complex internal cellular structure in 3D. [ Video clip of high resolution images from SEM with up to 60° tilt available on the NPL website]**

## CASE STUDY 2: SEM / FIB imaging at micro-resolution level (in collaboration with University of Surrey)

The focused ion beam (FIB) technique was developed in the late 1980's from device transplanted techniques and operates on the same principles as a conventional SEM, where a beam of charged particles is scanned across a specimen (with or without tilt) and the resulting images are constructed. FIB systems are widely used in microscopic-scale manufacturing operations because of their ability to image, etch, mill, deposit, and analyze very small features with great precision.

The SEM/FIB technique has the major advantage over conventional methods of TEM, in that sample can be extracted from specific locations. Furthermore, the range of materials from which samples can be extracted is enormous ranging from biomaterials samples to corrosive products at nano-meter resolution. A FEI-Dual beam system, including a FIB and a scanning electron microscope (SEM), was used in this work, which not only imaged the sample but milled the surface progressively (a destructive method) in order to show the true internal structure consisting of pores and complex bone structure. The sample was also tilted towards or away from each beam axis to facilitate various SEM and FIB milling operations (takes 5 minutes for a 5 micron mill depth). A video clip of the SEM-FIB investigation has been shown on the NPL website currently under construction.

(<http://www.draft.npl.co.uk/materials/biomaterials/z2lamb.html> ).

### SEM-FIB : Animal bone (Lamb)

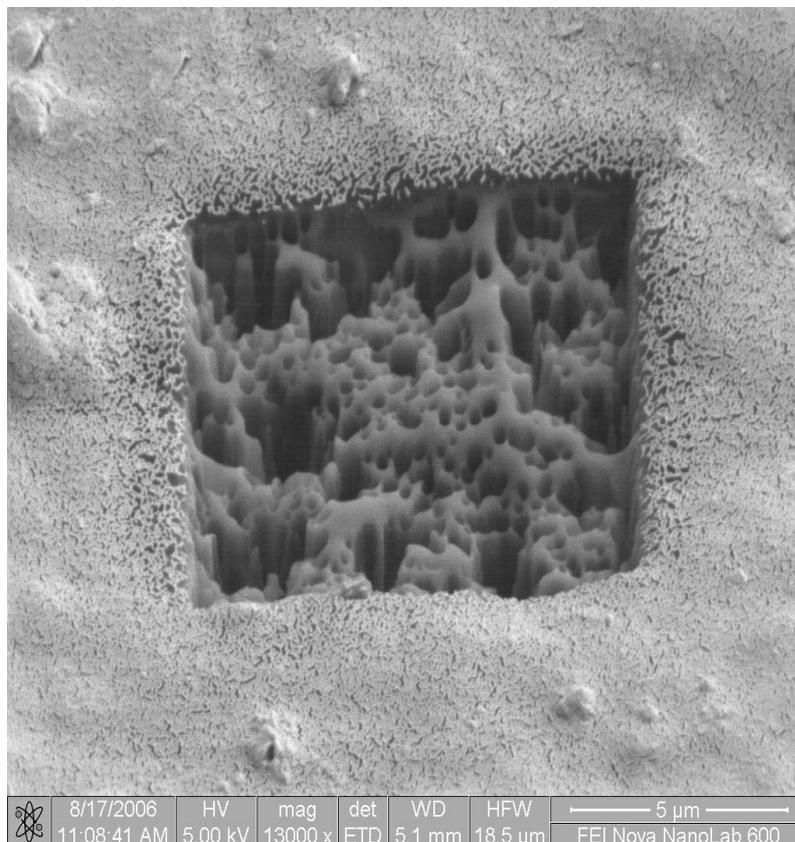


Figure 31: SEM/FIB image showing milled 3D internal structure of animal bone from a rib joint.

