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OSL dating of rock art

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Abstract

This article describes the principles of optical dating – an umbrella term for a family of related techniques based on the storage of radiation energy in light-sensitive traps in natural minerals – and its application to rock art. Optical dating has been applied predominantly to sand- and silt-sized grains of quartz (optically stimulated luminescence, OSL) and feldspar (infrared stimulated luminescence, IRSL) that were exposed to sunlight prior to deposition, where the age represents the time elapsed since the grains were last bleached by the sun's rays. Only a few studies have used OSL or IRSL dating to constrain the age of rock paintings and engravings, and these applications can be grouped under two broad headings: dating of associated sediments and dating of rock surfaces. These studies are briefly reviewed in this article, together with some comments on future directions and challenges for OSL and IRSL dating of rock art.

Keywords

optical dating, quartz OSL, feldspar IRSL, sunlight exposure, sedimentary deposits, rock surfaces

1. Introduction

Optical dating (Huntley *et al*., 1985) is based on the absorption of incoming radiation energy by naturally occurring, common minerals such as quartz and feldspar, and the storage of a small fraction of this energy as trapped electrons and holes (charge carriers) at light-sensitive defects in their crystal lattices. The trapped electrons can be evicted by heating the mineral grains to a high temperature or by exposing them to light, with the latter giving rise to optically stimulated luminescence (OSL) emissions. OSL dating is one of a family of closely related techniques – thermoluminescence (TL) dating being another – and usually refers to the eviction of electrons from light-sensitive traps in quartz using blue or green light. For feldspars, the term infrared stimulated luminescence (IRSL) in often used instead of OSL, as infrared photons can be used to evict the trapped electrons. Reviews of OSL dating for non-specialists include Aitken (1998), Jacobs and Roberts (2007), Duller (2008a) and Roberts *et al*. (2015), who also summarise the variety of alternative procedures and acronyms that archaeologists may encounter in the literature.

In the context of rock art dating, the age of primary interest is usually the time since grains of quartz or feldspar were last exposed to sunlight (a process referred to as bleaching). The energy of the sun's rays is sufficient to evict electrons from the light-sensitive traps, which then begin to refill once the mineral grains are hidden from further light exposure. The population of trapped electrons steadily increases over time as a result of the energy absorbed from environmental sources of ionising radiation (the most important being uranium, thorium – and the daughter products in their radioactive decay chains – and potassium) within about 30 cm of the sample. Additional contributions are due to the decay of these radioactive elements inside the mineral grains and cosmic rays from outer space. The rate of delivery of all these sources of ionising radiation to the grains is called the 'dose rate', which is estimated from measurements made on site and in the laboratory.

To calculate an OSL age, two quantities must be estimated: the dose rate and the 'equivalent dose', which corresponds to the amount of radiation energy stored in the light-sensitive traps since they were last emptied by sunlight. The age is obtained by dividing the equivalent dose by the dose rate.

The equivalent dose is estimated from the intensities of the OSL (quartz) or IRSL (feldspar) signals measured in the laboratory using instruments that are sensitive enough to detect the faint emissions from individual sand-sized grains of quartz and feldspar (Jacobs and Roberts, 2007; Duller, 2008b). The most light-sensitive OSL traps are bleached more rapidly and completely than are IRSL traps, so quartz is the preferred mineral for dating of sediments – especially those exposed only briefly to sunlight. On the other hand, feldspars can store much larger doses than quartz and are thus capable of dating much older events, provided that suitable IRSL signals are selected for dating (Li *et al*., 2014).

In their pioneering study, Huntley *et al*. (1985) used several replicates (aliquots) of a sample to determine its equivalent dose, but these multiple-aliquot procedures have given way to single-aliquot procedures over the past two decades (Roberts *et al*., 2015). These require only one aliquot to yield an estimate of the equivalent dose, so problems that may affect the accuracy of an OSL age (e.g., insufficient bleaching and sediment mixing) can be addressed before final age determination by examining the internal consistency of equivalent doses for each sample. Each aliquot might consist of thousands of silt-sized grains or just one sand-sized grain, with single-grain dating representing the extreme case of a single aliquot. Each grain in a deposit may have a unique bleaching and burial history, so a single grain is the smallest meaningful unit of analysis in OSL dating. Single-grain procedures were developed originally by Lamothe *et al*. (1994) and Murray and Roberts (1997) to obtain equivalent doses for individual sand-sized grains of feldspar and quartz, respectively. Single-grain dating avoids the uncertainties involved in measuring several grains simultaneously, whereas single aliquots composed of multiple grains may incorporate grains with mixed ages.

