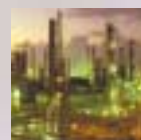# Neutrons and new materials

A review of ILL research
with potential industrial applications

NEUTRONS FOR SCIENCE

This booklet is the second one of a series devoted to the application of neutron techniques in different research areas. The next one, to be published in 2003, will focus on the use of neutrons in nuclear and particle physics.
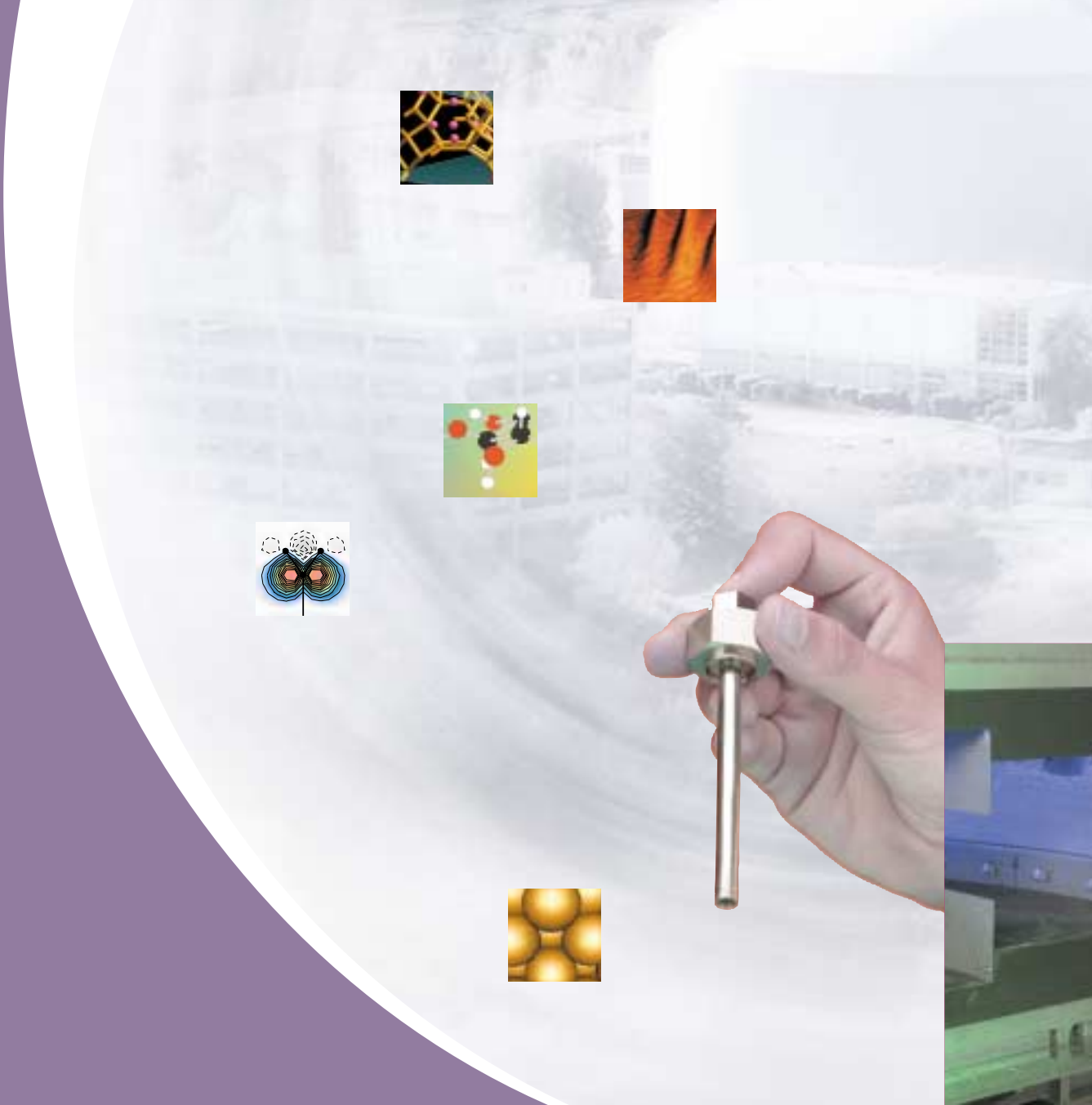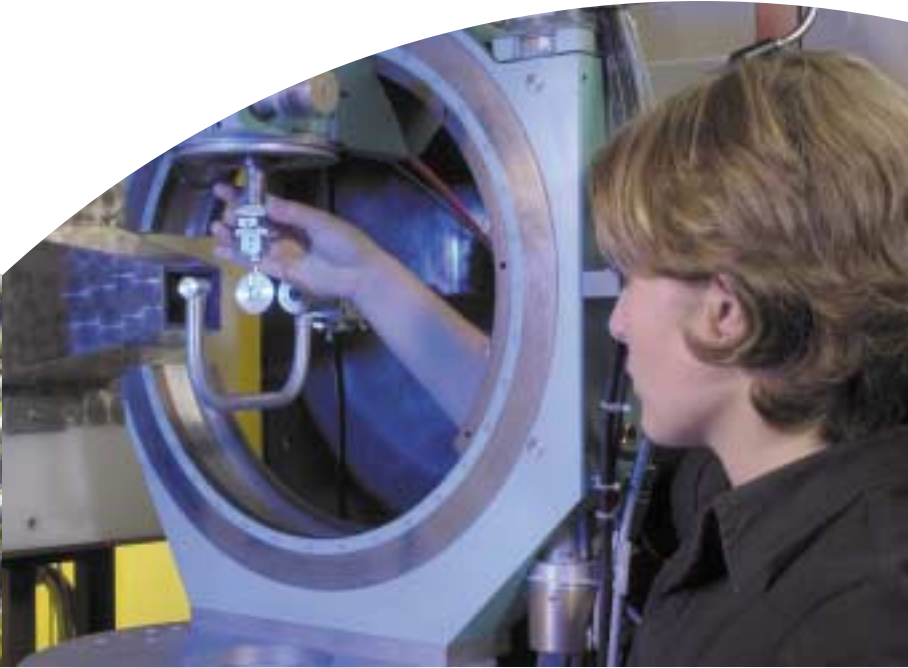
Foreword

T he development and engineering of devices and components with improved functionality is the key to technological progress, which in turn provides sustainable economic growth. To achieve this goal, scientists and engineers are developing smarter materials and components to make more efficient devices that benefit our daily lives.

Mastering the methodologies that lead to new and better materials requires the use of a wide range of experimental techniques and analytical tools – in particular, diffraction methods which provide a detailed knowledge of the structure of solids at the atomic or molecular level. Neutron and X-ray diffraction are essential and complementary tools: X-rays locate the atomic electrons, while neutrons pinpoint the position of nuclei as well as give information on microscopic magnetism. Owing to their unique properties, neutrons play a crucial role in resolving the arrangements of magnetic moments in complex magnetic systems or locating light atoms in intricate structures, or when assessing the residual stress distribution in bulky mechanical parts. Archetypal examples include hydrogen storage materials, electrolytes and battery materials, and magnetic films. In the field of organic chemistry, the possibility of isotopic substitution can be exploited in elucidating the precise location of hydrogen atoms, for example, in biopolymers.

This brochure presents some of the achievements that have been made in this field using the instruments at the Institut Laue Langevin in Grenoble, and which clearly demonstrate that neutrons are unrivalled in providing answers to an extremely wide range of problems related to materials science. The ILL user community can be proud of the results and achievements up to now. However, scientists are faced with new challenges related to more elaborate systems with higher complexity and smaller dimensions, which demand further progress in instrumentation. The ILL has undertaken a vast renewal programme of its instruments and infrastructure – the Millennium Programme – to meet the future needs of its users. It is our purpose to make sure that engineers and scientists can find the appropriate neutron methods and tools to optimise the technological processes and the materials that will be used by future generations. □

Dr Christian Vettier
Associate Director
Head of Science Division

Contents

Neutron diffraction is a powerful and often unique tool for studying the structure of materials used in everyday life

Introduction – Alan Hewat

Alan Hewat
Head of the
Diffraction Group

N ew materials are everywhere! Imagine all the tiny magnets in the electric motors of a new car, the lighter, longer-lasting batteries that power a mobile phone or computer, the selective catalysts used to create new fuels and clean up the environment. Imagine machines made from new kinds of fibres, with motors made from novel ceramics – both lighter and tougher than the metals they replace. Imagine storing greenhouse gases at the bottom of the ocean, or finding vast reserves of energy in this 'inner space'. But who could imagine, a few years ago, new superconducting materials that could conduct electricity without loss, and be used in more powerful magnetic scanners to map the human body in the finest detail? Who can imagine the future without working on the materials that will make it possible?

The properties of these materials are largely determined by their structure – structure on the atomic scale, or nano-structure (see Box). The distance between atoms is only about 0.1 of a nanometre (nm or one-billionth of a metre) much smaller than the wavelength of light (100 to 200 nm), so clearly we cannot use light. But neutrons, along with electrons and X-rays, can provide a new kind of 'microscope'. Neutrons are subatomic particles that act like waves – like X-rays, and electrons. Since the wavelength of 'thermal' neutrons is similar to the distance between atoms, they have the potential for providing images of structure on an atomic scale.

Neutrons – an ideal probe of materials
Neutrons have many useful properties:
- Neutrons are electrically neutral, unlike electrons, which have a negative charge. They can therefore penetrate deeper inside materials, and are even more penetrating than X-rays. This can be important for 'non-destructive' measurements of engineering components, and materials under extreme conditions such as inside pressure cells, furnaces and refrigerators;

- Neutrons interact with the tiny central nuclei of atoms, while X-rays are scattered by the surrounding clouds of electrons. Neutrons can then

locate atoms more precisely, and scattering is strong even at high scattering angles;

- Most important, neutrons act like tiny magnets, and are a unique tool for determining the magnetic structure of materials;

- Finally, the energy of thermal neutrons is similar to the energy of vibration of atoms in solids, and other types of 'excitations' involving magnetism. So neutrons can be used to study the dynamics of materials, and the forces between atoms.

