Analysis of lithic artefact microdebitage for chronological determination of archaeological sites

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Chapter six

In summary, the more varied and the more numerous the analogies that can be adduced, the more likely one is to find a convincing interpretation for an archaeological fact. The more numerous and the more detailed the parallels, the more likely one is to be able to assess the likelihood of a particular parallel being a significant one, and the greater the possibility of checking against the content and context of the archaeological material (Ucko and Rosenfeld, 1967: 157).

Conclusions and further considerations

Age determination of archaeological deposits has commonly been problematic. This has primarily been due to an insufficient understanding (or interest) by archaeologists of the dynamics and processes involved in sedimentary deposition. Greater consideration should be given to the possibility that archaeological deposits are bioturbated, and that post-depositional processes have been operative. Understanding the processes operating in archaeological deposits is fundamental for dating past events relevant to archaeology when excavating in pedogenic material. Each archaeological deposit is different, and the methodology devised in this research may be modified for site-to-site conditions.

This research has attempted to shift the reliance upon sedimentary quartz as the primary source of datable material for OSL age determination of archaeological deposits. Little is known of the movement of artefacts within sedimentary deposits, and even if the movement of artefacts is known, the age of the underlying sediment will only be relative to the position of the artefacts within the deposit. With the use of quartz microdebitage, the reliance on sedimentary quartz as the primary source for information relating to the age of archaeological deposits now has an alternative through direct OSL dating of the unheated quartz derived from the manufacture of lithic tools.

In this research, an attempt has been made to reconcile and understand the differences between the OSL ages of sedimentary quartz and microdebitage. This research has established that archaeologically relevant quartz artefactual material (microdebitage) can produce accurate and valid data regarding the timing of events within occupation deposits. However, this research did not provide sufficient evidence to fully
understanding the processes involved, due to the lack of sufficient data and the small sample sizes.

This research has been successful in identifying, isolating and determining an age for microdebitage. The methods proposed are valid and the possibility of continuing this research as routine is highly promising. The methods and techniques developed herein may be adopted for research into OSL age determination of archaeological sites through microdebitage dating and SEM combined or optical microscopy using large (500-1000 µm) microdebitage particles.

6.1 Discussion of results from the case studies

The case studies were chosen primarily for the extensive age determination programmes already undertaken, which allowed comparison of several methods of age determination with the results outlined in this research. Both archaeological sites are important for the understanding of human migration to northern Australia in the Pleistocene and Holocene.

In the case study of the age of two stratigraphic units from Jinmium rockshelter, it was found that the OSL techniques applied here encountered difficulties comparable to those reported in the existing literature on this site (Roberts et al., 1998a, 1999). The analyses showed that all the samples had palaeodose over-dispersions of well over 20%, which may be interpreted as the result of sediment mixing. The results show that the methodology developed here may be applied in the study of bioturbation and paedoturbation in archaeological deposits. In the Mushroom Rock West rockshelter case study, the microdebitage OSL age is 27,400 ± 2200 years (with an over-dispersion on the palaeodose of 0.1%) at a depth of 441 cm. The sedimentary quartz in the same level yielded an age of 34,000 ± 6000 (with an over-dispersion of 26%). This case (Laura 15 microdebitage) shows that the microdebitage, dated independently from the sediment, was not subjected to the same high percentage of over-dispersion. The ages of the two archaeological sites analysed are widely different, and this was also represented in the analysis of the microdebitage. None of the OSL age determinations of microdebitage was unrealistically outside the boundaries of pre-existing age control. This may be one of the indications of the validity of the experimental approach used here.
6.1.1 Microdebitage analysis

The analysis and OSL age determination of microdebitage used two distinct methods for particle surface morphology recognition. For OSL, the microdebitage was detected and isolated from sedimentary material with the aid of an optical stereomicroscope under red illumination. For particle sizes smaller than 500 µm in diameter, the particles were also observed under SEM prior to OSL analysis.

Under red incident light, deep shadows occur on the grains, and conchoidal fractures are the only features immediately diagnostic under those lighting conditions. Conchoidal fractures also occur on naturally broken grains. Although the incidences of broken quartz grains are a minority compared to the amount of microdebitage, this may be sufficient to make OSL analysis problematic. In the Mushroom Rock West samples, SEM was not used, and probably some of the particles in the smaller fraction (250-500 µm diameter) diagnosed as microdebitage may have been naturally broken grains. It was observed that under red illumination conditions, smaller grains (>500 µm in diameter) are difficult to assess for microdebitage, resulting in an error of identification between microdebitage and naturally occurring broken grains of ~40%. It is recommended that for the small size grains a SEM be used in conjunction with the optical stereomicroscopy. It was also observed that naturally broken grains were readily distinguishable from the microdebitage under stereomicroscopy when larger particles (500-1000 µm) were used for analysis. It is suggested that if a SEM is not readily available for analysis, than the samples used for OSL age determination of quartz microdebitage be in the range of 500-1000 µm in diameter.

