Scanning Acoustic Microscopy

A PHYSICIST'S TOOL

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Scanning acoustic microscopy (SAM) was born in the mid-1970's and a few hundred microscopes have been built, including commercially available units. Since contrast is determined by the acoustic properties of the specimen, several unique applications, involving both imaging and property measurement, are recognized. We shall describe a number of these applications to illustrate the potential of SAM as a tool for physicists.

The principle of the scanning acoustic microscope is extremely simple. Its heart is the acoustic lens which is a sapphire rod with a spherical cavity immersed in a fluid that makes contact with the specimen under observation. A piezoelectric transducer generates pulsed ultrasonic waves in the frequency range of 10 MHz to 2 GHz which propagate as acoustic plane waves along the lens rod (Fig. 1). The spherical interface between the sapphire and the coupling fluid at the opposite end of the rod acts as a lens. Focussed spherical ultrasonic waves are transmitted through the fluid (usually distilled water) and strike the surface of the specimen. A part of the acoustic wave is reflected by the specimen surface and this reflected wave is converted by the transducer into a useful signal as it makes its way back. If the lens is mechanically scanned in the *x*-*y* plane of the specimen surface, the reflected voltage *V* can be represented as a function of the *x*-*y* position, forming an acoustic image of the specimen surface.

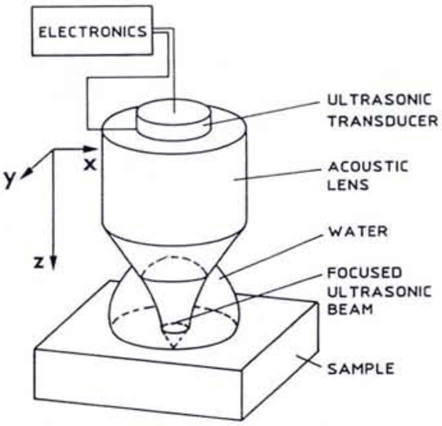

Fig. 1 — Principle of the scanning acoustic microscope (SAM). The spherical interface between the sapphire rod and the coupling fluid (usually water) acts as a lens for pulsed ultrasonic waves travelling down the rod. Spherical ultrasonic waves strike the sample and are reflected back into the transducer for conversion into a useful signal.

Several acoustic paths for the reflected waves are possible, including direct surface reflection, focal point reflection, reradiation by leaky surface acoustic wave (LSAW) propagation. Not only distances but also wave velocities are different for these different paths, so contrast in SAM for many applications is related to interference between the waves coming from these different paths. Interference between direct and LSAW reflections can be used for local measurements of the acoustic properties of the specimen, a method known as the *V(z)* technique which will be presented in the second section. But first we shall discuss imaging applications.

Imaging Applications

Low frequencies (10-100 MHz)

Working in the low frequency range, representing a bridge between non-destructive testing (NDT) and SAM, offers a tomographic technique allowing the examination of the interior of various types of specimens as acoustic attenuation in the low frequency range is not too high. The opening angle of the lenses used in the low frequency range is relatively small (about 10°), ensuring that primarily longitudinal waves are generated inside the specimen. The existence of one single acoustic path greatly simplifies the interpretation of images, although some instruments use several, narrow, time gates and receivers in order to produce simultaneously several images taken at different depths inside the specimen.

An example of a low frequency application is shown in Fig. 2 giving images

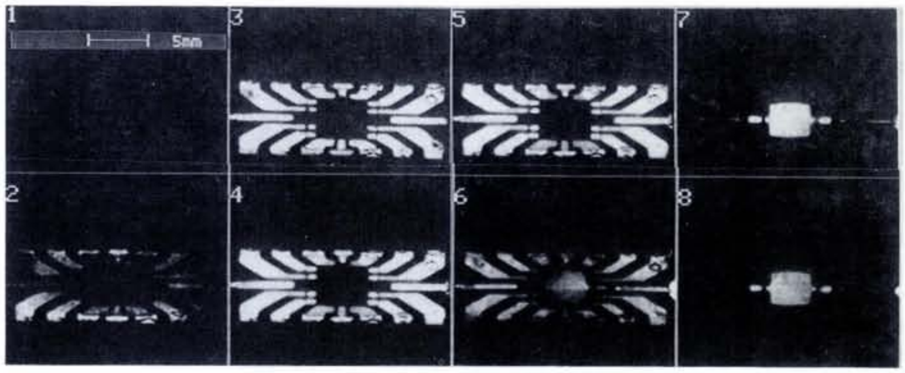

Fig. 2 — Low frequency (25 MHz) SAM images of a polymer encapsulated integrated circuit taken at increasing depths (1 to 8). Delaminated area is white on slice 2 (window in time electronic "gate" above the level of delamination) and dark on slice 6 (gate below the level of delamination). The scale bar in (1) represents 5 µm.