What kind of experiments can we do?
We generally use a large crystal monochromator to select a particular neutron wavelength, just as the different wavelengths of light can be separated using a prism or fine grating. The material to be studied is placed in this monochromatic neutron beam, and the scattered neutrons are collected on a large 2D detector. The sample can be a liquid, a bunch of fibres, a crystal or a polycrystal. A polycrystal is the usual form of solid matter, such as a lump of metal or ceramic, and is made up of millions of tiny crystals.

To understand how neutron diffraction works, imagine how light is diffracted by a regular grating or grid. Scattering from the different lines of the grid interferes to give diffraction 'spots' with spacing inversely proportional to the spacing of the lines. X-ray and neutron diffraction work the same way, but the grid is now the array of atoms in the material. By measuring the intensities and positions of the scattered X-ray or neutron spots, we can deduce the atomic structure.

Neutron diffraction experiments at ILL are thus really quite simple, and available to a wide variety of users – materials scientists, chemists, physicists and

biologists. The simplest is called 'powder diffraction', when a polycrystalline lump of material, often ground to a fine powder, is placed in the beam. Neutrons are scattered at specific angles, corresponding to the spacing between atomic planes, and by measuring these angles and intensities the atomic structure of the material can be deduced. If instead of a crystalline powder an amorphous or liquid sample is used, there are only broad peaks at specific angles corresponding to average interatomic distances.

To obtain more data, short neutron wavelengths are used, and sometimes one type of atom is replaced by its isotope – chemically identical, but with a different nucleus and different neutron-scattering power – this difference then gives information specific to that atom. So-called inelastic scattering is a little more complicated. Here the change in neutron energy is measured as well, and this gives information about the energies of vibrations and other excitations in the sample.

How can we obtain more neutrons ?

Although neutrons are the ideal probe for studying the structure of materials, the number of neutrons (even at the Institut Laue-Langevin, ILL, in Grenoble – the world's highest flux neutron source) is still only a tiny fraction of the number of electrons in the beam of an electron microscope, or the number of X-ray photons from X-ray sources.

It would be expensive to build a more intense neutron source, but cost-effective to make better use of the source that we already have. This is not as difficult as it might seem. Today, most neutrons are wasted because our detectors are small. New types of detectors, being developed at ILL and elsewhere, can increase the number of neutrons that we actually count by a factor of 10 or more.

The ILL is also developing new neutron optical elements, such as focusing monochromators and super-mirror neutron guides (which act like optic fibres for light), to bring more of the available neutrons to samples. These techniques are particularly important for the study of structure with neutrons, since new materials are often only available in small quantities when first produced. □

Why structure is important

The influence of structure is everywhere; the properties of water and ice, the hardness of metals, the strength of magnets, and even the biology of DNA or the effect of antibodies on viruses – all depend on structure. For example, the structure of gold consists of close-packed atoms, much like a stack of oranges in the local supermarket. The planes of oranges can easily slip over each other, and for largely similar reasons gold is readily malleable. There is another slightly less close-packed structure in which atoms are arranged at the corners of a cube, with another atom at the centre. The planes of atoms in this structure can slip less easily, and metals that adopt it, such as chromium, are less malleable. This is a trivial example, and there are other structural reasons why some metals are harder – to do with 'impurities' and imperfections, which also pin atomic planes and stop them from slipping. The articles in this booklet show many examples of the relation between the structure and properties of new materials.

The structure of gold, revealing its
'stack of oranges' arrangement

Zeolites are increasingly important materials
in industry, particularly as catalysts. Neutrons
are ideal probes of their behaviour

Marvellous zeolites

>> CARSTEN BAEHTZ
and HARTMUT FUESS

Zeolites are used as catalysts in the petroleum industry

Z eolites are remarkable minerals with a crystal structure consisting of a porous aluminosilicate framework which creates a system of linked channels and cavities. Atoms, ions (charged atoms) and molecules can enter the framework, and this property also makes zeolites extremely useful.

In naturally-occurring zeolites, the cavities are filled with water and possibly lightweight metal ions such as those of sodium, potassium or calcium, which can be exchanged for other ions. One of the first applications of zeolites was as water softeners to remove calcium ions from hard water. The minerals' absorptive and ion-exchange abilities are, in fact, the basis for many washing powders.

Zeolites, however, have many other important uses, particularly in the chemical industry. Zeolites with straight channels less than a nanometre across are widely used as 'molecular sieves' to separate molecules of different sizes and shapes such as similar hydrocarbons but with either a straight-chain or branched structure.

Perhaps the most exciting applications are as catalysts, bringing about specific and selected chemical reactions. They are used in the petroleum industry to break down or 'crack' heavy, oily hydrocarbons into lightweight products like gasoline. Zeolites are considered to be much more environmentally friendly than other traditional catalysts. They are also increasingly being used to make various organic chemicals. Ionic exchange can introduce catalytically active metal ions like those of copper or the rare earths, which are then constrained by the geometry of the zeolite framework to carry out only specific reactions giving compounds in high yields.

The new synthetics

Natural zeolites are not adequate for that many applications. A large number of synthetic zeolites have therefore emerged on the market, the most common being zeolites X and Y (with the structure of the mineral faujasite) and ZSM-5. A great deal of effort has also gone into fabricating new materials with different pore systems, like the mesoporous MCM-systems developed by the American company Mobil.

Establishing how and where molecules are absorbed in the zeolite structure is the basis for understanding the behaviour of such systems and their applications. Neutrons are particularly suited to studying both the structure and the dynamics of water or organic molecules in the voids and channels of zeolites. We can incorporate different 'probe' molecules and study them. For example, we used neutron diffraction to determine the location of an organic molecule 7,7,8,8-tetracyanoquinodimethane in the supercage of a zeolite Y, shown opposite. You can see how the molecule is attached to the zeolite framework.

The sensitivity of neutrons for hydrogen and other light atoms allowed us to locate the organic molecule. The results of structure determination is supported by molecular dynamics calculations. The dynamics of these 'guest' molecules can be followed by inelastic incoherent scattering, therefore complementing nuclear magnetic resonance experiments, which are on a different time-scale. Neutron scattering thus provides not only basic knowledge but also supports important industrial processes. ■

The arrangement of 7,7,8,8-tetracyanoquinodimethane in the pores of zeolite NaY

Research team C. Baehtz and H. Fuess *(Darmstadt University of Technology, Germany)*
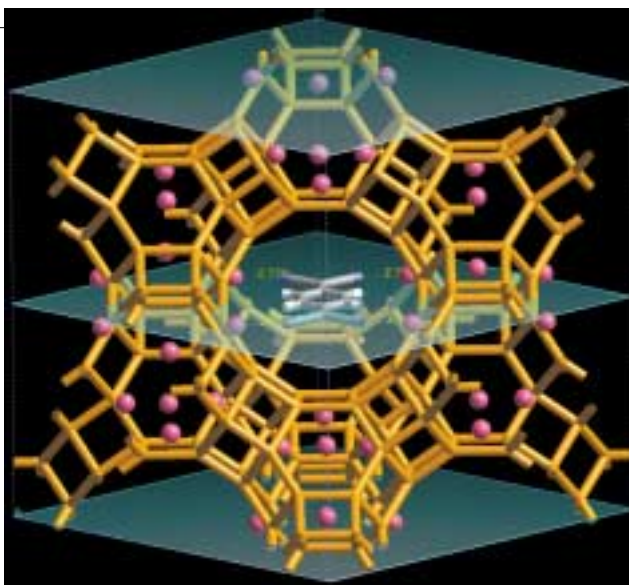
Novel ion-exchange materials are helping to remove toxic waste from the environment

New ions for old

>> JOHN PARISE

T oxic metals in the environment are dangerous for public health, and ways must be found to concentrate and remove them. This is a job for 'ion exchangers', which many people already use in their homes as water purifiers, or 'softeners'. The materials used are minerals or synthetic resins; they trap the unwanted ions – mostly positively charged metal ions – and replace them with harmless ions.

New types of mineral structures must be invented to trap or exchange specific ions. We use neutron and X-ray diffraction to understand the structure of these materials, and to guide the development of new and more efficient ion exchangers. For example, researchers at Sandia National Laboratories in the US have developed novel molecular sieves for removing ions such as the isotope strontium-90 found in radioactive waste.

Designer minerals

Sandia Octahedral Molecular Sieves (SOMS) consist of a hydrated niobium-titanium-sodium oxide arranged in an octahedral framework (see Figure 1). Some of the sodium ions reside in channels where they are relatively mobile. One of these materials, SOMS-3, can give up these harmless sodium ions in exchange for radioactive strontium-90 ions, which are then trapped because they are larger. This material was designed to have channels large enough to release sodium, but small enough to trap strontium-90.

Alumino-silicate minerals, or zeolites, are also used extensively as ion exchangers (see p.6). Their aluminium (Al) silicon (Si) oxide framework structures contain similar channels. Conventionally, the ion-exchange capacity is proportional to the relative amounts of aluminium and silicon, since replacing silicon ions, Si^{4+}, with Al^{3+}, which has one less positive charge, allows other positively charged ions to be accommodated – while still retaining the correct balance of positive and negative charges (from the oxide ions) in the crystal. However, the maximum Al/Si ratio that can be obtained is 1. We have found that the ion-exchange capacity can be further increased by exchanging singly-charged lithium ions (Li^+) for Si^{4+}; the lithium oxide components are more flexible and can more easily accommodate other positively charged ions.