Use of the new Risø OSL reader, which includes a mini X-ray generator (Andersen et al., 2003) as an alternative to the $^{90}$Sr/$^{90}$Y beta source, will avoid the problems encountered in delivering a beta dose from the source to particles of large size (500-1000 µm). Unlike the beta-source, the X-ray generator will deliver a uniform radiation dose to the entire volume of large particles, rather than to a dose gradient. This will allow the use of large particles to be used effectively with the OSL reader. However, the reader’s source still requires calibration for different size grains.
6.1.2 OSL age determination

OSL age determination on all samples of microdebitage was successful insofar that it has provided evidence that the technique used can be as reliable as using sedimentary quartz grains for age determination. In this research, at least one sample of microdebitage (Laura 15, 500-1000 µm size range) produced the lowest over-dispersion of any of the samples analysed, lending confidence to the accuracy of the palaeodose.

The OSL single-grain SAR technique works well for age determination of microdebitage. The modifications used for the dose rate (due to particle size and shape) and for the calibration of the beta source (due to particle size) did not produce any inconsistencies or anomalous results. The analysis and interpretation of the data from microdebitage has three major difficulties. The first is related to the optical microscopy of microdebitage within the 250-500 µm fraction. Misidentification of naturally broken grains as microdebitage may well lead to the possibility of including partially bleached grains that are mixed within the sample (theoretically, microdebitage does not suffer from partial bleaching). In this case, the minimum age model may be the most appropriate. The second relates to the possibility of a mixed-age sample, where the deposit suffers from bio-turbation. If the sedimentary material has mixed ages (and/or partially bleached grains), then there is the possibility that the microdebitage will be mixed as well. In this case (without knowing the nature of the sedimentation of the deposit), the minimum age may be used if the over-dispersion is over 20%. The third is related to the sample size, where the amount of microdebitage found in the sample, and the data remaining after OSL–SAR analysis, may be insufficient for accurate age determination. In general, the use of the OSL central and minimum age models applied to the sedimentary material in this research follows the ‘rule of thumb’ of 20% over-dispersion, as applied by Olley et al. (2004b).

6.2 Analysis of the methodology

In Table 3.1, the conceptual model was designed to take into consideration every possible variation in the methods used. The methods were tested and used in different combinations to find the most suitable. It was found that the model is best served by being modified to suit the individual needs and conditions of the investigation at hand. In the Jinmium case study, the microdebitage sample COOR 1/3 was exposed to a SEM
prior to OSL age determination, while all the other samples only used the SEM for information relating to the existence of microdebitage within the sample.

In the Mushroom Rock West case study, SEM was only used for roundness index analysis of the sedimentary quartz, while the microdebitage was discerned from the sediment by visual recognition under stereomicroscopy. It is believed that the use of SEM will allow a far better resolution for microdebitage recognition, especially for smaller (< 500 µm) particle sizes considering that the error of identification may be as high as 40% when using an optical stereomicroscope under dim red illumination.

The issue of sample size also has a bearing on the OSL results. While sedimentary quartz is plentiful in the samples, and the number of grains (data) retained after analysis can easily reach at least 100, the microdebitage found in the samples had a minimum of 5 to a maximum of 39 particles that have passed all of the SAR tests criteria. It is recommended that samples of microdebitage be sufficiently large as to produce at least 100 microdebitage particles with viable OSL data. This research was constrained by the size of the available bulk and laboratory samples.

6.3 Evaluation of the techniques used

In answering the questions posed as aims at the beginning of the thesis, the following provides a synthesis of observations for possible fine-tuning of the methodology.

a. Can quartz microdebitage be identified in archaeological deposits?

Microdebitage can be identified in both ambient light and under red illumination with stereomicroscopy. Under red illumination (for samples to be used for OSL age determination), particles in the size range between 250 and 500 µm diameter are not easily resolved, requiring the aid of SEM to discern between microdebitage and sedimentary quartz. The use of a preliminary analysis on a separate sub-sample in ambient light helps in identifying the sediment samples containing microdebitage.

b. Does quartz microdebitage have a discernable OSL signal, as natural quartz grains do?