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of an integrated circuit encapsulated in a moulded plastic case: delamination between the metal and plastic is easily distinguished as dark areas. Phase sensitive detectors can be used for the same purpose since the signal reflected from a solid-air (unbonded) interface has an opposite phase to the signal coming from a solid-solid interface.

Medium frequencies (100-800 MHz)

Increasing the working frequency increases the resolving power of the microscope but the penetration depth is decreased at the same time. However, from our experience, most SAM imaging applications involve medium frequencies. Careful sample polishing is necessary in order to suppress the contrast coming from the topography of the specimen surface. Two typical applications are presented here, both for composite materials. The first concerns a multilayer specimen, namely a piece of packing material used in the food industry. Acoustic waves were focussed at the interface between the coating polymer and the aluminium substrate and Fig. 3a shows clear evidence for several unbonded areas at the interface. The delamination problem was solved by applying the appropriate heat treatment, whereupon only contrast originating at the rough surface of the polymer film could be detected. The second example involves the photograph in Fig. 3b showing interference fringes between reflected waves coming from glass fibres emerging at an angle of 45° to the surface of an epoxy matrix: the longitudinal wave velocity in the matrix can be determined by measuring the distance between the fringes.

High frequencies (0.8-2 GHz)

High resolution SAM becomes especially difficult in the GHz range owing to the high level of ultrasonic attenuation in water. The acoustic path in water and the lens radius have to be small (usually 50 μm) in order to decrease losses in the coupling fluid. Attenuation in the sample also becomes important, resulting in a small penetration depth. Typical lenses used in the high frequency range are designed with a large opening angle (50-60°), guaranteeing that surface acoustic waves are generated in most materials. Contrast due to SAW dominates in this frequency range and the sensitivity of the method to small perturbations of the SAW propagation becomes important. This means in practice that structural defects (microcracks, microporosity) can be detected even well below the theoretical resolution of about one wavelength λ (detectabilities of $\lambda/30$ and better have been reported).

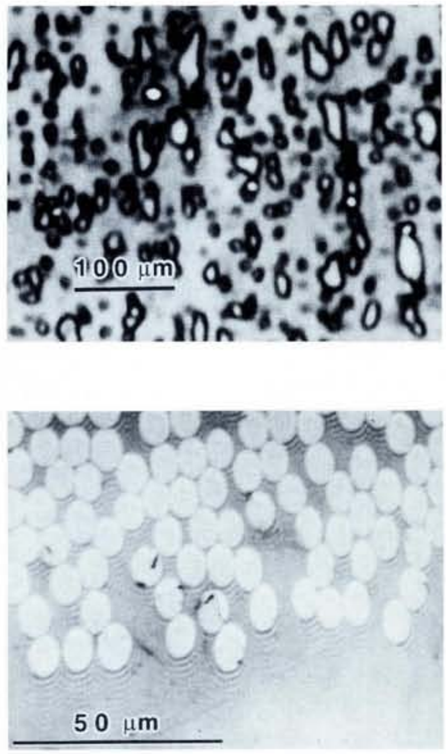


Fig. 3 — Medium frequency SAM images. (a, upper) Acoustic waves (200 MHz working frequency) focussed on the interface between a polymer coating on an aluminum foil: unbonded area are revealed as contrast-free zones.
(b, lower) Reflected waves from glass fibres emerging at 45° from the surface of a glass-fibre reinforced epoxy composite produce interference fringes in the lower half of each fibre (1 GHz working frequency).

Interference between the SAW path and the direct acoustic path are heavily perturbed by even small variations in the propagation constants, a feature that gives a high sensitivity of SAM to the grain structure, which can be easily resolved without etching the specimen surface. The generated SAW are mostly of the Rayleigh type in semi-infinite (thick) specimens, but other types of SAW can be generated in layered media. In thin specimens, Lamb waves can be propagated and these give important acoustic contrast.

