# THE POWER OF X-RAYS IN MATERIALS SCIENCE

THE X-RAY MATERIALS SCIENCE (XMaS) PROJECT USES A SYNCHROTRON FACILITY, A MASSIVE DOUGHNUT-SHAPED STRUCTURE THAT ACCELERATES ELECTRONS TO HIGH VELOCITIES. THESE ELECTRONS EMIT X-RAY RADIATION, WHICH CAN BE USED TO STUDY THE TINIEST INTRICATE STRUCTURES WITHIN ALL KINDS OF MATERIALS. WHAT DOES THIS MEAN FOR SOCIETY AND OUR FUTURE?

#### GLOSSARY

EUROPEAN SYNCHROTRON RADIATION FACILITY (ESRF) - a synchrotron research facility in Grenoble, France

**SYNCHROTRON** – a machine that accelerates electrons inside a circular ring to speeds close to the speed of light and bends their paths using magnets. As the electrons are accelerated along a curved path, they emit electromagnetic radiation whose energy spectrum is proportional to the speed of the electrons

X-RAY – a form of high-energy electromagnetic radiation

**BEAMLINE** – there are three definitions of a beamline: 1) a physical space within the ESRF's experimental hall, consisting of an optics cabin, an experimental cabin and a control cabin; 2) equipment that brings the X-ray beam to the material being studied and records the findings; 3) a research space in which experiments are

On the edge of the city of Grenoble in the

French Alps can be found a huge ring-shaped

accelerate electrons to extremely high speeds.

When these high-energy electrons change

direction, thanks to magnets within the ring,

they emit electromagnetic radiation which, due

high-power X-rays being produced. These X-rays

to the speed of the electrons, results in many

are directed into structures off the main ring,

called beamlines, and are used in experiments.

Each beamline is configured slightly differently

and targeted at specific scientific research areas.

structure. This is the European Synchrotron

Radiation Facility (ESRF), designed to

done in collaboration. For a more detailed explanation, visit: www.esrf.fr/about/synchrotron-science/beamline

**BLUE SKY RESEARCH** – also known as blue sky science or fundamental research, this is driven by curiosity and the outcomes or practical impact are uncertain. However, the blue sky research of 100 years ago underpins the whole of modern technology and scientific research

DIFFRACTION - the way that electromagnetic waves (at the ESRF, X-rays) bend and interfere when they encounter a particular obstacle

**ELECTRON** – a subatomic, negatively charged particle

SCATTERING – in physics, this is where an electromagnetic wave (at the ESRF, an X-ray)

"The 'acceleration' within the storage ring is not to make the electrons go faster (like a car entering a highway), but to bend them in a circular trajectory, like the acceleration, or force (F = m a), you feel when you take a sharp turn in your car," explains Dr Didier Wermeille, who is based at the ESRF. "It is the acceleration of the electrons (the bent trajectory of a charged particle) that produces the X-rays we use."

Researchers from across Europe use the X-rays to shed light on the position and motion of atoms in any substance of interest, like a 'super-microscope'. Why is this exciting? Well, synchrotrons can help scientists explore the structures of both inorganic and organic substances in fine detail. For instance, they have revealed the structures of proteins and viruses, helped develop more efficient catalysts and batteries, and identified the chemical states of heavy elements such as uranium.

"Modern technologies are underpinned by advances in our knowledge and function of the materials of which they are made," says XMaS Co-Director, Professor Tom Hase. "In many cases, researchers are trying to understand

changes its path or direction when encountering physical objects like atoms

**SPECTROSCOPY** - the study of the absorption and emission of light and other radiation by matter. It strongly depends on the energy (or wavelength) of the radiation and includes the study of the interactions between particles, such as electrons, protons and ions, as well as their interaction with other particles. See www.britannica.com/science/spectroscopy

XMaS - a research facility based at the ESRF that supports the UK materials science communities and is funded by the Engineering and Physical Sciences Research Council (EPSRC): www.ukri.org/councils/epsrc/ facilities-and-resources/find-an-epsrc-facilityor-resource/ the correlation between a material's useful properties and its structure. This information can help us make better and more efficient devices and materials." Given that materials behave differently under certain conditions, this involves creating complex sample environments to test their properties under realistic operating conditions.

