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Formation processes and context of complex stratigraphic features at the MSA archaeological sites of Pinnacle Point Site 5-6 and Mertenhof during MIS 3 in southern Africa

Abstract
The Middle Stone Age (MSA) is a crucial time period for the understanding of the origins and development of modern human behaviour and their expansion out of Africa into the rest of the world. There are numerous models that strive to explain the emergence of behavioural modernity, however these are inhibited by weak or poorly resolved evidence for modern human behaviour and occupation of many key MSA sites during Marine Isotope Stage 3 (MIS3).

Pinnacle Point Site PP5-6 (PP5-6) and Mertenhof Rock Shelter (MRS) are two MSA sites with rich evidence of modern human occupation during the MSA. These two sites contain complex stratigraphic features. Understanding the sedimentary context of these features, and their formation processes will provide a better understanding of human occupation of these sites during MIS 3, and provide a means for conducting similar analysis at other MSA sites.

Optical dating of single quartz grains was conducted at both sites, to provide chronological scaffolds for both the complex sedimentary features, and the evidence of modern human occupations during MIS 3 and into the Later Stone Age (LSA). Sediment analysis and geological observations of the sedimentary contacts were also conducted to better understand formation processes of specific stratigraphic features. These methods were used to create and test different hypotheses for formation of these complex features.

A total of 13 optical ages were obtained from the two sites. These were placed within their stratigraphic contexts to determine how and when the complex stratigraphic features were deposited in relation to surrounding archaeological stratigraphy.

The ages provided meaningful results for determining modern human occupation of MRS between ~51 ka and 22 ka ago, when compared to the artefact evidence contained within the stratigraphy where the optical ages were taken. In southern Africa this is the period that falls after the precocious complexity of the Howiesons Poort and Still Bay and before the appearance of the LSA. At PP5-6, the optical ages provided evidence of modern human occupation between ~31–21 ka. Further studies are required to better understand when the complex stratigraphic feature occurred at PP5-6. These ages, and the lack of mixing associated with them demonstrate occupation of these sites during the crucial later phases of MIS 3, and provide a temporal framework within which the archaeological material they contain can be interrogated. The data provided in this thesis can now be integrated into full site stratigraphy for an improved understanding of the site formation processes at both sites.

Degree Type
Thesis

Degree Name
BSc Hons

Department
School of Earth & Environmental Sciences
Advisor(s)
Zenobia Jacobs

Keywords
OSL dating, MIS 3, complex stratigraphic features

This thesis is available at Research Online: https://ro.uow.edu.au/thsci/159
Formation processes and context of complex stratigraphic features at the MSA archaeological sites of Pinnacle Point Site 5-6 and Mertenhof during MIS 3 in southern Africa

Matthew Paul Williams

A thesis submitted in partial fulfilment of the requirements of the Honours degree of Bachelor of Science (Advanced) in the School of Earth and Environmental Sciences, Faculty of Science, Medicine and Health.

University of Wollongong
2017
The information in this thesis is entirely the result of investigations conducted by the author, unless otherwise acknowledged, and has not been submitted in part, or otherwise, for any other degree or qualification.

Matthew Paul Williams

18 October 2017

M. Williams
Abstract

The Middle Stone Age (MSA) is a crucial time period for the understanding of the origins and development of modern human behaviour and their expansion out of Africa into the rest of the world. There are numerous models that strive to explain the emergence of behavioural modernity, however these are inhibited by weak or poorly resolved evidence for modern human behaviour and occupation of many key MSA sites during Marine Isotope Stage 3 (MIS3).

Pinnacle Point Site PP5-6 (PP5-6) and Mertenhof Rock Shelter (MRS) are two MSA sites with rich evidence of modern human occupation during the MSA. These two sites contain complex stratigraphic features. Understanding the sedimentary context of these features, and their formation processes will provide a better understanding of human occupation of these sites during MIS 3, and provide a means for conducting similar analysis at other MSA sites.

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Acknowledgements

First and foremost, I would like to express my deepest gratitude to my two supervisors, Zenobia Jacobs and Alex Mackay. You have both shown a supernatural amount of patience with me, always willing to answer my questions. The experience and knowledge that I have gained from you both has been invaluable. Zenobia, thanks for giving me the opportunity to travel to South Africa, and see an archaeological dig for the first time. It is a memory I’ll never forget.

I would also like to thank Terry Lachlan and Yasaman Jafari for helping me learn the ins and outs of the OSL instruments and sample preparation labs. I have learnt skills from you both that I am sure will come in very handy in the future.

Sally and Jake, you have both played a very large part in my success over the past 3-4 years. You have both driven me to perform and succeed at a level that I didn’t even believe was possible. It has been a hard but mostly fun journey, and it has been a pleasure sharing the honours room with you both this year, the countless coffees and endless conversations and encouragement.

Finally, but definitely not least, I would really like to express my love and gratitude to my wife Holly. It is only through your support, and your ability to encourage and believe in me that this year, and in fact this entire degree has been possible.
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Chapter 1 – Introduction
Chapter 1

1.1 Introduction

Over the past several decades, significant effort has been invested to determine when our species *Homo sapiens* evolved, where we evolved and when we began exhibiting traits of behavioural modernity consistent with the behaviour of humans today. A significant amount of this effort has elicited evidence of behavioural modernity as far back as ~100 ka, and showing varying amounts of evidence up until ~50 ka (Conard, 2005, 2008). Numerous models have been proffered to explain the emergence of behavioural modernity in *Homo sapiens*, of which three will be discussed in Section 2.2.

Marine Isotope Stage 3 (MIS 3), presents a period between ~60–25 ka, and is very important in understanding behavioural modernity, because it represents a window of time between sporadic displays of behavioural modernity and the preceding widespread use of the full complement of behavioural traits associated with behavioural modernity (McBrearty and Brooks, 2000; Conard, 2005; Lin et al., 2016). Interrogation of this crucial time period is inhibited by a paucity of evidence of human occupation, with most of the well-known sites in southern Africa containing little to no evidence of occupation, or reduced or no sedimentation during MIS 3 (Brown et al., 2009; Brown, 2011; Brown et al., 2012; Mackay et al., 2014a; Mackay et al., 2014b; Karkanas et al., 2015; Mackay et al., 2015; Mackay, 2016, 2017 unpublished).

In this project two southern African MSA sites, Mertenhof and PP5-6 (Figure 1.1), with rich evidence of modern human occupation during the MSA, are interrogated using optically stimulated luminescence (OSL) dating, geological interpretations and sedimentary analysis to determine stratigraphic contexts pertaining to complex stratigraphic features. In this chapter, the aims, significance and structure of the investigations conducted in this thesis will be discussed.

1.2 Aims

This project had 5 primary aims:

- Describe and identify complex stratigraphic features in the field using geological techniques.
- Formulate and test hypotheses for how these features formed, and their context within the site they occupy.
• Determine ages of sediment samples using single-grain OSL dating to constrain when these features formed.
• Provide a more robust chronology for human occupation of rock shelters during MIS 3.
• Integrate age and field data to augment existing understanding of site formation processes at both sites.

The first aim focuses on data collection using geological field techniques, such as field sketches, *in-situ* analysis of sedimentary relationships and characteristics of complex sedimentary features at PP5-6, and a similar virtual approach from data collected at MRS. The second aim requires abstract thinking, to determine how these complex features were deposited, when they were deposited and what their relationship might be with respect to other stratigraphic layers and aggregates of each site. The third aim involves the construction of a *chronological framework* for the assessment and interpretation of features and their surrounding stratigraphy, as well as providing a means to validate *stratigraphic contexts* for site formation processes at each site. The first three aims are used to help fulfil aims four and five.

*Figure 1.1:* Map of the two study sites, MRS and PP5-6, with respect to Cape Town.
1.3 Significance

Evidence of human occupation and behavioural modernity during MIS 3 is sparse and inconsistent – much more so than in the phases that precede and follow it (Mackay et al., 2014a). This project will provide some of the first absolute ages for human occupation at MRS, whilst contributing to the understanding of site formation processes during this little understood period of the MSA. These chronological results can be compared to artefact evidence discovered at MRS, to contribute to the debates on behavioural modernity during MIS 3. At PP5-6, extension of the existing chronological frameworks will augment understanding of human occupation of the site, and can later be compared to the artefacts and anthropogenic sedimentary structures discovered within and around the complex stratigraphic feature. The results will help drive future excavation efforts, and provide a framework for how to best conduct analysis of complex stratigraphic features at other MSA sites.

1.4 Project structure

Chapter 2 will provide background information on the current debates surrounding behavioural modernity, the complications arising from the often-weak evidence of occupation and behaviour during MIS 3, and the importance of understanding sedimentary relationships. Chapter 3 presents the different measurement, observational and analytical methods used to untangle the complex sedimentary relationships at MRS and PP5-6. Chapters 4 and 5 present both study sites, MRS and PP5-6 respectively, the hypotheses created to explain the complex sedimentary contexts, and the results of interrogation of these contexts using sediment analysis and OSL dating. Chapter 6 provides the chronological ages, and how these relate to the hypotheses proposed in Chapters 4 and 5. It also provides a discussion about what these ages and the results of the hypotheses mean for enhancing understanding of site formation processes, and a better understanding MSA archaeological sites during MIS 3.
Chapter 2 – Background
Chapter 2 – Background

2.1 Introduction

Our own species, *Homo sapiens*, have a unique behavioural range – building more elaborate and complex structures, information systems and social practices that surpass those of any other species that have previously existed in the past or present (Conard, 2005). *Homo sapiens* are generally portrayed to be ‘unique’ in our possession of high levels of cognitive brain function which led to linguistic and reasoning capabilities beyond those of any other species (d'Errico, 2003; Zilhão, 2007; Conard, 2010; Zilhão et al., 2010).

The current understanding is that anatomically modern humans (AMH) evolved in Africa more than 190 ka (White et al., 2003; McDougall et al., 2005; Oestmo et al., 2014; Wilkins et al., 2017) and possibly even as early as ~315 ka, based on fossil and stone tool evidence from Morocco (Hublin, 2017) and recent genetic evidence (Schlebusch et al., 2017). However, the early archaeological record of AMH in Africa has been determined to be little different from those records associated with species such as the Neandertals which became extinct with the expansion of AMH into Europe (d'Errico, 2003; d'Errico et al., 2008; Zilhão et al., 2010; Muller et al., 2017). It thus seems possible that the evolution of our anatomy and behaviour followed different pathways.

Furthermore, the major pulse of human expansion from Africa only occurred within the last ~100,000 years (Mellars, 2005, 2006; Higham et al., 2011; Clarkson et al., 2017), meaning our species was constrained to Africa for up to ~200,00 years before rapidly colonising the rest of the world – and eliminating or incorporating other species of hominin. The past 100,000 years also witnesses a marked increase in the production and utilisation of personal ornamentation, art, symbology and complex manipulation of material such as heat treatment and advanced lithic technologies and traits, many of which seem unique to our species (McBrearty and Brooks, 2000; Henshilwood et al., 2001; d'Errico, 2003; Conard, 2005; d'Errico et al., 2005; d'Errico and Henshilwood, 2007; Conard, 2008; Henshilwood et al., 2009; Henshilwood et al., 2011; Henshilwood, 2012).

2.2 Behavioural modernity in early humans

Mismatches in the timing of *Homo sapiens* biological appearance, behavioural evolution and their subsequent expansion out of Africa all contribute to a key debate in the archaeological and palaeoanthropological community. The cultural evolution of modern humans, coined the modernity debate, focusses on patterns of change in the development of
material culture during the Middle Stone Age (MSA) of Africa, and moving into the Later Stone Age (LSA) somewhere between 40-20 ka ago (Opperman, 1996; Ambrose, 1998; Mercader et al., 2012; Villa et al., 2012). The current, widely accepted view, is that behavioural modernity evolved during the MSA, and that the onset of the LSA delimits the point when the full spectrum of behaviourally modern markers became entrenched in our species. However, understanding how behavioural modernity arose in modern humans is complicated by competing models of behavioural modernity, and a critical weakness is the archaeological record during MIS 3 – the key period in which the MSA ends and the LSA emerges (Mitchell, 2008; Mackay, 2016).

2.2.1 Models of behavioural modernity

There are numerous models on the emergence of behavioural modernity in our species, of which three are briefly considered here. Other models, such as the multiple species model (d'Errico, 2003; Zilhão, 2006, 2007; Zilhão et al., 2010; Villa and Roebroeks, 2014) and the mosaic polycentric model (Conard, 2005) are not discussed.

2.2.1.2 The ‘demographic’ model

The demographic model argues that behavioural modernity arose in response to increases in population size (Henrich, 2004; Powell et al., 2009). Fluctuations in the size of a population impact its chances of discovering and sustaining complex behavioural variants (Henrich, 2004; Powell et al., 2009). In this view, Homo sapiens were always capable of complex behaviours, but did not display them until population size began to increase. The transition from the MSA to the LSA represents some threshold value in population size and cultural complexity.

2.2.1.3 The ‘revolution’ model

The second model of behavioural modernity argues that cognitive complexity arose through rapid biological evolution or mutation of the human brain at the start of the LSA, ~40-50 ka ago (Klein, 1995). In this model, markers for behavioural modernity should not occur in any significant quantity until after this time (Klein, 1995; Tattersall, 2009; Klein, 2013). Proponents of this view also argue that rapid emergence of behavioural modernity is what led to our species dispersing out into Europe and much of the Old World during the Upper Palaeolithic (Hoffecker, 2009; Klein, 2013).
2.2.1.4 The gradual accumulation model

The gradual accumulation model views human behavioural modernity as the result of a gradual process of accumulating ‘modern’ behavioural traits over tens of thousands of years from when our species first emerged (McBrearty and Brooks, 2000; Henshilwood et al., 2003; Hublin, 2017). This model allows for early examples of complexity – such as that witnessed in the Still Bay (SB) and Howiesons Poort (HP) industries during the MSA – but ultimately culminating with the transition to the LSA (Henshilwood et al., 2001; Marean et al., 2007; Watts, 2009; Henshilwood, 2012).

2.3 The wealthy southern African record

A significant amount of the understanding of AMH evolution, and the emergence of behavioural modernity comes from past and present excavations and studies in southern Africa. This geographical focus has been the driving force behind the models presented in section 2.2. This section will discuss some of the key archaeological finds and evidence in this geographical area, including some of the knowledge gaps.

2.3.1 The south of southern Africa

Significant effort has been invested in uncovering our species origins through archaeological studies focused on the southern cape of South Africa, particularly on the coastal regions. Archaeological excavations conducted at Pinnacle Point 13B (PP13B; Figure 2.1), revealed a long but discontinuous record of occupation dating as far back as 166 ± 9 ka ago starting during MIS 6, with evidence of behavioural modernity in the form of pigment use, symbolism, advanced bladelet tools and heat treatment of lithics. PP13B was occupied intermittently from ~166–90 ka ago, at which point the cave was blocked for human occupation due to dune intrusion (Karkanas and Goldberg, 2010; Marean, 2010; Marean et al., 2010; Karkanas et al., 2015). To the east of PP13B, excavation of PP5-6 unveiled evidence of sporadic small group human occupation starting from ~90 ka ago (during MIS 5) to denser occupation throughout MIS4 and MIS 3 up to ~51 ka (Karkanas et al., 2015). Little to no evidence for occupation thereafter has so far been uncovered and/or dated.

Blombos Cave (BBC) (Figure 2.1) is another site that has provided important insight into behavioural modernity (d’Errico et al., 2001; Henshilwood et al., 2001; Jacobs et al., 2003; d’Errico et al., 2005; Jacobs et al., 2006b; Henshilwood et al., 2009; Henshilwood et al., 2011). Studies at BBC have presented evidence of behavioural modernity through the storage of ochre in shells as far back as ~100 ka, along with re-engraved ochres and bead production dated to
~75 ka ago (Henshilwood et al., 2011; Henshilwood, 2012). Other significant south coast sites include Klasies River Mouth (KRM), Klipdrift Shelter (KDS), Die Kelders (DK) (Figure 2.1) and Nelson Bay Cave (Volman, 1984; d’Errico and Henshilwood, 2007; d’Errico et al., 2012; Henshilwood et al., 2014). All of these sites have rich archives from >100 ka to ~60–50 ka, but little to no signs of occupation between ~50–25 ka.