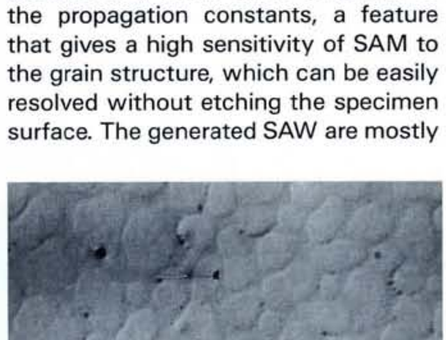


Fig. 4 — High frequency (1.3 GHz) SAM image of a specially prepared epoxy bond between gold and stainless steel imaged from the gold side. The cellular structure of the polymer accounts for deterioration of the bond.

An interesting example relates to a study of the interface between two metallic components bonded by an epoxy glue. This is a typical application where both an adequate penetration depth and high resolution are necessary. Since these two requirements are incompatible, samples were prepared as follows: a thin ($\approx$ 100 nm) gold layer was deposited on a flat piece of optically polished synthetic sapphire. An adhesive bond was then made between the gold-plated sapphire and a stainless steel plate. After hardening of the epoxy glue, the joint was cleaved at the gold-sapphire interface. Using this preparation technique, a three-layer sample was obtained (stainless steel/glue/gold layer) which could be examined from the gold layer side using a scanning acoustic microscope operating at 1.3 GHz. Typical images (Fig. 4) show a clearly defined cellular structure — an important observation that helps to explain the progressive deterioration of thin epoxy joints in the presence of water.

Measurement Applications

Contrast theory

To understand how SAM can be used to measure physical properties we first need to analyze the nature of the contrast in acoustic microscopy, where it is essential to know the expression giving the amplitude of the reflected signal V as a function of the distance z between the lens and the sample, the so-called $V(z)$ curve. A wave which is generated by the transducer and focussed on the sample is reflected back by the sample. It is modified during reflection by the sample's reflectance function $R(\theta)$ describing the amplitude and the phase of reflected waves as a function of the incident angle θ. At the focus, the signal received by the transducer is obtained by summing, over the area of the transducer, the reflected acoustic waves making their way back to the transducer:

$$V(0) = \int_0^\infty P(\theta)R(\theta)2\pi r dr$$

where r is the radius coordinate of the transducer and $P(\theta)$ is the pupil function of the lens. Assuming that the focal length is f, then $r = f \sin\theta$ at the focus, and $V(0)$ may be written as:

$$V(0) = 2\pi f \int_0^{\pi/2} P(\theta)R(\theta) \sin\theta d\theta$$

The phase of the waves on the transducer is changed by a factor $2kz \cos\theta$,

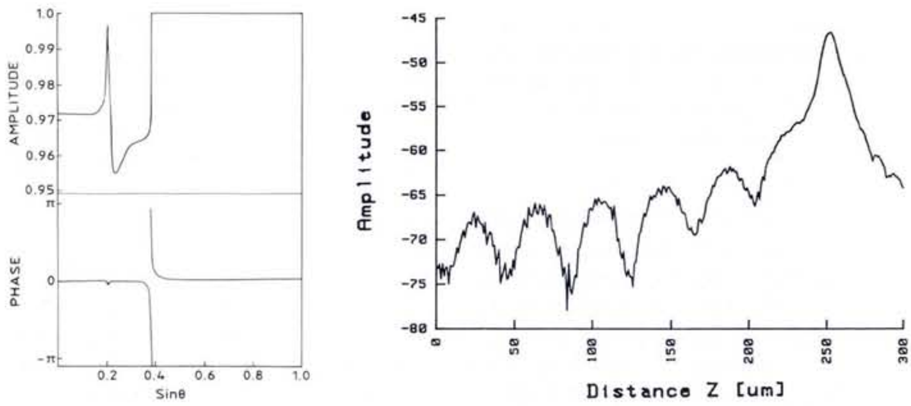


Fig. 5 — The basis for using SAM as a measuring technique. (a, left) The amplitude and phase of a typical reflectance function $R(\theta)$ of a sample showing a phase change of 2π near a critical incident angle θ_R of the acoustic wave, called the Rayleigh angle.
(b, right) Substituting the function $R(\theta)$ in an expression (see text) for the transducer output V as a function of the sample-lens distance z gives the classical V(z) curve for tungsten carbide at a working frequency of 237 MHz: the main periodicity of the oscillations is directly related to the surface acoustic wave velocity.