#### THE XMAS BEAMLINE

Each beamline at the ESRF is home to a particular research team who uses the X-rays produced by the synchrotron for their own areas of research. One such beamline hosts the XMaS (X-ray Materials Science) research project, which is run by two UK universities. Tom directs the project from the University of Warwick, alongside Professor Chris Lucas from the University of Liverpool. "It is our job to oversee, monitor and manage this multi-millionpound project," says Tom. "This includes both overseeing the research and ensuring the project remains within budget."

On the ground, the XMaS beamline is run by a multinational team of four scientists who support and help users to tackle a wide range of scientific challenges. "Our research involves scattering, diffraction and spectroscopy," says Didier, who is known as the 'Beamline Responsible'. "I facilitate these experiments, supervise technical upgrades to the beamline, organise the team, liaise with the ESRF's wider facility, and take care of health and safety."

Indeed, if high-powered X-ray radiation encounters living organisms, it can be very harmful – but this is not an issue at the ESRF. "The X-ray beam and all equipment and samples are contained in lead-lined, interlocked rooms called hutches, through which no radiation can escape," says Didier. "Radiation levels can get dangerously high within these rooms, but strict operational safety protocols make it impossible for any person to be in there when the beam is on."

#### SYNCHROTRON HISTORY

Synchrotrons have been around in some form for over half a century. They have helped scientists of many disciplines make grand strides in their knowledge, using X-rays to examine the structures and behaviour of minute and hidden objects. "Synchrotrons can produce much more intense sources of X-rays compared to conventional lab-based equipment," says Tom. "This means that experiments can be performed incredibly quickly. It also means that it's possible to perform experiments that otherwise would be inconceivable." The ESRF was built in 1989, first used in 1994, and world-leading in its capabilities. "The ESRF was built by a consortium of European countries, including the UK, but competition for its use was inevitably fierce," says Tom. "It was decided to allow individual nations to add their own user facilities, or beamlines, onto the ESRF, and the UK managed to secure a place." The negotiated deal means that the XMaS beamline is dedicated to UK-led science for two-thirds of the time. Other international ESRF researchers can also use it in the remaining time available.

# INTERDISCIPLINARITY AND COLLABORATION

Though the XMaS beamline principally investigates materials science, this does not mean the research exclusively involves materials scientists. "Materials science can be a surprisingly broad term," says Tom. "At XMaS, we have embraced dental researchers studying hierarchical teeth structure, and scientists investigating the decay of museum artefacts such as the Mary Rose, which was King Henry VIII's war ship, or baroque organ pipes. We often work at the interface between disciplines, exploiting advances in one scientific area to impact others."

Currently, XMaS is investigating energy-related issues, including the behaviour of catalysts, the operation of batteries, and the photovoltaics used in solar panels. The findings could assist the transition to clean energy, which is needed to help mitigate climate change. Excitingly, these energy-related projects are working alongside blue-sky research, too. "There are studies taking place into unusual magnetic materials, which might one day have some practical use," says Didier. "There is also research into substances with zero electrical resistance and this could have great commercial applications if they can be made to operate closer to room temperature."

As a pan-European facility, the ESRF (and XMaS) is a vital hub for attracting cuttingedge research and the finest minds from across Europe. "The best science emerges when the brightest scientists meet and discuss what they are doing," says Tom. "These conversations can help stimulate new ideas and approaches that would not have emerged if they were working in isolation, leading to productive collaborations."

