

# Humble neutron is valuable tool in geology

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Artistic interpretation of a geyser erupting on Neptune's icy moon Triton. Scientists at UCL used neutrons at ISIS and the ILL to explain this activity by studying the behaviour of methanol monohydrate, a known constituent of outer solar system moons, under conditions thought to be present within these bodies.

With the ability to analyse the properties of the Earth's internal components to the atomic scale in conditions only found kilometres below our feet, recent studies have allowed geoscientists to study our planets internal working, as well as those of worlds further afield, at new fundamental levels. And all from the relative comfort of the laboratory! Only last year, for example, researchers from Bath University used neutron techniques to map structural changes within oxide glasses and liquids - research that could provide a new tool to investigate the environmental conditions that melt the Earth's interior and produce volcanism at the surface.

With instruments able to recreate deep sub-surface conditions up to 7 GPa and 1500 K, neutron sources such as those found at the UK's ISIS and nuclear reactor-based sources (like the internationally owned Institut Laue-Langevin (ILL) in Grenoble) are helping support a small but growing community. Data models which they are developing will shine a new light on how melt structure and density change with depth through the Earth - vital clues as to how our current layered internal planetary structure formed billions of years ago and how it continues to evolve today.

## **Neutrons as a tool**

The application of neutron science techniques in the field of geology did not occur in isolation. Their use followed several decades after the construction of the first x-ray sources, which perform similar types of analyses but in very different ways.

The application of x-rays was first discussed in geoscience circles almost 100 years ago, and very quickly early adopters of the technique were able to work out the atomic crystal structure of all the major components of the Earth, as they did for many other materials across condensed matter physics. Neutrons, by contrast, are a far newer proposition, whose

history in geoscience dates back only half as long as that of their light-based compatriots.

In 1932, James Chadwick first proved the existence of a neutrally charged particle within the atom. Once confirmed, thoughts immediately turned to its potential use. The basis of this interest lay in the observation that, like all particles, neutrons demonstrate some wave-like behaviour, and when they encounter obstacles whose size is comparable with their wavelength they are deviated and scatter along well-defined angles. This is the property that allowed scientists to analyse the scattering patterns and so to infer the structure of the material the neutrons have passed through.

The usefulness of neutrons was further enhanced by a number of highly desirable properties which helped open up their application across a wide range of sciences - including the geosciences. First, the neutron's lack of charge means that it can penetrate more deeply into matter than x-rays or their electrically charged cousins. They also do so in a non-destructive manner, allowing researchers to study changes in structure as a function of time, relating to changes in temperature or pressure.

## **Forecasting melts**

The data neutrons provide allow geoscientists to create highly accurate vertical profiles of the behaviour and structure of different interior components at different levels of the Earth's interior.

One recent study of this kind was carried out by a team led by Prof Phil Salmon (Bath University), which showed how the tight packing of oxygen atoms in common silicon-based glasses under extreme pressure leads to larger-scale structural changes that affect the material's properties. Under ambient conditions these oxide glasses have quite an open structure. However under high pressure conditions, the collapse of

their atomic arrangement brings the oxygen atoms closer together. Eventually, the packing of oxygen creates changes in the connectivity of the atomic network so that silicon atoms convert from having four oxygen-atom tetrahedral arrangements to six.

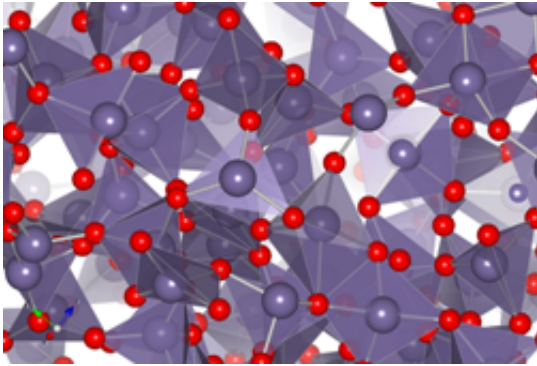
As a result of the sensitivity of oxygen atoms to their surrounding environments, and the impact these structural transformations have on materials' bulk properties, it was believed that analysing the structural arrangements could be used to gauge the conditions under which different materials form. However, an exact understanding of how pressure produces these transformations has so far been lacking.

To investigate one potential indicator of these changes, Salmon and his team tracked the close packing of oxygen atoms within oxide glasses under extreme pressures using neutron diffraction. Their analysis showed that the overall network structures and associated physical properties of a wide variety of disordered oxides can be categorised - and therefore predicted - by the material's 'oxygen-packing fraction' (the space within a liquid or glass structure occupied by [oxygen atoms](#)), which increases under pressure. This analysis also extends to liquid basalt at deep mantle conditions.