The microdebitage OSL signal behaves similarly to that of sedimentary quartz grains, and is subject to the same problems as quartz. The only difference is that microdebitage is assumed to be not subject to partial bleaching. Because the
production of microdebitage from the manufacture of lithic tools is presumed to be
done mainly in daylight, this should allow for full bleaching of the OSL sensitive traps
within minutes. Microdebitage may also remain on the surface of the deposit until
buried, providing further opportunity for adequate exposure to sunlight. All of the
processes that produce an OSL signal in natural quartz grains are valid for quartz
microdebitage.

c. Can SEM and OSL dating be used in conjunction for microdebitage analysis?
The tests undertaken in Chapter 3 show that no adverse effects are measurable from
the OSL signal after exposure to SEM, provided that the electron beam is kept at, or
under, 10 keV. The exposure electron voltage of the beam controls the penetration of
electrons into the sample. If the beam is kept under 10 keV, the penetration is lower or
comparable to that of alpha radiation, which is usually eliminated from the quartz
grain surface by etching the grain with HF for ~40 minutes prior to OSL analysis.

The combined application of SEM and OSL provided new data from the analysis of
microdebitage from the Jinmium rockshelter. The OSL data is comparable to the
results obtained from the sedimentary material, and from other dating methods, and
the microdebitage OSL analysis did not show anomalies for the particles used for age
determination.

d. What advantages over existing techniques and models does direct age determination of
microdebitage brings to the study of archaeological deposits?
Microdebitage analysis has been used for many years as a method to assess areas of use
within archaeological sites. Spatial analysis is usually used in combination with
microdebitage concentrations to assess lithic manufacture areas as separate from areas
of use and retouch of the lithic tools. The addition of SEM to microdebitage analysis
allows a better resolution for quartz as a manufacturing raw material, which was partly
neglected because of the difficulty of discerning naturally broken quartz from quartz
micro artefacts under optical stereomicroscopy. The addition of OSL dating as a
technique for direct age determination introduces a new use for the analysis of
microdebitage.

The OSL age of microdebitage is derived from the last exposure of the artefact to
sunlight. Although bound within sediment, microdebitage is subjected to any post-
depositional processes that occur within the deposit. It may not necessarily be bound
to the stratigraphic sequence of the sediment, and may not behave in the same manner
as larger-sized artefacts. Microdebitage may be seen as being independent from the
sediment, providing an age that may or may not correspond to the sedimentary level; it
will produce a direct age for the archaeological event.

e. Can the combination of techniques be used routinely in archaeological investigations and
age determinations?
Provided the equipment for analysis of microdebitage is available, it is possible to
replicate any of the tests conducted in this research. The routine use of the
methodology proposed will enhance the availability of data concerning age
investigations of archaeologically relevant sedimentary deposits. Having sufficient
data will also allow the quantification of post-depositional processes by using OSL
dating of microdebitage as an independent source of information from that provided
by the sediments. Cross-examination with other dating techniques will further
enhance the resolution of these processes. Further, the application of the OSL
technique to crypto-crystalline materials (such as chert, siltstone, and, quartzite) merits
further consideration, as a means of widening the lithic microdebitage materials
dateable by OSL. New tests and protocols will have to be devised for any new
materials investigated.

6.3.1 Advances and advantages of using this model

This research has demonstrated that quartz microdebitage can be isolated from other
sedimentary material and that it has luminescent properties comparable to those of
quartz grains used routinely for OSL age determination.

The issues related to archaeology may be far reaching. To return to the overview from
Chapter 1 about age determination sedimentary deposits and artefacts; Richardson
(1992) and Villa (1982) applied the technique of conjoining lithic flakes to assess the
extent of vertical and lateral movement in the sediment, resulting in confusion about
the age of the occupation layers derived from radiocarbon dating. Their assessment of
vertical movement may well indicate the extent of stratigraphic integrity (or the lack
thereof), resulting in further inaccuracy of age determination.
The OSL technique combined with the isolation of quartz microdebitage is an advance over the age determination of sediment surrounding the lithic manufactured material. In this manner, the OSL technique can be used not only for age determination of sediments and heated quartz (hearth-derived heated stones and pottery), but also for unheated (or heated), identifiable micro-artefacts.

6.4 Reliability and applicability of the methods

The techniques used are mostly standard. The adaptation of quartz microdebitage to OSL dating has been the novel focus of this research, providing useful data to explore the possibility of using this method for any archaeological deposit. The method can also be used retrospectively, provided that sediment samples from archaeological deposits are still retained in conditions consistent with the use of OSL. Bulk samples of archaeologically relevant sediments (not etched with HF) retained by OSL laboratories can be reviewed using the techniques developed in this research, as the usual grain size used for OSL dating is smaller than the microdebitage which is, therefore, some times still available. This research has shown that samples retained in dark conditions may be used for OSL age determination of microdebitage many years after the archaeological investigation has ended. The addition of new information on the age of archaeological deposits based on microdebitage will refine the definition of the age of the artefacts.