#### TACKLING SOCIETY'S GRAND CHALLENGES

Much of the equipment at XMaS is expensive to replace, but after 10-20 years of use, XMaS has been upgraded. "The ESRF was updated to produce higher-brilliance X-rays, which meant it was time to update the equipment on the XMaS



#### **PROFESSOR TOM HASE**

XMaS Co-Director, Department of Physics, University of Warwick, UK



#### **DR DIDIER WERMEILLE**

XMaS Beamline Responsible, European Synchrotron Radiation Facility (ESRF), France; Department of Physics, University of Liverpool, UK

#### FIELD OF RESEARCH

Materials Science

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#### RESEARCH PROJECTS

Using high-powered X-rays to investigate the minute properties and behaviour of a range of materials under different conditions

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#### **FUNDER**

Engineering and Physical Sciences Research Council (EPSRC)

beamline," says Didier. "This means we can investigate materials and matter in much finer detail and at an even faster pace. And now there is room for more scientists!"

As Tom goes on to explain, this step-change will enhance XMaS's capabilities for the next influx of researchers: "XMaS enables UK scientists to do experiments at the forefront of international science. The young students and researchers being trained on the beamline now will become research leaders in time, tackling the greatest challenges our society faces."

# MEET SOME OF THE RESEARCHERS USING XMaS

# PROFESSOR MAISOON AL-JAWAD



PROFESSOR OF BIOLOGICAL PHYSICS AND BIOMINERALISATION, SCHOOL OF DENTISTRY, UNIVERSITY OF LEEDS, UK I am interested in how teeth and bones grow; what happens to their structure when they are broken or damaged; and how we can design better ways to repair or replace them when damaged.

I carry out 2D synchrotron X-ray diffraction and X-ray fluorescence experiments at the XMaS beamline to study tooth and bone tissues. Teeth and bones are made of soft tissue (a mixture of proteins, water and other biological molecules) and hard tissue (calcium and phosphate ions calcified into a crystalline mineral called hydroxyapatite). Combined, the soft and hard tissues give teeth and bones the properties they need to function correctly.

Bones comprise around 60% mineral and 40% organic molecules, which means they are strong but have elastic properties, so they don't break under load. In contrast, dental enamel (the outer coating of a tooth) is made up of 96% mineral and 4% organic components. This makes enamel hard and resistant to wear, such that your teeth should last a lifetime. The mineral within these hard tissues is organised in a very precise way, from nano-sized building blocks to microscopic bundles of mineral, to a whole macroscopic tooth or bone. This is called hierarchical structure and I am interested in how this hierarchical structure grows biologically. This process is called biomineralisation and I'm interested in how it can be destroyed e.g., through tooth decay, which breaks down the mineral structure of the tooth enamel, and how it can be restored through careful remineralisation treatments.

XMaS enables us to look in great detail, deep inside tooth and bone samples, to probe structural and chemical features from length scales smaller than a nanometre ( $x10^{-9}$  metre), to micron-scale ( $x10^{-6}$  metre).

We have found out that, with careful remineralisation treatments, it is possible to restore the complex hierarchical crystal structure of dental enamel when it has been damaged. This is encouraging because we might be able to suggest better ways to prevent tooth decay in the future, which, in turn will save the health service a lot of money.

We have also found that bone from different sites (e.g. skull bone versus limb bone) looks very different at the sub-nanometre and nanometre length scales. This gives us an insight into the role that crystal structure might play in the effects of osteoporosis on bones in later life.

# DELVE INTO DENTISTRY AT THE UNIVERSITY OF LEEDS

- Open Wide, a student-led project based at the University of Leeds' School of Dentistry, works with children aged 4-18 and prioritises pupils from backgrounds that are under-represented in dentistry: www.openwidedentists.co.uk
- The Dentistry Summer School is designed to give Year 11 and 12 students an insight into life as part of a dental team, and the science behind dentistry: www.healthsciences.leeds.ac.uk/events/dentistry-summer-school-2021

### PROFESSOR WUGE BRISCOE



PROFESSOR OF PHYSICAL CHEMISTRY, SCHOOL OF CHEMISTRY, UNIVERSITY OF BRISTOL, UK At XMaS, my team and I have studied how molecules (called surfactants, lipids and polymers) organise at interfaces – surfaces that form the boundary between different matter. This interfacial structural information is important for nanoscience, understanding biological processes, and industrial applications.