Of course, lithium is a very light element, and is much more easily located with neutrons than with X-rays, so a combination of the two techniques must be used to understand these new ion-exchange materials. We synthesised several new porous litho-silicates, including the complex framework structure called RUB29 shown in Figure 2. Here the Si-Si (blue) and Li-Li (grey) frameworks only are shown. Notice the large channels surrounded by the flexible lithium oxide structure; these channels can accommodate large metal ions such as caesium, removing them from the environment. This type of material is again of potential interest for cleaning up radioactive waste, with the removal of radioactive caesium isotopes.

These two examples illustrate how neutron diffraction can be used to understand such structures, and in particular the mechanism whereby ions enter and leave the pore spaces shown in the figures. This information will help to develop new materials for cleaning up the environment. ■

1 SOMS-1, a new ion exchanger for removing unwanted ions from the environment

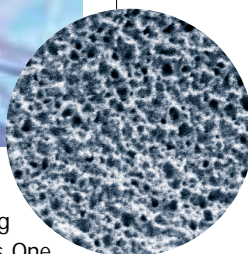2 RUB29, a new lithium-containing zeolite for cleaning up radioactive caesium

1

2
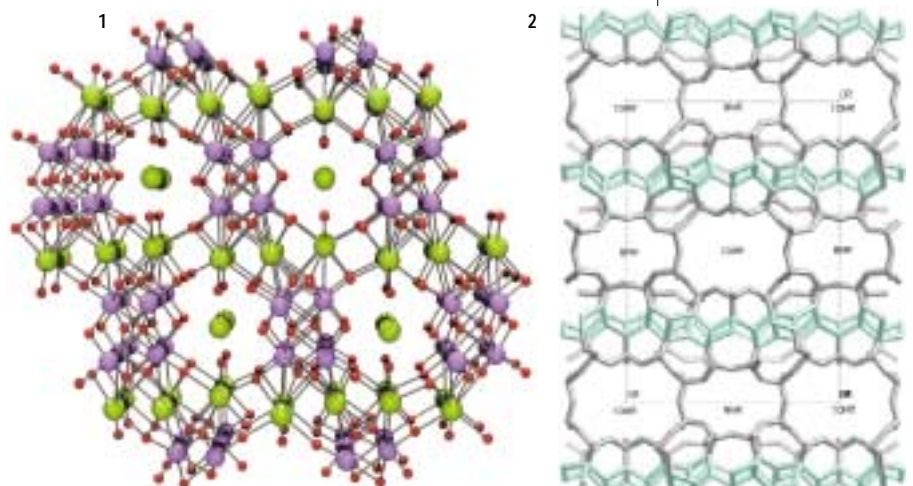
Icy sponges filled with natural gas may be
the next source of the world's energy

Fuel from the ocean floor

WERNER KUHS <<

An electron micrograph of methane hydrate showing its sponge-like structure

The detector bank of the high resolution powder diffractometer D2B

I ndustrialised countries are constantly searching for new sources of fossil fuels. One potential candidate are gas hydrates found on the ocean floor and in arctic regions. These crystalline compounds consist of networks of water molecules in which are caged small gas molecules – methane, for example.

People have known of gas hydrates, also called clathrate hydrates, for almost 200 years, but it was only a few decades ago that they were discovered in Nature – and in great abundance! In fact, marine sediments probably contain 10,000 billion tonnes of methane hydrate – considerably exceeding sources of coal, oil and gas (methane, of course, is natural gas). Methane hydrates are therefore likely to be of major economic importance, once we know how to extract them safely.

Gas hydrates are also interesting for other reasons. They can cause blockages in gas pipelines. Hydrates containing carbon dioxide could be used to trap the gas at the bottom of the ocean thus reducing levels in the atmosphere; in fact, it is likely that water on Mars is

largely stored as carbon dioxide hydrate. Air hydrates found in the deeper parts of polar ice sheets reveal how the composition of air has changed over the past million years.

Not surprisingly, there is intense research interest in gas hydrates. One important aspect is to understand how their stability changes with pressure and temperature. Gas hydrates exist only at high pressure and/or low temperatures – as are found at depths of several hundred metres in the sea or in permafrost regions. Experiments on gas hydrates, therefore, have to be done under similar conditions. This has proved extremely challenging and has meant that a number of properties of gas hydrates have not been well established.

How stable are gas hydrates?

For this reason, our research group at the University of Göttingen decided, a few years ago, to investigate the stability of gas hydrates. We first prepared the hydrates as polycrystalline materials containing large and small 'cages' (below left). Since the constituent atoms are lightweight, neutron powder diffraction (see Introduction, p.4) is the ideal technique to study them.

One of the major unknowns is how the filling up of the large and small cages with gas molecules depends on pressure and temperature. We had assumed that it obeyed a well-established thermodynamic theory but it had never been rigorously proven. Using the D2B instrument at ILL, we were able to test the theory by following the changes in composition of several gas hydrates with increasing pressure.

We found that although the theory predictions were broadly followed, there were deviations in all cases, and in some cases the theory failed completely. We were very surprised to find that the large cages in the nitrogen hydrate contained two nitrogen molecules, violating one of the basic assumptions of the theory, although it can be modified to allow for this double occupancy. In other cases such as the carbon dioxide hydrate, the behaviour was quite different, and the theoretical description has to be changed considerably. These findings are extremely important to chemical engineers developing techniques to handle gas hydrates. We also looked at the compressibility of gas hydrates, which is important in detecting them using seismic profiling of the ocean floor.

Neutron techniques will continue to probe these and other questions such as unravelling how their fascinating structures form and decompose. ■
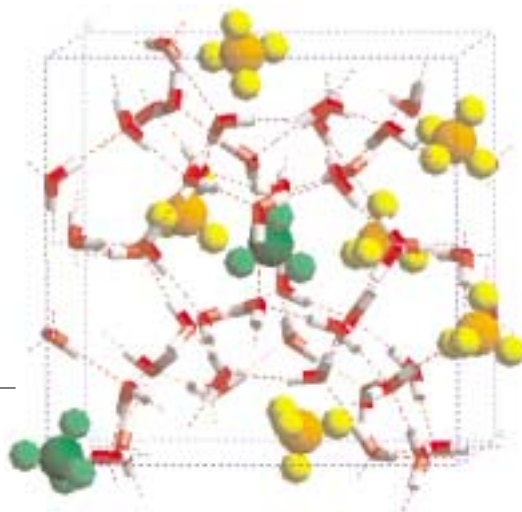
The structure of methane hydrate. Methane molecules occupying small and large cages are shown in green and yellow respectively

Research team B. Chazallon, A. Klapproth, D. Staykova, W. F. Kuhs *(University of Göttingen, Germany)*

Metal hydrides hold the key to
green energy

Hydrogen could be an ideal ecological fuel once researchers have found the right material in which to store it

>> KLAUS YVON

F or several decades, people have been exploring alternative fuels to petroleum that could be used to provide power. Hydrogen is one possibility. It is environmentally friendly, burning in an internal combustion engine to produce only water. It is also possible to extract hydrogen from water (H_2O) with electricity made, say, with solar energy.

Hydrogen has one big snag, however. As a gas, it takes up a large volume and is very explosive, so is difficult to store and handle. Fortunately, there are a range of metal alloys (based on copper, manganese and titanium, for example) that can soak up huge amounts of hydrogen gas, which is then released on gentle heating. They provide a safe, effective way of storing and transporting hydrogen.

The hydrogen-storage materials tested so far are expensive and rather heavy, so our research team in Geneva is investigating alternatives which are lighter and can absorb even more hydrogen. These are the so-called complex metal hydrides which may contain up to four times as much hydrogen as the conventional hydrogen-absorbing alloys. In fact, two of our compounds, the barium-rhenium and magnesium-iron complexes shown below, hold the world record for hydrogen absorption. However, the complex hydrides studied so far have disadvantages too. They do not release their hydrogen easily but require heating to 300°C, which is commercially uneconomic; the barium-rhenium compound does release its hydrogen at just above room temperature but is too heavy and expensive.

We haven't yet found the ideal storage material, but neutron experiments play an important part in our search by helping us to characterise the compounds we make and predict more effective ones.

High resolution at ILL

Neutron diffraction is ideal for investigating the crystal structures of metal compounds containing light elements like hydrogen which scatter neutrons strongly but are almost invisible to X-rays. Nevertheless, our polycrystalline complex hydrides, which are analysed using ILL's powder diffraction instruments (see p.4), present a formidable challenge. The smallest repeating unit of each crystal (unit cell) contains many atoms, resulting in very crowded, overlapping diffraction patterns. The ILL neutron source is the only facility in the world that provides an intense-enough neutron beam to resolve these patterns and pinpoint the location of the atoms.

Cutting grass with Professor Yvon's own hydrogen-powered lawnmower is child's play

Klaus Yvon

On the practical side, several car companies have been experimenting with hydrogen-fuelled vehicles employing metal hydrides. In Switzerland, we have been demonstrating the technology at home! I have been using a hydrogen-powered lawnmower for 10 years, and an architect, Markus Friedli, has equipped his house in central Switzerland with solar panels to generate the electricity for recovering hydrogen from water. This is then used to power the family van.