### **CASE STUDY 3: X-ray Micro-CT imaging at micro- and nano- resolution levels (in collaboration with Universities of Exeter, Hull & Simpleware Ltd.)**

The X-ray-CT method is a non-destructive X-ray inspection technique that produces high resolution 3D internal maps of samples / artefacts. Typical reconstruction times for a 512x512x512 volume are 5 - 15 minutes depending on the speed of the CPU in the computer. This is similar to the time taken to acquire low noise images from all angles. After reconstruction the sample can be viewed from any 3D angle, sliced in any direction, measured and even animated in a virtual workspace. This enables detailed analysis of the internal structure of a wide range of components.

Normal test conditions are: X-ray sources from 160kV micro-focal sources with a focal spot size down to one micron, up to 450kV mini-focus sources and 8MeV betatrons to gain more penetrating power. For real-time applications cameras are either mounted on an x-ray image intensifier or viewed on a flat fluorescent screen. The image acquisition and subsequent processing are done under computer control.

What does it achieve?

- Verify complex internal structures
- Isolate and inspect included components
- Measure dimensions and angles within the sample without sectioning it.
- Automatically detect and measure internal voids / volumes.
- Remove / strip external surfaces from view to ease inspection

Applications:	Examples
Advanced materials research	Analysis of ceramic and metal foam structures
Verification of complex structure after manufacture	Electronic package development and failure analysis
Dimensional measurement of internal components	Measuring internal wall thicknesses and hole diameters
Fault detection (esp. cracks), location and sizing	Porosity and crack detection in castings / ceramics
Analysis of the action of complex mechanisms	Studying the action of complex switches
Measurement of the distribution of one material in another	Measurement of impurities/inclusions in composite materials
Analysis of the structure of biological materials	Studying the internal structure of teeth, bones, insects.

A brief assessment of the X-ray micro-CT technique in generating powerful 3D images has been carried out on human cortical bone (from femur area) with help from University of Exeter and in association with University of Hull and Simpleware Ltd.

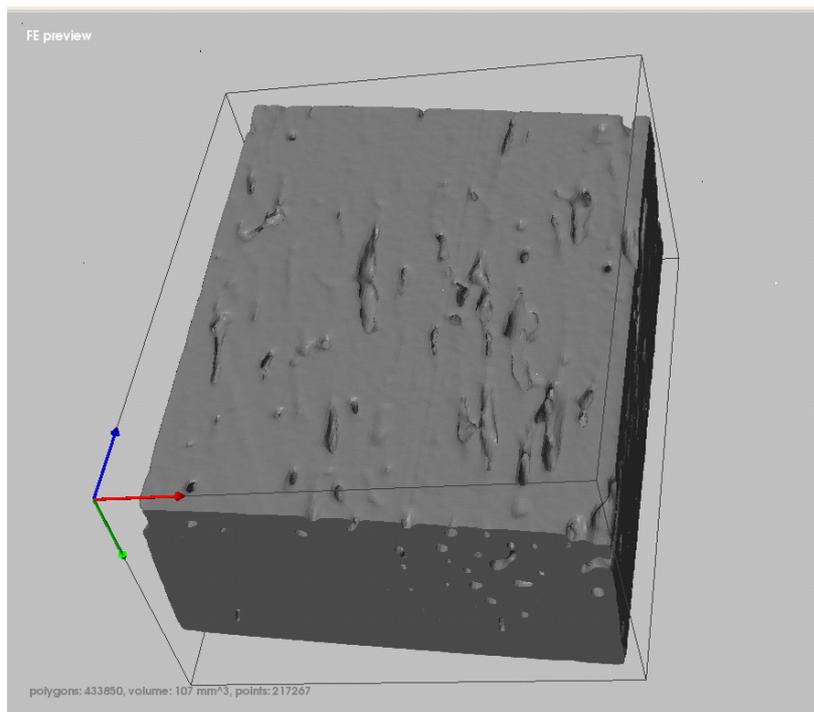
Traditional material characterisation techniques typically require the preparation of homogeneous samples of prescribed dimensions/geometry. This can often be difficult with biomaterials- for example, skin or bone- as sample preparation is more involved

and properties vary rapidly over small length scales. An alternative approach, coupling high-resolution scanning and numerical simulation, has been explored in this study to characterise high resolution 3D imaging of bone. A number of samples from a cadaveric bone had been taken and scanned in an “XTEK” cone beam X-ray micro-CT scanner at resolutions of less than 20 microns (see Figure below).