Typically, we shine an X-ray beam at the interfaces and the reflected X-ray beam can be analysed to obtain information, such as the thickness, density and packing in the molecular layer at the interface.

XMaS uses the world's brightest light, and we can make use of it to probe buried interfaces that cannot **be accessed through other methods**. The molecular details of the structures are sometimes surprising, and this helps us advance our understanding of how molecules behave at interfaces.

Beyond scientific curiosity, such knowledge has wide ranging implications. For instance, we have applied our research findings and knowledge to formulate eco-friendly artificial snow, which has been used in many Hollywood films and public events. We are also helping people in industry understand how interfacial molecular layers can be optimised for their functionalities in personal care products and lubricants for future transport.

### DISCOVER BRISTOL ChemLabS

- The Bristol ChemLabS Outreach programme engages with teachers, schools and students of all ages: www.chemlabs.bris.ac.uk/outreach/chemnet
- Bristol ChemLabS also has a YouTube channel, courtesy of Dr Jonny Furze, which includes teacher training videos: www.youtube.com/channel/UCD7Gud4DucBvmw\_zCTKe70g
- The Bristol Outreach Project has a website aimed at post-16 students, which showcases the research done at the University of Bristol: **bristoloutreachproject.wordpress.com**

### PROFESSOR MARKYS CAIN



TECHNICAL DIRECTOR ELECTROSCIENCES LTD, UK I have always loved physics and dreamt of running my own research company from a young age. I managed to secure a dream job at the UK's National Physical Laboratory (NPL), setting up a new research group devoted to 'functional' materials. These are materials that have useful sensing or actuation properties. They're sometimes called 'smart materials' because they respond in a useful way to changes in their local environment.

I left NPL and launched my new company, Electrosciences Ltd, in 2015. My main interests are piezoelectric, ferroelectric and other smart/active materials used for medical scanning systems, sonar, haptic touch screen technology, nanometre precision actuators and other sensors for a whole range of industrial applications.

I have collaborated with the wonderful team at XMaS

for probably a couple of decades now. My research is centred on the development of *in situ* or operando methods of characterising functional materials. I use X-rays to probe the materials' electrical, dielectric and functional performance. The neat thing is that we have worked out ways of synchronising all these measurements. This is important because we can now start to paint a picture of why a material behaves in the way that it does.

Electrosciences Ltd. works with many industrial companies that are developing new materials or using novel materials in new ways. One example is the use of piezo-materials in the treatment of arthritis. All of these materials benefit enormously from discovering the links between their function and fundamental crystalline structure, as we test them in conditions that match their real applications (stress, temperature, strain, magnetic and electric fields, and so on).

# CONTACT INDUSTRIES NEAR YOU

Markys has demonstrated the use of smart materials to a class of 4–5-year-olds, which shows that many companies are dedicated to sharing their work with students and schools. It is well worth contacting companies near you to see what they can offer.

# PROFESSOR MARK DOWSETT



EMERITUS PROFESSOR IN PHYSICS, THE UNIVERSITY OF WARWICK, UK

HONORARY FELLOW IN CHEMISTRY. GHENT UNIVERSITY, BELGIUM

# EXPLORE **KING HENRY VIII'S MARY** ROSE

The Mary Rose Museum has everything you need to know about King Henry VIII's warship - including a virtual museum:

Since I was a small boy, I've been fascinated by making things. Back then (in the 1950s) it was Meccano<sup>™</sup> cranes, wooden model boats and spaceship interiors inspired by Dan Dare (from The Eagle comic). In the 60s, my interest evolved into making real sailing boats, rockets, radio sets and hi-fi. Now, it's scientific instruments!

