DATING TECHNIQUES

Illuminating the past

The technique of optical dating was first reported 30 years ago, and has since revolutionized studies of events that occurred during the past 500,000 years. Here, two practitioners of optical dating assess its impact and consider its future.

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Thirty-year anniversaries are traditionally associated with pearls, which are renowned for the lustre produced by the reflection, refraction and diffraction of light. It is fitting, then, that in this International Year of Light and Light-based Technologies, we also celebrate the dawn of the optical dating technique, first reported three decades ago by David Huntley and colleagues in *Nature*[1]. Optical dating was proposed by the authors as a method for determining the time since wind-blown and water-borne mineral grains were last bleached by the Sun's rays before becoming buried, for example in a sedimentary landform. It has since become an essential arrow in the quiver of scientists worldwide, enabling geological, biological and archaeological events to be placed on a timescale extending from the present to half a million years ago or earlier — well beyond the 50,000-year limit of radiocarbon dating, and without the need for subsequent calibration corrections.

Optical dating exploits the physical properties of light-sensitive electron traps in ubiquitous minerals — chiefly, quartz and feldspar — as atomic 'time capsules'. These traps are rapidly emptied when exposed to sunlight, but steadily refill if mineral grains are buried within a deposit and concealed from light, because of the energy received from background environmental radiation (Fig. 1). The time elapsed since the grains were last bleached by sunlight is calculated from a laboratory estimate of the past radiation dose divided by the rate at which ionizing radiation from environmental sources is absorbed by the grains after burial[1–7].

Huntley, together with Ann Wintle, had previously been pivotal in developing reliable procedures for thermoluminescence dating of unheated sediments[8]. This technique is closely related to optical dating, except that thermoluminescence traps are emptied by heating grains — a process that evicts electrons from both optically inert traps and light-sensitive ones. Optical dating, by contrast, enables the latter to be accessed directly. Huntley *et al.*[1] achieved this using the green beam from a powerful argon-ion laser to induce dim, optically stimulated luminescence (OSL) from quartz grains[3]. They then compared this signal with the OSL obtained from grains that had been dosed with radiation in the laboratory, to estimate the past radiation dose and therefore the burial time of the grains.

Their approach was promptly implemented by another team using a similar laser[9], but optical dating spread more widely only after it was discovered that feldspars are acutely sensitive to infrared stimulation[10], enabling the convenient use of infrared light-emitting diodes (LEDs). By the late 1990s, the technique had matured into a powerful tool for dating sediments from the Quaternary period (the current geological period, which began about 2.6 million years ago), shedding light on the evolution of desert dunes and other landforms and on the timing of past human activities, particularly in Australia and Europe[2].

Applications proliferated after the turn of the millennium, following a decade of development of 'single-aliquot' procedures[2] to determine the burial dose — an idea originally proposed by Huntley and colleagues[1]. The adoption of optical dating by laboratories worldwide was then spurred by several advances: the advent of single-aliquot regenerative-dose (SAR) procedures[11] (which involves making repeated OSL measurements on individual grains or separate groups of grains to obtain many independent estimates of the burial dose for a sediment sample); the use of established statistical methods to analyse OSL data[12]; and the incorporation of sufficiently bright LEDs and compact solid-state lasers to stimulate quartz and feldspar grains in purpose-built, automated instruments[13].

