

Looking into the distant past with neutrons from TU Delft's nuclear reactor



[FOTO: ALFRED MOLON, UNTERHACHING]

Panorama of the pyramids at Giza, which were built around 2600 B.C.

BY ASTRID VAN DE GRAAF

How the ancient Egyptians managed to raise a seven million tonne pyramid in a time span of only twenty years remains a mystery. Every now and then discussions flare up as to how exactly the pyramids at Giza in Egypt were constructed. How could they cut stone without the necessary chisels? How did they manage to drag 2.5 million blocks of stone, each weighing several tons, as high as 140 metres above the desert? Or did they? Perhaps the ancient Egyptians were brilliant engineers who poured broken up limestone and cement into moulds to form made-to-measure? Cut or cast? That is the question which Dr Menno Blaauw, head of Facilities & Services at the Reactor Institute Delft, subjected to the latest analysis techniques.



The pyramids were built using blocks of limestone varying in weight from 3 to 30 tonnes. The main pyramid at Giza stands 140 metres high.



[PHOTO: INSTITUT GÉOLOGYÈRE, PARIS]

French professor Joseph Davidovits is convinced that the pyramids were built using blocks of cast stone made in much the same way as concrete.

with neutrons

Six years ago Menno Blaauw came into possession of a piece of stone from Cheops' pyramid. At the time he was still working as a researcher investigating neutron scattering at the TU Delft Interfaculty Reactor Institute (IRI). He found the fragment among the effects of his late father, with whom he had visited Egypt in 1995. Egyptology had come to fascinate Blaauw senior, a chemical researcher by profession, upon his retirement.

"He felt that he was nearing the end of his days, and the Egyptian culture being so highly focused on the afterlife, this was his way of coping with the concept of death," his son says. "Having visited the country on a number of previous occasions, he was able to give me a tour of the most interesting places." One of the places they managed to see was the Queen's chamber inside Cheops' pyramid, which is seldom opened to the public. Inside the chamber Blaauw senior, demonstrating the inquisitiveness of a true researcher, crawled into a crack at the back of an alcove, where he reached as high and as far as he could, and dislodged a piece of stone which he slipped into his pocket. "Just to be on the safe side, he kept the fragment in his pocket to guide it safely through customs, since suitcases are all routinely scanned for artefacts," Blaauw admits with a slightly guilty look on his face.

Inexplicable Infected by his father's fascination with all things Egyptian, Blaauw junior also became interested in the mystery of the pyramids. "In 2500 BC there were no chisels harder than stone. You simply cannot use copper to cut or break away limestone. So how did they manage to cut those

Dr Oebele Blaauw with his regular taxi driver. In the background is the pyramid of Chefred, the tip of which is still clad in alabaster.

The sample retrieved by Dr Oebele Blaauw from the crack in an alcove in the Queen's chamber of the Cheops' pyramid.





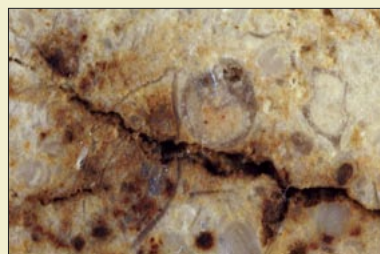
A section through the Cheops limestone sample used for tests using electron microscopy and thin section techniques.



Close-up view of the pyramid fragments in which the fossils are clearly visible.



If we zoom in on the sectional plane, several small cavities can be seen which give the impression that the stone might have been cast, since such a process could easily trap air bubbles inside the material.

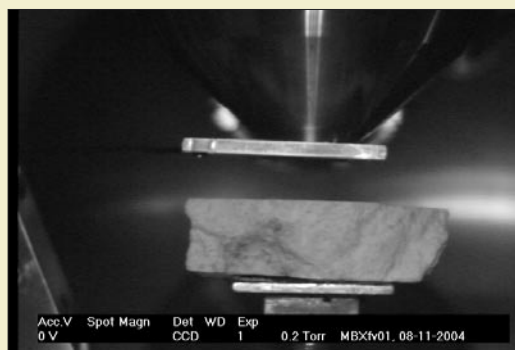


This crack divides a fossil in half and is reminiscent of shrinkage.