So far, none of the applications of hydrogen gas storage materials has been commercialised. This is partly because we are still searching for that 'miracle material'. However, we have barely started to look at all possible combinations of elements, and neutron experiments will continue to make a significant contribution. ■

The neutron diffraction patterns and structures for (A) magnesium iron hydride (Mg_2FeH_6) and (B) barium rhenium hydride ($BaReH_9$). Note that the magnesium-iron compound contains twice the density of hydrogen atoms (150 grams per litre) compared with that in liquid hydrogen (70 grams per litre). The barium-rhenium compound contains 4.5 hydrogen atoms per metal atom, which is greater than the ratio of hydrogen to carbon in natural gas, methane (CH_4)

Mg_2FeH_6

$BaReH_9$

$[FeH_6]^{4-}$

A

$[ReH_9]^{2-}$

B
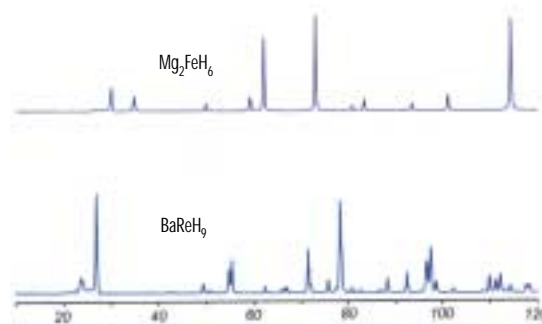
Neutron studies of materials in real operating batteries are helping to improve their performance

Towards a
better battery

>> YVES CHABRE,
MICHEL LATROCHE and
GWENAELLE ROUSSE

Our electrochemical cell assembly developed for studying rechargeable batteries

Inner nickel electrode

Silica fibre separators

Metal hydride electrode

Silica vessel with deuterated potassium hydroxide (KOD) electrolyte

3cm

0cm

Capillary for a reference electrode

Outer nickel electrode

M aterials for storing electrical energy have been the subject of intense research in recent years, as a result of the rapid development of portable electronic equipment such as mobile phones and laptop computers. These devices depend for their operation on rechargeable batteries, which work by converting chemical energy stored in electrodes, into electrical energy, via electrochemical reactions. These reactions correspond to the loss or gain of electrons and ions in the electrode materials. The electrical current is carried by electrons outside the battery and by ions (charged atoms) in the electrolyte between the electrodes inside the battery. The electrochemical reactions are reversible, so when all the energy has been delivered the battery can be charged up again from an external electrical source.

There are two types of rechargeable batteries currently used in portable devices: nickel-metal hydride (Ni-MH) batteries, which involve hydrogen ions, and are replacing the kind based on nickel and toxic cadmium; and lithium-ion batteries.

These batteries still have shortcomings, however, and are continually being improved in terms of performance – power capacity and output, and lifetime (the number of charge-discharge cycles the battery goes through without degrading). One of the keys to making improvements is a detailed understanding of the crystal structure of the electrodes and how the ions diffuse in it. Here, neutron powder diffraction (see p.4) is a powerful tool and gives more information than X-ray diffraction. Neutrons can easily penetrate the sample so as to give accurate information about the bulk material, and the hydrogen and lithium ions trapped in the electrodes scatter neutrons well so they can be readily located.
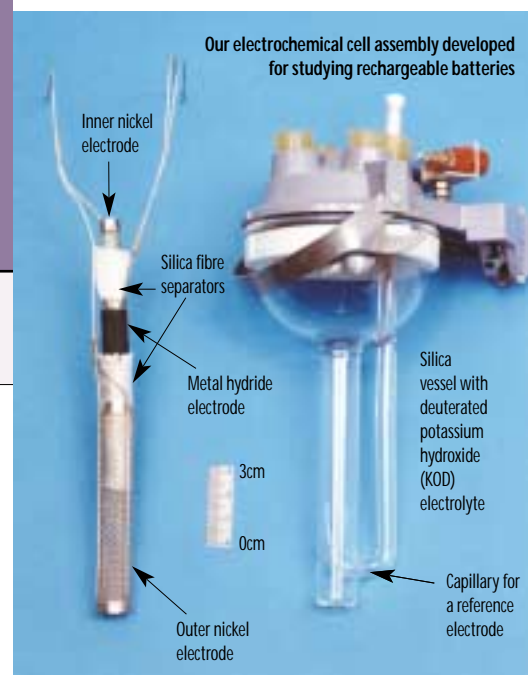
Studies in real time

We have been carrying out extensive studies on nickel-lanthanum hydrides used as electrode materials in Ni-MH batteries. The continual charge-discharge of the standard electrode involves a cyclic transformation between two crystalline phases called α and β. These have unit (cell) volumes that differ by 20 per cent, which induces heavy constraints in the material and causes its crystal structure to fragment. That leads to surface corrosion and reduces the battery lifetime.

We carried out experiments using the D1B diffractometer at ILL, which followed the structural changes during cycling on different electrode materials. These experiments showed that a transitory intermediate γ phase with a cell volume between that of the α and β phases may appear (see left) for some peculiar alloy compositions. The appearance of this intermediate γ phase at the boundary between the α and β phases significantly reduces the constraints during the cycling process, leading to a better lifetime.
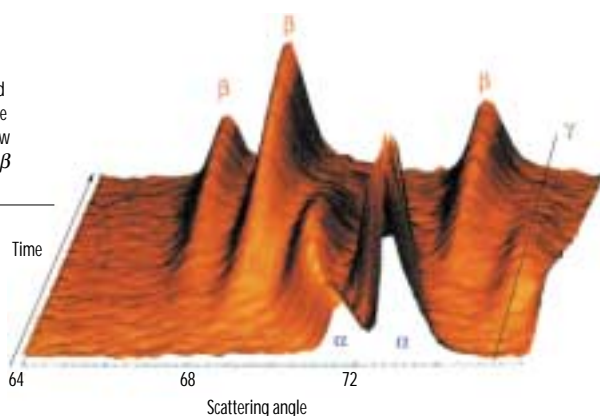
Another problem is related to the high charge/discharge rates required by many applications of these batteries. Using a newly designed electrochemical cell (above), with an electrode arrangement similar to that in real batteries – in conjunction with the high neutron flux available on the D20 diffractometer – we could also follow changes in the electrode at high cycle rates. We showed that the cycle rate was limited by the speed of associated transitions in the electrode's crystal form, rather than the rate of diffusion of hydrogen ions in the electrode material.

The next challenge will be similar studies of lithium-ion battery electrode materials. ■

A 3D plot of the neutron diffraction patterns recorded for a metal hydride electrode as it discharges. It shows how different crystal phases (α, β and γ) form over the cycle

Time

Scattering angle

Research team Y. Chabre *(CNRS & UJF-Grenoble)*, M. Latroche *(CNRS)*, M.R. Palacin *(ICMAB–CSIC, Spain)*, O. Isnard *(CNRS & UJF-Grenoble)*, G. Rousse *(ILL)*

The benefits of stress

A unique neutron technique for mapping residual stresses in engineering components can help extend their life

THILO PIRLING <<

Thilo Pirling aligning the neutron strain imaging instrument with a cog as a sample

E very mechanical component is stressed to some extent, even when it is not experiencing a load. This is due to various stages in its manufacture like rolling or machining, heat treatment or welding. These residual stresses remain inside the material, influencing its characteristics, and thus the performance and lifetime of the component.

For ever-more reliable hi-tech products – especially in applications where safety is a priority, it is important to know the distribution and the size of stresses in each component. Tensile stresses can cause cracks to develop, while compressive stresses can prevent them starting. In fact, hardening surfaces by various surface treatments such as 'peening' (shot is fired at a surface so that it is plastically deformed) introduces beneficial compressive stresses.

Neutron strain imaging

Neutrons provide a unique tool for determining residual stresses deep inside matter. The principle is quite simple: compressive stresses reduce the spacing between atoms in the material while tensile stresses stretch them. Since neutron diffraction can measure atomic distances, it can probe the stresses in an object. This 'neutron strain imaging' technique does not destroy the material and can map stresses with a spatial resolution of a cubic millimetre, or even smaller. The high penetration power of neutrons allows measurements to be carried out to several centimetres deep in steel.

ILL's neutron strain imaging instrument is used in many applications for examining alloys and composites, and various engineering components. The stresses in joints introduced by different welding techniques can also be characterised. We have studied the tungsten-copper brazed components for a fusion reactor, as well as titanium tanks and new composite materials for satellites.

One everyday application, which illustrates how the neutron strain imaging technique helps to develop better products, is in strengthening the crankshafts of car engines. A crankshaft can oscillate when rotating, thus shortening its lifetime. One way of overcoming this problem is to use a process called deep rolling, in which three whee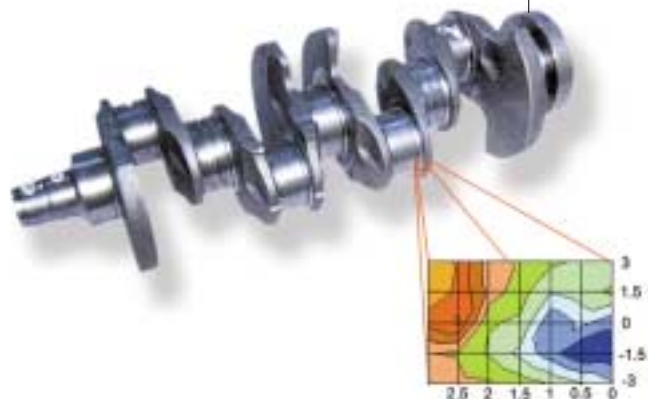ls are turned around the workpiece while applying force in the radial direction. This introduces compressive residual stresses into the material which strengthen the piece and may reduce the amplitude of oscillation by 200 times, provided that the force and number of turns in the process are optimised. Engineers perform calculations to find the optimum parameters, but only experiment can verify their exactness and the efficiency of the deep rolling procedure. The neutron strain imaging technique provides the determination of the stress distribution, which can be directly compared with calculations.