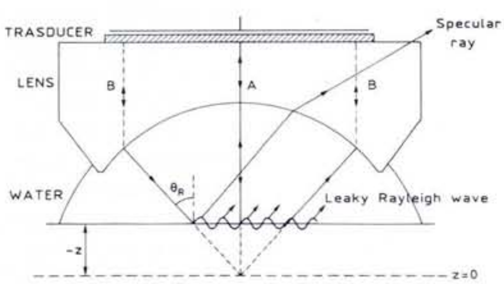


Fig. 6 — Simple ray model illustrating how the oscillatory behaviour of the V(z) curve can be understood. The main periodicity is related to destructive and constructive interference between two important acoustic paths, the direct reflection path (A) and the path associated with the surface acoustic (leaky Rayleigh) waves (B).

where k is the wavenumber in the coupling fluid, when defocussing is performed by changing the distance z between the lens and the sample. One can then derive the following formula for the $V(z)$ curve:

$$V(z) = 2\pi f\int_0^{\pi/2} P(\theta)R(\theta)\exp(-i2kz\cos\theta)\sin\theta\cos\theta d\theta$$

The reflectance function $R(\theta)$ of the sample plays a very important role in this expression. It is complex and can be represented by its amplitude and phase (Fig. 5a), the most important feature being a sudden phase change of 2π near a critical angle called the Rayleigh angle θ_R. Introducing such a reflectance function in the expression for $V(z)$ leads to the typical oscillatory behaviour which is observed for the $V(z)$ curve (see Fig. 5b). The main periodicity Δz of the $V(z)$ curve can be calculated and is given by:

$$\Delta z = \pi/[k(1-\cos\theta_R])$$

with $k = 2\pi\nu/c_w$, in which ν is the acoustic frequency and c_w the wave velocity in the coupling fluid.

The oscillatory behaviour of the $V(z)$ curve can be understood from the simple ray model presented in Fig. 6. The main periodicity Δz is related to the appearance, as a function of z, of constructive and destructive interference occurring between the waves coming back normally to the transducer from two different paths, namely, the direct reflection acoustic path in the middle of the lens and the acoustic path associated with surface acoustic waves (leaky Rayleigh waves) generated at the critical angle of incidence θ_R. The periodicity can be used to determine the SAW velocity v_r as the velocity is related to the critical angle θ_R by:

$$\sin\theta_R = c_W/v_r.$$

One can also measure the SAW attenuation — albeit somewhat less precisely — from the rate of decay of V as a function of z. An important advantage of the $V(z)$ technique is related to the fact that the measurement is localized within the diameter of the lens cavity. In practice, this means that point-to-point measurements can be performed on the sample surface in order to study the properties of small inclusions, different phases or other inhomogeneous features such as porosity, microcracks, *etc.* Investigating small samples is also possible and this becomes very useful when large samples cannot be produced (*e.g.* quasicrystals).

Continuous wave

In layered media, surface acoustic waves become dispersive (the velocity depends on frequency). In such cases we use the continuous wave (CW) $V(z)$ method (Fig. 7), which gives results si-