### Image Processing

CT scan data was then imported into ScanIP™ as a stack of images and the image processing was carried out. The 3D image was segmented using the original data before re-sampling to allow a greater accuracy when identifying material boundaries. The segmentation of the image involved identifying the materials that are required for the model. Masks are produced which cover the area of the image occupied by the required material.

The Figure below represents 3D image data of the cortical bone, which has been rendered in Simpleware “ScanIP” software.



**Figure 32: Internal bone structure of Cortical bone in 3D from femur with 20 micron resolution, X-ray micro-CT. Size and distribution of bone porosity are evident.**

## CASE STUDY 4: Atomic Force Microscopy (AFM) at nano- resolution level (at NPL)

The atomic force microscope (AFM), or scanning force microscope (SFM) was invented in 1986. Like all other scanning probe microscopes, the AFM utilises a sharp probe moving over the surface of a sample in a raster scan. In the case of the AFM, the probe is a tip on the end of a cantilever, which bends in response to the force between the tip and the sample.

AFMs employ an optical lever technique. As the cantilever flexes, the light from the laser is reflected onto the split photo-diode. By measuring the difference signal, changes in the bending of the cantilever can be measured. Since the Cantilever obeys Hooke's Law for small displacements, the interaction force between the tip and the sample can be found. The movement of the tip or sample is performed by an extremely precise positioning device made from piezo-electric ceramics, most often in the form of a tube scanner. The scanner is capable of sub-angstrom resolution in x-, y- and z-directions. The z-axis is conventionally perpendicular to the sample.

### Image display

The height image data obtained by the AFM is three-dimensional. The usual method for displaying the data is to use a colour mapping for height, for example black for low features and white for high features. Specific colour mappings can be used for non-topographical information such as phase or potential.

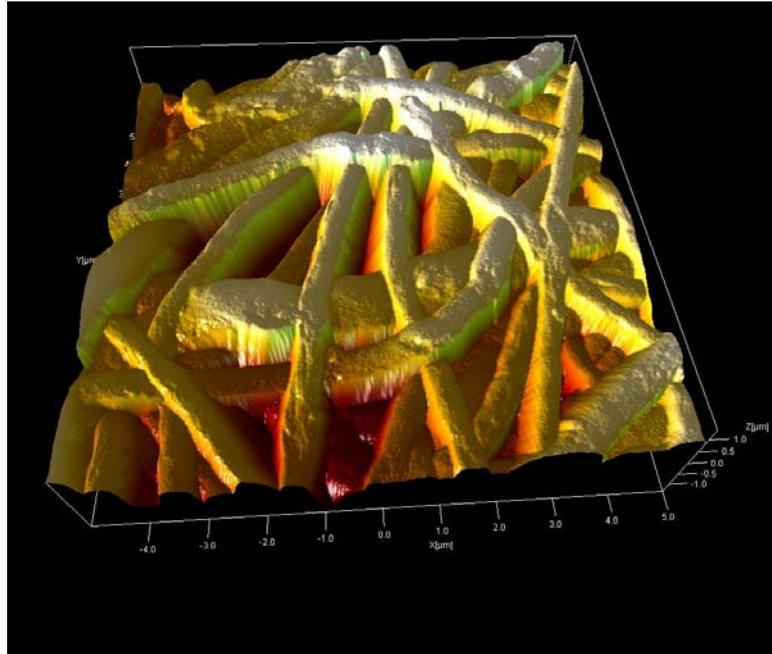
### Tip effects

One of the most important factors influencing the resolution, which may be achieved with an AFM is the sharpness of the scanning tip. The first tips used by the inventors of the AFM were made by glueing diamond onto pieces of aluminium foil.