Figure 2.1: Locations of some influential archaeological sites in southern Africa. PP = Pinnacle Point PP5-6 and PP13B, DRS = Diepkloof Rock Shelter, BBC = Blombos Cave, KRM = Klasies River Mouth, SC = Sibudu Cave, NBC = Nelson Bay Cave, MRS = Mertenhof Rock Shelter, KDS = Klipdrift Shelter, BPA= Boomplaas, PL8&1 = Putslaagte 8 and Putslaagte 1, KKH = Klein Kliphuis, KFR = Klipfonteinrand and Die Kelders (DK).

2.4.2 The eastern southern African record

Sibudu Cave (Figure 2.1) provides a perspective of MSA occupation by AMH on the eastern side of southern Africa, located slightly inland on the banks of the Tongati river. The site has a long MSA occupation history shown by the SB and HP complexes found within its stratigraphy, as far back as >70 ka (SB industry) (Wadley and Jacobs, 2006; Wadley, 2007; Lombard, 2008; d’Errico et al., 2012). This evidence of advanced tool working, supports models of behavioural modernity much older than the ~50 ka revolution model (Klein, 1995; Klein, 2013). Sibudu Cave also contains a rich MIS 3 archaeological record, with evidence of hearth structures containing high frequencies of cemented ochre and powdered ochre during the post-Howieson’s Poort (Wadley, 2013; de la Peña and Wadley, 2017).
2.4.3 The western southern African record

Diepkloof Rock Shelter (DRS) (Figure 2.1) located on the western side of southern Africa provides evidence of advanced tool industries, predominantly those of the SB and HP industries linked to behavioural modernity in AMH. Initial ages for the SB industry of ~ 72 ka, and the HP industry of ~64–59 ka were proposed by Jacobs et al. (2008) and later confirmed by Jacobs and Roberts (2015). The HP industry at Diepkloof is split into three components, the upper, middle and lower HP. The HP industry at Diepkloof exhibits technologies that have been defined as markers for behavioural modernity, such as the selection and movement of production materials long distances, production of blades by marginal soft hammer percussion, manufacturing of geometric backed pieces and the use of adhesives (Porraz et al., 2013). Also of note is the presence of engraved ostrich shells, which are present from ~100–60 ka ago, but disappear between ~60–50 ka, not reappearing until the Holocene (Texier et al., 2010). The post-HP at Diepkloof is characterised by unifacial point production, and dated to between ~55.4 ± 2.0 ka (Jacobs et al., 2008) and ~52 ± 5 ka (Tribolo et al., 2013). However, apart from an isolated age of ~43.6 ± 1.6 ka, Diepkloof has an extremely weak habitation signal after ~50 ka ago until ~20 ka ago (Jacobs and Roberts, 2017).

Another archaeological site in this region, is Elands Bay Cave (EBC) which has two representative phases of occupation; the early MSA phase, and the MSA to early LSA phase. Of particular interest at this site is the post-HP assemblages indicating that occupation of EBC occurred during MIS 3. The post-HP assemblages at EBC consist of discoidal reduction of bladelets, bifacial and truncated blade technology dating to between ~39–30 ka (Porraz et al., 2016).

Klein Kliphuis (KKH) also contains evidence of post-HP occupation during the late MSA from bifacial blades, dating to ~33.3 ± 1.3 ka ago with OSL dating, however the density of these finds during most of the MIS 3 period, between ~50–20 ka are of extremely low density, when compared to those found between ~60–50 ka at the beginning of MIS 3 (Jacobs et al., 2008; Mackay, 2010).

2.4.4 Inland southern African record

Compared to the coast and coastal fringes of southern Africa, the inland regions are understudied (Mackay, 2016). There appears to have been a trend of study that focuses much more on the rich savanna, grassland and fynbos biomes that make the Cape Floristic Region (CFR; Figure 2.2), which is located on the very southern tip of South Africa, is one of the...
richest regions for biodiversity in the world (Marean, 2010). The more arid to semi-arid landscapes, which are not contained within the CFR, have been sorely neglected. Considering that one of the markers for behavioural modernity within AMH is the ability to adapt to different environmental conditions, study of these environments is equally important. Some excavation is underway at Mertenhof Rock Shelter (Figure 2.1) to determine linkages between in-land arid environments and the coast (Mackay, 2016; Schmidt and Mackay, 2016), which provides evidence of advanced tool industries (SB and HP), however thus far, no chronometric dating has been conducted to substantiate these observations.

Sites such as Boomplaas and Klipfonteinrand have SB and HP industries, however evidence of occupation ceasing completely after the HP industries, with distinct lack of engraved pieces and advanced tools, fuelling debates on the trajectory of behavioural evolution in southern Africa (Mackay et al., 2014a), and represents a trend of substantially decreased occupation of caves and rock shelters in this region during ~50–25 ka (Mackay et al., 2014a).

Other inland sites include Putslaagte 8 (PL8) which has evidence of weak pulses of occupation during the post-HP of MIS 3 (Mackay et al., 2015), and differs from the finds at Putslaagte 1 (PL1) located ~2 km away, which suggest occupation of the PL1 was relatively consistent during MIS 3. This difference has been attributed to re-organisation of land use during MIS rather than complete abandonment of the region (Mackay et al., 2014b).

Figure 2.2: The Cape Floral Region (CFR), outlined in red, represents an extremely biodiverse region covering a very small area.
2.4 South Africa and Marine Isotope Stage (MIS) 3

Complications begin to arise when using the southern African dataset to test the behavioural models outlined in Section 2.2. Early MSA has limited evidence for ornaments or complex tools, though there is the ochre processing at Blombos Cave and the heat treatment at Pinnacle Point. Arrival of the SB brings complex tools, engravings and heat treatment. This is sustained during the HP with engraving systems becoming more elaborate and widespread (Texier et al., 2010; Henshilwood et al., 2014). Much of this evidence seems to dissipate into early MIS 3 during the post-HP, with arguments for declines in technological complexity (Soriano et al., 2007). While this evidence re-emerges with the LSA (Villa et al., 2012) we currently know very little about behaviour in the critical intervening phase. This complicates, among other things, assessment of the models outlined in Section 2.2. Specifically, there is a consistent paucity of available human occupation data during MIS 3 during which the MSA ends and the LSA emerges (~60-25 ka: Siddall et al. (2008)). MIS 3 is central to all models that were outlined. The gradual accumulation (Section 2.2.1.4) model implies constant increases in complexity during MIS 3 (Henshilwood et al., 2001; Marean et al., 2007; Watts, 2009; Henshilwood, 2012). The revolution model (Section 2.2.1.3) suggests little complexity in behaviour during the MSA components of MIS 3 and a sudden increase in complexity during the LSA (Klein, 1995; Tattersall, 2009; Klein, 2013). The demographic model implies continuous tracking of complexity with respect to population size through MIS 3 (Henrich, 2004; Powell et al., 2009).

Many of the major sites in southern Africa have little to no evidence of sedimentation and human occupation between ~50-25 ka. This includes Blombos Cave, the Pinnacle Point cave complex, Klasies River, Nelson Bay Cave, Diepkloof, Elands Bay Cave, PL8, PL1 and many more. Where sites do exhibit evidence of sedimentation and occupation during this period, the sedimentation rates tend to be slow (Mackay, 2016, 2017 unpublished). In sites with smaller sedimentary profiles, exacerbation of bioturbation can serve to limit confidence in the relationship between the sediment and the cultural archaeological data it contains (Mackay, 2010, 2017 unpublished). Due to there being so few sites with substantial occupation in this period, and because those that were occupied often have very little evidence, it is essential to our understanding of human behavioural evolution that we develop and apply effective methods for understanding site formation, particularly in MIS 3.
2.5 Importance of sediment and sediment context in archaeological analysis

Sediment and sediment context are crucial in understanding the site formation processes that result in the preservation of archaeological sites and the evidence they contain. Understanding how sediment was deposited, when the sediment was deposited and where the sediment came from can provide crucial context for disentangling how the site formed and what it means for the debates regarding the evolution of modern human behaviour generally, and more specifically to the occupational history of southern Africa (Goldberg and Berna, 2010).

Sediment at archaeological sites can be deposited through geogenic, biogenic or anthropogenic processes; meaning that they can be derived through geological, animal/plant and human related means (Goldberg and Sherwood, 2006). Determining the context of these sediment deposits can be just as important as looking at the artefacts discovered at the site being studied (Goldberg and Sherwood, 2006). Detailed analysis of sediment at some of the key sites in the modernity debate in southern Africa, such as the Pinnacle Point cave complex, Blombos Cave, Sibudu Cave to mention a few, has provided invaluable contributions to the modernity debate (d'Errico et al., 2005; Backwell et al., 2008; Henshilwood et al., 2011). These contributions come from unravelling the sediment context with respect to depositional mechanisms, stratigraphic relationships, time of deposition of the sediment using verified dating techniques and differentiating between geogenic and anthropogenic sedimentation or bioturbation. For example, at Sibudu Cave, intensive study of the complex sediment stratigraphy at the macro and microscopic levels confirmed that dominant sources of sedimentation at the site were anthropogenic, with little or no water action. Geochemical analysis of the sediment determined that the major component of the anthropogenic sediments was ash, indicating evidence of intensive human occupations (Wadley and Jacobs, 2006; Wadley, 2007; Backwell et al., 2008; d'Errico et al., 2008). OSL dating was used at Sibudu Cave to interrogate the sediment to determine when these occupation events occurred, isolating three of the youngest periods and identifying two significant sedimentary hiatus’s (Wadley and Jacobs, 2006; Wadley, 2007). Similarly, at Blombos Cave, OSL dating was used to determine modern human inhabitancy of the site during high sea stands based on evidence of sedimentation during these periods, by placing the periods of sedimentation or lack thereof in context with the archaeology (Jacobs et al., 2006b). At PL8, in the vicinity DRS and MRS, OSL dating was used to determine the extent of post depositional mixing, and its impact on stratigraphic integrity. It was discovered that low sedimentation rates, and pulses of more
intense occupation at the site led to significant mixing within the sedimentary profile during MIS 3 between 33.2 - 41.5 ka (Mackay et al., 2015).

Understanding that sedimentation can be caused by human occupation of a site, such as those created from hearth structures, has provided crucial evidence of habitation where physical artefacts are lacking in the sediment profile (Goldberg and Berna, 2010; Goldberg and Macphail, 2012; Goldberg et al., 2017). Alternatively, understanding the context of sedimentation at known MSA sites can shed light on why sites may have been abandoned for long periods of time, due to palaeoclimatic variability such as those that occurred <90 ka at PP13B (Karkanas and Goldberg, 2010; Marean, 2010; Marean et al., 2010).

The law of superposition dictates that deposits lower down in the sequence are older than deposits higher in the sequence (Lyman et al., 1998). Whilst generally in undisturbed sedimentary facies, this can be held true, it does not consider post-depositional alteration. Human interaction, animal bioturbation, tectonic activity and environmental interactions are broad examples of post depositional processes that can impact the preservation of archaeological sites (Goldberg and Sherwood, 2006; Shahack-Gross, 2017). Detailed study of the sedimentary facies at archaeological sites can help disentangle complex stratigraphic features such as deformation, cut-and-fill and erosional truncations (Shahack-Gross, 2017; Stephens et al., 2017), and place them within the context of the archaeology at the site being studied.

Interrogating the sedimentary record at sites that exhibit possible evidence of human occupation during MIS 3 can contribute to the understanding of modernity by shedding light on site occupancy during this poorly understood period of the MSA.

2.6 Sedimentary complexities from depositional and post-depositional sources

Understanding the types of processes that can affect the integrity of stratigraphic layering at archaeological sites should not be discounted if one strives to provide the most holistic picture of events in time at the site being studied.

Post-depositional alteration comes in multiple forms including: sediment mixing, deposition or removal through erosional cut-and-fill features (rills/channels), anthropogenic trampling, burial or waste disposal pits and by bioturbation from burrowing or root systems, which can be common depending on where the site is located geographically (Stephens et al., 2017); mass-movement or mass wasting of the sediment profile if situated on a hillslope (Karkanas and Goldberg, 2010; Karkanas et al., 2015; Shahack-Gross, 2017; Stephens et al.,
Mixing may also be caused by roof-spall events, caused by erosion of the overhanging cave or rock shelter. Roof-spall can often be large fragments (Karkanas et al., 2015), and as they fall and strike unconsolidated sediment, this sediment can be displaced out of context of the archaeology of the site.

To delve further into the understanding of these events, a range of techniques can be employed such as: dating of the sediment using a proven dating technique such as OSL dating; geological observations and interpretations of sedimentary facies to determine modes of deposition, evidence of anthropogenic sedimentation and alteration (Goldberg and Sherwood, 2006; Goldberg and Berna, 2010; Goldberg and Macphail, 2012; Karkanas et al., 2015; Goldberg et al., 2017; Shahack-Gross, 2017; Stephens et al., 2017); micromorphological study of the sedimentary facies to determine what is going on at the smaller scale (Goldberg and Berna, 2010; Karkanas and Goldberg, 2010; Karkanas et al., 2015; Stephens et al., 2017) as well as geochemical analysis of the sediment (Stephens et al., 2017).

This thesis will focus principally on the application of one of these methods – OSL dating – to complex stratigraphic features at two sites. PP5-6 on the south coast of South Africa, and Mertenhof in the western Interior zone, both containing rich early records of human behaviour. Both of these sites contain sediment that may – based on their stratigraphic position relative to other facies – relate to the later phases of MIS 3. By clarifying the age and formation of these strata, the thesis will enable us to understand the significance of the cultural materials they contain to the broader debates highlighted above.

2.7 Synopsis

This chapter provides a brief introduction into the literature of AMH origins, and the emergence of behavioural modernity, along with the models for how and when this occurred. Also discussed is the difficulty in validating any of these behavioural models due to the paucity of data around the crucial MSA period of MIS 3, caused by lack of sedimentation and artefact evidence during this time. Understanding of site formation processes, sedimentary context and post depositional alteration can help contribute to the clarity of MIS 3 with respect to modern human occupations. Finally, the techniques that will be employed to achieve these clarifications is provided. More detail on these techniques is provided in Chapter 3.
Chapter 3: Methodology
Chapter 3 – Field and laboratory methods

3.1 Introduction

The primary analytical method used in this thesis is OSL dating of sediment samples collected from complex sedimentary features at PP5-6 and Mertenhof Rock Shelter MRS, South Africa (Figure 1.1). All analyses, apart from in-situ gamma spectrometry, was conducted in the OSL laboratories at the University of Wollongong (UoW). A combination of field geological techniques and standard laboratory measurement and preparation techniques were used to accurately determine the context of the sediment collected and age derivation of those sediments. This chapter details these techniques.

3.2 OSL dating

OSL dating has seen a growth in popularity over the past few decades by geologists, physical geographers, palaeoanthropologists and archaeologists to analyse sedimentary and geological contexts, that contain objects linked to material culture and occupation by hominin species during the Quaternary (Huntley et al., 1985; Feathers, 1996; Feathers, 2003; Jacobs and Roberts, 2007; Duller, 2008; Jacobs et al., 2008; Wintle, 2008a; Roberts et al., 2015).

OSL dating provides an estimate of when a mineral grain, such as quartz or potassium feldspars, were last exposed to sunlight prior to burial (Wintle and Huntley, 1979; Wintle, 1993; Wintle, 1996; Aitken, 1998; Jacobs and Roberts, 2007; Wintle, 2008b). Following burial of the mineral grain, when the grain is hidden from sunlight, it begins accumulating radiation energy in the form of electrons which become trapped within defects or holes in the crystal lattice structure of the mineral grain (Aitken, 1985; Aitken, 1998). Electrons are moved into the defect traps as a result of exposure to background ionising radiation in the form of alpha, beta, gamma and cosmic radiation (Aitken, 1989) derived from radioactive decay of uranium (\(^{238}\text{U} & 235\text{U}\)), thorium (\(^{232}\text{Th}\)), and the decay of their daughter products as well as potassium (\(^{40}\text{K}\)). The accumulation of electrons in sufficient quantity for viability of OSL dating requires that the traps be of suitable ‘depth’ to avoid loss of the accumulated electrons over long geological time periods (Aitken, 1998). The electron traps within the mineral grains are sensitive to ultra-violet light (UV), with exposure resulting in stimulation of the electrons and ‘emptying’ of the accumulated charge (Aitken, 1985; Aitken, 1998).