Because the potential of the technique is so high, ILL is constructing a new strain imager with improved characteristics and an extended range of applications. It will enable measurements to be carried out on specimens from 1 centimetre up to 2 metres in length and weighing more than 500 kilograms. ■

The picture below shows the result of a neutron strain-imaging measurement ± 1.5 millimetres around the deep rolled region and down to a depth of 4 millimetres into the material. Compressive stresses are marked in blue and it shows that the deep rolling process modifies the stress field of the workpiece down to a depth of about 2 millimetres

Research team T. Pirling *(ILL)*, Giovanni Bruno *(ILL and University of Manchester, UK)*

Neutrons explore how to make high-performance
materials that behave both like ceramics and metals

New ceramics for
jet engines

ERICH KISI <<

Rolls-Royce

Ti atoms
Si atoms
C atoms

The crystal structure
of titanium silicon
carbide (Ti$_3$SiC$_2$)

T itanium silicon carbide (Ti$_3$SiC$_2$) is a layered
material. Its crystal structure, shown left, has
double layers of titanium carbide interleaved with
single layers of silicon. This laminated structure leads to
an unusual combination of properties that is both
ceramic and metallic. It is stable at high temperatures,
as in the case of ceramics, but also conducts heat and
electricity like a metal. Furthermore, because the layers
can slide over each other, the material is not brittle like
other ceramics and is thus readily machined with
ordinary machine tools. Other useful mechanical
properties include excellent resistance to sudden
changes in temperature, good strength at high
temperatures and resistance to oxidation. It is also
relatively resistant to fracturing. All these attributes
make titanium silicon carbide an excellent candidate
material for high-temperature applications such as
jet-engine turbine blades.

One drawback, however, is that the material is
difficult to make in a pure form. It tends to contain
unacceptably large amounts of other compounds –
titanium carbide (TiC) and titanium silicides (Ti$_x$Si$_y$).
This makes it harder, less machinable and difficult
to determine the properties of the pure material.

Ceramics like titanium silicon carbide are
traditionally made by heating the components, either
as elements or compounds, in the correct ratios.
However, a large amount of heat is generated in the
synthesis of titanium silicon carbide, and this
heat of reaction can actually be exploited to
make the material. This is called self-
propagating high-temperature synthesis
(SHS). Once ignited by an ignition source
(laser, electron beam, furnace or electric
arc), the reaction becomes self-sustaining and
converts the reactants to the product very
rapidly – in less than 100 seconds.
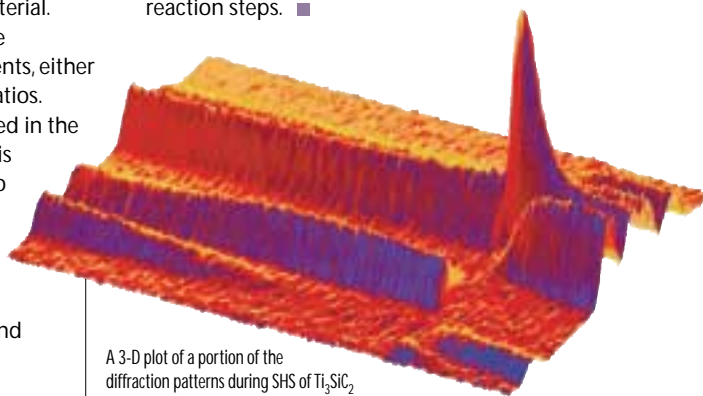
Reactions caught in the act

We have been studying SHS reactions in the Ti-Si-C
system so as to understand better how to make the
pure layered material. Until recently, the extreme speed
of the reactions had prevented us from obtaining a
clear understanding of the reaction mechanism.
However, using the powder-diffraction instrument –
D20 – at ILL, we have been able to follow the detailed
structural changes as the reaction proceeded.

We first heated cold-pressed pellets of the reactants
(titanium, carbon and silicon carbide) in a furnace at
a rate of 100°C per minute through the critical range
of 800 to 1000°C – to initiate the reaction – and
simultaneously recorded diffraction patterns every
0.9 seconds, as shown below.

The SHS reaction proceeded in five stages. Up to
880°C, the reactants simply heat up. Then, there is a
change in the crystal structure of the titanium, which
goes on to react with the free carbon in the sample. It
is this reaction that gives off the heat needed to start
ignition. The fourth stage is the formation of a single
solid intermediate phase (in less than 0.9 seconds) by
the true SHS reaction. It is stable for only about 6
seconds. The reaction temperature was estimated at
2200°C. We believe that the intermediate phase is a
solid solution of silicon in titanium carbide which is
relatively stable at the combustion temperature but
becomes unstable as the temperature drops. The final
stage is the rapid development and growth of the
product phase, titanium silicon carbide. We are now
working on measuring the rates of the different
reaction steps. ■

A 3-D plot of a portion of the
diffraction patterns during SHS of Ti$_3$SiC$_2$
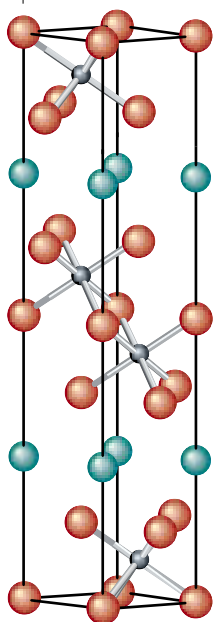as the reaction progresses (left to right)

Research team D. P. Riley and E. H. Kisi *(University of Newcastle, Australia)*, T. C. Hansen and A. W. Hewat *(ILL)*

Magnetic shape memory alloys

Metals that change their shape in a magnetic field have tremendous potential as actuators, sensors and other devices

>> JANE BROWN

T he shape memory effect is the ability of some alloys to remember, and return to, the form which they had at one temperature after being plastically deformed into another shape at a lower temperature. This property can be exploited in many ways, for example, in actuators controlled by heat. Devices based on shape memory alloys are being developed in fields as far apart as astronautics and medicine.

In order for an alloy to have the shape-memory property, it must undergo what is called a martensitic transition. This derives its name from a change in crystal structure when steel is cooled rapidly to form so-called martensite (after its discoverer Adolf Martens), which has a variety of characteristic microstructures. The transition involves small displacements, or slips, between planes of atoms in the crystal at a certain temperature.

The shape memory alloy is first formed at a temperature above that of the transition and then deformed below it. The memory arises because residual stresses in the structure introduced during the forming process, influence which slips then occur in the martensitic transformation, and thus which variants of the martensitic phase are present at the lower temperature.

Shape memory alloys scatter neutrons very effectively, so neutrons are an almost ideal probe with which to view the martensitic transformation at a microstructural level. Neutron diffraction can follow the evolution of different martensite variants as the temperature is changed.

Magnetic possibilities

Combining the shape memory property with ferromagnetism, vastly increases the range of applications. Magnetic fields can also influence the martensitic transition, and the possibility of controlling shape memory properties using a magnetic field is currently receiving much attention. An alloy made of nickel, manganese and gallium (Ni_2MnGa) is one of the rare ferromagnetic shape memory alloys. It undergoes a martensitic transition from a cubic structure to a tetragonal variant at a temperature around 200K.

At ILL, we carried out neutron diffraction experiments on single crystals of this alloy using the 4-circle diffractometer (D9). On this instrument a small position-sensitive detector records the scattered neutrons. The figure below shows how the diffraction pattern evolves before, during and after the martensitic transition. At 235K, above the transition, the scattering shows a single compact peak associated with the cubic phase. At 206K, the martensitic transformation is under way, and the original single peak has broken up into seven smaller peaks, each corresponding to a different martensite variant. On further cooling, two of the variants grow at the expense of the others so that at 200K there are just two peaks in the pattern. On reheating the process is reversed and the original cubic single crystal restored.

Experiments of this kind, carried out on crystals which have been subjected to different mechanical treatment, allow the transformation processes that give rise to the shape memory effect to be studied in great detail. ∎

Positioning a sample on the 4-circle diffractometer D9
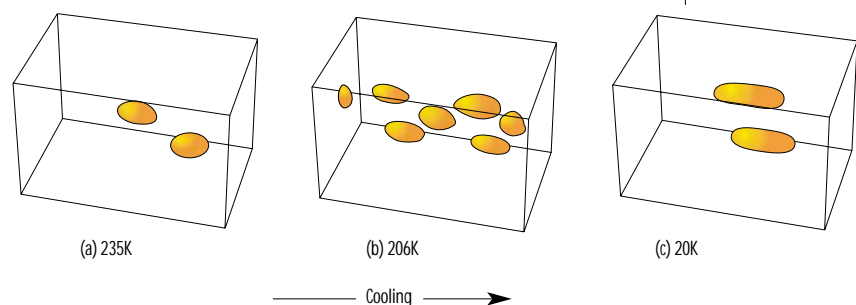
A 3D representation of the neutron scattering peaks as the crystal of the ferromagnetic shape memory alloy is cooled through the martensitic transition

(a) 235K   (b) 206K   (c) 20K

Cooling ⟶

Inside high temperature superconductors

Neutrons have helped uncover exciting new materials that conduct electricity without resistance

ALAN HEWAT <<

An artist's impression of a German Transrapid maglev train being built in Shanghai using superconducting technology

O xides are normally insulators because their electrons are intimately associated with the atoms. Metals can conduct because the electrons are free to move. But there are some oxides that become electrical superconductors, having zero electrical resistance above the temperature of liquid air – cold but easy to produce and handle. This is truly amazing, and a few years ago would have been thought impossible.

Neutrons were important for understanding why one of the most famous of these materials, YBCO ($YBa_2Cu_3O_7$) could be either insulating or superconducting, depending on how it was produced. The difference was found to be the amount of oxygen, and oxygen is much more easily seen with neutrons than with X-rays. Neutron diffraction at ILL and elsewhere found that the superconducting layers were 'doped' by so-called 'charge reservoir' layers – the square copper oxide chains in the figure opposite.