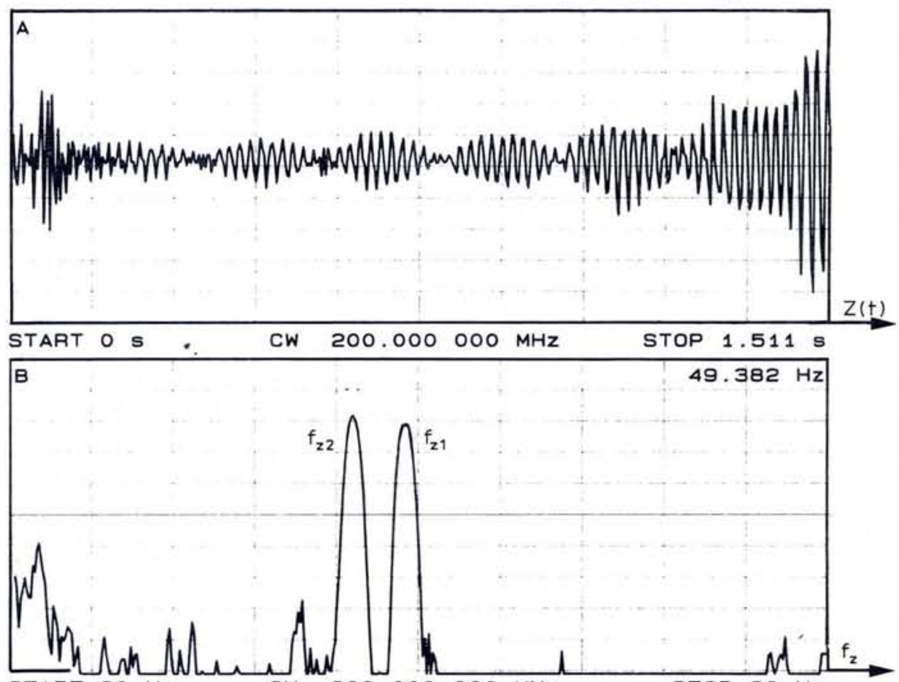


Fig. 7 — Continuous wave V(z) method used for layered media where the SAW velocity depends on the frequency. (a, upper) Transducer output amplitude as a function of z during continuous radiation of the specimen with 200 MHz acoustic waves: oscillatory behaviour similar to that for a pulsed field is observed.
(b, lower) Fourier transform of the CW V(z) curve showing two peaks: f_{z1} related to the velocity of sound in water (the coupling fluid) and f_{z2} related to the SAW velocity.

milar to those obtained by the classical pulsed method, but allow in addition the working frequency to be changed. Dispersion curves can easily be obtained, from which elastic constants of the layers can be calculated. Fig. 8a shows the dispersion curves of two SAW modes measured in a thin layer of gold (1.2 μm) deposited on a substrate of $LiNbO_3$.

Using a cylindrical lens ensures unidirectional SAW propagation perpendicular to the axis of the cylinder. Repeating the *V(z)* measurements while turning the sample around the *z*-axis allows the determination of the angular dependence of the SAW velocity, this being in fact a measure of the anisotropy of materials. For example, this method was used to determine whether Al_6Li_3Cu quasicrystals are elastically isotropic (Fig. 8b), and to measure the angular dependence of the velocity of SAW propagating along the (100) plane of a single crystal of Ni (Fig. 8c), permitting a comparison between estimates from the SAM measurements, theoretically computed SAW velocities and Brillouin scattering measurements.