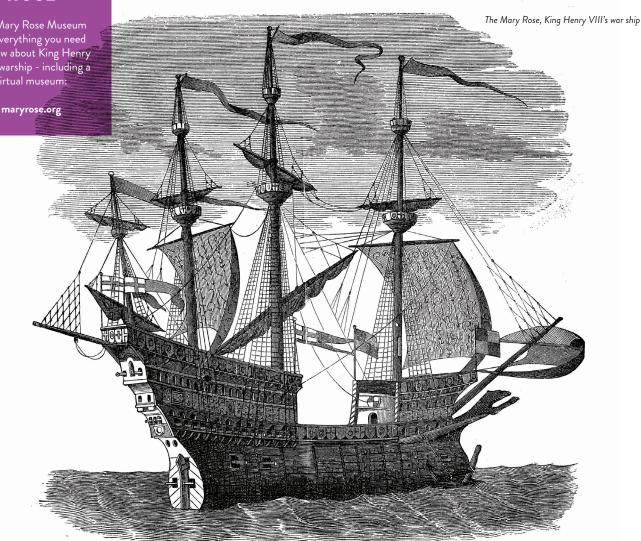
Through XMaS, I work with Professor Mieke Adriaens from the Electrochemistry and Surface Analysis Group at Ghent University. She and I use XMaS to watch chemical reactions on surfaces in real time: that is, as they happen. We can see intermediate reactions, how reactions start and when they come to an end. We use a special electrochemical and environmental cell (eCell) to watch how surfaces change when they are exposed to gases, in corrosive liquids, or coated with protective coatings.

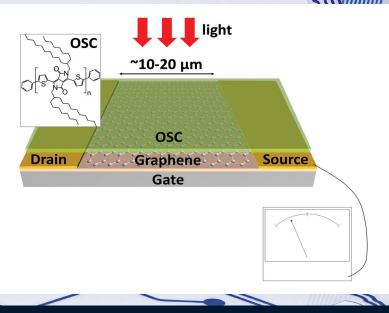
These experiments are important in a huge range of applications, from the study of contraception devices to the corrosion of metals (especially heritage metals), and the development of protective coatings.

Recently, we had the privilege of working with scientists from the Mary Rose Trust to examine metal artefacts recovered from the seabed over 30 years ago. When objects are taken from the sea they rapidly corrode in air and need immediate special treatment if they are to survive. This is even more important if they're put on long-term display in a museum or used for archaeological study. Using X-ray diffraction, we were able to identify unusual compounds on the surfaces of the metal artefacts, show that their conservation had been very effective, and discover traces of other metals - possibly from the tools used by Tudor metalworkers.

Another study we are involved in looks at the design of less intrusive and more effective intra-uterine devices (IUDs). IUDs, also known as the coil, are used for birth control and release copper in the womb. In a PhD project, we made a version of the eCell to simulate the chemical environment of the uterus and measured the chemical changes on the surface of the copper used to make the device. It's important to know how IUDs actually work before you can improve them!

It's demanding but enormous fun to work on XMaS with the XMaS team. Between us, we have come up with a couple of new instruments to try and, lately, I've also been writing software codes to help other researchers on XMaS analyse their data.





Hybrid photodetector consisting of an organic semiconductor (OSC) on top of graphene. Incoming light gets absorbed by the OSC and causes an electrical signal to be detected.

# DR JAKOB KJELSTRUP-HANSEN



ASSOCIATE PROFESSOR, UNIVERSITY OF SOUTHERN DENMARK, DENMARK

#### DR MATTI KNAAPILA



DEPARTMENT OF PHYSICS, NORWEGIAN UNIVERSITY OF SCIENCE AND TECHNOLOGY, NORWAY We are interested in organic electronics, which is a new branch of electronics based on plastic materials. In contrast to most electronic devices today, which are hard and rigid, organic electronics enable devices to be mechanically flexible (bendable and possibly stretchable). These materials could be used to create wearable devices that are integrated into clothing, or stretchable OLED displays that wrap around objects. OLED stands for organic light-emitting diode, which is basically a light source used widely in electronics, like smartphones.

**Organic electronic devices are still at an early phase**. We develop and study such devices to find out how we can improve these to a level where they can be used in real-world applications.