3.2.1 Determining age using OSL dating

The luminescence signal can be measured in the laboratory, which provides an estimate of stored energy within a single mineral grain since its last exposure to sunlight. The equivalent
Dose ($D_e$) signal can be determined by applying known doses of radiation to mineral grains in a controlled laboratory environment and comparing the known laboratory doses to the measured natural dose received in nature. The rate of exposure to ionising radiation received in nature can be determined using either field or laboratory generated environmental dose rates ($D_r$). The optical age of a sample can be determined by dividing the $D_e$ (Gy) by the environmental dose rate (Gy/ka), using the following equation:

$$\text{Age estimate (ka)} = \frac{\text{Equivalent dose estimate (Gy)}}{\text{Environmental dose rate (Gy/ka)}}$$

The age of artefacts and fossils contained within an archaeological site can be inferred from the age of their enclosing sediments (Feathers, 1996; Feathers, 2003; Duller, 2004; Wintle, 2008a). The longer a mineral grain has been buried, the higher its accumulated burial dose and the standard measurement unit or SI is Grays (Gy) where 1 Gy = 1 J/kg-1.

3.3 OSL sample collection

A total of thirteen sediment samples were collected from two sites in South Africa for the purposes of OSL age analysis for this thesis. Five samples were collected in June 2015 from the semi-arid inland site of MRS (Figure 4.1) and eight samples were collected in March 2017 from the coastal site of PP5-6 (Figure 5.1) by the author of this thesis. A schematic reconstruction of sampling locations for both MRS and PP5-6 can be viewed in Figures 4.8 and 5.8, respectively.

Collection of each sample was conducted in a way that prevents exposure to light, which can result in zeroing or ‘bleaching’ of the OSL signal (Duller, 2004; Roberts et al., 2015). To accomplish this, samples were collected at night using only head-lamps equipped with infra-red LEDs. The initial ~2 cm of sediment was scraped from the surface of the profile wall, and a hand coring tool was inserted into the non-light exposed sediment to prevent collection of ‘zeroed’ material. An additional sediment sample was taken from each sample location for moisture content and environmental dose rate measurements (Section 3.6). The sediment was placed and sealed inside air-tight plastic sampling bags to ensure minimal loss of moisture content, before being placed within double black sealed bags for transport. Samples were not opened until arrival in the OSL laboratory at UoW.
3.4 OSL sample preparation

Sample preparation of OSL samples were conducted using standard laboratory procedures outlined by Wintle (1997). The following flow diagram (Figure 3.1) is the process by which each sample was prepared:

**Figure 3.1:** OSL sample preparation procedure from start to finish. Full process is explained in detail in Appendix 1.

3.5 $D_e$ determination

3.5.1 Introduction

Section 3.2 outlined the relationship between the $D_e$ and *environmental dose rates*. The $D_e$ value of an individual mineral grain, in this instance quartz, is determined by stimulation of the grain with a specific wavelength of light, resulting in release of the trapped charge in the form of photons of light (Jacobs and Roberts, 2007). This OSL signal is observed as an optical decay curve like the one in Figure 3.2 (Huntley et al., 1985; Feathers, 1996; Duller, 2004), where the signal decays as a function of time whilst the stimulation light source is held at a constant power.
Figure 3.2: Example of optical decay curve, with the normalised decay curve shown in the inset panel. The Y axis represents OSL counts per 0.02 s, and the X axis indicates length of stimulation of the grain (Image from Jacobs and Roberts (2007))

The ‘natural’ signal measured from the release of photon charge trapped during burial is measured during initial stimulation with a light source. This signal can then be compared to OSL signals that result from radiation doses of known amounts administered in the lab using an artificial radiation source. The natural signal can then be compared to these laboratory generated signals through the construction of a dose response curve (Huntley et al., 1985; Feathers, 1996; Duller, 2004; Jacobs and Roberts, 2007; Jacobs et al., 2015). An example of a dose response curve can be seen in Figure 3.3. The ‘natural’ signal is projected horizontally from the Y axis on to the dose response curve, and where the natural OSL signal intersects the dose response curve, a straight line down to the dose axis gives an estimation of the $D_e$ value in Gy.

For this project, single-grain analysis was used over single-aliquot, as advancements over the past two decades have led to improvements in age determination (Murray and Wintle, 2000; Duller, 2003; Murray and Wintle, 2003; Wintle and Murray, 2006; Duller, 2008). Single-grain analysis has several inherent advantages over single aliquot analysis. These are:

1. Confirmation of adequate bleaching of grains before burial (Duller et al., 2000; Duller, 2008).
2. Determining beta-dose rate variability in individual grains in the environment they were buried in (Jacobs et al., 2011).
3. Determine stratigraphic integrity of the archaeological or geological sequence, including the identification of post-depositional alteration or mixing (Roberts et al., 1999; Jacobs and Roberts, 2007).
4. Validating reproducibility of single-grain $D_e$ measurement for the same sample (Jacobs and Roberts, 2007).

5. Identification and isolation of single-grains with abnormal physical properties before age determination, which may result in reduced scatter in a single-grain $D_e$ distribution (Roberts et al., 1999; Feathers, 2003; Galbraith and Roberts, 2012).

Figure 3.3: Dose response curve showing an increase in the OSL signal with an increase in applied dose to determine $D_e$. The black dots indicate regenerative doses applied to the grain (Jacobs and Roberts, 2007).

3.5.2 Single-grain OSL equipment

An automated Risø TL/OSL-DA-20 reader with single-grain laser attachment was used for all OSL measurements conducted in this project. Aluminium single-grain discs with a 10 x 10 grid of 100 depressed chambers were loaded with 100 individual grains of quartz (Figure 3.4d) for all samples analysed from MRS and PP5-6. The depressed chambers are 300 μm in diameter and depth, easily accommodating a single 212-180 μm in diameter grain (Bøtter-Jensen et al., 2000). These single-grain discs were loaded on to a 48-holder carousel and placed within the measurement chamber of the reader. The reader is equipped with a heater plate, which serves to preheat and maintain elevated temperatures of individual grains to help isolate the electron traps of interest. The reader is also fitted with a $^{90}\text{Sr}/^{90}\text{Y}$ beta source which is used to give artificial doses of known amounts (Bøtter-Jensen et al., 2003).

Optical stimulation of the mineral grains is conducted using a 10 mW 532 nm Nd:YVO4 solid-state diode-pumped green laser, with a power density of ~45 mW/cm$^2$ at 90% power (Bøtter-Jensen et al., 2003). The laser beam is targeted onto a location of ~10 μm
diameter and is directed at the target at a precision of ~3 μm, using three lenses and two mirrors driven by motors (Figure 3.4c) (Bøtter-Jensen et al., 2003).

**Figure 3.4:** Risø TL/OSL reader with attachment for single-grain analysis by Risø National Laboratory in Denmark. a) Risø TL/OSL reader fitted with blue-light emitting diodes for single-aliquot, b) same instrument with green laser attachment for single-grain analysis, c) single grain laser attachment under magnification, d) single-grain OSL disc seated in carousel (Jacobs and Roberts, 2007).

The emitted luminescence signal is measured using a photomultiplier tube (PMT; vertical elongate cylinder in Figure 3.4b). Emitted photons are detected and counted by the PMT whilst interacting with a photosensitive cathode contained within a vacuum tube. Isolation of UV emissions between 270-370 nm is achieved when the particles pass through the two Hoya U-340 filters.

### 3.5.3 Single aliquot regenerative-dose (SAR) protocol

The SAR protocol was implemented for the OSL dating of single quartz grains at MRS and PP5-6. The process was developed by Murray and Wintle (2000), and is the preferred method for determining the $D_e$ value of quartz grains. The SAR protocol provides a method by which one can track possible changes in sensitivity that may occur as a result of differences between natural and laboratory generated $D_e$ values (Murray and Wintle, 2003). The SAR
protocol involves the use of the OSL signal that arises from the unknown natural dose ($L_N$) being measured, and several OSL signals generated by applying known laboratory doses ($L_X$). The values $L_N$ and $L_X$ are divided by corresponding test doses, $T_N$ and $T_X$, to generate the sensitivity-corrected signal ($L_X/T_X$ or $L_N/T_N$). The SAR protocol is distinguishable from other measurement protocols because it makes use of the response of single-grains to a test dose that is applied following measurement of the natural signal and every regenerative dose to monitor for sensitivity change (Murray and Wintle, 2003).

A 0 dose is used for recuperation monitoring, which determines whether the dose-response curve passes through the origin. This can occur if there is residual thermal signal not released by the bleaching and subsequent preheating of the grain before the measurement of the $T_N$ or $T_X$ and is instead released during preheat before $L_N$ or $L_X$ (Murray and Wintle, 2000). A repeat regenerative dose is given to the grain to monitor and calculate the recycling ratio; which is the ability for the same OSL signal to be recorded from the same given dose in a single-grain, and tests the efficiency of administered sensitivity monitoring and corrections (Aitken, 1998; Murray and Wintle, 2000, 2003). Individual grains are stimulated at 50°C (room temperature) and exposed to infra-red (IR) light emitting diodes and then given a final laboratory dose equivalent to the first test regenerative dose (44 and 58 Gy for PP5-6 and MRS respectively) to determine if there are any feldspar inclusions or grains present. Following this IR depletion, the IR depletion ratio can be determined by monitoring the depletion of the OSL signal after IR stimulation (Duller, 2003). These three ratios; that is, the recuperation ratio, recycling ratio and IR depletion ratio were used in the single-grain rejection criteria (Section 3.5.4.3). The SAR protocol used in this project can be viewed in Table 3.1.

3.5.4 Single-grain analysis
3.5.4.1 Dose recovery test

Dose recovery tests were performed in this project to determine the appropriate preheat combinations for single-grain analysis. A dose recovery test uses a light source to zero the grains of their natural luminescence signals, in this case natural sunlight for ~4 days per sample. The same sample is then rested for several days (>3) in the dark. The sample is then administered with known laboratory dose using a $^{90}$Sr/$^{90}$Y beta source (Roberts et al., 1999; Jacobs and Roberts, 2015). A suitable SAR protocol, with a selection of preheat combinations and known laboratory doses, is designed to accurately measure the dose given to the sample, and the amount of this dose recovered when the signal is measured upon bleaching (Wintle and Murray, 2006). Typically, the dose recovery test is displayed as a measured over given dose
ratio (Galbraith et al., 1999) with the expectation that a successful dose recovery will have a ratio consistent with unity.

**Table 3.1: Example of generic SAR protocol used for analysis of single-grains of quartz.**

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<thead>
<tr>
<th>Step</th>
<th>Treatment</th>
<th>Observed</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Dose (Natural and regenerative – excluding second repeat dose)</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>Preheat (PH-1) to x°C for 10 s.</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>OSL measurement for 2 s at 125°C.</td>
<td>LN or LX</td>
</tr>
<tr>
<td>4</td>
<td>Test dose.</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>Preheat (PH-2) to x°C for x s.</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>OSL measurement for 2 s at 125°C.</td>
<td>TN or TX</td>
</tr>
<tr>
<td>7</td>
<td>Repeat step 1 through 6 for natural and all regenerative doses.</td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>Second repeat dose.</td>
<td></td>
</tr>
<tr>
<td>9</td>
<td>IRSL stimulation for 40 s at 50°C.</td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>Repeat step 2-6.</td>
<td></td>
</tr>
</tbody>
</table>

### 3.5.4.2 Optical decay curves

Analysis of OSL decay curves, resulting from laboratory stimulation of quartz mineral grains, allow the operator to make informed decisions about the optical stability of various components contained within the OSL signal of a quartz mineral (Bailey et al., 1997). By targeting a quartz grain with a green laser at a constant power (e.g. 90%), a continuous wave (CW) OSL decay curve is generated as the OSL signal is depleted as a function of stimulation time (Jacobs and Roberts, 2007). An example of an optical decay curve can be seen in Figure 3.2, with the inset displaying the decay curve normalised to the first data channel. This allows multiple decay curves to be compared at a normalised decay value to determine the reproducibility of decay signals between grains.

### 3.5.4.3 Rejection criteria for single-grain analysis

One of the inherent strengths of single grain analysis is the ability to remove aberrant grains, which are grains described as having inherent physical properties unsuitable for age analysis. Grains with these aberrant physical properties, if not removed may provide $D_e$ values that could be mistaken as evidence of sediment mixing, insufficient bleaching, or roof-spall contamination.

All quartz grains analysed in this project were subjected to the five primary rejection criteria following those proposed by Jacobs et al. (2006a), and two additional criteria. Grains that failed the criteria were rejected:
1. When the test natural \((T_N)\) luminescence signal of the grain was less than 3 times that of the background signal.

2. If the recycling ratio sensitivity test where grains were not within 2\(\sigma\) of unity; repeat test dose is applied at the end of the measurement cycle to monitor the grains for sensitivity changes. If this process does not work, recycling ratio will fail.

3. If the IR depletion ratio test is \(>2\sigma\) less than unity. This test determines if there are feldspar contaminants that made it through density separation mentioned in Section 3.4.1, or feldspar inclusions are present.

4. If an error of \(>20\%\) with the \(T_N\) signal

5. Where \((L_0/T_X)/(L_N/T_N)\) was \(>5\%\), the recuperation test failed.

Additional criteria:

1. When the \(L_N/T_N\) ratio does not intercept with the dose response curve, the grain displays Class-3 behaviour (Yoshida et al., 2000) or is likely saturated. In both instances a \(D_e\) value cannot be obtained.

2. If grains were exhibiting poor fit characteristics to saturating exponential or saturating exponential plus linear curves, due to dim luminescence signal or scatter of the dose response curve.

3.5.4.4 Radial plots and overdispersion (OD) values.

Radial plots were utilised in this project to visually assess the distribution patterns of individual \(D_e\) values that passed through rejection criteria and were deemed to have suitable \(D_e\) values for age determination. OD values represent the amount of scatter present in single-grain \(D_e\) distributions beyond what is expected of measurement uncertainties alone (Galbraith, 1990).

A radial plot visually displays all grains with acceptable \(D_e\) values with their related precision to determine, primarily, if there is more than a single population (simple distribution) or if the deposition of the sediment was more complex containing multiple distributions or very broad distributions. Figure 3.5 is an example of a radial plot displaying a typical single \(D_e\) population. This figure represents relative error and precision of each grain, which can be measured by linking a straight line vertically down to the relative error or precision axis. The grey bar indicates a \(\pm 2\) standardised estimate, where 95\% of grains should fall within this grey bar assuming standard measurement errors explain the scatter, i.e. no overdispersion (Jacobs
and Roberts, 2007). The $D_e$ value of a grain is read from the plot by extending a straight line from the origin of standardised estimate axis, onto the righthand radial axis.

![Radial plot with associated OD value, representing a single population of $D_e$ values.](image)

**Figure 3.5:** Radial plot with associated OD value, representing a single population of $D_e$ values.

The OD value was generated using the central age model (CAM; Section 3.5.4.5). The higher the OD value, the more scatter within the $D_e$ distribution, which could indicate that the sample has been affected by post-depositional alteration such as bioturbation or erosional mixing of the sediment, inadequate bleaching prior to final burial or contamination by external grains during sample collection (Olley et al., 2006; Jacobs et al., 2008) among other reasons.

### 3.5.4.5 Central age model (CAM)

The CAM can be used to generate the weighted mean $D_e$ values and associated OD values. The CAM is the best and simplest method for generating weighted mean $D_e$ values for age analysis in samples that show little or no evidence of post-depositional alteration resulting in scatter and higher OD values (Galbraith, 1990). These distributions consist of a single relatively well-defined population of $D_e$ values, distributed around a central mean $D_e$ value, with no evidence of a divergent population. $D_e$ values are assumed to be clustered around a single central weighted mean value, and OD values for the population are incorporated into the uncertainty estimate for the $D_e$ value (Galbraith et al., 1999).
3.6 Determining dose rates

The total environmental dose rate introduced by the equation in Section 3.2.1 is a representation of the rate at which electrons are transferred into the traps as a result of exposure to ionising radiation (Section 3.2) from the alpha, beta, gamma ionizing radiation of the surrounding material caused by radioactive decay of parent isotopes $^{238}$U, $^{235}$U, $^{232}$Th and the subsequently formed daughter products in the U and Th decay chains and also $^{40}$K and $^{87}$Rb. Added to this is the contribution from cosmic-radiation (Aitken, 1985). Each of these main radiation sources have different spheres of influences; that is, the emitted particles can only travel certain distances in solid material. Alpha particles travel ~0.02 mm, beta particles ~2-3 mm, gamma rays ~300 mm and cosmic-radiation can travel the greatest distance up to several 10s of meters (Aitken, 1985).

Alpha particles have the smallest travel distances, and thus the external contribution is removed during the HF etching procedure carried out in Section 3.4. The contribution from beta radiation was determined using a GM-25-5 beta counter in the laboratory. The gamma radiation contribution was determined using in-situ gamma spectrometry. Finally, the contribution from cosmic-radiation was determined using geomagnetic latitude, site altitude, sediment density and depth in profile to the collected samples at both sites using the equation presented in Prescott and Hutton (1994).