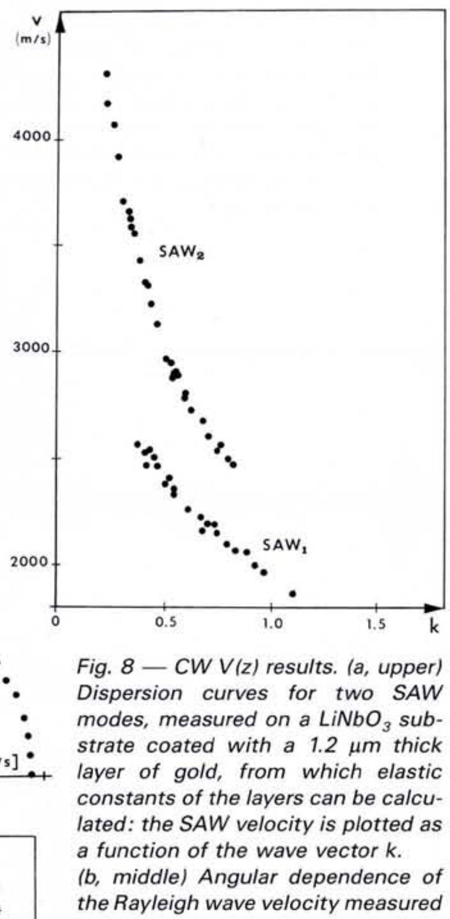
Future Prospects

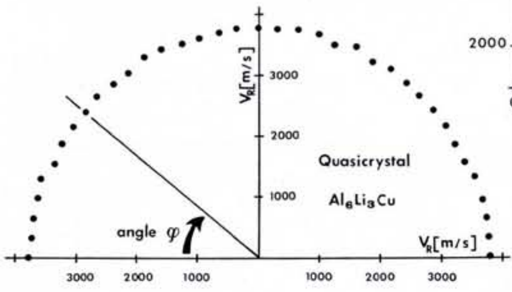
High resolution microscopy

SAM resolution at room temperature is comparable to that of a good optical microscope. Since a limit is imposed by the attenuation in the coupling fluid, major improvements are not expected. A more than tenfold increase was obtained using liquid helium as coupling fluid, but such techniques are not suitable for everyday use. In the field of high resolution imaging, one of the most interesting techniques involves exploiting the extremely good defect detectability owing to the artifacts produced by wave interference.

Defect detection

The first and most logical step in the field of defect detection is phase sensitive detection in the high frequency domain. The most significant achievement belongs to IBM, where phase-sensitive SAM was successfully used at 670 MHz to visualize internal stresses in solids. But the separation of reradiated SAW and direct reflection by signal analysis is also an interesting high sensitivity technique for detecting defects. Such separations have already been realized at low frequencies and only technical limitations need to be overcome in order to generalize the approach in the high frequency range.





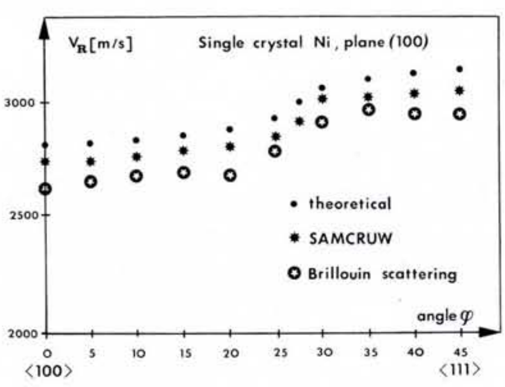


Fig. 8 — CW V(z) results. (a, upper) Dispersion curves for two SAW modes, measured on a $LiNbO_3$ substrate coated with a 1.2 μm thick layer of gold, from which elastic constants of the layers can be calculated: the SAW velocity is plotted as a function of the wave vector k.
(b, middle) Angular dependence of the Rayleigh wave velocity measured on a single grain of a Al_6Li_3Cu quasicrystal by rotating the sample (cylindrical lens at 200 MHz): the data show that the crystal is elastically isotropic.
(c, lower) Angular dependence of Rayleigh wave velocity measured on the (100) plane of a nickel single crystal: results agree with numerical calculations and Brillouin scattering measurements.

Acoustic power is extremely high near the focal point, leading to nonlinear acoustic behaviour, especially in biological samples. Nonlinear parameters can be of great interest in the field of biology and medicine, by allowing discrimination between normal and pathological biological tissues.

Property measurement

Ultrasonic measurements are applied in different fields of solid state physics whenever a physical phenomenon leads to an acoustic loss or to a change of the propagation velocity. Acoustic measurements are currently used to study the mechanical properties of crystalline materials (elasticity, inelasticity, plasticity), structural (martensitic) and several types of solid state phase transformations (amorphous, superconducting, ferromagnetic). Appropriate ultrasonic measurements can be performed locally on a sample surface using an acoustic microscope; they can also be carried out on very small samples or in very thin layers. In this field of potential applications of SAM, the sensitivity and the accuracy of determinations of wave velocity and wave attenuation must be improved. Very interesting applications in solid state physics can also be imagined by using different coupling fluids to allow measurements over an extended temperature range.

FURTHER READING

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100 T Workshop

Professor Laurie Challis of Nottingham University, UK, who helped draft the 1990 report of the CEC Study Panel on High Magnetic Field facilities (see *Europhysics News* **22** (1991) 158), writes to say that the EC Advisory Committee for the Large Installations Plan recommended last February that a Steering Committee should be set up to prepare and supervise the design of a new semi-continuous facility.

A committee has now been established from members of the Study Panel. Its first task will be to organize in May 1992 a 2/3 day workshop to discuss the science which could be carried out at a 100 T facility, the instrumentation that would be needed and possible applications, including materials development. The workshop report will form the basis of the scientific case for a full feasibility study.

These plans were reported on 31 August at the plenary session of the 3rd International Symposium on High Magnetic Field Facilities, and were warmly endorsed.