The resulting studies have addressed

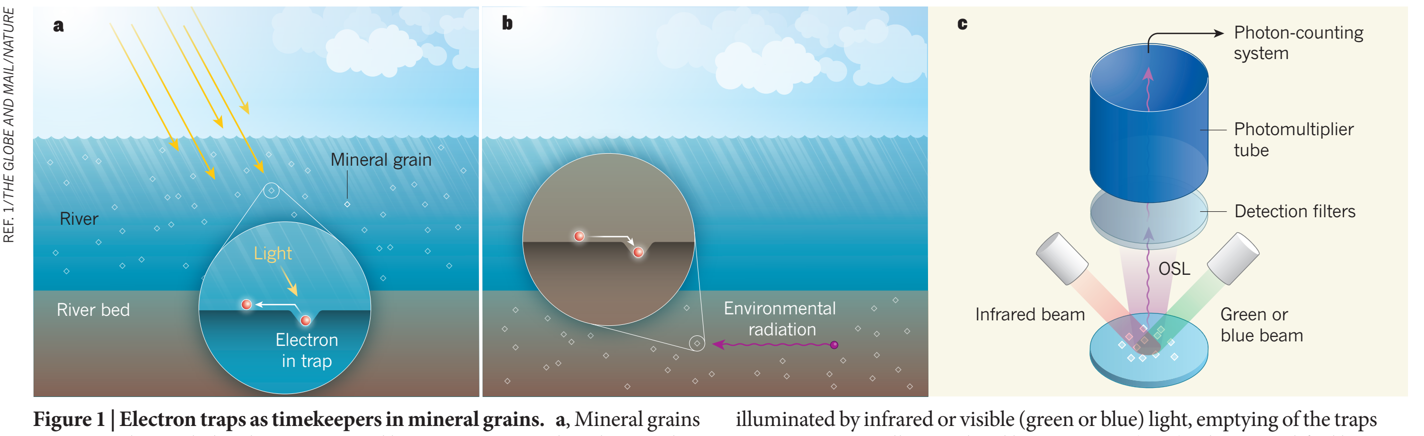

Figure 1 | Electron traps as timekeepers in mineral grains. **a**, Mineral grains are exposed to sunlight when transported by air or water, or when deposited on the ground. Electrons caught in light-sensitive traps in the crystal lattices of the grains are evicted by the light and return to their normal atomic sites. **b**, When grains are buried and hidden from sunlight, environmental radiation causes electrons to leave these sites and be captured by the traps. **c**, If the grains are collected (concealing them from daylight), prepared in the laboratory and illuminated by infrared or visible (green or blue) light, emptying of the traps gives rise to optically stimulated luminescence (OSL). This is amplified by a photomultiplier tube and measured using a photon-counting system. The past radiation dose — from which the burial time of the grains is determined — is estimated as the equivalent dose of laboratory radiation needed to produce an OSL signal of the same intensity[1]. The OSL signals are separated from unwanted emissions and light from the stimulation beam using filters.

questions on topics ranging from landscape dynamics, climate change and soil development to human evolution and dispersal over the past few hundred millennia, as well as more recent archaeological events[3–7]. For example, optical dating has revealed that symbolic markings, personal ornaments and innovative technology associated with early modern humans appeared more than 70,000 years ago in southern Africa, and were widespread across the region by 60,000 years ago[14–16] — some 15 millennia before modern humans entered Europe. The technique has also had a key role in establishing that humans had arrived in Australia by around 50,000 years ago[17] and that the last of the 'megafauna' (the giant marsupials, reptiles and flightless birds that once roamed the continent) perished soon after[18], during a period of increasing aridity but preceding a protracted phase of much drier climate[4,17].

Methodological and instrumental developments continue to drive advances in optical dating. Many quartz grains have physical properties that are ill-suited to SAR procedures, and two further possible complications are insufficient bleaching of grains before deposition and mixing of sediments after burial. Measurements of individual grains — the fundamental unit of analysis in optical dating — allow each of these factors to be investigated for sand-sized grains using SAR procedures[12]. This has helped to improve the accuracy of optical ages by reducing the uncertainties inherent in measuring composite OSL signals from multiple grains[7,15].

Nonetheless, other constraints on optical dating remain, and will keep researchers busy searching for solutions. A key limitation is the time range, which is governed by the maximum number of electrons that can be caught in light-sensitive traps and by the long-term stability of the traps at environmental temperatures. Optical-dating applications have largely been restricted to the past 200,000 years, and efforts to push the maximum limit beyond the 800,000-year timespan investigated by Huntley *et al.*[1] have mostly ended in disappointment. But new vistas are opening up, with the recent identification of longer-range optical-dating signals in quartz and feldspar[5–7]. If these are confirmed as reliable chronometers, then optical dating of major events in Earth and human history during the Early Pleistocene — the period from about 2.6 million to 0.8 million years ago — has a bright future.

Developments are also afoot to map the distribution of optical ages for individual grains on the cut surfaces of intact sediments and artefacts[7]. The ability to acquire such spatially resolved ages would be an advance over the current practice of disaggregating samples to extract grains for OSL measurements, which results in the loss of valuable contextual information. The fundamental insights obtained would be on a par with those gained from single-crystal dating in other branches of geology and in single-cell analysis in biology.