3.6.1 Sample preparation for dosimetry and water content

The 13 dosimetry samples collected and described in Section 3.3 were prepared using the following steps for both MRS and PP5-6:

1. Plain glass 250 mL beakers were thoroughly cleaned and dried to prevent sediment contamination.

2. The empty beakers were weighed.

3. The dosimetry bags were cut open, and 1 sample was allocated to its own beaker. This beaker was then weighed again together with the ‘wet’ sediment sample

4. The sample was placed into a 100°C oven until the sample was completely dry. This process generally took 1-2 days. The sediment was deemed dry when it would no longer clump together, and individual grains would separate easily.

5. The samples plus beaker were weighed again to determine the amount of moisture content within the sediment.
Water content was calculated using the following equation, where mass 1 = beaker weight, mass 2 = wet sample – mass 1 and mass 3 = dry sample – mass 1:

\[
\text{Water content (\% dry mass)} = \frac{(\text{mass } 2 - \text{mass } 3)}{(\text{mass } 3)} \times 100
\]

### 3.6.2 Beta dose rates

Beta dose rates were determined for both sites using a GM-25-5 beta counter (Mejdahl, 1979; Bøtter-Jensen and Mejdahl, 1988). Beta emissions from U, Th and K decay within a sample are directly estimated for each sample. The machine consists of five GM detectors and a guard counter, surrounded by a lead shield to minimise cosmic-ray interference. Samples are presented before the GM detectors simultaneously to measure the emitted beta radiation from the sediment samples for a 24-hour period. Each time a beta particle is emitted from the sediment samples, it is recorded by the GM detector as a pulse. Particles measured simultaneously to the guard counter were rejected as background interference. The samples were prepared by first being crushed to a fine powder to homogenise the sediment, and placed within ~25 mm diameter plastic pod sealed with clear film. Three pods were measured for each sample, and compared to a standard loess sample (Nussi) with a known beta dose rate of 1.5399 ± 0.02766 Gy/ka based on estimates derived from high resolution gamma spectrometry (Kalchgruber et al., 2002). A magnesium oxide (MgO) was also measured to determine background dose rates due to its non-radioactive nature.

Uncertainties in beta dose rate measurements using a GM-25-5 beta counter were calculated using the equations in the supplementary information of Jacobs and Roberts (2015), which account for daily fluctuations in equipment behaviour, differences in counting efficiency of the 5 detectors, reproducibility of the 3 sub-samples, laboratory standard and background blank. Four systematic errors are taken into account; imprecision of laboratory standard at ~1.8%, uncertainties in dose rate conversion factors (~2%) outlined by Guérin et al. (2011) and ~2% for beta particle attenuation outlined by Murray and Olley (2002). Finally, a 25% uncertainty of the water content of the sediment sample was factored in.

### 3.6.3 Gamma dose rates

Gamma dose rates for all samples were collected using in-situ gamma spectrometry, because this method allows for measurement of the entire ~300 mm sphere of influence of gamma rays acting on the quartz grains, considering inhomogeneity of the sediment being measured (Aitken, 1985; Jacobs and Roberts, 2007). The measurements were conducted using
a Digidart handheld gamma spectrometer with a 1, 1.5 or 2-inch NaI (T) crystal detector. Calibration was conducted using the ‘threshold’ technique outlined by Mercier and Falguères (2007). The ‘threshold’ technique is used as it records a wider part of the spectrum which significantly reduces the measurement time in the field, whilst reducing the impact of temperature fluctuations as well (Mercier and Falguères, 2007).

Uncertainties in gamma dose rates calculated using in-situ gamma spectrometry include measurement errors of ~2.2%, which is based off large numbers of repeat estimates, a 3% calibration ratio error and a 25% relative uncertainty associated with moisture content (Mercier and Falguères, 2007; Guérin et al., 2011).

3.6.4 Cosmic dose rates

Cosmic dose rates have the greatest spheres of penetration, but inherently low overall contributions when compared to beta and gamma radiation contributions. For accuracy of age analyses these still need to be included (Aitken, 1985). Cosmic-ray dose rates were calculated using the equation presented by Prescott and Hutton (1994). The equation considers the geomagnetic latitude of the site which is calculated using the longitude and latitude of the site, and the elevation of the site. Also taken into account is the density of the sediment being analysed and the depth at which the sediment was taken from the profile. Other parameters include the density and extent of the rocky overburden and angular distributions of the cosmic-rays entering the site.

3.6.5 Internal dose rates

All samples collected from MRS and PP5-6 used an internal dose rate of $0.031 \pm 0.011$ Gy/ka due to the very minute amounts of U and Th present within quartz grains (Aitken, 1985).

3.6.6 Final ages

Final ages for the sediment samples collected at both MRS and PP5-6 were calculated using the equation outlined in Section 3.2.1. This equation divides the weighted mean $D_e$ value obtained by the CAM by the total environmental dose rate, which is a combination of the internal, beta, gamma and cosmic dose rates.

3.7 Field and geological techniques

In March 2017 PP5-6 was visited for one week to collect samples and make field observations about the stratigraphic context of the cut-and-fill feature described in Section 1.1. A virtual approach was undertaken for MRS, using photographic evidence and site descriptions provided by Dr. Alexander Mackay, the site director at MRS.
The primary geological techniques used in the field included:

1. Geological observations for rock shelter formation at PP5-6 by studying the geology of the overhanging cleft of rock (Goldberg and Sherwood, 2006; Goldberg and Berna, 2010; Karkanas and Goldberg, 2010).

2. Sketching and observations of stratigraphy and stratigraphic relationships – involved detailed measurements of stratigraphic thicknesses, extent of stratigraphic layers and an assessment of the composition of the sediment contained within each StratAgg or StratLayer. For MRS, this involved studying high resolution photography and field notes collected by Dr. Alexander Mackay.

3. Generation of hypothetical models to explain the two complex stratigraphic features; the cut-and-fill feature at PP5-6 and the apparent wedge of sediment at MRS.

3.7.1 Laboratory geological techniques

Two laboratory methods were used to analyse the sediment at MRS. This analysis was conducted to give insight into the deposition of the sediment and potential models for the formation of complex stratigraphic features at MRS.

3.7.1.1 Minerological analysis

Analysis of sub-samples of sediment collected next to the OSL sample locations at MRS were conducted using X-ray diffraction at the School of Earth and Environmental Science at UoW. A total of 7 samples were crushed to a very fine powder using mortar and pestle. Mineralogical analysis was conducted using a Phillips 1150 PW Braggs-Brentano diffractometer with CuKα radiation. Phases for each mineral type were identified using SiroQuant™ software by analysing diffraction peaks and comparing to reference peaks for given minerals. Accuracy was determined using chi-squared values.

3.7.1.2 Grain size analysis

A Malvern Mastersizer 2000 was used at UoW to determine the grain size distributions for the same sediment samples mentioned in Section 3.7.1.1. For each sample, ~2 g aliquots of sediment were sonicated for 45 s to separate sediment that had formed clasts. The grain size distribution was determined by averaging 5 consecutive measurements and reporting these as a % fraction for sand (2000 – 63 μm), silt (63 – 4 μm) and clay (<4 μm). These distributions were plotted as bimodal plots to determine the proportion of fine to coarse grained fractions in the individual StratAggs and StratLayers at MRS. This information was used to determine
patterns of sediment deposition, and infer site formation processes at MRS for both the complex stratigraphic feature (Section 1.1) and the greater site formation context.

3.8 Synopsis

In this chapter, the different measurements and techniques used to analyse the OSL samples and additional geological sediment samples were discussed for both MRS and PP5-6. Included, are sample preparation procedures for OSL samples to separate quartz grains from the rest of the sediment, rejection criteria for single grain OSL, graphical displays of quartz $D_e$ values using radial plots, decay curves and dose response curves. Methods for calculating total dose rates, including alpha, beta, gamma and cosmic-ray dose rates were discussed. The combination of environmental doses rates and equivalent doses for final age determination were presented. The results of these analyses are presented in Chapter 4 (MRS) and Chapter 5 (PP5-6). Additional geological techniques, including field sketches, stratigraphic relationship interpretations, mineralogical analyses and grain size analyses were also introduced.
Chapter 4: Mertenhof Rock Shelter (MRS)
Chapter 4 – Mertenhof Rockshelter

4.1 Introduction

This chapter describes Mertenhof Rock Shelter (MRS) within both its archaeological and geological contexts. This includes a brief description of the site history and the excavation area, followed by an introduction to the sediment context and two hypotheses relating to the formation of the complex stratigraphic feature being analysed within this site, as outlined in the aims in chapter 1. Also provided are the existing 14C ages for the sequence, along with estimated ages based on the stratigraphic distribution of artefact types with known or predicted age ranges.

The results section of this chapter begins with discussion of the geochemical and grain size analyses. This is followed by detailed OSL dating results including: dose recovery results, single grain quartz selection/rejection results, \(D_e\) determination, analysis and interpretation, dosimetry results and final ages for five sediment samples.

4.1.1 Site setting and geology

MRS (~32°08'58.9"S 19°14'15.3"E) is located in western South Africa in the Cederberg mountains ~300 km north of Cape Town and 27 km east of Clanwilliam, at ~600 m.a.s.l. Figure 4.1 shows a map of the site location with respect to Cape Town and PP5-6 (Chapter 5). MRS is situated close to the mouth of a narrow canyon where the Biedouw River emerges from the massive sandstones of the Nardouw formation onto the more open terrain of the Bokkeveld shales and sandstones (Shone and Booth, 2005; Schmidt and Mackay, 2016). The Biedouw River flows year-round, which is rare in the semi-arid region in which it is located (Schmidt and Mackay, 2016). This availability of water is one of the main reasons proposed for strong occupation of MRS in the past (McBrearty and Brooks, 2000; Thomas, 2002; Mitchell, 2008; Mackay, 2016).
To gain a better understanding of our species behavioural evolution, it is important to study evidence from sites located within the full range of ecological and climatic zones, including arid inland sites such as MRS (Mackay, 2016). MRS’s location within the arid zone, and its unusual position adjacent to a perennial water source places it in an ideal position to understand how *Homo sapiens* survived away from the coast and the more productive inland regions of South Africa, and to determine potential interactions with coastal counterparts (Guglielmino et al., 1995; Will et al., 2015). Both the limited existing $^{14}$C chronology and aspects of the technological sequence imply occupation during MIS 3, which is one of the least understood periods during the late Pleistocene with regards to human occupation and behaviour, specifically so in the western Cape (Mitchell, 2008; Mackay, 2017 unpublished). MRS is, therefore, a pivotal site for contributing to further understanding of modern human adaptation and behaviour during MIS 3, interaction between coastal and inland foraging groups, and their responses to palaeoclimatic variability within the region (Ziegler et al., 2013).

Cave and rock shelter formation within this unit of rock is made possible by exploitation of rock fractures or faults planes within the quartzite bedrock, created by internal post depositional stress (Shone and Booth, 2005). MRS is situated in a quartzite cliff face with a northern aspect entrance situated ~25 m above the Biedouw River.

**Figure 4.1:** Location map of MRS, with respect to Cape Town and PP5-6 (From Google Earth, 2017).
4.1.2 Site history and excavation area

MRS is ~10 m wide at the mouth, ~9 m deep from the dripline to the rear wall at its deepest point, and up to 4.2 m high, with a floor area of ~70 m². The site was originally inspected in 2012, where stone artefacts comparable to those of the LSA Robberg phase (~14-22 ka) were discovered on the surface of the rock shelter floor (Mackay 2017, unpublished). The interior rock walls of the shelter are abundantly covered in rock art depicting human activity alongside various depictions of regional fauna and more abstract images.

The site was excavated over five field seasons between 2013 and 2017, in a 2 x 3 m grid subdivided into six metre squares (Figure 4.2). The location of the excavation area was chosen following successful application of ground penetrating radar technology which indicated a depth of deposit of ~1.5-2.2 m (Mackay 2017, unpublished). Excavation followed natural stratigraphic units (StratLayers), which were later grouped into major stratigraphic aggregates (StratAggs). All cultural materials >20 mm were piece plotted, and sediment residues were sieved on-site.

Samples collected and analysed in this study were collected from excavation squares 4 (MRS OSL 7-9) and 6 (MRS 5-6). Figure 4.4 shows the OSL sample locations for the south wall of square 6, and Figure 4.3 shows the OSL sample locations for the east wall of square 4. Sample locations are shown with respect to profile depth.

![Figure 4.2: Photograph displaying the excavation grids at MRS consisting of 7 squares at the end of season 3, full depth of profile not shown (after Mackay 2017, unpublished).](image-url)
Figure 4.3: OSL sample locations shown with respect to the complex stratigraphic feature (Wedge) along the East wall of the current excavation. MRS 6 and 5 are display in their relative horizontal positions on the East wall as a reference to the complex feature (Photograph: Alexander Mackay).

Figure 4.4: OSL Sample locations shown with respect to complex stratigraphic feature (Wedge) along the South wall of the current excavation. The wedge feature is visible on the left side of the image to provide perspective on where it intercepts with the south wall (Photograph: Alexander Mackay).
4.2 Sedimentological Context

The sedimentary deposits contained within MRS are stratigraphically complex. A total of 460 *StratLayers* were identified within excavated sediments to a depth of ~1.5 m, and include two human burials, five mongoose burials and several pit features (Mackay 2017, unpublished). These contexts were grouped together into nine major stratigraphic aggregates (*StratAggs*). This study focuses on four of these *StratAggs*, along with two *StratLayers* that have not yet been allocated to a *StratAgg*, to provide the stratigraphic context of the wedge-like sedimentary feature shown in Figures 4.3 and 4.4, and to provide a more robust chronology for the interpretation of MIS 3 related archaeology at MRS. The targeted *StratAggs* and *StratLayers* overlie the Howiesons Poort / post-Howiesons Poort components of the site and underlie the oldest documented LSA (*StratAgg R/GBS*). Table 4.1 provides information for each *StratAgg/StratLayer*, including the estimated ages deduced from the detailed analysis of ~18000 artefact finds from the entire site. Figure 4.5 is a schematic representation of the *StratAggs* identified along the E-W (Figure 4.5a) and N-S section walls (Figure 4.5b).

![Figure 4.5a](image)

**Figure 4.5a:** Schematic of the *StratAggs* identified along the E-W section of the South Wall at the rear of the cave. Approximate thicknesses are provided for *StratAgg/StratLayers* to infer scale (Photograph: Alex Mackay).
In Figure 4.5a and Figure 4.5b, the complex nature of the sediment becomes apparent when trying to interpret the stratigraphic relationships of the site. Figure 4.5b shows *StratAgg* LGS being truncated by the pit feature, with continuation into the rear of the cave unknown. *StratAgg* LRS appears to be truncated by the pit in Figure 4.5b, and may potentially re-appear above CWGS manifested as VDGS in Figure 4.5b.

**4.2.1 Hypotheses for complex sedimentary feature (wedge)**

A primary aim of this thesis, is to develop and test different hypotheses for how and when complex stratigraphic features occurred within each site. This will allow for a more complete understanding of site stratigraphy and site formation processes.

The complex sedimentary feature at MRS appears as a wedge of sediment, visible in both the east and south profile walls. This wedge is delineated in Figures 4.3 and 4.4. Two hypotheses were formulated to explain how this wedge feature was potentially formed.

**Hypothesis 1** – Figure 4.5a shows a diagram of the sedimentary relationships on the South Wall of MRS and Figure 4.5b shows a diagram of the sedimentary relationships on the East Wall of MRS. In this hypothesis, the sediment contained within *StratLayer* VDGS (Figure 4.5b) is analogous to one of two *StratAggs*: 

---

**Figure 4.5b**: Schematic of the *StratAggs* identified along the N-S section on the east side of the cave.
a) *StratAgg* LRS – In this case VDGS is a continuation of LRS, which means that chronologically they should be of the same age. OSL sample MRS 6 was taken from *StratLayer* VDGS, in the South Wall (Figure 4.3), and MRS 8 was taken from *StratAgg* LRS on the East Wall (Figure 4.4). In this case, we would need to validate that the age of MRS 8 and MRS 6 were of the same age to confirm this hypothesis.

b) *StratAgg* LGS – In this case, CWGS is a continuation of LGS, which is truncated by the pit feature (Figure 4.5b). LRS in this scenario thins out closer to CWGS until it disappears. For this to be validated, it would be expected that MRS 5, collected from CWGS in the South Wall (Figure 4.3) would have a similar age to that of MRS 7 collected from LGS in the East Wall (Figure 4.4).