2. Applications to rock art

OSL dating has been applied only rarely in rock art contexts, in part because the method is best suited to sedimentary deposits and not the typical constituents of paint or the rock surfaces at engraving sites. Some attempts have been made to constrain the age of rock paintings and engravings with OSL dating, and these are grouped below under two broad headings: dating of associated sediments and dating of rock surfaces.

a. Dating of associated sediments

The most straightforward application of OSL to rock art is through dating of sediments that have partly or completely buried a painted or engraved rock surface. In such cases, standard OSL dating methods can be used to estimate the time of the deposition of the juxtaposed sediments and, hence, a minimum age for the rock art. In northern Australia, for example, circular engravings (pecked cupules) on the wall of Jinmium rock shelter were traced below ground level to a depth of about 1 m, from where an engraved sandstone fragment was also recovered. These engravings were thought to be at least 50,000–75,000 years old, based on TL dating of quartz grains from the covering sediments (Fullagar *et al*., 1996). This controversial finding was subsequently overturned by accessing the most light-sensitive traps in quartz using optical stimulation. OSL dating of single aliquots and individual grains, together with radiocarbon dating of charcoal fragments, showed that the rock engravings were buried by sediments within the last 10,000 years (Roberts *et al*., 1998).

Single-grain OSL dating was also used at Lapa do Santo in central Brazil, where an anthropomorphic figure was found pecked into bedrock at the bottom of the 4 m-deep archaeological deposit. OSL dating of the overlying sediments revealed that the petroglyph was buried between about 11,700 and 9900 years ago (Neves *et al*., 2012).

Possibly the earliest OSL application to rock art was the direct dating of the Uffington White Horse – a large, stylised equine figure carved into the chalk hills near Oxford in the UK (Rees-Jones and

Tite, 1997). It was constructed by cutting a series of ditches and filling them with chalk, on to which silty sediments were occasionally washed and then buried by chalk added during repairs. The OSL and IRSL ages indicated that the hill figure was first made about 3000 years ago, during the late Bronze Age or early Iron Age. This dating study was conducted before the advent of single-aliquot procedures and silt-sized grains are, anyway, not amenable to single-grain analysis. Nevertheless, agreement between the multiple-aliquot OSL and IRSL ages suggests that the sediments were exposed to sufficient sunlight at the time of deposition, given the differential bleaching rates of quartz and feldspar (Roberts, 1997).

Another unusual OSL application to rock art is the dating of mud-dauber wasp nests formed on top of Aboriginal rock paintings in the Kimberley region of northern Australia (Roberts, 1997; Roberts *et al*., 1997). The nests ranged from a few millimetres to several centimetres in size and consisted of mud gathered by wasps from the margins of local streams and pools and carried to the rock shelters. The quartz grains would have been exposed to sunlight during mud collection, transportation and nest construction, and the OSL traps steadily refilled after the grains were concealed inside the nest. Several late Holocene nests were dated by Roberts *et al*. (1997) and an age of about 16,400 years obtained for the residual stump of an indurated nest overlying the head-dress of a faded anthropomorphic painting. The latter was the first OSL age ever published for single grains of quartz and showed the feasibility of dating very small samples.