The optically active minerals can be made visible using a pair of polarising filters at cross angles in what is known as the thin-section technique.. There is no layered structure, which there would be in a natural sedimentary rock according to Professor J. Davidovits' theory.



At the Microlab of the Civil Engineering faculty, the ESEM (environmental scanning electron microscope) was used to scan the elementary composition of the Cheops stone. A special feature of this device is that it can be used to measure the composition of a very small local part of the surface area.



blocks so accurately to size? Limestone is a relatively brittle type of stone, and it contains faults that can develop into cracks. Producing a large, solid block of limestone will inevitably leave you with an identical volume of rubble. No such mountains of rubble have ever been found."

What we did manage to find was the quarries where the stone came from. At least ten quarries all over Egypt have been traced as the suppliers using trace element analysis. Apart from cutting and stacking the blocks, transporting each 3 to 30 tonne block on sleds must have been a vast logistical operation. All in all the pyramids are still veiled in mystery.

The Blaauws have also dug up calculations on the subject. One human being can provide a certain power to drag a block of stone up an incline. We know that a pyramid took about twenty years to build. Construction commenced as soon as a new pharaoh came to the throne, because the building had to be ready when he died. This means that 20,000 to 250,000 workers had to spend each and every day dragging blocks of stone.

"The resulting construction yard would have been the size of a city, and that's just including the dragging teams, without any of the other activities. It is almost impossible to imagine," says Blaauw junior.

Cast stone In his insatiable thirst for knowledge about the pyramids Blaauw senior picked up a book by the French geochemical engineer, Joseph Davidovits. According to this scientist, the building theory of cut blocks of stone being dragged up inclines is cracking at the seams. He proposes that instead the giant pyramid stones were artificially made by pouring limestone granulate into moulds and allowing it to set, much in the way that we make concrete today.

The cast stone theory appeared plausible to Menno Blaauw.

"It is much easier to imagine. Limestone is made up mostly of calcium carbonate, a perfect basis for cement. You would leave part of the extracted limestone in granule form, grind down the other part, fire it up in a kiln, and then add water. According to Davidovits the pyramid as a whole should contain 15% water, although he makes a secret of the actual recipe for the artificial stone, since he holds a patent on the geopolymer, as it is known. After some time you end up with artificial stone that cannot be distinguished from the real thing. The treatment does not affect the trace element pattern. A simple test will not help you determine whether you are dealing with artificial stone or not. At the time when the accepted hypothesis on the construction of the pyramid was postulated, the late nineteenth century, concrete was not the common construction material we know today. So it is hardly surprising that the archeologists back then never considered the option. Today we can look at things in a different light. Who knows, we may have overlooked something," Blaauw says. His new job as head of Facilities & Services at the Reactor Institute Delft (as the facility half of the IRI is known since 1 January 2005) gave him the idea of taking a closer look at the fragment that was now his.

Searching for water At the RID, Blaauw has an instrument at his disposal that will let him take a closer look at the Cheops stone. It is a neutron scattering device, the rotating crystal spectrometer (RCS). It can be used to investigate the water content of cement stone. Concrete sets as the result of a slow chemical reaction with water that changes the cement powder into a stone-like solid material. Concrete therefore contains crystal water that is firmly bonded to the cement by hydrogen bridges. Just as astronomers scan the surface of Mars for water to detect possible signs of life, so can Blaauw use his neutron scatterer to look for signs of engineering in the age of the pharaohs.

Neutrons are uncharged particles with a mass identical to that of a hydrogen nucleus. When a neutron collides with a hydrogen nucleus head-on, it can transfer all its kinetic energy to the nucleus. But if the neutron strikes a water molecule, the latter will only absorb certain portions of energy to match the vibration frequencies of the molecule itself. The largest part of these portions will be equivalent to 70 meV. If the difference in energy between the incoming and the reflected neutrons does not peak in the 70 meV region, there are almost certainly no water molecules in the material.