In variation 1a, MRS 6 is older than MRS 7, MRS 6 is approximately the same age as MRS 8 and MRS 5 is older than MRS 8. In variation 1b, MRS 6 is younger then MRS 7, MRS 5 is approximately the same age as MRS 7 and MRS 5 is older than MRS 8 (Figure 4.4).

**Hypothesis 2** – In this hypothesis, neither VDGS or CWGS (Figures 4.5b and 4.5a) are continuations of either LGS or LRS, as proposed above. Neither LGS or LRS make it into the South wall, and were at some stage truncated towards the rear of the shelter. Following the truncation of these two *StratAggs*, CWGS is deposited over the top truncation surface, which is dipping toward the rear of the cave, followed by deposition of VDGS (Figure 4.5a). In this scenario, it is expected that the age of the MRS samples is analogous to the posited deposition of the *StratAggs* and *StratLayers* (Table 4.1). That is, MRS 6, collected from VDGS is the youngest, followed in succession by MRS 5, MRS 7, MRS 8 and MRS 9 (Figures 4.3 and 4.4).

One of the confusing features of the complex sedimentary feature is that it appears to be dipping on its lower contact between CWGS and DGS (Figure 4.5b). If hypothesis 2 is correct, then it is expected that there would be a pattern of dipping beds towards the rear of the cave that had not been observed so far during the excavations that had been completed during seasons I to IV. Further excavation and interrogation of the stratigraphy toward the rear of the cave would need to be carried out to provide stronger evidence in support of hypothesis 2.
Table 4.1: Sediment characteristics of StratAggs and StratLayers and inferred ages or time periods from archaeological finds in stratigraphic sequence from top to bottom.

<table>
<thead>
<tr>
<th>Stratigraphic Aggregate (Top to Bottom)</th>
<th>Stratigraphic Layer</th>
<th>Age est.</th>
<th>Avg. Thickness (cm)</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>ULBD</td>
<td>&lt; 2 ka</td>
<td></td>
<td></td>
<td>Loose sandy surface sediment with discontinuous ‘dung’ crust overlying variable thickness of burnt and unburnt sediments. Contains pottery and adzes suggesting an age of &lt;2100 years, as well as glass beads suggesting occupation during the past 500 years.</td>
</tr>
<tr>
<td>R/GBS</td>
<td>16-22 ka</td>
<td></td>
<td></td>
<td>Heavily burrowed sandy/silty sediment, representing a probable hiatus in deposition between R/GBS and ULBD. Artefacts dominated by Robberg industry based on prevalence of silcrete, bladelet technology and distinctive core types.</td>
</tr>
<tr>
<td>Unresolved Aggregate – Sediment wedge</td>
<td>VDGS (OSL 6)</td>
<td>16-22 ka</td>
<td>9</td>
<td>Moderately compact, very dark grey coloured gritty fine sand/silt. High density artefacts of the Robberg industry. Truncated on N-S section by large overlying burrow feature, continuous along rear E-W section, but thins in centre due to animal burrowing.</td>
</tr>
<tr>
<td>Unresolved Aggregate – Sediment wedge</td>
<td>CWGS (OSL 5)</td>
<td>MIS 3</td>
<td>8</td>
<td>Moderate compaction, bright white inclusion sediment, with increasing silt content towards the base, and sandy sediment towards the top. Truncated by numerous animal burrows in south-west corner. Thickness of layer increases toward rear of cave.</td>
</tr>
<tr>
<td>LGS (OSL 7)</td>
<td>MIS 3</td>
<td>8</td>
<td></td>
<td>Medium grain sand dominated sediment. Presence of decayed white stone due to decay of hornfels, with age estimate based on blade and bladelet technology, though some mixing could have resulted from overlying R/GBS sequence.</td>
</tr>
<tr>
<td>LRS (OSL 8)</td>
<td>MIS 3</td>
<td>8</td>
<td></td>
<td>Red coloured sandy sediment. Less abundant roof-spall material within matrix compared to overlying layers. Distinguishable from LGS by increased abundance of decayed white stone and MSA markers such as Levallois and discoidal flakes and cores.</td>
</tr>
<tr>
<td>DGS (OSL 9)</td>
<td>50-55 ka</td>
<td>10</td>
<td></td>
<td>Dark grey sand/silt rich sediment. Abundant quartzite, bipolar cores and unifacial points suggest a late post-Howiesons Poort archaeology. Clear dip of stratigraphy toward back of cave (south side of cave).</td>
</tr>
</tbody>
</table>
4.2.2 Sediment analysis and results

Analysis of the sediment collected near the OSL samples was conducted using X-ray diffraction (XRD) and grain-size analysis using a master-sizer (section 3.7.1.3). This data was collected and analysed to determine if there were any significant patterns within the geochemistry or the grain size of the sediment to help determine how the sediment was deposited.

Table 4.2 provides the results from the XRD analysis, which shows no distinct patterns separating the samples from each other geochemically. The samples were predominantly quartz-rich, with quartz contents ranging between 91.3% and 84.6%. The quartz-rich composition can be explained by the dominance of quartzite source rock within the local area that the sediment likely formed from. Sample #3489 which is taken from the base of CWGS (Figure 4.5b), and sample #3493, taken from VDGS (Figure 4.5a) located just above the sedimentary wedge, contained slightly higher (5.3%) average K-feldspar content compared to other samples (2.0–3.2%) (Table 4.2). Sample #3648, from LRS contained a relatively higher content of plagioclase (7.5%) compared to the rest of the samples (0.5–3.6%). However, due to the small amount of sample that was analysed (~10–15 g), it was determined that the data had the potential to be biased. Insufficient sediment remained for further analysis.

Table 4.3 provides the results obtained from grain size analysis. These results show a definitive pattern of coarsening grain size toward the front of the cave (samples #3647–#3649), and fining toward the rear of the cave (samples #3479–#3493). Figures 4.6a to 4.6e show grain size distributions for each of the samples. All samples show bimodal grain size distributions, with some showing a dominance of coarse (Figures 4.6a, 4.6b and 4.6d) or fine (Figure 4.6c and 4.6e) modes. There is also a trend of fining upwards within the stratigraphic sequence, shown by the dominance of the coarse mode in the lower samples (Figure 4.3; MRS 7-9) followed by dominance of the fine mode (Figure 4.4; MRS 5-6) (Table 4.3).

Inferences can be made on the distribution of grain size within the cave, and are further discussed in Chapter 6.

4.3 Existing chronological framework

Limited chronological work has so far been conducted at MRS. Analysis of artefacts collected within each stratigraphic layer, when spatially allocated using geographic information systems, allowed for an archaeological stratigraphy to be constructed. Approximate ages were then inferred by correlation to other dated archaeological assemblages
within the region (Mackay and Welz, 2008; Mackay, 2010; Porraz et al., 2013; Högberg and Högberg, 2014; Mackay et al., 2014a; Mackay et al., 2014b; Mackay et al., 2015; Lin et al., 2016; Mackay, 2016). Table 4.1 shows the estimated ages or time periods for each StratAgg.

Radiocarbon ($^{14}$C) dates on charcoal samples from MRS were obtained by Alex Mackay prior to this thesis. Table 4.4 presents the $^{14}$C ages and their stratigraphic associations. At the completion of Season I at MRS, a series of range-finder AMS $^{14}$C ages were obtained from the deep sounding in square 3 (Figure 4.2a), which had, at that stage been excavated into the late MSA. All charcoal in the square occurred as isolated fragments, with no intact hearths discernible. This probably reflects both the extensive bioturbation in the upper deposits in this part of the excavation, and ‘anthro-turbation’ in the form of multiple pits, like those in Figure 4.5a and 4.5b. The pits include the interment of two juvenile individuals in what was likely a single grave, the boundaries of which were hard to identify. As can be seen from the ages and the associated age-depth profile (Figure 4.7) there is no clear correlation between age and depth (~30 cm) in the dated charcoal fragments from this part of the deposit. This may reflect the above-mentioned disturbance, and the fact that the charcoal used was likely disaggregated and transposed from its initial deposition location. All, but one of the ages range between ~18-23 ka cal BP, corresponding to early MIS 2 (Table 4.4). A similar suite of ages dominates the MIS 2 sequence at nearby Klipfonteinrand (Mackay, 2016). The outlier in the group is an age of 36914-38241 cal yr BP, which may relate to the latest MSA in the sequence. The fact that the ages are so strongly clustered is unsurprising given that charcoal preservation declines appreciably with depth, such that there is generally very little preserved charcoal in the MSA layers of the site. The situation here is thus comparable to that at Klipfonteinrand, Putslaagte 8 and Klein Kliphuis in all of which, sequences of charcoal preservation was generally poor beyond the LSA (Mackay and Welz, 2008; Mackay, 2010; Mackay et al., 2014a; Mackay et al., 2014b; Mackay et al., 2015; Lin et al., 2016; Mackay, 2016).
**Table 4.2:** A geochemical breakdown of the composition for the sediment collected from MRS for OSL analysis.

<table>
<thead>
<tr>
<th>Sample Name</th>
<th>#3647</th>
<th>#3648</th>
<th>#3649</th>
<th>#3489</th>
<th>#3479</th>
<th>#3491</th>
<th>#3492</th>
<th>#3493</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Lower Profile (OSL 7-9)</td>
<td></td>
<td></td>
<td>Upper Profile (OSL 5-6)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mineral Phase</td>
<td>Weight (%)</td>
<td>Weight (%)</td>
<td>Weight (%)</td>
<td>Weight (%)</td>
<td>Weight (%)</td>
<td>Weight (%)</td>
<td>Weight (%)</td>
<td>Weight (%)</td>
</tr>
<tr>
<td>Quartz</td>
<td>91.3</td>
<td>84.6</td>
<td>90.3</td>
<td>89.9</td>
<td>88.8</td>
<td>85.8</td>
<td>90</td>
<td>88.5</td>
</tr>
<tr>
<td>Plagioclase</td>
<td>2.5</td>
<td>7.5</td>
<td>1.3</td>
<td>1.2</td>
<td>1.3</td>
<td>4.6</td>
<td>3.8</td>
<td>3.6</td>
</tr>
<tr>
<td>K-Feldspar</td>
<td>2.3</td>
<td>2</td>
<td>1.7</td>
<td>5.1</td>
<td>2.1</td>
<td>5.3</td>
<td>3.2</td>
<td>5.3</td>
</tr>
<tr>
<td>Kaolin</td>
<td>0.4</td>
<td>1.2</td>
<td>0.1</td>
<td>0.3</td>
<td>3.3</td>
<td>1.4</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Illite</td>
<td>2.8</td>
<td>2.7</td>
<td>1.1</td>
<td>1.9</td>
<td>3.3</td>
<td>2</td>
<td>1.7</td>
<td>2.3</td>
</tr>
<tr>
<td>Chlorite</td>
<td>1.3</td>
<td>0.9</td>
<td></td>
<td>1.2</td>
<td>4.1</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Calcite</td>
<td>0.5</td>
<td>0.1</td>
<td></td>
<td>0.2</td>
<td>0.4</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Gypsum</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Halite</td>
<td>0.6</td>
<td>0.3</td>
<td>1.0</td>
<td>0.4</td>
<td>0.2</td>
<td>0.4</td>
<td>0.4</td>
<td>0.3</td>
</tr>
</tbody>
</table>

**Table 4.3:** Grain size analysis results obtained using a Master-sizer. D[4, 3] refers to centre of mass frequency distribution of the volume of measured sediment. Sediment samples were collected around the OSL samples, where below and above refer to sub-samples of sediment collected either below or above the OSL sample.

<table>
<thead>
<tr>
<th>Sample Name</th>
<th>Sand (%)</th>
<th>Silt (%)</th>
<th>Clay (%)</th>
<th>Clay (&lt;2 um)</th>
<th>Volume weighted mean (D[4, 3])</th>
<th>Coarse Mode</th>
<th>Fine Mode</th>
</tr>
</thead>
<tbody>
<tr>
<td>3647 - MRS 9</td>
<td>54.99</td>
<td>41.49</td>
<td>3.52</td>
<td>0.48</td>
<td>247.33</td>
<td>538.30</td>
<td>48.54</td>
</tr>
<tr>
<td>3648 - MRS 8</td>
<td>57.64</td>
<td>39.90</td>
<td>2.46</td>
<td>0.31</td>
<td>312.79</td>
<td>55.61</td>
<td>846.81</td>
</tr>
<tr>
<td>3649 - MRS 7</td>
<td>63.41</td>
<td>34.22</td>
<td>2.37</td>
<td>0.33</td>
<td>337.50</td>
<td>715.46</td>
<td>58.76</td>
</tr>
<tr>
<td>3479 - Below MRS 5</td>
<td>57.76</td>
<td>37.62</td>
<td>4.62</td>
<td>0.85</td>
<td>222.60</td>
<td>466.33</td>
<td>88.37</td>
</tr>
<tr>
<td>3491 - MRS 5</td>
<td>50.02</td>
<td>45.64</td>
<td>4.34</td>
<td>0.63</td>
<td>192.14</td>
<td>49.42</td>
<td>483.24</td>
</tr>
<tr>
<td>3492 - MRS 6</td>
<td>51.58</td>
<td>44.32</td>
<td>4.10</td>
<td>0.58</td>
<td>195.39</td>
<td>55.33</td>
<td>460.41</td>
</tr>
<tr>
<td>3493 – Above MRS 6</td>
<td>50.40</td>
<td>45.11</td>
<td>4.49</td>
<td>0.71</td>
<td>156.28</td>
<td>61.57</td>
<td>419.85</td>
</tr>
</tbody>
</table>
Figure 4.6: Plots of frequency % by volume as a function of grain size for the 5 sub-samples collected from the OSL samples analysed, a) #3491 (MRS 5), b) #3492 (MRS 6), c) #3649 (MRS 7), d) #3648 (MRS 8) and e) #3647 (MRS 9).
To date, no charcoal has been dated from the eastern half of the excavation, where stratigraphic integrity in the upper deposits is thought to be better. The stratigraphic context of these results is not directly relatable to those from which the OSL samples were collected in squares 4 and 6, as they are not from the same StratAggs/StratLayers.

**Table 4.4:** $^{14}$C ages for charcoal fragments from Sq.3 (Figure 4.2) at MRS. Note, they do not correspond with the stratigraphy analysed for the sediment wedge. Ages were calibrated using SHCal13 (Hogg et al., 2013).

<table>
<thead>
<tr>
<th>StratLayer (Square 3)</th>
<th>Arbitrary Z height (depth in profile)</th>
<th>Estimated Time Period</th>
<th>Age_mid (Yr BP)</th>
<th>Age_err (Yr BP)</th>
<th>Calibrated minimum age (Yr BP)</th>
<th>Calibrated maximum age (Yr BP)</th>
</tr>
</thead>
<tbody>
<tr>
<td>MDBCWP</td>
<td>98.297</td>
<td>MIS 3 with small bladelet</td>
<td>16265</td>
<td>54</td>
<td>19386</td>
<td>19817</td>
</tr>
<tr>
<td>MDBCWP</td>
<td>98.315</td>
<td>MIS 3</td>
<td>18637</td>
<td>65</td>
<td>22321</td>
<td>22633</td>
</tr>
<tr>
<td>GBL</td>
<td>98.361</td>
<td>Robberg</td>
<td>33344</td>
<td>138</td>
<td>36914</td>
<td>38241</td>
</tr>
<tr>
<td>LGCI</td>
<td>98.428</td>
<td>MIS 3?</td>
<td>15731</td>
<td>57</td>
<td>18780</td>
<td>19097</td>
</tr>
<tr>
<td>MDBC</td>
<td>98.498</td>
<td>Robberg / MIS 3</td>
<td>18586</td>
<td>73</td>
<td>22245</td>
<td>22598</td>
</tr>
<tr>
<td>CSS</td>
<td>98.559</td>
<td>Robberg</td>
<td>16084</td>
<td>56</td>
<td>19165</td>
<td>19564</td>
</tr>
<tr>
<td>LBGS2</td>
<td>98.599</td>
<td>Robberg</td>
<td>15186</td>
<td>71</td>
<td>18172</td>
<td>18609</td>
</tr>
</tbody>
</table>

**Figure 4.7:** Age-depth plot for $^{14}$C ages, using minimum calibrated ages in Years BP. Ages were calibrated using ShCal13.
4.4 OSL results

OSL dating of single grains of quartz is one of the primary methods used in this thesis to investigate the stratigraphic integrity and depositional ages of the sedimentary deposits at MRS. In this chapter, the data analysis and results of five sediment samples (MRS 5, 6, 7, 8 and 9) will be discussed. Sample locations are shown in Figures 4.3 and 4.4. The 5 samples were collected by Dr. Zenobia Jacobs in 2015 following the sampling procedures outlined in Section 3.5.1. Figure 4.8 shows a site schematic with sampling locations of the five samples, collected in squares 4 and 6. The five samples were prepared for analysis using the laboratory procedures discussed in Section 3.4.