This neutron picture of the structure immediately suggested a search for other materials containing different charge reservoirs – a very successful search since much higher superconducting temperatures were soon found in materials containing bismuth or mercury oxide charge reservoirs. There are many uses for materials as strange as superconductors, apart from simply making very efficient power cables and faster, cooler computer chips.

3D brains and levitating trains

One of the most important applications for these new superconductors is in making more efficient electromagnets, such as those used in hospitals for magnetic resonance scanners. Instead of using X-rays, a patient is placed in a strong magnetic field and signals from atoms in the body tissue are recorded to reconstruct a 3D computer image of vital organs. These images can reveal blood clots in the brain, tumours and other conditions that can be precisely located and treated.

Magnets and superconductors might also be used to make more efficient maglev trains, floating above electrical coils in the 'rails'. This is possible because superconductors exclude magnetic fields from their interior – the Meissner effect. When a magnetic field is applied to a superconductor, spontaneous electric currents flow in it resulting in an induced magnetic field that exactly opposes the applied field, resulting in a repulsive force. The train can then be supported, guided and even propelled using a kind of linear electric motor in the rails. ■

The layered structure of yttrium barium copper oxide showing the square copper-oxide chains
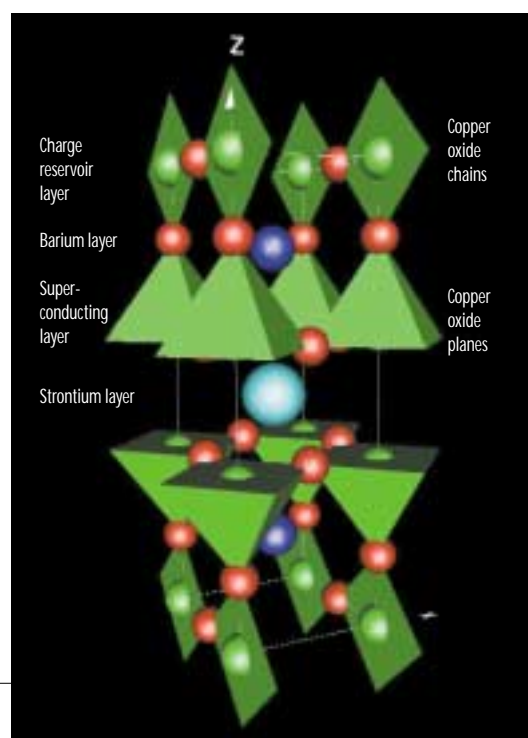
Understanding colossal magnetoresistance

Neutron studies are helping researchers explore a recently-discovered electronic effect that will lead to new devices

>> TAPAN CHATTERJI

T he complex manganites are derived from a structure consisting of a rare-earth element such as lanthanum combined with manganese and oxygen ($LaMnO_3$). These electrically conducting compounds have caused great excitement in the past decade because they show a huge change in resistivity when a magnetic field is applied. This colossal magnetoresistance (CMR) effect could be the key to the next generation of magnetic memory devices, magnetic-field sensors, or transistors.

Like their chemical cousins, the high-temperature superconductors (see p.14), they possess complicated electronic structures. Yet unravelling their behaviour – over a range of temperatures or in magnetic fields – is essential to understanding CMR and finding suitable CMR materials for commercial application.

Hidden electron orbitals

Our contribution to this active research area has been to investigate, in these materials, an attribute of electronic structure that has hitherto been difficult to probe experimentally – the electron orbitals. Electrons involved in chemical bonding have three defining properties laid down by quantum mechanics, their charge, spin and orbital. The orbital refers to the shape of the volume that the electron is likely to occupy (the electron cloud). In transition metals such as manganese, it is the outer 'd' orbitals that are involved in bonding and give heavy metal compounds their electronic and structural characteristics.

In the CMR manganites, the manganese atom is triply charged (Mn^{3+}) and is bound to six neighbouring oxygen atoms in an octahedral shape as shown in Figure 1. Mn^{3+} has one d electron involved in bonding. This 'valence' electron occupies one of two possible d orbitals, as shown in Figure 2. These orbitals are thought to be significant in CMR. Each can mix, or 'hybridise' with the oxygen orbitals to form bonding orbitals; each is geometrically different and is associated with differing bond lengths.

The lanthanum ions, which are also triply charged (La^{3+}), sit between the layers of the manganese-oxygen octahedra. If a percentage of the lanthanum ions is replaced with metal ions such as strontium (Sr^{2+}) which are doubly charged, then there is a redistribution of electrons resulting in the formation of some Mn^{4+} ions,

now with an empty d orbital (a hole). This is called 'hole-doping' and allows the remaining valence electrons to hop from manganese to manganese atom so that the material conducts like a metal. Furthermore, when the 30 or 40 per-cent hole-doped manganite is cooled below a certain temperature, the spins of these electrons align so that the material becomes ferromagnetic. It is this that increases the resistivity of the material because the aligned electrons scatter oppositely-aligned electrons trying to pass through.

Using a combination of X-ray and neutron diffraction, we investigated the interplay between the ordering of the spins, the charge and the orbital occupancy in the 50 per-cent hole-doped manganite ($LaSr_2MnO_7$). Neutron studies enabled us to measure the manganese-oxygen bond lengths, which indicated which d orbitals were occupied. We found that this compound has equal numbers of Mn^{3+} and Mn^{4+} ions arranged alternately, and a 'staggered' arrangement of bonding orbitals (see Figure 3) below 225K. At 170K this charge-orbital order 'melts' as antiferromagnetic order (spins alternately aligned) sets in. ∎

1 | The structure of lanthanum strontium manganite

- A-cation
- BO_6

A

$x^2 - y^2$

$3z^2 - r^2$
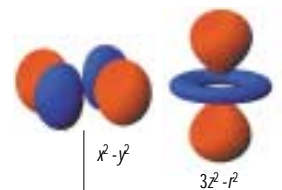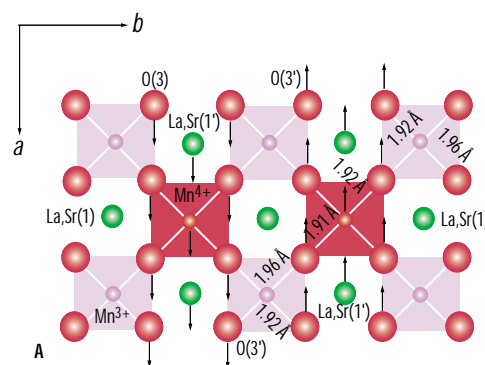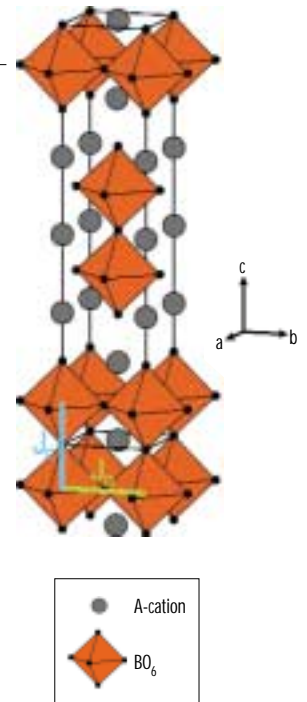
2 | The d electron orbitals of manganese

B

3 | The structure of 50 per-cent hole-doped lanthanum strontium manganite ($LaSr_2Mn_2O_7$), at a temperature of 165K, looking down through the manganese-oxygen octahedra. Neutron diffraction allowed us to determine the position of both manganese and oxygen atoms and thus the Mn–O bond distances. This revealed the ordering of the Mn^{3+} and Mn^{4+} ions (A) and the ordering of the hybridised orbitals (B)

Research team T. Chatterji *(ILL)*, G.J. McIntyre *(ILL)* and A. Stunault *(ILL)*

Ultrathin layers of magnetic materials have huge potential for the electronics industry but show complex behaviour uniquely studied by neutrons

Magnetic multilayers

>> JON GOFF

Jon Goff working at ILL
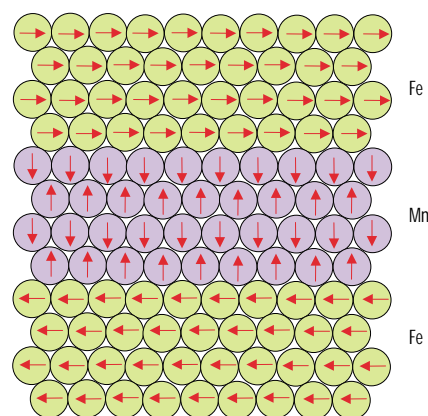
The magnetic structure of the iron manganese $[Fe_9/Mn_4]_{300}$ multilayer

O ver the past 20 years, physicists have developed techniques that allow them to deposit, in a controlled way, sequential layers of atoms in regular crystalline planes on a surface. One such method is molecular beam epitaxy (MBE) in which beams of atoms are fired onto a surface. As a consequence it is now possible to create, for example, multilayers of metals with exotic magnetic properties. Magnetic multilayers have a panoply of practical applications. They can be used to produce devices that exploit the spin rather than the charge of the electron. This new field of magnetoelectronics looks set to revolutionise the electronics industry.

One type of device made of magnetic layers, called a spin valve, is already employed as a read head for computer hard disks, and this has led to dramatic improvements in data-storage densities. Arrays of spin valves have also been used to create non-volatile random access memory (meaning the information is not lost when the computer is switched off).

Exploring magnetic ordering

Studies of magnetic multilayers are also opening a new window on our understanding of magnetism. By alternating layers of metals with different magnetic properties, for example, we can explore how magnetic ordering (the precise arrangement of the electron spins in the layers) propagates across the layers. The coupling mechanism between the layers is often intricate, and neutron scattering techniques are ideal for investigating them. Instrument D10 at ILL has been used to study a number of magnetic systems which aim to throw light on important problems in condensed matter research.