The plastic materials used for organic electronics consist of microscopic molecules that form a thin layer on a substrate. The performance of the organic electronics component strongly depends on how the molecules are organised on the substrate – the so-called microstructure. Our research at XMaS uses X-rays to characterise the microstructure to better understand how we should fabricate thin layers. Our findings will help us develop organic electronics components that perform well.

XMaS allows us to use very intense X-ray beams and sophisticated detectors, beyond what is possible with small-scale equipment in university laboratories. Using a technique called grazing incidence X-ray diffraction (GIXRD), we can study the features and functions of thin layers; in particular, how the organic molecules are organised when they form thin layers on the substrate.

Our research projects are mostly fundamental and curiosity driven, but the intended impact is to improve organic electronic devices for the future. Our research at XMaS connects us with other researchers who have similar focus areas, which means we can work on exciting collaborations that go way beyond what is possible in individual universities alone.

# STUDY AT A UNIVERSITY IN DENMARK OR NORWAY

• Many of the engineering programmes at The University of Southern Denmark and all the master's programmes at the Norwegian University of Science and Technology are taught in English. Students from all over the world are welcome.

www.sdu.dk/en/uddannelse/ingenioer

www.sdu.dk/en/uddannelse/moedsdu/studerende\_for\_en\_dag/ingeniorstuderendeforendag

www.ntnu.edu

#### **DR PETER WELLS**



ASSOCIATE PROFESSOR, SCHOOL OF CHEMISTRY, UNIVERSITY OF SOUTHAMPTON, UK

#### **GEORGE TIERNEY**



PHD STUDENT SCHOOL OF CHEMISTRY, UNIVERSITY OF SOUTHAMPTON, UK The main focus of our research is the study of catalytic processes. Catalysis underpins vast aspects of modern society: it is essential for transportation, the production of food, the development of active pharmaceuticals, and many other important industries. Furthermore, new catalytic technologies are essential as we transition towards a net-zero carbon future.

A catalyst promotes chemical transformations by lowering the energy input required, whilst not being consumed in the process. However, there is a significant distinction between not being consumed and remaining unaltered.

Our projects at XMaS focus on two major strands: i) the upgrading of waste plant matter (i.e., biomass) to useful products, and ii) automotive exhaust aftertreatment applications for clean air.

Many current chemical commodities – that are needed for our material world – are based on fossil resources. However, there is a need to develop a new supply chain, based on recycling waste material into useful chemical building blocks. Here, waste biomass is a plentiful and renewable feedstock. A feedstock is any renewable, biological material that can be used directly as a fuel, or converted to another form of fuel or energy product.

NOx emissions from diesel cars contribute globally to many premature deaths. For exhaust aftertreatment, we have been investigating the role of palladium (Pd) nanoparticles for the selective oxidation of ammonia – by selective, we mean to only produce the benign products of nitrogen and water.

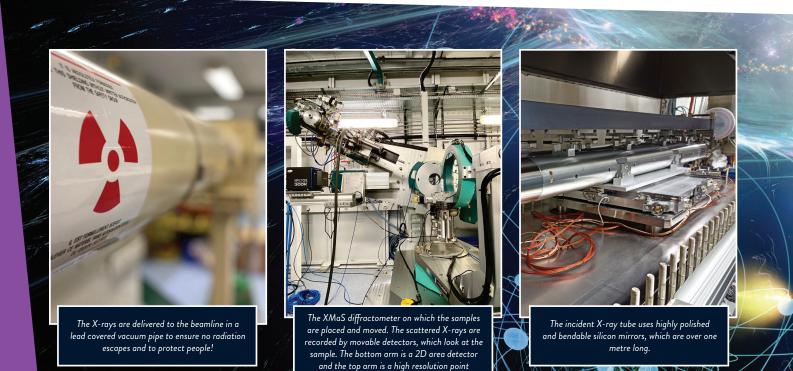
Our results on supported nanoparticle catalysts that were measured at XMaS are very exciting. The work on ammonia oxidation confirmed the presence of a type of Pd structure that had not been reported previously. This interstitial Pd nitride structure – where N sits within the voids of the metallic Pd sponge – had a very important role in stopping the formation of harmful NOx products.