![Schematic Reconstruction of Sampling Locations at MRS](image)

**Figure 4.8:** Schematic reconstruction of sampling locations at MRS (after field sketch by Dr. Robert Roberts).

4.4.1 Single grain OSL measurements

OSL dating of single quartz grains was conducted at MRS using the SAR protocol (Section 3.5.3) by stimulating each grain with a green laser operating at 90% power and an elevated temperature of 125°C for 2 seconds following a preheat (PH-1) of 260°C for 10 s for measurement of L_N and L_X signals and a preheat (PH-2) of 160°C for 5 s for measurement of T_N and T_X signals. Section 3.5.2 provides more detailed information about the equipment used.
for this process, and Section 3.5 provides a detailed account of how $D_e$ values are determined using the SAR protocol.

### 4.4.1 Dose recovery test

Prior to the measurement of the natural signals from the MRS samples, a dose recovery test was conducted on MRS 5 to determine the optimal preheat temperature combinations (PH-1 and PH-2) for single grain OSL measurement of all MRS samples. Enough grains for 15 single grain discs (Section 3.5.2) were exposed to natural sunlight for ~4 days to deplete the natural dose (Murray and Roberts, 1998; Roberts et al., 1999) and were then given a laboratory dose of ~73 Gy. Fifteen single grain discs (1500 grains) were tested at three preheat combinations (5 discs per combination): (1) 260°C for 10s and 220°C for 5s, (2) 240°C for 10s and 160°C for 5s, and (3) 260°C for 10s and 160°C for 5s. They were given regenerative doses of 58, 87, 115, 173, 229, 0 and a repeat dose of 58 Gy. Table 4.5 shows a breakdown of grain rejections for the samples analysed during dose recovery, and Figure 4.9a-c show the individual measured/given dose ratios for each PH combination as a radial plot. All three combinations resulted in high % of useable grains (32-35%). Combination 3 generated the best measured over given dose ratio of 0.97 ± 0.02, compared to combination 2 (0.93 ± 0.01), and combination 1 (0.91 ± 0.01). Interestingly, the OD value associated with combination 3 is significantly higher (23 ± 2%) due to outlying ratios that are consistently greater than ~1.4 (Figure 4.9c). The reason for their presence is currently unknown and can also be seen in the distribution for combination 1 (Figure 4.9a). Combination 3 was used as the optimum combination for measurements of all samples from MRS.

### Table 4.5: Results of dose recovery tests for all 3 combinations, including PH-1 and PH-2 temperatures, measured to given dose ratio, OD values, rejected and accepted grains.

<table>
<thead>
<tr>
<th>Preheat 1 (PH-1)</th>
<th>Preheat 2 (PH-2)</th>
<th>Measured to given dose ratio</th>
<th>OD values (%)</th>
<th>No. of grains measured</th>
<th>Sum of rejected grains</th>
<th>Accepted Grains</th>
</tr>
</thead>
<tbody>
<tr>
<td>240°C/10s</td>
<td>160°C/5s</td>
<td>0.908 ± 0.013</td>
<td>12.3 ± 1.3</td>
<td>500</td>
<td>340</td>
<td>160 (32%)</td>
</tr>
<tr>
<td>260°C/10s</td>
<td>220°C/5s</td>
<td>0.927 ± 0.013</td>
<td>14.5 ± 1.3</td>
<td>500</td>
<td>334</td>
<td>166 (33%)</td>
</tr>
<tr>
<td>260°C/10s</td>
<td>160°C/5s</td>
<td>0.970 ± 0.020</td>
<td>23.2 ± 1.7</td>
<td>500</td>
<td>325</td>
<td>175 (35%)</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>1500</td>
<td></td>
<td>501</td>
</tr>
</tbody>
</table>
4.4.2 Single grain measurements and rejection

All samples were measured using the preheat combination of 260°C for 10s and 160°C for 5s. The same range of regenerative doses of 58, 87, 115, 173, 229, 0 and a repeat dose of 58 Gy used in the above dose recovery tests were given to the actual samples to generate dose response curves. A repeat dose of 58 Gy was applied to each sample after it had been exposed to infra-red diodes for 40s at 50°C to obtain the OSL-IR depletion ratio (Section 3.5.3). A test dose of 11 Gy was applied after measurement of each natural dose ($L_N$) and regenerative dose ($L_X$) of each sample to test for sensitivity changes.

Figure 4.9: Radial plots of measured over given dose ratios for all 3 PH-1/PH-2 combinations.
Table 4.6: Summary of rejected grains from MRS 5-9 following criteria outlined in Section 3.5.4.3

<table>
<thead>
<tr>
<th>Sample name</th>
<th>No. of grains measured</th>
<th>( T_N ) signal &lt; 3xBG</th>
<th>( T_N ) error &gt; 20%</th>
<th>Recycling ratio &gt;2( \sigma ) from unity</th>
<th>IR depletion ratio &gt;2( \sigma ) from unity</th>
<th>Zero ( L_X/T_X ) &gt;5% of ( L_N/T_N )</th>
<th>Poor fit of saturated exponential curve</th>
<th>Sum of rejected grains</th>
<th>Acceptable individual ( D_e ) values</th>
</tr>
</thead>
<tbody>
<tr>
<td>MRS 5</td>
<td>1000</td>
<td>249</td>
<td>161</td>
<td>215</td>
<td>36</td>
<td>5</td>
<td>36</td>
<td>12</td>
<td>714</td>
</tr>
<tr>
<td>MRS 6</td>
<td>1000</td>
<td>315</td>
<td>178</td>
<td>199</td>
<td>59</td>
<td>14</td>
<td>14</td>
<td>9</td>
<td>788</td>
</tr>
<tr>
<td>MRS 7</td>
<td>1000</td>
<td>458</td>
<td>195</td>
<td>104</td>
<td>28</td>
<td>9</td>
<td>45</td>
<td>16</td>
<td>855</td>
</tr>
<tr>
<td>MRS 8</td>
<td>1000</td>
<td>444</td>
<td>171</td>
<td>141</td>
<td>29</td>
<td>6</td>
<td>17</td>
<td>27</td>
<td>835</td>
</tr>
<tr>
<td>MRS 9</td>
<td>900</td>
<td>349</td>
<td>168</td>
<td>110</td>
<td>51</td>
<td>7</td>
<td>27</td>
<td>36</td>
<td>748</td>
</tr>
<tr>
<td>Total</td>
<td>4900</td>
<td>1815</td>
<td>873</td>
<td>769</td>
<td>203</td>
<td>41</td>
<td>139</td>
<td>100</td>
<td>3940</td>
</tr>
</tbody>
</table>

Table 4.7: Summary of weighted mean \( D_e \) values and overdispersion values for MRS OSL 5-9, with their respective error values attained from using the CAM.

<table>
<thead>
<tr>
<th>Sample name</th>
<th>Number of Grains</th>
<th>Weighted Mean ( D_e ) (Gy)</th>
<th>OD (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>MRS 5</td>
<td>286</td>
<td>70.2 ± 1.4</td>
<td>28.5 ± 1.6</td>
</tr>
<tr>
<td>MRS 6</td>
<td>212</td>
<td>44.1 ± 1.2</td>
<td>31.0 ± 2.0</td>
</tr>
<tr>
<td>MRS 7</td>
<td>145</td>
<td>71.0 ± 2.0</td>
<td>32.0 ± 2.0</td>
</tr>
<tr>
<td>MRS 8</td>
<td>165</td>
<td>83.0 ± 2.0</td>
<td>26.0 ± 2.0</td>
</tr>
<tr>
<td>MRS 9</td>
<td>152</td>
<td>87.0 ± 2.0</td>
<td>27.0 ± 2.0</td>
</tr>
<tr>
<td>Total</td>
<td>960</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
A total of 4900 grains of quartz were measured (Table 4.6) and were subjected to the rejection criteria outlined in Section 3.5.4.3. A total of 960 (19.6%) grains passed the rejection criteria. This proportion of returned grains is quite high, but similar when compared to other sites in the region such as Diepkloof Rock Shelter (Jacobs and Roberts, 2017). A breakdown of the number of grains rejected based on the five primary criteria, and two additional criteria are shown in Table 4.6. Removal of these grains ensured that only suitable grains were used for $D_e$ determination and age calculation.

4.4.3 Decay curves and normalised decay curves

Figure 4.10 shows five representative T$_N$ decay curves for each sample after following a T$_D$ of 11 Gy and PH-2 of 160°C for 5s. Grains were selected to include two dim grains (<200 cts/0.02s), one moderately bright grain (200-900 cts/0.02s) and two bright grains (>900 cts/0.02s). Inset into each decay curve plot are the same five decay curves that have been normalised to the first data channel. Each decay curve is dominated by the ‘fast’ component (Bailey et al., 1997) and in most cases, after ~0.2s less than ~10-25% of the OSL signal remains. Li and Li (2006) characterise this rapid bleaching of grains as a desirable attribute in OSL dating. Most samples show grains with reproducible curve shapes between grains from the same sample. However, in some instances, some grains exhibit a slower rate of decay than others (e.g. yellow curve in Figure 4.10d). This characteristic is independent of grain brightness, as a similar pattern of slower decay is exhibited by the moderately bright grain (red) in Figure 4.10a.

4.4.4 Dose response curves

Representative dose response curves for the same five grains from each sample in Figure 4.10 are shown in Figure 4.11. The sensitivity-corrected dose points ($L_X/T_X$) were fitted using a saturating exponential or saturating exponential plus linear curve (Section 3.5.4.3). There are no inherent patterns between grains within each OSL sample that can be made in respect to the dose response curve shapes, as each grain has its own saturation characteristics, which vary independently from the brightness of the grain. However, some samples such as MRS 7 (Figure 4.11c) show all five grains with similar dose response curve shapes. If we consider the dimmest grains for each sample in Figure 4.11, indicated as the green curves, we can see that in Figure 4.11a, the dimmest grain saturated early, while in Figure 4.11e, the dimmest grain had the highest growth rate.
Figure 4.10: Decay curves for 5 representative single grains from a) MRS 5, b) MRS 6, c) MRS 7, d) MRS 8 and e) MRS 9. All grains were accepted for final $D_e$ determination. Grains are colour coded from dimmest to brightest (Green → Blue → Red → Yellow → Grey).
Figure 4.11: Dose response curves for the same 5 representative single grains for which the decay curves are plotted from Figure 4.10: a) MRS 5, b) MRS 6, c) MRS 7, d) MRS 8 and e) MRS 9. All grains were accepted for final D_e determination. Grains are colour coded from dimmest to brightest with Green > Blue > Red > Yellow > Grey.
Similarly, in Figure 4.11d, the moderately bright grain, indicated by the red curves, saturated much earlier than all other grains in MRS 8, but in MRS 5 the moderately bright grain had the highest growth for its dose response with increased dose.

4.4.5 $D_e$ determination and results

The individual $D_e$ values for each sample (MRS 5-9) were plotted as radial plots (Figure 4.12). In each radial plot, the grey band denote a 95% confidence interval, with the width of the grey band demonstrating a $\pm 2$ standardised estimate on each side of the weighted mean $D_e$ value. Overdispersion (OD) values (Table 4.7) quantify the spread of the data (scatter) after allowance was made for measurement uncertainties mentioned above (Galbraith et al., 1999). The number of $D_e$ values presented in each radial plot are indicated in Table 4.7 and Figure 4.12a-e. The radial plots provide a visual representation of the distributions of each sample, to determine the precision and potential patterns of each population of grains.

$D_e$ values for MRS 6 and MRS 7 shown as radial plots in Figure 4.12b and 4.12c have the highest OD values (~31% and 32%, respectively), when compared to the remaining three samples (Figures 4.12a, d, e). However, all five samples exhibit similar $D_e$ distributions, with the spread of each sample greater than can be explained by measurement uncertainties alone (Galbraith et al., 1999). Greater than 5% of grains fall outside the grey bands, with OD values ranging between 26 ± 2% (MRS 8) and 32 ± 2% (MRS 7). This extra scatter within the samples can be explained by many inherent and external factors, including differences in beta dose rate received by individual grains (beta micro-dosimetry), bioturbation caused by natural, anthropogenic and biogenic processes, different responses of individual grains during measurement such as the preheat conditions (Section 3.5.3). The majority of grains for all samples do fall within the grey bands, suggesting consistency. When examining the distributions in Figures 4.12a-e, there is no detectable difference between the sediment samples collected within the sedimentary wedge on the south wall (Figure 4.4), and the samples collected from the intact StratAggs outside the wedge on the east wall (Figure 4.3).

Weighted mean $D_e$ values were calculated using the CAM for final age determination. The CAM infers that all $D_e$ values for all grains are centred around the same average value. The uncertainties, such as the OD values, are incorporated. The final $D_e$ values and respective errors for all 5 samples are presented in Table 4.8.
Figures 4.12: Individual $D_e$ values and 1σ errors shown as radial plots for the 5 MRS samples. Also shown are the number of grains used and the final $D_e$ values. a) MRS 5, b) MRS 6, c) MRS 7, d) MRS 8, e) MRS 9, f) Summary of weighted mean $D_e$ values in stratigraphic order.
4.4.6 Environmental dose rate determination

Total environmental dose rates are determined from the beta (β) and gamma (γ) and cosmic radiation external to the individual grains, along with a minor contribution of alpha (α), radiation from the U and Th decay chain (Section 3.6) occurring inside sand sized quartz grains. For the calculations in this thesis, an α dose rate of $0.031 \pm 0.011 \text{ Gy/ka}$ was assumed for all samples. The β dose rates were estimated using a GM-25-5 multi counter (Bøtter-Jensen and Mejdahl, 1988). An allowance was made for beta-dose rate attenuation due to the impact of moisture content in each sample (Aitken, 1985). The measured moisture content for each sample is provided in Table 4.8. For these samples however, an assumed long-term value of moisture content of $5 \pm 2\%$ at 1σ is assumed.

An allowance for attenuation factors for a grain size of 180-212 μm was used for all samples (Mejdahl, 1979). The beta dose rates for all samples are presented in Table 4.8, and are also shown in stratigraphic order in Figure 4.13a. The values range from $0.93 \pm 0.04 \text{ Gy/ka}$ (MRS 9) to $1.20 \pm 0.05 \text{ Gy/ka}$ (MRS 5), and show that samples from the South Wall (Figure 4.4), located inside the stratigraphic wedge feature have beta dose rates ~27% higher than the samples collected along the East Wall (Figure 4.3). This may be a by-product of the increased potassium feldspar contents noted in Table 4.3, resulting in a greater contribution to the beta dose rate from potassium.

Gamma dose rates were measured in the field for 1800s (30 min) per sample with an in-situ gamma spectrometer that utilizes a 1-inch NaI (T) crystal. The gamma dose rates were estimated using the ‘threshold’ technique outlined by Mercier and Falguères (2007). This method gives an estimate of the combined dose rate of gamma ray emitters in the U and Th decay chains, as well as from $^{40}\text{K}$. Allowances were made for moisture content mentioned above. The γ dose rates are listed in Table 4.8, with values ranging from $0.68 \pm 0.03 \text{ Gy/ka}$ (MRS 9) to $0.81 \pm 0.03 \text{ Gy/ka}$ (MRS 6). The γ dose rates exhibit a similar pattern (Figure 4.13b) to those presented for the β dose-rates, where samples collected along the South Wall (Figure 4.4) have values higher than those collected from the East Wall (Figure 4.3).

Cosmic dose rates were calculated using a site altitude (~600 m.a.s.l) and geomagnetic latitude (~ -31°), density (1.8 g/cm³) and thickness of rock and sediment overburden (~8 m thick) (Prescott and Hutton, 1994). The calculated value used for all samples was $0.06 \pm 0.01 \text{ Gy/ka}$ (Table 4.8).
The total dose rate for all five samples were calculated by adding together the β, γ and cosmic dose rates from Table 4.8. These values ranged from $1.68 \pm 0.05\text{ Gy/ka}$ (MRS 8) to $2.06 \pm 0.06\text{ Gy/ka}$ (MRS 5). The total dose rate values are presented in Figure 4.13c, showing the higher total dose rates for samples collected from the South Wall (Figure 4.4) compared to those collected from the East wall (Figure 4.3). Further work needs to be done to understand the source of the higher dose rates along the South Wall.