This Pleistocene age has been widely debated, largely questioning the stratigraphic relationship between the nest and the art, rather than the OSL age of the nest itself (Bednarik, 2002; Aubert, 2012; David *et al*., 2013). A pattern of similarly old ages for rock paintings is ultimately required to settle this issue. To this end, a series of single-grain OSL ages were reported recently for late Holocene nests overlying and underlying rock paintings in the same region, as well as a minimum age of about 16,000 years for a yam-like motif (Ross *et al*., 2016). Mud-wasp nests can survive for at least 30,000 years, as shown by OSL and radiocarbon dating of embedded quartz and pollen grains, respectively (Yoshida *et al*., 2003), so older paintings could be dated if overlain by suitable nests.

b. Dating of rock surfaces

OSL dating of rock art has also been extended to painted or engraved rock surfaces, although only two case studies have been published thus far: Greilich *et al*. (2005) and three related reports (Chapot *et al*., 2012; Sohbati *et al*., 2012; Pederson *et al*., 2014). The use of OSL dating in such contexts can trace its roots to TL dating of limestone building blocks in ancient Greece (Liritzis, 1994) and IRSL dating of quartzite pebbles from a Palaeolithic site in central Siberia (Richards, 1994). In these cases, the age represents the time since surface grains on the undersides of the stones were last exposed to sunlight – that is, when the Mycenean wall was built and the river pebbles were deposited. For dating of exposed surfaces, a critical step is to establish how far daylight penetrates into the rock, just as Roberts *et al*. (1997) used a ‘microstratigraphic’ approach to show that grains in the outermost 3 mm of a mud-wasp nest may be bleached. Sunlight can penetrate up to 1 mm through limestone and several millimetres through quartz, with a strong attenuation of light with increasing depth (Laskaris and Liritzis, 2011; Liritzis, 2011).

The most straightforward approach is to extract grains of quartz or feldspar from close to the surface, as they will have been bleached to the fullest extent. At Canyonlands National Park in Utah, successive rockfall events have resulted in boulder accumulations at the foot of the Great Gallery rock art panel and some of these boulders retain traces of Barrier Canyon style paintings (Pederson *et al*., 2014). Using multi-grain, single-aliquot dating procedures, Chapot *et al*. (2012) obtained an OSL age of about 890 years for quartz grains extracted from the outermost 1 mm of the buried surface of a boulder preserving pigment of broken figures. The boulder was lifted at night to avoid bleaching of the grains during sampling, and the sediment grains directly beneath the boulder were also collected for dating. These gave an age of about 815 years using single-grain OSL procedures, while a leaf squashed against the underside of the boulder yielded a calibrated radiocarbon age of around 930 calibrated years (with all ages expressed in years before AD 2010 for consistency). These three ages are within analytical error of each other and indicate that the boulder fell on to the ground around 800–900 years ago, representing a minimum age for the rock painting on its buried surface.

The same boulder was also examined by Sohbati *et al*. (2012), with the aim of measuring the bleaching profile through the outer few millimetres of its buried surface to estimate the duration of daylight exposure on the cliff wall prior to collapse and burial and, thereby, a maximum age constraint for the rock art. Quartz grains were extracted in 12 successive layers of 1 mm thickness and analysed using multi-grain, single-aliquot OSL procedures. From the shape of the bleaching profile, they concluded that the surface of the boulder had been exposed on the cliff face for about 700 years before becoming detached. This gives an age bracket for the painted figures on the fallen boulder of about 900–1600 years ago, when combined with the minimum ages of Chapot *et al*. (2012).

This study in Canyonlands National Park illustrates the potential for dating the exposure history of rock surfaces using OSL, but the use of bleaching profiles involves several assumptions that require validation. The approach assumes that the OSL traps are refilled at a negligible rate during daylight exposure, and this limits the effective time range to the last few millennia. The exact mathematical form of a bleaching profile also depends on many site- and sample-specific factors that affect the transmission of light: these include the spectrum, intensity and duration of sunlight exposure, the optical properties of the mineral grains and any other materials present on or within a few millimetres of the rock surface (such as organic or mineral accretions and discolourations) and the effects of weathering and removal of bleached grains from the rock surface. Consequently, a calibration sample is needed of the same rock type with a known exposure history – Sohbati *et al*. (2012) used rock exposed in the wall of a local road-cut made 80 years earlier – but such samples may not be readily available. Multiple exposure events may also be inferred from the shape of the bleaching profile (Polikreti, 2007; Freiesleben *et al*., 2015), provided there is a suitable means of exposure calibration.