Blaauw: "Lots of experiments on cement have been done in this way, so we know all we need to know about these analyses. After a full week's measuring we had still not found any sign of water. Of course, this is hardly conclusive evidence, but it gives an indication that the Cheops sample was not manufactured with

a water-binding cement as we know it. If it contains any water, it is certainly rigidly locked away.”

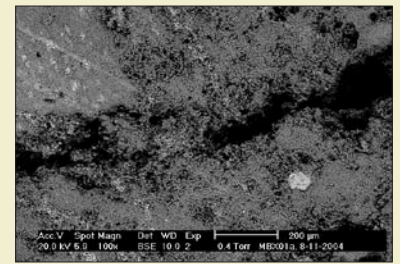
Pore structures Cement is usually made with a slight excess of water to make it easier to pour and spread. The water eventually evaporates, leaving a specific type of pore structure. A highly ingenious method that can measure such pore structures with sizes ranging from 30 nanometres to 10 micrometres is known as small angle scattering. The device used at TU Delft is of the spin echo variety, and it is used to measure the size and arrangement of the pores. Blaauw: “This device is the brainchild of Dr Theo Rekvelde, at what was then the department of Neutrons and Mössbauer Spectrometry. The smaller the angle at which the device can see, the larger the components and structures that you can observe. Our small angle scattering device is the only one in the world that can measure structures up to 10 microns. Standard equipment cannot handle anything over one tenth of a micron. In addition, our spin echo technique speeds up measurements 10,000 times, so experiments that would otherwise take a whole day can now be finished in ten seconds.” Just like the rotating crystal spectrometer, the spin echo system employs neutrons, but in a slightly different way. This device uses not only the scattering properties of the particles, but also takes advantage of the fact that neutrons in a magnetic field behave like compass needles. Although neutrons do not carry an electric charge, they do have a magnetic moment, or spin. As the neutrons come flying out of the nuclear reactor, their spins all face in different directions. A polariser is used to straighten out the neutrons so they all spin in the same direction, the orientation of which is controlled by four magnetic fields of identical strength. In Rekvelde’s small scattering angle spectrometer, this is used to translate scattering into depolarisation, which is easy to detect. To assess the pore structure of the Cheops stone, the team of Prof. Dr Ir. Klaas van Breugel of the faculty of Civil Engineering and Geosciences supplied two types of reference cement, Portland cement and blast-furnace cement. The experiments were a success according to Blaauw. The only thing was that the distribution of the pore size in the fragment from the pyramid was nothing like that of the two modern cement types. But Blaauw has some more neutron tricks up his sleeve.

Trace elements Neutrons can cause reactions in which the nuclei become unstable when struck. The radioactive nuclei thus produced show a specific pattern for each different element. “Activating a sample with neutrons makes practically the whole thing radioactive, so we can use a high-resolution gamma spectrometer to determine which elements, and how much of each, the sample contains,” Blaauw explains. “This method made it fairly simple for us to carry out a trace element analysis on the stone sample.” Blaauw found that the sample contained mostly calcium carbonate (CaCO_3) with traces of other elements. This makes it no different from natural limestone. “It did contain 1% common salt (NaCl), but this is pretty normal for sedimentary rock. And, as my father discovered to his horror, the crack in the alcove was used for exactly the purpose that secluded corners in tourist attractions tend to be used for when nature calls.” The trace of iridium they found, a material that must have come from outer space, he dismisses as the result of meteor impacts at the end of the Cretaceous Period. He does not believe in visiting aliens. Although an element analysis can never provide conclusive evidence to show whether a stone was cut or cast, Blaauw did get other useful information, in the form of traces of uranium, thorium, and potassium. “It is quite normal to find these elements, as they are found throughout the Earth’s crust. In addition to cosmic radiation, these traces form a major source of our daily dose of radiation. However, they also form an interesting source of radioactive information for dating the pyramid fragment.”