**Figure 4.13:** Wet and attenuated a) beta, b) gamma and c) total dose rates for OSL samples collected at MRS, in relative stratigraphic order. Errors are at 1σ.

### 4.4.7 Mertenhof Rock Shelter OSL results

The final ages are presented in Table 4.8, along with the dose rate information, the weighted mean $D_e$ values and overdispersion values for each of the MRS samples. The aim of applying OSL dating for this site was to untangle the stratigraphic relationships presented by the complex sedimentary wedge. Two hypotheses were presented in Section 4.2.1 for how the sedimentary wedge was formed.
Hypothesis 1 had two variants, allowing for different linkages between the samples in the east and south sections. In hypothesis 1a, sample MRS6 (VDGS, south section) would be equivalent in age to MRS8 (LRS, east section), MRS6 would be older than MRS7 (LGS, east section), and the age of MRS5 (CWGS, south section) would fall between MRS8 and MRS9 (DGS, east section). Hypothesis 1b required that MRS5 was equivalent in age to MRS7, and thus that MRS6 would be younger than any samples in the east section.

In Hypothesis 2, the sediment within the wedge, represented by south section samples MRS 6 and 5, would be younger than all of the samples in the east section. In this scenario the wedge was deposited between the StratAggs LRS and RGB/S.

Five OSL ages were generated for MRS to test the 2 hypotheses for site formation. MRS 6 has the youngest OSL age of 22.4 ± 1.0 ka (Table 4.8), which was taken from the upper part of the sedimentary wedge in StratLayer VDGS (Figure 4.5b). MRS 5 has the second youngest age of 34.1 ± 1.4 ka (Table 4.8), representative of the lower part of the sedimentary wedge in StratLayer CWGS (Figure 4.5b). There is only ~8-10 cm between the two samples which would indicate a period of either extremely low sedimentation, no sedimentation, or erosional events over ~ 12,000-year period between the two samples.

MRS  7 is the next oldest sample in the sequence (StratAgg LGS, Figure 4.3), with an age of 40.8 ± 1.8 ka, followed by MRS 8 at 49.4 ± 2.0 ka (StratAgg LRS, Figure 4.3) and MRS 9 (StratAgg DGS, Figure 4.5b) at 51.2 ± 2.2 ka. The sediment samples from MRS 7-9 are significantly older, showing an increase in age from MRS 7 to MRS 8-9 (same age within error) inferring the presence of good stratigraphic integrity.

With the stratigraphic ages of MRS 6 and 5 being younger than those of MRS 7, 8 and 9, neither scenario from Hypothesis 1, presented in Section 4.2.1, can be validated. In the case of Hypothesis 2 however, MRS 5 and 6 have been proven to be younger than MRS 7, 8 and 9 (Table 4.8). It is probable that the sediment located within the wedge (StratLayers VDGS and CWGS) were their own sedimentary layers that at some point prior to 22.4 ± 1.4 ka dipped upward and continued over the tops of LGS (Figure 4.5b) before being truncated, removing the section that comprised the representative StratLayers VDGS and CWGS further toward the entrance of the rock shelter.
Table 4.8: Dose rate, $D_e$ values and single-grain quartz OSL ages for MRS.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Layer/ depth of layer</th>
<th>Adj. Water Content (Measured WC) (%)$^1$</th>
<th>Beta$^2$</th>
<th>Gamma$^3$</th>
<th>Cosmic$^4$</th>
<th>Total Dose Rate (Gy ka$^{-1}$)$^5$</th>
<th>$D_e$ (Gy)$^6$</th>
<th>No. Grains$^7$</th>
<th>$\sigma_d$ (%)$^8$</th>
<th>OSL age (ka)</th>
</tr>
</thead>
<tbody>
<tr>
<td>MRS 5</td>
<td>CWGS/~67cm</td>
<td>5 ± 2 (3.0)</td>
<td>1.20 ± 0.05</td>
<td>0.80 ± 0.02</td>
<td>0.06 ± 0.01</td>
<td>2.06 ± 0.06</td>
<td>70.2 ± 1.4</td>
<td>286</td>
<td>28.5 ± 1.6</td>
<td>34.1 ± 1.4</td>
</tr>
<tr>
<td>MRS 6</td>
<td>VDGS/~52cm</td>
<td>5 ± 2 (4.1)</td>
<td>1.08 ± 0.04</td>
<td>0.81 ± 0.03</td>
<td>0.06 ± 0.01</td>
<td>1.97 ± 0.06</td>
<td>44.1 ± 1.2</td>
<td>212</td>
<td>31.0 ± 2.0</td>
<td>22.4 ± 1.0</td>
</tr>
<tr>
<td>MRS 7</td>
<td>LGS/~38cm</td>
<td>5 ± 2 (3.5)</td>
<td>0.93 ± 0.04</td>
<td>0.73 ± 0.03</td>
<td>0.06 ± 0.01</td>
<td>1.74 ± 0.05</td>
<td>71.0 ± 2.0</td>
<td>145</td>
<td>32.0 ± 2.0</td>
<td>40.8 ± 1.8</td>
</tr>
<tr>
<td>MRS 8</td>
<td>LRS/~55cm</td>
<td>5 ± 2 (3.6)</td>
<td>0.93 ± 0.04</td>
<td>0.67 ± 0.03</td>
<td>0.06 ± 0.01</td>
<td>1.68 ± 0.05</td>
<td>83.0 ± 2.0</td>
<td>165</td>
<td>26.0 ± 2.0</td>
<td>49.4 ± 2.0</td>
</tr>
<tr>
<td>MRS 9</td>
<td>DGS/~69cm</td>
<td>5 ± 2 (4.1)</td>
<td>0.93 ± 0.04</td>
<td>0.68 ± 0.03</td>
<td>0.06 ± 0.01</td>
<td>1.70 ± 0.05</td>
<td>87.0 ± 2.0</td>
<td>152</td>
<td>27.0 ± 2.0</td>
<td>51.2 ± 2.2</td>
</tr>
</tbody>
</table>

1. Water contents were adjusted from laboratory measurements to better account for current and past levels of water content within the site.
2. Beta dose rates were determined using beta counting as outlined in section 3.6.2. The value is a mean ± 1σ standard error.
3. Gamma dose rates were determined in the field using in-situ gamma spectrometry.
4. Cosmic dose rates were determined using geomagnetic latitude, thickness of rock/sediment overburden and height above sea level.
5. Total dose values calculated through addition of beta, gamma and cosmic dose rates.
6. Mean $D_e$ values were determined using the central age model (CAM) created by Galbraith et al. (1999). Errors ± 1σ include laboratory uncertainties from beta-dose calibration.
7. Total number of grains measured from those grains with acceptable $D_e$ values after rejection criteria are applied.
8. Over-dispersion values represent the spread of $D_e$ values within a given sample population.

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This hypothesis is well supported by the OSL ages, and is the most likely scenario for how the wedge formed. It is also extremely pertinent that for the most part the OSL ages corroborate the estimated qualitative ages from the archaeological finds (Table 4.1).

4.5 Synopsis

This chapter contains a brief introduction to the archaeological site of MRS, including the sedimentological context of the sediment wedge being analysed within the greater site context, the importance of untangling the complex nature of this sedimentary wedge and the limited existing chronological analysis that had been conducted prior to the OSL analysis conducted in this thesis. Results for the sediment analysis, through grain size and geochemical data interrogation, along with optical dating results including: $D_e$ determination, environmental dose rates and final optical ages are also presented, providing some of the first ever chronometrically significant results for the site and its occupation during the MSA, particularly MIS 3. The implications of these findings will be discussed in Chapter 6.
Chapter 5: Pinnacle Point 5-6 North Rock Shelter
Chapter 5 – Pinnacle Point Site 5-6 (PP5-6)

5.1 Introduction

This chapter will describe Pinnacle Point Site 5-6 (PP5-6) within both its archaeological and geological contexts. This is followed by a brief description of the site history and excavation area. The significance of the site is then addressed, followed by an introduction to the sediment context of the site and scenarios created to place the complex stratigraphic feature (cut-and-fill) being studied within the site (Section 1.1) into its whole site context. The existing chronological framework for the site is provided, to show the current published extent of ages at PP5-6.

A detailed results section is presented on the OSL age determination of eight sediment samples (PP5-6 OSL 2,4,6-11) including: dose recovery results, single-grain quartz selection/rejection results, equivalent dose ($D_e$) determination, analysis and interpretation of decay and dose response curves for grain behaviour. Results for total dose rates, including beta, gamma and cosmic dose rate determination are also discussed. The equivalent dose rates and environmental dose rates are then used (Section 3.6) to provide ages for the eight analysed sediment samples. Finally, these age results are used to determine the validity of the hypotheses for the cut-and-fill.

5.1.1 Site setting and geology

PP5-6 (~34°12’20” S 22°05’29” E) is located on the southern coast of South Africa, ~3 km south of Mossel Bay and ~335 km east of Cape Town, beginning at an elevation of ~15 m.a.s.l. Figure 5.1 shows a map of the site location with respect to Cape Town and MRS. PP5-6 is nestled directly above the coastline, with the rock shelter opened dominantly to the east overlooking the Southern Indian Ocean. PP5-6 is situated within the species rich Cape Floristic Region and is dominated by the Fynbos biome and diverse shellfish populations, argued to be one of the primary reasons for its popularity for occupation by AMH during the past ~164 ka (McBrearty and Brooks, 2000; Marean et al., 2007; Bar-Matthews et al., 2010; Marean, 2010).

PP5-6 was formed from a coastal outcrop of the Skurweberg formation, which is part of the Nardouw subgroup of the Table Mountain Sandstones (TMS), the same subgroup in which MRS was formed in the Western Cape (Shone and Booth, 2005; Tham and Johnson, 2006; Brown, 2011).
The TMS consists of dominantly sandstone layers, with interbedded mudstones and shales generated through both marine and fluvial sedimentation processes (Tham and Johnson, 2006). PP5-6 is one of a collection of six excavation locations within the Pinnacle Point complex (Figure 5.2). As with the other caves and rock shelters along the coastline, PP5-6 was formed from weathering of brecciated fault zones within the quartzite bedrock. The weathering of the brecciated zones at Pinnacle Point was caused by multiple high stand sea level events, with the main cutting of the caves along the coast estimated to have occurred during the MIS 11 interglacial (Karkanas and Goldberg, 2010; Pickering et al., 2013; Karkanas et al., 2015). This was followed by sequential infill and erosion of soft sediment during repeated sea level transgressions/regressions until the start of sediment accumulation of the basal archaeological sediments at PP5-6, starting from ~90 ka, during MIS 5 (Brown, 2011). Successive anthropogenic and geogenic sedimentation continued to occur, with episodic truncational events due to dripline runoff during heavy rain, resulting in the erosional scouring of the sediment along the dripline. This erosional action resulted in a cone of sediment being preserved (Brown, 2011; Karkanas et al., 2015).
5.1.2 Site history and excavation area

Excavation began at PP5-6 in 2006 after a partially exposed outcrop of MSA archaeology covered by a modern sand dune was discovered (Brown, 2011). A ‘long-section’ of the sediment cone was excavated layer by layer into in-situ sediments, in 1x1 m squares (Figure 5.3). From December 2006 until March 2017, regular excavation seasons were conducted at PP5-6. As of 2015, the dimensions of the excavated long-section were ~8 m wide, ~30 m long and 14 m deep. Excavated squares are the filled white squares in figure 5.3 (Karkanas et al., 2015).

Figure 5.3: Excavated grids of the ‘long-section’ as of 2015, with the sampling of PP5-6 OSL 2,4,6-11 used in this paper, collected from the squares outlined in red (image after Karkanas et al. (2015).
Excavation of four squares was carried out for the samples collected in this thesis (OSL 2, 4, 6-11), with Figure 5.3 indicating the squares excavated in the red box. Whilst excavating these squares, a complex stratigraphic feature was identified.

### 5.1.3 Site significance

PP5-6 was first occupied by modern humans ~ 90 ka, during MIS 5. Initial occupation is thought to have been episodic, with only small groups staying for short periods of time (Karkanas et al., 2015). It has been suggested that low occupancy may have been because humans mostly exploited shorelines in close proximity to the cave system, resulting in a finite amount of resources (Karkanas et al., 2015). With the onset of glacial conditions during MIS4, PP5-6 appears to have been occupied more frequently, with evidence of longer and more dense populations at the site up until ~ 50 ka (Brown et al., 2012; Karkanas et al., 2015). However, later excavation during the 2015-2017 excavation seasons on the upper section (BBCSR StratAgg) (Table 5.1) revealed evidence for human occupation at PP5-6 during MIS3 (Karkanas et al., 2015). This marks the current extent of knowledge for human occupation at PP5-6. Brown et al. (2012) presented OSL ages of ~71-60 ka from SADBS, DBSC and OBS1 (Table 5.2) for the existence of advanced bladelet technology. The backed bladelet microlithic technology from SADBS were found to be different from those of other HP sites in South Africa, as well as in other African sites (Brown et al., 2012).

### 5.2 Sedimentological context

The sedimentary deposits at the site are generally well bedded, stratigraphically intact units, with some small-scale evidence of bioturbation and post depositional alteration (Karkanas et al., 2015). The stratigraphy has been separated into Stratigraphic Aggregates (StratAggs), and smaller StratUnits that make up the StratAggs. Table 5.1 lists and describes the main StratAggs. In this project, there are three main StratAggs that are focused on which are BBCSR, BAS and the OBS2.

In places, the BAS StratAgg occasionally outcrops directly overlying the OBS2 StratAgg. Communication with the site geologist, Panagiotis Karkanas, suggests that the BBCSR and BAS which originally represent upper and lower sections of the same StratAgg, were in-fact two separate StratAggs. This stratigraphic relationship is complicated by the presence of a complex stratigraphic feature, or ‘cut-and-fill feature’ (CAFF). Part of the problem to be addressed in this thesis, is to determine where exactly this CAFF is positioned in context to the upper stratigraphy of the site. The CAFF was excavated in January 2017,
exposing a North (Figure 5.4) and East (Figure 5.5) facing profiles. There is some evidence of occupation, represented by an intact hearth structure (Mary) in Figure 5.4. For the purposes of this study, the StratUnits within the CAFF were given arbitrary names. Table 5.2 provides descriptions of the individual stratigraphic layers that make up the CAFF. Figure 5.6 is a field sketch of the North section and stratigraphic layers that make up the CAFF. Figure 5.7 is a field sketch of the East section and stratigraphic layers that make up the CAFF. Appendix 2 provides photographs of the sections with the OSL samples annotated with their field sample numbers.

**Table 5.1:** Description of documented sediment *StratAggs* at PP5-6 in relative stratigraphic order, including the approximate thickness of the *StratAggs*, composition and depositional mechanisms. Weighted average mean OSL ages for existing *StratAggs* are also shown.