This research is unlikely to be used for commercial applications because costs associated with this research are high and subject to the availability of the equipment needed. In recent years, advances in OSL equipment have reduced the cost so that several universities and other institutions around the world have dating laboratories. To distinguish between the activities and costs involved, the methodology may be divided into several, separate areas of research:

1. Cores of sediment for OSL dating can be taken at the time of excavation (including samples for high-resolution gamma spectrometry, or measurements made in situ with a portable spectrometer).

2. Microscopy to find microdebitage in white ambient light can be done by a team at the excavation site to maximise the likelihood of finding microdebitage in the cores used for OSL age determination.

3. In a darkroom, the archaeologists should be able to find microdebitage in the core sample, provided that the OSL protocols for lighting and sample cleaning are followed.
SEM is not necessary for microdebitage particles larger than 500 µm in diameter, which are easily distinguishable from naturally broken grains. For microdebitage particles smaller than 500 µm in diameter, SEM should be used if possible.

The microdebitage isolated for OSL dating should be sent to the OSL laboratory and analysed separately from the sedimentary quartz.

The remaining sediment sample should be dated by OSL.

This research is time-consuming, expensive, and should be restricted to those occasions where the archaeological deposits need clarification and better resolution, because of difficulties in the identification and interpretation of post-depositional processes.

6.5 Application of the methods to archaeological sites

This research has shown that the methods used for diagnosis of microdebitage and their age determinations have been generally successful. The combination of standard methods will allow application to any archaeological deposit, with the exception of sites in regions with modern glacial sediments. This limitation is due to the similarity between the surface morphology of glacier-crushed quartz grains surface and that of microdebitage. In archaeological terms, the microdebitage dated with the OSL technique on a single-grain basis posed some questions as to the origins of the material. Each particle of microdebitage may be the result of the same event, or the sample may be composed of multiple events being buried at the same time or being buried at different times and then mixed together at a latter date.

Although this research did not study in depth the possibility of investigating possible interpolation of different age populations within the same sample. It should be feasible, given sufficient data (i.e., larger amounts of microdebitage particles) to statistically examine whether the microdebitage palaeodoses belong to different palaeodose populations (e.g., Roberts et al., 2000a).

6.6 Further research

Further studies are required into the combination of microdebitage grain surface morphology and OSL dating analysis. These techniques can be gainful when applied to chronological and environmental reconstructions, and to improve the understanding of site formation processes and post-depositional disturbance.
During the analysis of quartz microdebitage, it was noted that particles of broken quartz and siliceous binder derived from sandstone were diagnosed as microdebitage. This microdebitage is derived from the manufacture of rock engravings on the surface of the rockshelter. Further studies will be undertaken to distinguish between this microdebitage and other material to provide an age relationship between the engraving events and the microdebitage from the sub-surface archaeological deposits. Use of the recently manufactured Risø OSL reader with a mini X-ray generator (Andersen et al., 2003) as an alternative to the $^{90}$Sr/$^{90}$Y beta source, will avoid the problems encountered in delivering a uniform radiation dose to particles of large size. Unlike the beta-source, the X-ray generator will deliver a homogeneous radiation dose to the whole volume of large particles, allowing large particles to be used effectively with the OSL reader.

The use of combined SEM/OSL techniques may also be useful in geology. By using SEM to recognise quartz grain surface features it is possible to determine the specific environmental provenance of different sediments. In combination, these techniques can be applied to a sample to isolate quartz grains originating from different environments, and use OSL to provide age estimates for different geological events that may otherwise be erroneously compounded in multiple-grain or single-grain studies that do not first segregate grains of different origin.

The application of the techniques developed in this thesis is to a large extent dependant on the type of information required by the investigator. With some modification, the techniques applied to microdebitage in this study can be used.

The ultimate implication for this research is to deliver a workable method for direct dating of microdebitage. Much of the applications in this research were developed by Fladmark (1982) — microdebitage analysis, Krinsley and Doornkamp (1973) — quartz grain environmental textures, and Murray and Roberts (1997) — single quartz aliquot OSL dating. The combined work of these scholars allowed the successful application of the methodologies discussed within this thesis.

Errare humanum est, perseverare autem diabolicum…