Whilst there are large numbers of papers that discuss catalyst design, very few of them provide an understanding to how these remnants from catalyst preparation influence catalyst operation. By using XMaS, we have been fortunate to add to the growing understanding of how minority components, not removed during catalyst preparation, can play a crucial role in the functioning of catalysts.

# VISIT SOUTHAMPTON UNIVERSITY - OR GET A RESEARCHER TO VISIT YOUR SCHOOL

Southampton University offers an extensive programme of outreach activities for all ages: www.southampton.ac.uk/chemistry/outreach/index.page

Contact a university near you to find out if it offers similar experiences.



detector.



# • BE PART OF THE XMaS SCIENTIST EXPERIENCE AND MORE...

XMaS is committed to training students and early career researchers and, as such, has a wide range of outreach activities to fulfil this goal. "Students are our future, the next generation of X-ray scientists," says Tom. "Moreover, they often have exciting new ideas to offer us, meaning we benefit from the interaction as well as providing students with a unique training experience."

The students who will become the next generation of materials scientists are not only those doing research degrees, but also undergraduate and school students. The **XMaS Scientist Experience** is a UK competition for Year 12 female physics students to win an all-expenses-paid four-day trip to the ESRF. "The competition is designed to encourage female students into scientific careers where they are seriously under-represented," says Tom. "Winners get the opportunity to visit the ESRF, see synchrotron beamlines in action, take part in lab experiments, and talk with successful female scientists." The competition involves completing an assignment on the achievements of a leading female scientist. For more information, visit: warwick.ac.uk/ fac/cross\_fac/xmas/impact/outreach/xmas\_ scientist\_experienceb.

The XMaS team at the University of Warwick organises a **Science Gala** every year. "The Gala showcases all physics, from astronomy to particle physics, as well as materials science," says Tom. "There are activities suitable for ages 6-18, all with the message that science is fun and exciting and can lead to a wide range of careers." More information can be found at: warwick.ac.uk/fac/cross\_fac/xmas/impact/ outreach/sciencegala.

The XMaS team and the latest research findings can be found on the XMaS website: www.xmas.ac.uk. The Twitter page also provides an insider view into the XMaS beamline: twitter.com/XMaSBeam. Accessing XMaS through the website and Twitter is a great way to keep up to date with outreach activities that might interest you.

The XMaS team is part of a wider ESRF network, which also offers its own range of outreach activities. This includes site visits for schools, internships and summer schools, and the **Synchotron@School** programme, a oneday programme of experimental workshops and site visits for high school students. Visit the ESRF website:

www.esrf.fr/home/education.html

The ESRF sits within the EPN campus, an international science hub in the French city of Grenoble. The institutions within the campus have a wide range of career opportunities, from core science to administration, outreach, publications, and many others. Site visits can often be arranged. If you want to learn more, visit: www.epn-campus.eu.

# PATHWAY FROM SCHOOL TO MATERIALS SCIENCE

Materials science is a broad-ranging subject based on investigating and manipulating the properties and behaviour of different artificial and natural materials. This involves understanding the physics and chemistry of matter, and the manufacturing and engineering of materials.

At school, key subjects to take to prepare for a degree in materials science include physics, engineering, mathematics and chemistry. Other scientific subjects such as computer science and biology could also prove useful. As Tom says, "Many people do not start out as material scientists. A 'materials scientist' can cover a multitude of disciplines; it's really about working at the interface between different subjects and understanding how fundamental processes work in real systems."

Career Explorer offers a good explanation of what a materials scientist does, including the salary range (in US dollars): www.careerexplorer.com/careers/materials-scientist

"The good news is that the materials industry is well-regarded as it contributes £200 billion each year to the UK economy," says TheUniGuide, which gives a great introduction to studying materials science at the university level: www.theuniguide.co.uk/subjects/materials-science

Prospects provides a list of careers open to graduates with a degree in materials science: www.prospects.ac.uk/careers-advice/what-can-i-do-with-my-degree/materials-science-and-engineering