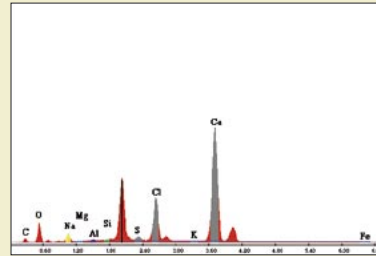
Luminescence dating Practically every type of rock contains bits of feldspar or quartz. When a piece of rock is subjected to ionising (radioactive) radiation, the tension in the crystal structure of these minerals increases. The Cheops stone contained mostly feldspar. The increased tension resulting from the ionising radiation is recorded as if by a photographic film. This enables us to determine the age of the stone.



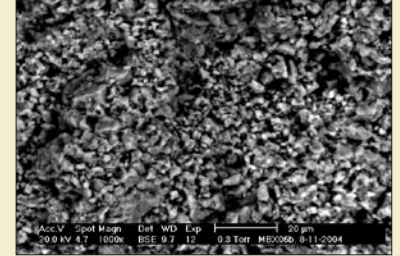
Blaauw and his team were very interested in the composition of the red colour in this crack.



The area of the red crack as seen by the ESEM.

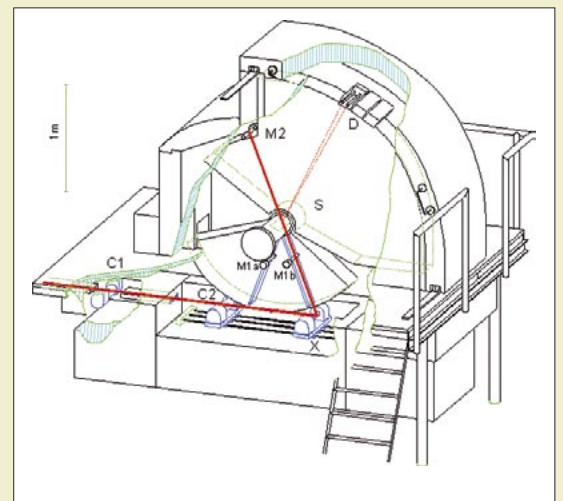


The X-ray spectrum of an ESEM measurement shows the composition of the various elements making up the stone. As was expected, calcium has a dominating presence. By comparing two readings, the red pigment is shown to be iron chloride.



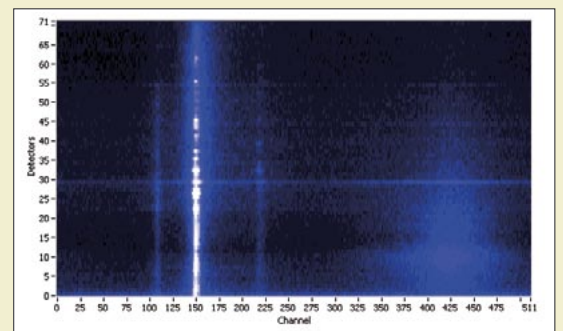
Zooming in even further on the Cheops stone yields no new information.

Using the rotating crystal spectrometer (RCS) developed by IRI, Blaauw was unable to prove the presence of water in the Cheops stone (S). The operating principle of the RCS is as follows. Neutrons from the reactor enter the device at the lower left. Two choppers (C1 and C2) chop up the neutron flow into pulses of an approximately known wavelength. When the pulses strike the crystal (X), only the neutrons

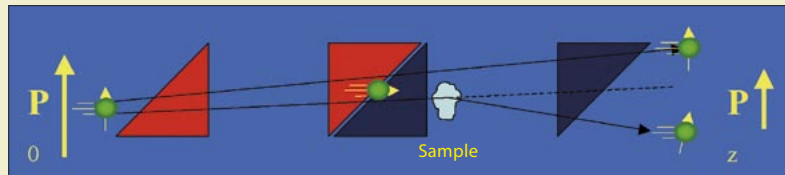


with an exactly known wavelength are registered. The orientation of the crystal determines the wavelength. By rotating the crystal, neutrons with a wide spectrum of wavelengths in time are reflected. The purpose of the system is to measure the fractions of neutrons that the sample scatters in a certain direction and with a certain velocity. Any neutrons that fly straight on are counted by monitor M2, while the scattered neutrons are detected by hundreds of detectors (D). The exact time of detection is also recorded to enable the flight time to be measured. The detectors are all based on the absorption of neutrons by ^3He .