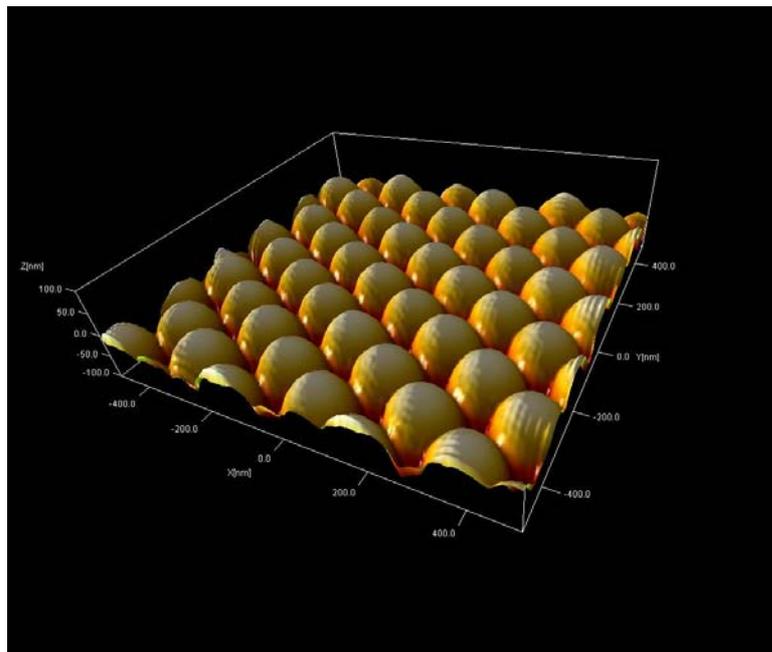
Commercially fabricated probes are now universally used. The best tips may have a radius of curvature of only around 5nm. The need for sharp tips is normally explained in terms of *tip convolution*. This term is often used to group together any influence, which the tip has on the image. The main influences are :

- broadening
- compression
- interaction forces
- aspect ratio

A brief investigation on two material artefacts has been carried out at NPL using AFM technique to examine its effectiveness as a measurement tool in capturing true 3D images at nano-resolution level. The two images below represent pseudo-3D views of a polymer nano-fiber ( Polypractam) at 10 micron scan (**image a**), and 150 nm Al gratings on Si, with 500 nm scan (**image b**). The images are further enhanced in the computer to generate false colouring.



**Figure 33: AFM study: Image “a” Polypractum**



**Figure 34: AFM study: Image “b” Al gratings on Si.**

Although, AFM technique is regarded to be a powerful imaging tool to produce images at nano- and micro- levels for a wide range of materials, this has remained thus far to be a most successful technique in providing reliable topographical / morphological images up to a limited depth. For acquiring images of true 3D internal structure in this project, the AFM technique is therefore seen to offer limited value.

## 4 CONCLUSIONS

3D imaging tools are rapidly finding their strong position in the “high-tech” industries in the UK and elsewhere by successfully demonstrating their inherent technological and commercial advantages over traditional methods in evaluating precision novel products. The key questions that these internal imaging methods successfully address are: true shape, true size, spatial distribution and exact location (of particles, objects, defects etc. within a finite volume).

The demand for characterising intricate internal structures at micro- and nano-levels are rising fast and the challenges posed are steadily turning out to be complex. It is estimated that the following premium sectors are likely to benefit most in the next 5-10 years from such challenging studies: Electronics, Bio-technology, Nano-technology Aerospace, Automotive, Packaging, Construction and Pharmaceuticals.

Collaboration and interactions that have been recently generated with the scientific and industrial communities in the UK are expected to raise general awareness and appreciation in true 3D internal imaging techniques. In particular, their future applications in characterising complex material properties including nano- and bio-technology areas are strongly evident.