Until now there has been no reliable guide for predicting the conditions under which transformations occur. However Salmon's study has shown that if you have a material whose packing density is known to approach its random close-packing upper limit (where 64% of the volume is taken up with oxygen) this is a strong indication you are going to get a change in structure and therefore a change in physical properties. These properties include the material's compressibility and its viscosity, which determine flow behaviour.

This is important information. A better understanding of how local subsurface conditions affect viscosity could help model the speed of

magma turnover, which in turn affects how slowly or quickly you can get heat out, and therefore how quickly the Earth's ancient magma ocean crystallised, billions of years ago.



The arrangements of polyhedra in a compacted glass under high-pressure conditions.

## Other materials

However, glasses are not the only materials that can be analysed. Because neutrons 'see' the nucleus of atoms (unlike x-rays which 'see' electrons) they are particularly good at identifying the position of light atoms - such hydrogen, a very important component of many minerals, particularly hydrous minerals - and so can be used in studying the movement of water within geological process.

Some minerals, such as clays and zeolites, contain significant quantities of water in pores and between atomic layers. Near-surface geology is in fact dominated by the effects of water acting as the 'grease', enabling the convection of minerals in the inner Earth that drives plate tectonics. The sensitivity of neutrons to hydrogen allows precise measurements to be made of the structure, synthesis and dynamics of these hydrous mineral

phases.

With regards to scale and resolution of analysis, the wavelengths of neutrons lend themselves to very precise measurements at the atomic and molecular scale. While the wavelength of your average lab-sourced x-ray corresponds roughly to that of an atomic bond, neutrons can be produced with wavelengths six times as small - providing far greater resolution. Neutron wavelength can also be tweaked by altering the energy they carry. At facilities like the ILL they are produced over a wide variety of energies, from hot thermal neutrons (with an energy of about 0.025 eV - which whizz around at a couple of kilometres per second) to ultra-cold neutrons (with energies less than  $3 \times 10^{-7}$  eV, which move more slowly than most people can run).

This range of energies corresponds to wavelengths that vary over four to five orders of magnitude. Although ultra-cold neutrons are used particle physics experiments to study the properties of the neutron itself, these large facilities produce a sufficient range of neutron energies to allow scientists to study both the atomic and molecular structure of many common geological minerals.

Finally we have the neutron's ability to investigate magnetism. Being made up of three electrically charged quarks, neutrons act like small magnets themselves and so interact with the electron spins in magnetic materials. This makes them an ideal probe for understanding the structure and dynamics of magnetic minerals such as haematite and magnetite.

## **First analysis**

Despite possessing such unique properties, neutron studies were initially slow to take off. It was not until the advent of nuclear reactors, around 1945, that high neutron fluxes, capable of in-depth structural

investigations, became possible. Even then, researchers had to wait until the 1960s for specially developed high-flux research reactors to be built and optimised. This development culminated in the high-flux reactor at the ILL (in operation since 1972), which has achieved the highest neutron flux to date.

Today there are over 20 neutron active science facilities across the world, and they come in two forms. Research reactors, such as the ILL, use nuclear fission to produce a steady, reliable source of neutrons. Spallation sources, such as the STFC ISIS Neutron Source in the UK, accelerate protons into target material, prompting the emission of neutrons. A lot of my own research has taken place at these two facilities. The story of how I discovered the usefulness of neutron science as a tool for geoscience studies follows a similar path to the emergence of the technique more widely - with initial struggles and obstacles to overcome.

During my PhD work, I used single-crystal and powder x-ray diffraction. However, in my first post-doc position, I repeated some of my x-ray powder diffraction studies using neutron powder diffraction at ILL, and was amazed at how this alternative technique provided so much more detail for my particular samples.

I soon became a convert to neutron scattering and have since carried out a large number of studies at ISIS on geophysical and related materials, including structural studies of minerals such as quartz, leucite and calcite, measurements of phonons in minerals like calcite to investigate phase transitions, and using incoherent quasielastic scattering to explore the motions of water in minerals. Much of my recent effort has concerned using neutrons to help understand the structure of disordered materials (including disordered crystalline materials), using a measurement of all the scattering processes at once combined with a modelling technique.