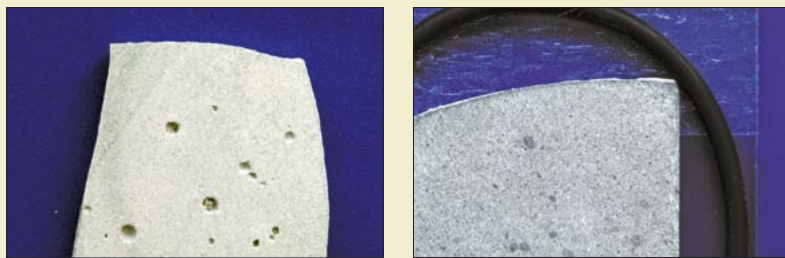
Result of the RCS test: the X axis plots the neutron velocity, and the Y axis plots the scattering angle. The colour indicates which fraction of the neutrons arrived at which angle and at which velocity.



Blaauw used the SESANS to determine the pore structure and the arrangement of the pores.. This device was developed at IRI in recent years by the team of Dr Wim Bouwman. The set-up consists of a monochromator, a polariser, four electromagnets, an analyser, and a detector.

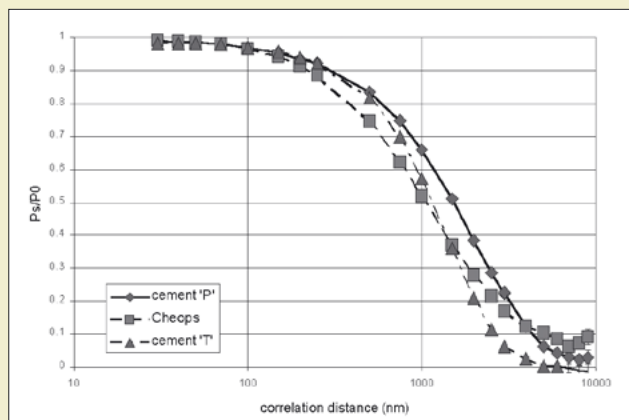


The operating principle of SESANS is as follows. Neutrons passing through the monochromator (a crystal) all have the same wavelength. A mirror selects neutrons of identical spin (polarisation). Four electromagnets turn the spin directions clockwise or anticlockwise. When the neutrons have passed through the sample, the distribution of the spin direction of the scattered neutrons is measured using the detector and analyser.

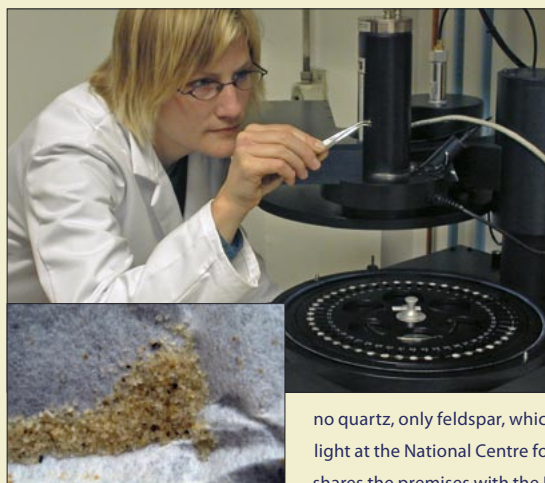


These two samples of fundamentally different types of cement were prepared at the Microlab of the Civil Engineering faculty. The sample on the left was made using Portland cement, while the one on the right was made with blast-furnace cement.

The results of the SESANS experiments. The X axis plots the distances within the material, while the Y axis plots



the probability of finding solid material at a distance X from any point within the solid material. The plots indicate that the two types of cement differ only in the coarseness of the pore structure, whereas the Cheops sample plot shows markedly different properties around 10 μm , so the plots intersect.