New frontiers for optical dating also include the use of OSL signals to investigate the long-term exhumation of landscapes and evolution of mountain ranges[19], and the *in situ* dating of minerals on Mars using robotic devices, which would propel optical dating into space[20]. These applications are extremely challenging, but if the past 30 years of progress are any guide, we can expect optical dating to illuminate much more of the history of this planet — and perhaps that of others — before we celebrate its fiftieth anniversary. ■

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1. Huntley, D. J., Godfrey-Smith, D. I. & Thewalt, M. L. W. *Nature* **313,** 105–107 (1985).
2. Aitken, M. J. *An Introduction to Optical Dating* (Oxford Univ. Press, 1998).
3. Lian, O. B. & Roberts, R. G. *Quat. Sci. Rev.* **25,** 2449–2468 (2006).
4. Prescott, J. R. & Robertson, G. B. *Aust. J. Earth Sci.* **55,** 997–1007 (2008).
5. Rhodes, E. J. *Annu. Rev. Earth Planet. Sci.* **39,** 461–488 (2011).
6. Liritzis, I. *et al. Luminescence Dating in Archaeology, Anthropology, and Geoarchaeology: An Overview* (Springer, 2013).
7. Roberts, R. G. *et al. J. Archaeol. Sci.* **56,** 41–60 (2015).
8. Wintle, A. G. & Huntley, D. J. *Nature* **279,** 710–712 (1979).
9. Smith, B. W., Aitken, M. J., Rhodes, E. J., Robinson, P. D. & Geldard, D. M. *Radiat. Protect. Dosim.* **17,** 229–233 (1986).
10. Hütt, G., Jaek, I. & Tchonka, J. *Quat. Sci. Rev.* **7,** 381–385 (1988).
11. Murray, A. S. & Roberts, R. G. *Radiat. Meas.* **29,** 503–515 (1998).
12. Galbraith, R. F., Roberts, R. G., Laslett, G. M., Yoshida, H. & Olley, J. M. *Archaeometry* **41,** 339–364 (1999).
13. Bøtter-Jensen, L., McKeever, S. W. S. & Wintle, A. G. *Optically Stimulated Luminescence Dosimetry* (Elsevier, 2003).
14. Henshilwood, C. S. *et al. Science* **295,** 1278–1280 (2002).
15. Jacobs, Z. *et al. Science* **322,** 733–735 (2008).
16. Brown, K. S. *et al. Nature* **491,** 590–593 (2012).
17. Bowler, J. M. *et al. Nature* **421,** 837–840 (2003).
18. Roberts, R. G. *et al. Science* **292,** 1888–1892 (2001).
19. Herman, F., Rhodes, E. J., Braun, J. & Heiniger, L. *Earth Planet. Sci. Lett.* **297,** 183–189 (2010).
20. Kalchgruber, R., Blair, M. W., McKeever, S. W. S., Benton, E. R. & Reust, D. K. *Planet. Space Sci.* **55,** 2203–2217 (2007).

STRUCTURAL BIOLOGY

Pain-sensing TRPA1 channel resolved

The TRPA1 ion channel activates pain pathways in response to noxious compounds. The structure of TRPA1 has now been solved, providing insight into how it functions. **SEE ARTICLE P.511**

DAVID E. CLAPHAM

Rooted in place, many plants resort to chemical warfare to survive predation by bacteria, fungi, insects and mammalian herbivores. They produce pungent natural chemicals, such as capsaicin, which makes chilli peppers 'hot', and the thiosulfinates that make onion chopping a tear-jerker. Reactive chemicals in onions, wasabi and related spices activate the ion channel TRPA1, a relative of the capsaicin receptor TRPV1. On page 511 of this issue, Paulsen *et al.*[1] follow up their description of TRPV1 (refs 2, 3). Using electron cryo-microscopy techniques, they define the full-length, single-particle structure of TRPA1 to around 4 ångströms, a level of resolution that reveals its general features.

TRP channels are found in almost all cell types in eukaryotes (organisms that include plants, animals and fungi). There are 27 human TRP-channel genes, which mostly encode weakly selective cation channels that enable ion flux across membranes in response to the binding of extracellular or intracellular ligands, or to changes in temperature or membrane voltage. On opening, TRP channels reduce the voltage across membranes and enable cations such as calcium ions (Ca^{2+}) to flow into the cytoplasm[4].

TRPA1 is found in the plasma membranes of pain-detecting sensory nerves[4]. It activates pain pathways that trigger avoidance behaviours and pathways that promote longer-lasting biological responses, such as inflammation[5]. Blocking TRPA1 function is therefore a promising strategy for treating pain. Pungent agents from wasabi, and other TRPA1 triggers, are known to be electrophiles, activating the channel by forming covalent bonds with specific cysteine or lysine