It is important though that companies have access to easy and cost-effective equipment, which would provide **traceable** and **validated** true 3D imaging measurements in the future. These techniques will enable them to grow, be successful and innovate novel products more effectively. Opportunities for such collaborative research within the NMS (e.g. NPL) should facilitate this goal.

## 5 RECOMMENDATIONS FOR FUTURE DEVELOPMENT

Recommendations for any 3D imaging techniques are to be primarily based on the following key factors: cost, availability, resolution and accuracy, direct or indirect method; invasive or non-invasive method and ease of operation. The adopted technique should also meet the requirements of type of artefacts, scale of operation and other variables as listed in Tables 1 and 2.

In making an overall assessment, all reviewed major 3D imaging techniques are being split into two main groups. Methods included in **Group A** have gained or are continuing to gain wider popularity & true recognition in the recent years for generating true internal images in 3D for a wide range of materials (mainly in Bio- and Organic-materials). Other methods, as listed in **Group B** are gaining gradual adoption in the materials research work (e.g. in high-resolution nano-technology work), but are generally capable of producing pseudo-3D images only i.e. 3D images of the surface morphology /topography of materials up to a limited depth (with or without computer image enhancement).

**Group A: True 3D internal imaging methods**

**MRI**  
**X-ray-CT**  
**Nuclear-CAT**  
**Ultrasound -CT**  
**Thermograph in 3D**  
**SEM / STEM + FIB .....destructive**  
**Optical (OCT, Serial Sectioning...destructive )**

**Group B: Pseudo-3D imaging methods**

**AFM**  
**SEM**  
**SAM**  
**TEM**  
**SOM**  
**Optical – Stereo Microscopy**  
**Optical - Laser Profilometry**

Existence of some of the 3D imaging facilities (mostly pseudo- methods) in NPL is listed in Table 1. Taking account of NPL's present position, it is important that for a NMI such as NPL further evaluation of the following three increasingly popular 3D imaging techniques should be given careful consideration. Strengthening NPL's 3D measurement facilities (including true imaging, characterisation and prediction) in the materials research in the next 3-5 years should be a top priority.

**The three recommended true 3D imaging techniques are:**

**X-ray Micro-focus CT; SEM/STEM + FIB; Ultrasound CT**

[some superior 3D internal imaging techniques, such as MRI, NMR, PET, EBCT are excluded from this recommended list due to high investment, challenging operation and difficult accessibility. Others including 3D holographic images are excluded due to their invasive or pseudo nature, slow speed or operator intensive output].

There are inevitable risks associated with applying /developing novel 3D imaging techniques at the boundaries of modern science. However, with the help of experts with key skills both within and outside NPL (e.g. Universities, equipment manufacturers, other NMIs), the potential risks and difficulties of evaluating recommended 3D imaging techniques and interpreting complex results can be minimised. In certain areas (particularly where the investment is significantly high), suitable "buy-in" arrangements with key Universities, equipment suppliers or internationally recognised external specialists in specific 3D measurement techniques need to be considered (particularly in the use of MRI, PET, EBCT, NMR techniques, but also including recommended Microfocus X-ray CT or Ultrasound-CT methods).

A range of awareness meetings and dissemination activities (within the remit of this project) with relevant industries, supplier companies, distributors, software manufacturers and some hospitals, that are due to take place in the coming months

should help promote NPL's profile in this research area. As an additional benefit, these events could generate future collaborations and co-funding opportunities from industrial partners. Interactions with other NPL scientific groups, other NMIs, VAMAS, ISO, Universities, centres of excellence, industrial experts and instrument suppliers are to continue in the foreseeable future, which are likely to bring further valuable inputs into this feasibility study and help the authors to form key links with the major players in UK. It is anticipated that the planned initiatives will help to progress the present work into the next phase (with validated quantitative analysis, traceability and prediction modelling) and strengthen the proposed strategy on developing true 3D imaging capabilities within the NMS.

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