Feldspar and quartz are minerals that store energy when exposed to ionising radiation. Daylight frees the stored energy in the form of visible light. The Cheops stone contained no quartz, only feldspar, which was read using infrared light at the National Centre for Luminescence Dating, which shares the premises with the Reactor Institute Delft.

Blaauw: "It is like a film that has been exposed, but not yet developed. To free the feldspar, the stone is pulverised in darkroom conditions and dissolved, leaving the feldspar, which is insoluble. The darkroom conditions are necessary because normal daylight is sufficient to relax the crystal tension. We then shine an infrared beam onto the feldspar and get visible light in return. The level of the returned light provides a measure of the tension and therefore of the radiation dose the rock has received since it last saw daylight. Since reading the stone in this way will simultaneously relax the crystal tension, this is an experiment that you can do only once."

The strength of the radiation source (uranium, thorium, potassium) being known, and thus the speed at which 'the damage is done', by measuring the damage, the time at which the Cheops stone last saw daylight can be calculated. Unfortunately the experiment showed this to be 400,000 years ago.

"This is a minimum value by the way. The material could be older, as the tension slowly relaxes of its own accord, and the feldspar moves back a bit to its former state. If the stone had been made from pulverised limestone, the counter would have read 2600 BC. Even if the feldspar had not been subjected to daylight during the pulverisation stage, it would most certainly have resettled during the production of cement from limestone, most of which takes place in kilns at high temperatures. Even Davidovits assumes that the ancient Egyptians used this method to prepare their cement."

Conclusive Even though Blaauw couldn't prove the theory favoured by his father, which he would have loved to do, he is certain of his findings. "Additional research will of course be necessary if we are to have unambiguous results for the stone used in Cheops' pyramid, for example comparative tests with other materials such as the artificial stones created by Davidovits. However, our particular stone is a normal, natural limestone, not a manufactured cast. To my mind the luminescence dating has provided conclusive evidence."

The halved piece of stone from Cheops' pyramid and its many fragments have now been stored in a plastic sample bag. Blaauw will be sending his findings to Davidovits to disprove the cast stone theory. Recently a new advocate of the theory appeared on the scene in the person of Belgian physicist Guy Demortier, who takes the presence of arsenic and unusual silicon oxide compounds as indications for the cast stone theory. However, using his activation analysis, Blaauw managed to find only 1 mg arsenic per kg of material, and hardly any silicon. So the theory proposed by Demortier is not supported by Blaauw's results either. He does not think much of Davidovits' claim that the fossils in artificial stone are arranged in a haphazard fashion either.

"The same applies to the material I experimented on, but petrologists can see nothing wrong with it. It just indicates that we are talking about the same type of material. According to Demortier and Davidovits, the cement should contain sodium hydroxide (NaOH), but sodium is present in the same molar concentration as Cl, in other words, as common salt, which is nothing unusual in sedimentary rock."

Of course, Blaauw used only a very small fragment from an enormous pyramid, so how representative can his results be?

"That question is difficult to answer. In Davidovits' book there is a drawing of a fossil structure that is identical to what we see in our fragment, so that makes it more plausible. Also, the fragment came from the core of the pyramid. I would not have thought there would be much chance of taking a 50 gram sample of natural limestone from 7 million tons of artificial stone, but it must be said that we used only a very small piece of stone from a giant pyramid, so the last word on the subject has yet to be said."

For more information please contact Dr Menno Blaauw, phone +31 (0) 15 278 3528, e-mail m.blaauw@tnw.tudelft.nl.

More information about the theory of Prof. Joseph Davidovits can be found in:
 - La Nouvelle Histoire des Pyramides d'Egypte, ed. Jean-Cyrille Godefroy, Paris, 2004, ISBN 2-86553-175-9.
 - Ils ont bâti les pyramides, ed. Jean-Cyrille Godefroy, Paris, 2002, ISBN 2-86553-157-0.
 - The pyramids: an enigma solved, Hippocrene Books, New York, 1988, ISBN 0-87052-559-X