The extraction of grains from the bleached zone of a rock surface necessarily involves some degree of physical destruction of the sample. An alternative approach is to image the OSL or IRSL emitted from the surface of an intact slice of rock using a sensitive charge-coupled device (CCD). The use of such instruments offers some benefits for dating, such as measuring grains in their original spatial locations, but reliable estimates of the *in situ* dose rates are challenging to obtain (Roberts *et al*., 2015). A creative use of CCD imaging in the context of rock art dating is the application to the pre-Columbian Nasca lines in southern Peru (Greilich *et al*., 2005; Greilich and Wagner, 2006). These geoglyphs were made by removing the dark brown stones of the desert pavement to reveal the underlying pale silt. The stones were then placed on the ground, shielding their bottom surfaces from further light exposure, so dating the undersides of the stones should indicate when they were last moved during construction of the geoglyph (provided they have not been disturbed since).

Several stones were carefully lifted at night and cores drilled into their shielded surfaces, after which the stones were put back into their original positions. The cores were then cut into 2 mm-thick slices and the uppermost slice used for high-resolution CCD measurements. The sampling procedure used in this pilot study is no less destructive than that used in Canyonlands National Park, but an intact stone could, in principle, be imaged using a CCD without the need for coring. Six stones were examined at the same spatial resolution as an individual grain of sand (100 × 100 μm). A wide spread of ages was obtained for the feldspar grains, from as young as 50 years ago to more than 50,000 years ago for two of the stones, which highlights the need to date several stones for such structures (Greilich *et al*., 2005). The other four stones yielded a tight cluster of ages, with the most reliable estimates lying between about 1300 and 2100 years ago; these ages were not corrected for anomalous fading (the leakage of electrons from IRSL traps at a much faster rate than expected from kinetic considerations), which is ubiquitous in feldspars and gives rise to age underestimates unless corrections are made or non-fading signals are selected for dating (Li *et al*., 2014).

3. Future directions

OSL dating has played only a limited role in developing a timeline for rock art, with applications restricted largely to buried paintings or engravings that have been discovered during excavation. OSL can enhance its value to the field if procedures are developed for *in situ* application to a wider range of materials associated with rock art on the walls and ceilings of rock shelters and at open-air sites. Insects and birds construct sedimentary structures in rock shelters, and some of these overlie or underlie rock paintings – mud-wasp nests being one such example. Applications to biogenic sedimentary deposits are never likely to become commonplace, however, as modern sunlight will bleach grains in the outermost few millimetres. Consequently, samples amenable to OSL dating must be many millimetres thick, so that grains can be extracted from the light-safe inner portions, and sufficiently large samples are rare.

Further refinements to the bleaching profile approach for rock surfaces could provide some useful maximum age constraints for rock paintings and engravings, using CCD imaging technology to reduce the extent of site disturbance and sample destruction. It might also be feasible to estimate the time elapsed since a rock painting was last exposed to sunlight if the paint residue is sufficiently opaque to conceal the grains beneath from further sunlight exposure (Bednarik, 1996), but collecting the grains would involve destruction of the overlying art. Alternatively, opaque mineral precipitates or other accretions covering at least part of a painting or an engraving may have grains embedded within or beneath them that are suitable for OSL dating (Bednarik, 1996; Roberts, 1997) and that could be extracted without damaging the art. Liritzis *et al*. (2013) used the latter strategy to obtain a tentative age of about 3300 years for quartz grains extracted from the interface between a sandstone rock surface and the overlying calcite accretion at a site with numerous petroglyphs in southern Saudi Arabia. The age obtained from two multi-grain single aliquots represents the last time that the rock surface was exposed to daylight prior to precipitation of the 3 mm-thick calcite crust, but it does not provide a direct date for the petroglyphs as the quartz grains were collected from below the rock art.

While none of these suggested approaches are straightforward to implement, they may become less daunting with future technological and methodological advances in OSL dating (Roberts *et al*., 2015).

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