One of the beamlines at the ILL where neutrons are fired at samples of materials to investigate their structure under a wide range of temperatures and pressures

For new applications in geosciences we pioneered the development of methods to measure neutron diffraction at simultaneous high pressures (up to 10 GPa) and high temperatures (up to 1500 K). We needed to develop a new method to measure temperature, because normal methods are too fragile for high pressures; we eventually settled on a method of using the neutron absorption spectrum, giving us a precision of  $\pm 20$  K. We are now looking to stretch this range of temperatures and pressures.



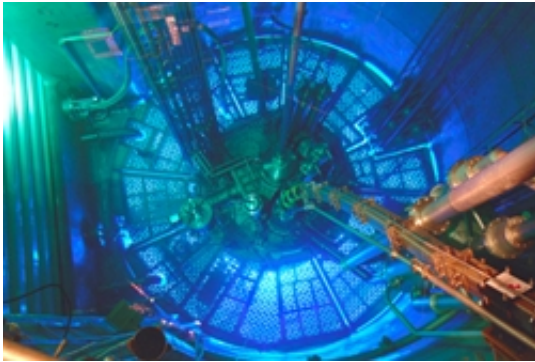
## Global

As a global community, these types of instruments and techniques have delivered important crystallographic insights into minerals as diverse as zeolites, feldspars, magnetite and various carbonates. Silicate minerals are a rich source of potential study. The transition from one molecular arrangement to another through the application of pressure (as each silicon atom, normally surrounded by four neighbouring atoms, moves to a more compact structure with two extra neighbouring atoms) is well known. However the exact conditions and the process involved in this transition has been little understood and is now being explored with the latest neutron techniques. Such studies should yield a far deeper understanding of a variety of Earth processes.

The application of these 'geological' studies made possible by neutron science has not been restricted to our home planet. In 2009 a science team led by Dr Dominic Fortes (University College London) working with the ILL and ISIS explored the internal structure of icy moons, such as the Neptunian satellite, Triton. The aim was to explain the icy eruptions seen by passing spacecraft by using neutron scattering to study the behaviour of methanol monohydrate, a known constituent of outer Solar System moons, under conditions thought to be present within these bodies.

The Fortes team's analysis showed that at room pressure the methanol crystals would expand enormously in one dimension while shrinking in the other two dimensions - whereas heating under an even pressure expanded them in two directions, while compressing in the third! With this new understanding the London based team were able to model their role in surface volcanism.

## Opportunities



Top view of ILLs high flux reactor

With such a wide range of applications relevant to the geoscience community, there should be much excitement around the potential of neutron science. Continuing upgrades, and new instrument development at institutes like ISIS and ILL, are bringing new capabilities and allowing researchers to recreate new, ever more extreme conditions and are giving the technique never-before reached levels of credibility within the neutron science community. This awareness and recognition, supplemented by the ability to match computer simulation and neutron scattering, will continue through plans for 'next generation' neutron facilities being pioneered by the European Spallation Source (ESS).

Based in Lund in Sweden, ESS is one of the biggest science projects under construction anywhere on the planet. It promises to provide researchers from various scientific disciplines with a new super microscope, powered by the world's most powerful neutron source. For the geology, geochemistry and volcanology community, its backers (including the UK government) hope its opening (due in the early 2020s) will offer access to specially-developed new instrumentation for in situ measurements of structure, reactivity and physical properties of multi-component melts and fluids under a variety of extreme conditions.

Despite the impressive investment and planned scale of ESS, and the continued improvements, new capabilities and world-class level of expertise at existing facilities, the potential impact of this fundamentally important work has yet to be fully appreciated. The community of advocates and those with the experience in preparing and analysing samples with neutrons is still relatively small, because the discipline suffers from the mistaken perception that anyone using these instruments needs themselves to possess in-depth understanding of complex neutron instrumentation.



Overview of the winning design for the European Spallation Source (ESS) by Henning Larsen Architects, COBE and SLA

This slow rate of acceptance of the modelling and analysis capability of neutron science may not be surprising for other reasons too. Geologists are far more used to dealing with kilometre-scale models, while microscope analysis is at crystal rather than atomic level. This represents a missed opportunity.

Neutrons provide a unique tool to understand the detailed behaviour of common materials and minerals under very uncommon conditions within

our planet. While we can measure the viscosity and density of lavas at the surface, the deeper you go into the Earth the more difficult it is to quantify these properties. With [neutron](#) science, geologists could start to unlock the fundamental explanations of the deep-Earth processes that formed our planetary structure billions of years ago and which manifest themselves today at surface as some of the most violent and important events in nature.

Provided by University of Bath

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