A magnetic read head based on the spin-valve concept

Recently, we have been investigating multilayers made from transition metals, iron (Fe) and manganese (Mn), which will allow us to understand the basic physics underpinning the next generation of magnetoelectronic devices. Of particular interest is what happens at the interfaces between the layers.

Manganese at room temperature has an unusually complex crystal structure and we don't know how the spins are ordered in this form. However, at elevated temperatures, manganese forms much simpler cubic crystals. Fortunately, MBE provides a unique way of depositing this cubic form at low temperatures using an iron layer as a template. In this way a multilayer could be built up of composition $[Fe_9/Mn_4]_{300}$, where the subscripts labelling the elements refer to the number of atomic planes in the average bilayer and the last subscript to the number of bilayers.

Iron is, of course, ferromagnetic, with all spins aligned. We found, however, that the blocks of nine Fe layers coupled antiferromagnetically (spins oppositely aligned) across the manganese blocks at room temperature. The Mn itself orders with a simple antiferromagnetic structure below a temperature of 187K with spins pointing along the growth direction, perpendicular to those of the Fe (see figure above). Furthermore, the neutron scattering results show that the Mn blocks also couple across the iron blocks.

Theory predicts that the cubic manganese would form either an antiferromagnetic structure with spins in the same direction as the iron, or a helical structure. The fact that the moments in the Mn are perpendicular to those of the Fe may be due to conflicting magnetic interactions (frustration) at the interfaces. ■

Seagate

Research team J. P. Goff and S. Lee *(Liverpool, UK)*, R. C. C. Ward and M. R. Wells *(Oxford, UK)*, G. J. McIntyre *(ILL)*

Molecular magnets

Can we create permanent magnets which are light, transparent and bio-compatible?

>> EDDY LELIÈVRE-BERNA

The polarised hot neutron diffractometer D3

C onventional permanent (ferro)magnets, made of transition metals and rare-earths like iron and neodymium, are everywhere: in cars, kitchen gadgets, telephones, computers, and so on. Indeed, they generate the second largest cash flow in the global economy. Now, the market for magnetic devices is set to widen further, thanks to the creation of magnets made from molecular compounds.

Molecular magnets have many useful additional properties: lightness, transparency, solubility, magneto-optical properties (using light to change the magnetic state), and biocompatibility. They also offer the opportunity of being able to 'tune' the transition temperature at which the material becomes ferromagnetic – by tweaking the chemical structure. These molecular compounds will not only extend the use of permanent magnets but also open the way to a novel class of information storage systems.

The first ferromagnetic molecular compound – decamethylferrocenium tetracyanoethylenide – was discovered in 1986; its transition temperature was only 4.8K. Since then, huge progress has been made and room-temperature molecular magnets are now quite common. An example is a magnet consisting of a complex metal compound – a hexacyanometallate $L^{p+}[M(CN)_6]^{q-}$, where L and M are transition-metal ions. The electronic structure of this compound is well understood, so it is possible to tune its magnetic properties. Another example is the low-temperature magnet based on clusters of manganese atoms, Mn_{12}acetate, which can exist in a variety of subtle magnetic states that can be controlled by an applied magnetic field.

In the conventional compounds based on metals, the magnetic moments (which arise from the spins of unpaired electrons) are well localised on the metal atoms, with only a small part of the 'spin density', or magnetisation, transferred onto the groups of atoms attached to the metal. The distribution of the magnetisation is thus just the sum of contributions of individual magnetic atoms within the molecule.

In the case of a molecular compound, however, the situation is completely different. The unpaired electron responsible for its magnetism sits in a molecular orbital built up from the orbita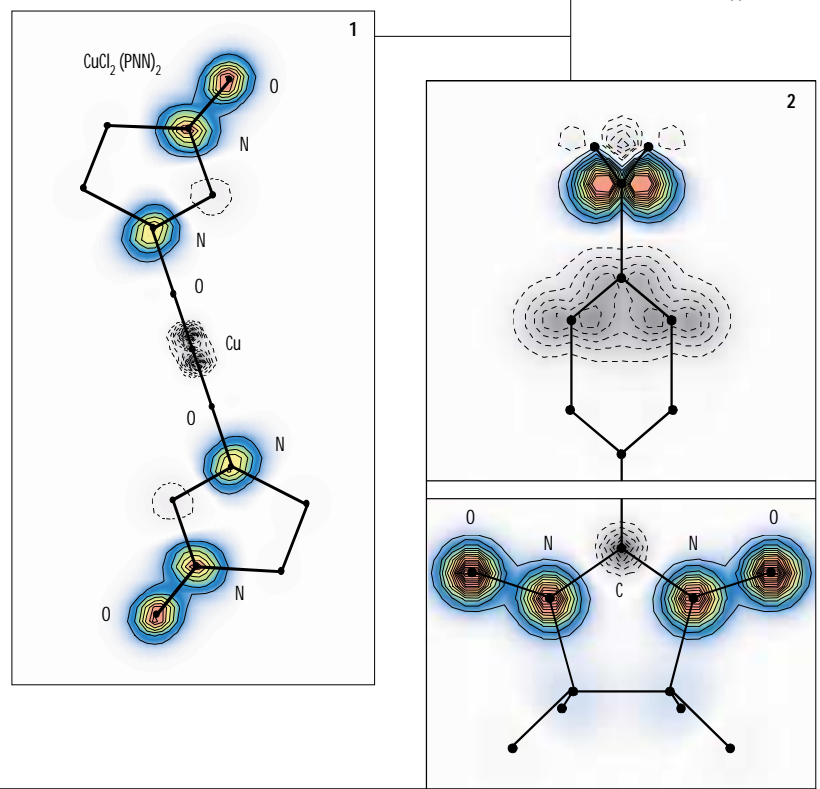ls of the atoms constituting the molecule. This means that the magnetisation tends to be smeared out across the molecule, though perhaps concentrated on certain atoms.

A uniquely sensitive probe

Polarised neutron diffraction is the only technique sensitive enough to investigate the magnetisation. Neutrons themselves have a magnetic moment, and polarised beams of neutrons (with spins aligned in the same direction) are a unique probe of magnetic behaviour. Measuring the magnetisation distribution across a molecule reveals precious information on the nature of the molecular orbitals responsible for the magnetism, the interactions with neighbouring molecules in the solid, as well the chemical bonding and how the electron spin is spread out and oriented. This allows us to test the underlying theories of molecular bonding and magnetism, and create new magnetic materials with predicted properties. ■

Figure 1 shows the spin density in the first genuine organic ferromagnet synthesised, the β phase of *para*-nitrophenyl nitronyl nitroxide (transition temperature 0.6K). Most of the spin density lies on the O-N-C-N-O group of atoms of the nitronyl nitroxide fragment; the contours reveal that the unpaired electron responsible for magnetism is mostly situated on the oxygen and nitrogen, but when on the central carbon atom it reverses its spin direction, giving a negative spin density

Figure 2 shows what happens to the spin density in a similar molecule, $CuCl_2(NitPh)_2$, (the phenyl groups are not shown) when a transition metal like copper is present. The spin density is positive on the nitroxides (NO) and negative on the copper. The lack of magnetisation on the oxygen nearest the copper shows that the spin distribution in the nitronyl nitroxide has been completely upset by the magnetic interaction with the copper

$CuCl_2 (PNN)_2$

Research team E. Lelièvre-Berna *(ILL)*, E. Ressouche and J. Schweizer *(CEA, Grenoble)*

Materials with a bright future

New organic compounds for optoelectronics are being engineered with the aid of neutron studies

Second harmonic generation in a crystal

JACQUELINE COLE <<

L asers can be found everywhere from light-shows to supermarket check-outs and CD players. They are also a key part of an optoelectronic revolution which is transforming communications. An important effect called second harmonic generation (SHG) plays a major role in how lasers are used in these technologies. SHG doubles the frequency of light and is used to change the colour of a laser beam.

The SHG effect results from certain subtle electronic changes in a crystalline material when a laser beam shines through it. The compounds used to generate the SHG effect have traditionally been *inorganic* – typically, potassium dihydrogen phosphate and lithium niobate – and rely on small perturbations of key ions in the crystal array. We are interested in investigating novel *organic* compounds which generate the same effect by transferring electronic charge across the constituent molecules. They generally have a flat structure with an asymmetric distribution of charge resulting in an electric dipole moment. Their optical response is much faster than that of the inorganic materials so we are hoping to identify much more efficient SHG materials.

Although both neutron and X-ray diffraction are core techniques in exploring the charge transfer process, neutrons are particularly effective at pinpointing hydrogen atoms, which are mostly positioned at the extremities of the molecules and may play a key role in the charge transfer process.

Mapping electron density

At the ILL, we have used a combination of X-ray and neutron diffraction data to map the electron density in several well-known organic SHG-active compounds such as methylbenzylamino-(di)-nitropyridine (MBADNP), below, in order to identify the critical regions of charge-transfer across the molecule. Moreover, these studies have also enabled us to evaluate the dipole moment. Experimental determination of not only the magnitude but also the sense of the dipole moment in the solid state is very difficult by any other means.

For second harmonic generation to work, the charge transfer must be propagated right through the crystal. This is promoted by a type of intermolecular interaction called hydrogen bonding. However, the ILL work has shown that some types of hydrogen bonding appear to be more favourable than others in enhancing SHG properties. The most promising structures often show a delicate and complicated compromise between a number of factors.

Our increasing understanding of the charge-transfer processes in organic compounds has led us to try to engineer better SHG-active compounds, based upon known, favourable structural attributes in a series of compounds. The ultimate goal is to design a material for a given optical application from the huge variety of possible organic compounds that can be ma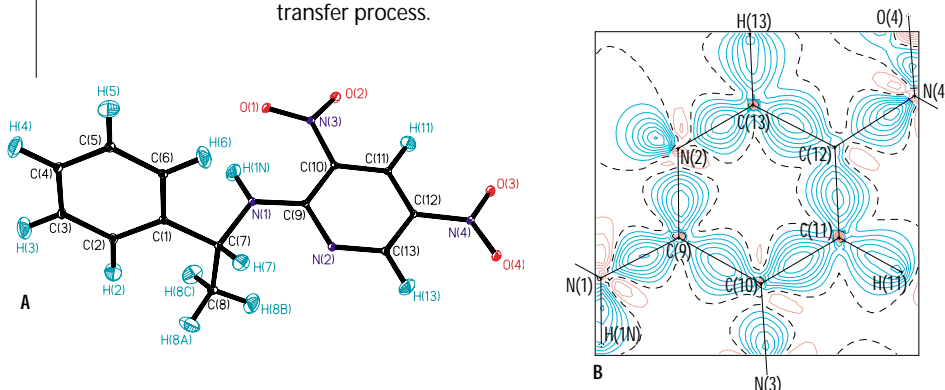de by substituting one small part of the molecule for a different one. The prospects for organic SHG materials are very good, although they are not as thermally stable as their inorganic counterparts. In recent years, however, there has been progress in overcoming this problem. In addition, organometallic materials show promise, since they possess the fast optical response of organic materials while having good thermal stability. Further structural studies on these and other materials will ensure that the future of opto-electronics continues to look bright. ■

Figure A
The molecular structure of MBADNP as determined by single-crystal neutron diffraction at the ILL

Figure B
A contour map of the bonding-electron density in the pyridine part of the optically active molecule, MBADNP. Each blue, red and black contour represents a positive, negative and zero electron density respectively at increasing levels of one-tenth electron density per cubic angstrom

Research team J. M. Cole *(University of Cambridge, UK)*, J. A. K. Howard *(University of Durham, UK)*, G. J. McIntyre *(ILL)*

Sweetness and life

How sugars dissolve in water may play an important role in biological processes

>> GEORGE NEILSON and PHILIP MASON

George Neilson

S ugars are important biological materials. They are key components of the genetic molecules DNA and RNA. They also play crucial parts in many biochemical cycles, some of which have been turning as long as life itself. These cycles include the photosynthetic production of sugar from sunlight, water and carbon dioxide – the method of energy storage in plants, and the use of sugar directly in metabolic processes in animals. Although such cycles are understood in general chemical terms, little is known about many of them at the molecular level.

Even our knowledge of molecules as simple as the hydrated structure of sugars in solution is fragmentary and usually inferred from X-ray or neutron crystallographic studies. How water molecules are arranged and bound around biological molecules such as sugars in cells is vital for understanding their function and behaviour.

Isotopic substitution

Neutron diffraction with isotopic substitution is the only method that can give information on all aspects of the hydration structure of ions and molecules at atomic resolution. The technique relies on the fact that different isotopes of the same elements have identical chemical structures but appreciably different neutron scattering properties.

In this experiment, three chemically identical aqueous solutions of glucose ($C_6H_{12}O_6$) with differing ratios of hydrogen and deuterium atoms were prepared at three different concentrations of the sugar. The glucose molecule has a ring structure of five carbon atoms and one oxygen atom with hydrogen and hydroxyl groups (OH) attached. Both hydrogen and deuterium atoms on the hydroxyl groups of the sugar molecules and the water molecules exchange rapidly with each other. Therefore, deuterium atoms can be incorporated into the sugar molecules themselves.

Relatively simple calculations on the measured diffraction patterns allow us to deduce structural aspects of the exchangeable hydrogens.

With our first results from the D4C diffractometer at ILL, we have proved that even in solutions that are as much as 50 per cent by weight glucose, certain geomet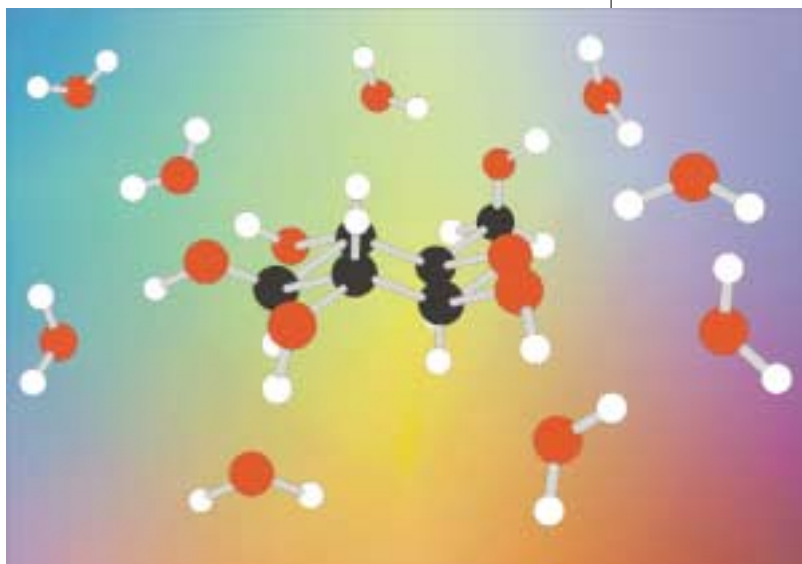rical aspects of the water structure are almost unperturbed! This contrasts strikingly with simple salt solutions such as lithium chloride in which the water structure around the ions is dramatically changed. The glucose results imply that the hydration structure around the exchangeable hydrogens in the hydroxyl groups is similar to the water structure itself. This is undoubtedly related to the biological role of sugars in metabolic processes.

Although our present studies have been applied only to exchangeable hydrogens in the sugar solution, we are confident that, in this ongoing programme, we will also be able to apply the same technique to each hydrogen atom in the sugar structure that is not exchanged in solution.

In this way we will be able to make a major contribution to understanding the hydration properties of sugars, and by extension provide a critical and quantitative assessment of computer simulation studies of hydrated sugar molecules. ■

The sugar molecule, glucose, depicted in a hydrated environment

Research team P.E. Mason, G.W. Neilson and A.C. Barnes *(University of Bristol, UK)*, M-L. Saboungi *(Argonne National Laboratory, USA)*, G.J. Cuello *(ILL)*

Glossary

Angstrom (Å)
10^{-10} metres (1Å = 0.1nm).

Antiferromagnetism
A type of magnetic order in which the electron spins are alternately oppositely aligned.

Colossal magnetoresistance
A phenomenon in which certain materials show very large changes in electrical resistance with temperature and magnetic field; they have potential applications for digital recording.

Deuterium
A heavier isotope of hydrogen having a neutron as well as a proton in the nucleus.

Doping
Adding or subtracting a small number of electrons to a material such as a semiconductor or superconductor.

Electric dipole moment
The electric dipole moment is the product of the magnitude and the distance between a pair of electrical charges.

Electron orbital
The quantum state of an electron orbiting an atom or ion.

Ferromagnetism
A type of magnetic order in which the electron spins are all aligned.

Inelastic coherent scattering
Scattering in which the neutrons gain or lose energy to the sample, thus giving information about the dynamics of the sample molecules or atoms. Coherent means that there is a phase relation between the neutrons scattered by different atoms, so that an interference pattern can be formed.

Ion
An atom or molecule that has gained or lost electrons so that it has become electrically charged.

Ion exchanger
A material that is used to exchange one type of ion for another, as in water softeners, for example.

Magnetic moment
A measure of the strength of a magnet.

Martensite transition
A transition between crystal structures that occurs gradually. The name derives from the transition from one type of steel, Austenite, to another, Martensite.

Metal oxide
A compound of a metal and oxygen, and the usual form of metals in nature.

Molecular orbital
The quantum state of an electron associated with a molecule.

Molecular sieve
Porous materials through which molecules or ions can selectively pass.

Nanometre
One-billionth of a metre (10^{-9} metres).

Neutron
One of the two particles found in the atomic nucleus. They have a characteristic wavelength depending on energy.

Neutron diffraction
Neutrons can be reflected, or scattered, off a material in which the interatomic distances are similar to the neutron wavelength. The scattered waves interfere to produce a characteristic diffraction pattern. In standard neutron diffraction, a single crystal is oriented over a range of angles to collect diffraction patterns from different planes of atoms in the crystal.

Neutron strain imaging
A method of using neutron diffraction to create a 3D image of the 'strain' in the interior of engineering structural components, obtained by measuring small changes in the spacings of planes of the constituent atoms.

Nuclear magnetic resonance
Another powerful analytical technique in which powerful static and radiofrequency magnetic fields induce magnetic transitions, usually in hydrogen atoms. It is complementary to X-ray and neutron diffraction in the information it provides, and can be also used to reconstruct 3D images.

Powder diffraction
Coherent scattering from a polycrystalline material.

Second harmonic generation
The doubling, or halving, of the frequency of monochromatic light when it impinges on certain materials.

Shape memory metal
A metal which when formed at one temperature, then cooled and deformed, will return to its original shape on re-heating.

Spin valve
A device that operates by allowing electrons with one spin state, but not the other, to pass a junction. Spin valves are used in data storage.

Superconductor
A material that has no electrical resistance below a certain temperature.

X-ray diffraction
A technique used to determine the structure of materials. X-rays are reflected, or scattered, off a material in which the interatomic distances are similar to X-ray wavelengths such that the scattered waves interfere to produce a characteristic diffraction pattern.

Zeolite
Natural or synthetic mineral with a porous aluminosilicate framework through which ions and molecules can pass. It comes from a Greek word meaning 'boiling stone' because zeolites swell and lose water from their porous structure when heated.

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