<table>
<thead>
<tr>
<th>Stratigraphic Aggregate (Top to Bottom)</th>
<th>Age (ka)</th>
<th>Thickness (m)</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>RBSR</td>
<td>51 ± 2</td>
<td>2.75</td>
<td>Reddish-brown aeolian sand. Varying roof-spall content.</td>
</tr>
<tr>
<td>BBCSR</td>
<td>52 ± 3</td>
<td>0.5</td>
<td>Dark compact sand. Very rich in burnt remains, and dense in archaeological finds. Sits above OBS2.</td>
</tr>
<tr>
<td>BAS</td>
<td>61 ± 3</td>
<td>0.5</td>
<td>The BAS was recently allocated as the upper section of the old BCSR <em>StratAgg</em>, sitting below BBCSR. It consists of intact layered ash and in some places directly overlies the OBS2 layer.</td>
</tr>
<tr>
<td>DBCS</td>
<td>62 ± 3</td>
<td>0.75</td>
<td>Dark brown compact sand. Lateral variation of BCSR, sitting at lower elevation than BBCSR. Includes eroded sediments of OBS2. Strongly dipping to SE of shelter. Extreme density of archaeological finds.</td>
</tr>
<tr>
<td>OBS2</td>
<td>63 ± 3</td>
<td>1.0</td>
<td>Beige and brown sands like OBS1, except it is dominated by decalcified sands and combustion SubAggs.</td>
</tr>
<tr>
<td>SGS</td>
<td>64 ± 3</td>
<td>0.3</td>
<td>Gray sand, with shell material. Aeolian dominated. High frequency lithic artefact and faunal fragments.</td>
</tr>
<tr>
<td>OBS1</td>
<td>69 ± 3</td>
<td>0.7</td>
<td>Beige and brown sand. Interrupted by tabular layers of sand rich ash bands. Intact hearth features, but most trampled and reworked. Aeolian, sheet wash and debris flow deposited.</td>
</tr>
<tr>
<td>SADBS</td>
<td>71 ± 3</td>
<td>0.7</td>
<td>Dark brown sand and white ash lenses. Rich in trampled combustion features, with no complete single hearth structures discernible. Aeolian deposited</td>
</tr>
<tr>
<td>ALBS</td>
<td>72 ± 3</td>
<td>0.8</td>
<td>Beige sand, draping collapsed quartzite roof-spall. Chaotic distorted appearance of coarser components. Aeolian deposited.</td>
</tr>
<tr>
<td>YBSR</td>
<td>89 ± 5</td>
<td>1.25</td>
<td>Yellow/brown coloured sand. Sharp contact with YBS. Consists of significant amounts of roof-spall, and white roof-spall rich ash. Interbedded lenses of combustion activity indicating AMH occupation. Small scale debris flow. Aeolian and sheetwash deposited.</td>
</tr>
<tr>
<td>YBS</td>
<td>96 ± 6</td>
<td>3.0</td>
<td>Beige coloured sand devoid of anthropogenic activity, deposited by aeolian activity.</td>
</tr>
</tbody>
</table>
Figure 5.4: Photograph of North Wall with stratigraphic layers indicated together with position of collected OSL samples.
5.2.1 Existing chronological framework

The sedimentary sequence at PP5-6 has been heavily dated using OSL (Brown et al., 2012; Karkanas et al., 2015). Table 5.1 lists the weighted mean OSL ages for each StratAgg in stratigraphic order. Ages range from ~96 ± 6 ka (StratAgg YBS) to ~51 ± 2 ka (RBSR), representing an almost continuous sedimentary record over an ~45 ky period.

The OSL ages provided in Table 5.1 show that no dating has yet been published for sediment younger then ~51 ± 2 ka. The sediment being dated in this thesis, if younger than this age, could prove to be the youngest samples analysed for PP5-6, potentially filling in information for human occupation (Mary) of PP5-6 during MIS3.
5.2.2 Hypotheses for context of complex sedimentary feature (CAFF) and hearth feature (Mary)

A primary aim of this thesis, is to develop and test different hypotheses for how and when complex stratigraphic features occurred at multiple sites. A second aim at PP5-6 was to determine the stratigraphic context of the isolated hearth structure (Mary – Figure 5.4), which should become clear after understanding when the CAFF formed. Accomplishing these aims will allow for a more complete understanding of site stratigraphy, site formation processes and enhance existing understandings of human occupation at PP5-6.

Table 5.2: Descriptions of StratLayers analysed to determine the context of the CAFF at PP5-6. Arbitrary names were created by the author and geologist Panagiotis Karkanis to help explain stratigraphic relationships within the context of the feature.

<table>
<thead>
<tr>
<th>Stratigraphic Layer (Top to Bottom)</th>
<th>Section</th>
<th>Thickness (m)</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zebra (OSL 2 &amp; 4)</td>
<td>E-W</td>
<td>-</td>
<td>Highly laminated and cross bedded sands giving indication of water flow processes.</td>
</tr>
<tr>
<td>Giraffe</td>
<td>E-W</td>
<td>0.5</td>
<td>Rich in circular mottled stones. Stones are randomly orientated. Evidence of iron oxide-rich red bands, but dominated by coarse to fine grey/brown sands with poor sorting.</td>
</tr>
<tr>
<td>Mary</td>
<td>E-W</td>
<td>0.7</td>
<td>Charcoal-rich hearth structure.</td>
</tr>
<tr>
<td>Caitlyn (OSL 6)</td>
<td>E-W</td>
<td>0.75</td>
<td>Fine-grained sand matrix surrounding highly chaotic stony fragments exhibiting no orientation patterns. Brown to orange in colour, with possible grey ash banding.</td>
</tr>
<tr>
<td>Panther (OSL 7 &amp; 8)</td>
<td>N-S</td>
<td>1.0</td>
<td>Extremely chaotic with dark bands, red bands, and brown bands of sandy/silt sediment. Significant clasts ranging up to 20 cm in size. Dominant clasts consist of quartzite likely derived from the overhanging quartzite rock shelter (roof-spal).</td>
</tr>
<tr>
<td>Lion (OSL 9)</td>
<td>N-S</td>
<td>0.3</td>
<td>Likely continuation of Hyena, with a lower abundance of large stones. Sediment is lighter in colour than Hyena, with less dark grey/black ash/charcoal material.</td>
</tr>
<tr>
<td>Hyena</td>
<td>N-S</td>
<td>0.7</td>
<td>Highly mottled and well-mixed dark coloured layer. Colour is brown to orange, containing abundant clasts indicating higher energy deposition, possibly by smaller scale debris flow. This section is part of the cut-and-fill sequence.</td>
</tr>
<tr>
<td>Leopard</td>
<td>N-S</td>
<td>0.7</td>
<td>Coarsening upward sand from Tiger to Leopard. Layered clasts of larger rock fragments become more noticeable indicating increased energy of deposition from that of Tiger. Also noticeable are rip-up clasts of red and black material, possibly derived from overlying sediment of Hyena. Evidence of laminated bedding.</td>
</tr>
<tr>
<td>Tiger (OSL 10 &amp; 11)</td>
<td>N-S</td>
<td>0.8</td>
<td>Evidence of laminated bedding of fine to medium grained sands, possibly deposited by channel/rill action. North side of boulder represented in Figure 5.7, the sediment is finer, containing some layers of larger rock inclusions imbricated toward the south. Could be roof-spal or rip-up clasts. The coarsening upward and larger fragments give evidence for channel deposition over aeolian deposition.</td>
</tr>
</tbody>
</table>
Figure 5.6: Schematic drawing of stratigraphic relationships and OSL sample locations on the East wall of Sq.3 and Sq.4.
Figure 5.7: Schematic drawing of stratigraphic relationships and OSL sample locations on the North Wall of Sq.1 and Sq.2 of PP5-6.
The complex sedimentary feature at PP5-6 is a CAFF, which is thought to have been created during heavy rain events, where water would run off from the rock shelter dripline, pool and run downslope cutting an erosional feature into the soft underlying sediment. Understanding how the feature formed was relatively straightforward, as the sediment, particularly in Tiger and Leopard StratLayers showed good examples of laminar bedding caused by surface flow of water (Table 5.2). To determine when the cut and the subsequent filling event occurred, two hypotheses were formulated and tested using OSL dating. The fill is believed to have been deposited rapidly in a single event. The CAFF was proposed in the field to consist of all of the StratLayers in Table 5.2, with the possibility that Panther was or was not part of the feature. The southern extent of the CAFF was proposed to be where Lion and OBS2 meet.

**Hypothesis 1** – Schematic depictions of the stratigraphic relationships between Panther, Lion, BAS and OBS2 (Figure 5.6) in the East Wall of the excavation for this project, show a complex relationship between the perceived CAFF and the surrounding stratigraphy. In this hypothesis, it is proposed that Panther is the upper most section of the fill, which was deposited after truncation of both BAS and OBS2. For this hypothesis to be confirmed, it requires that OSL 7-11 will be younger than the already posited age of OBS2 which was dated to 63 ± 3 ka (Table 5.1).

**Hypothesis 2** - In this scenario, Panther is considered to belong to BAS. The CAFF, therefore, occurred after deposition of OBS2 (63 ± 3 ka), but before deposition of BAS (61 ± 3 ka), therefore only truncating OBS2. This means, we would expect OSL 9-11 on the East Wall (Figure 5.6) to be older then OSL 7 and 8, which were taken from Panther (Figure 5.6). OSL 6 is a lateral equivalent sample of the base of the CAFF on the North Wall (Figure 5.7), therefore, it is expected that OSL 6 is equivalent in age to OSL 10-11. This infers that the hearth structure (Mary) on the North Wall (Figure 5.7) is only younger than OBS2, but older then the BAS because it sits above the base of the fill, which is posited to have been deposited rapidly in a single event.
5.3 OSL results

OSL dating of single grains of quartz is one of the primary methods used in this thesis to investigate the stratigraphic complexities imposed by the CAFF, and the depositional ages of the natural and anthropogenic sedimentary deposits at PP5-6. In this chapter, the data analysis and results of eight sediment samples (OSL 2, 4, 6-11) will be discussed. Relative sample locations are shown in Figure 5.8 and actual sample locations are shown in Figures 5.4 and 5.5. The eight samples were collected by the author of this thesis in March 2017 following the sampling procedures outlined in Section 3.3. Figure 5.8 shows a site schematic of sampling location with respect to the excavated squares. The eight samples were prepared for analysis using the laboratory procedures discussed in Section 3.4.

5.3.1 Single grain OSL measurements

OSL dating of single quartz grains was conducted at PP5-6 using the SAR protocol (Section 3.5.3) by stimulating each grain for 2 seconds with a green laser operating at 90% power and an elevated temperature of 125°C. PH-1 was 240°C for 10 seconds prior to the measurement of $L_N$ and $L_X$ and PH-2 at 160°C for 5 seconds prior to measurement of $T_N$ and $T_X$. 

Figure 5.8: Schematic reconstruction of sampling locations at PP5-6. Reconstruction indicates approximate layout of excavated pit for this project.
5.3.2 Dose recovery test

A large number of dose recovery tests had previously been conducted at PP5-6 and other sites at Pinnacle Point such as PP13B (Brown et al., 2012; Karkanas et al., 2015). Because of these previous dose recovery tests, a preheat combination of 240°C for 10s (PH-1) and 160°C for 5s (PH-2) were decided upon for all measurements. Following these measurements, a dose recovery test was conducted on OSL 6 to test validity of the preheat temperatures (PH-1 and PH-2) used for the single grain measurements of OSL 2, 4, 6-11. Enough grains for 5 single grain discs (500) were exposed to natural sunlight for ~4 days to bleach them of their natural dose signals (Murray and Roberts, 1998; Roberts et al., 1999) and given a laboratory dose of ~58 Gy. Five single grain discs of quartz were tested using the preheat combination of 240°C for 10s and 160°C for 5s. The sample quartz grains were given regenerative doses of ~29, 58, 88, 117, 146, 0 and a repeat dose of 29 Gy. Table 5.3 provides a breakdown of grain rejections for OSL 6, with eleven percent of grains accepted. The preheat combination of 240°C for 10s and 160°C for 5s for this dose recovery test had a measured to given ratio of 1.18 ± 0.02 (Table 5.3; Figure 5.10), which is an overestimation of the dose by ~18%. This measured to given ratio was a highly unexpected result, indicating that further dose recovery testing is required. In future, analysis of these samples at a higher PH-1 and PH-2 combination may be required to bring this value closer to unity and to test the effect of the different preheat temperatures on D_e and age calculations of these samples (Murray and Wintle, 2003; Preusser et al., 2009). The OD value for the dose recovery preheat combination was low (5 ± 3%).

Table 5.3: Results of dose recovery test including measured to given dose ratio, OD values, and total rejected/accepted grains.

<table>
<thead>
<tr>
<th>Preheat 1 (PH-1)</th>
<th>Preheat 2 (PH-2)</th>
<th>Measured to given dose ratio</th>
<th>OD values (%)</th>
<th>No. of grains measured</th>
<th>Sum of rejected grains</th>
<th>Accepted Grains</th>
</tr>
</thead>
<tbody>
<tr>
<td>240°C/10s</td>
<td>160°C/5s</td>
<td>1.18 ± 0.02</td>
<td>5 ± 3</td>
<td>400</td>
<td>356</td>
<td>44 (11%)</td>
</tr>
</tbody>
</table>
5.4.3 Single grain quartz rejection

The eight OSL samples were analysed using the preheat combination of 240°C for 10s and 160°C for 5s. Regenerative doses of 44, 66, 89, 131, 175, 0 and a repeat dose of 44 Gy were given to each sample to generate dose response curves. A repeat dose of 58 Gy was applied to each sample after it had been exposed to infra-red diodes for 40 s at 50°C to obtain the OSL-IR depletion ration (Section 3.5.3). A test dose of ~11 Gy was applied to each quartz grain after measurement of each natural dose and regenerative dose. A total of 7800 grains of quartz were analysed for eight samples at PP5-6, and were subjected to the rejection criteria outlined in Section 3.4.5.3. A total of 417 (5%) grains passed the rejection criteria. A breakdown of the grains rejected based on the 5 primary criteria, and two additional criteria are shown in Table 5.4. Removal of these aberrant grains ensured that only $D_e$ values were calculated for appropriate grains.

**Figure 5.10:** Radial plot of measured over given dose ratios for a preheat combination of 240°C for 10s and 160°C for 5s.

**Table 5.4:**

<table>
<thead>
<tr>
<th>PP5-6 OSL 6 240°C for 10s/160°C for 5s</th>
</tr>
</thead>
<tbody>
<tr>
<td>N = 44</td>
</tr>
<tr>
<td>OD = 5 ± 3%</td>
</tr>
<tr>
<td>Mean ratio = 1.18 ± 0.02</td>
</tr>
</tbody>
</table>
Table 5.4: Summary of rejected grains from OSL 2, 4, 6-11 following rejection criteria outlined in Section 3.5.4.3. Samples are not listed in stratigraphic order, but numerical order.

<table>
<thead>
<tr>
<th>Sample name</th>
<th>No. of grains measured</th>
<th>$T_N$ signal &lt; 3xBG</th>
<th>$T_N$ error &gt; 20%</th>
<th>Recycling ratio &gt;2σ from unity</th>
<th>IR depletion ratio &gt;2σ from unity</th>
<th>Zero $L_X/T_X$ &gt;5% of $L_N/T_N$</th>
<th>Poor fit of saturated exponential curve</th>
<th>Saturated</th>
<th>Sum of rejected grains</th>
<th>Acceptable individual $D_e$ values</th>
</tr>
</thead>
<tbody>
<tr>
<td>PP5-6 OSL2</td>
<td>1000</td>
<td>606</td>
<td>246</td>
<td>24</td>
<td>12</td>
<td>57</td>
<td>27</td>
<td>6</td>
<td>978</td>
<td>22</td>
</tr>
<tr>
<td>PP5-6 OSL 4</td>
<td>1000</td>
<td>610</td>
<td>223</td>
<td>30</td>
<td>14</td>
<td>46</td>
<td>25</td>
<td>10</td>
<td>958</td>
<td>42</td>
</tr>
<tr>
<td>PP5-6 OSL 6</td>
<td>1000</td>
<td>607</td>
<td>233</td>
<td>35</td>
<td>20</td>
<td>12</td>
<td>29</td>
<td>12</td>
<td>948</td>
<td>52</td>
</tr>
<tr>
<td>PP5-6 OSL 7</td>
<td>900</td>
<td>486</td>
<td>233</td>
<td>55</td>
<td>8</td>
<td>16</td>
<td>25</td>
<td>26</td>
<td>849</td>
<td>51</td>
</tr>
<tr>
<td>PP5-6 OSL 8</td>
<td>1000</td>
<td>542</td>
<td>271</td>
<td>40</td>
<td>18</td>
<td>18</td>
<td>27</td>
<td>31</td>
<td>947</td>
<td>53</td>
</tr>
<tr>
<td>PP5-6 OSL 9</td>
<td>900</td>
<td>514</td>
<td>224</td>
<td>27</td>
<td>12</td>
<td>1</td>
<td>27</td>
<td>48</td>
<td>852</td>
<td>48</td>
</tr>
<tr>
<td>PP5-6 OSL 10</td>
<td>1000</td>
<td>552</td>
<td>266</td>
<td>35</td>
<td>9</td>
<td>9</td>
<td>28</td>
<td>20</td>
<td>919</td>
<td>81</td>
</tr>
<tr>
<td>PP5-6 OSL 11</td>
<td>1000</td>
<td>578</td>
<td>229</td>
<td>37</td>
<td>11</td>
<td>8</td>
<td>45</td>
<td>24</td>
<td>932</td>
<td>68</td>
</tr>
<tr>
<td>Total</td>
<td>7800</td>
<td>4495</td>
<td>1925</td>
<td>283</td>
<td>104</td>
<td>167</td>
<td>233</td>
<td>177</td>
<td>7384</td>
<td>417</td>
</tr>
</tbody>
</table>
5.4.4 Decay curves and normalised decay curves

Figure 5.11 shows five representative T\textsubscript{N} decay curves for each sample following a T\textsubscript{D} of 11 Gy and a PH-2 of 160°C for 5s. Grains were selected to include two dim grains (<150cts/0.02s), one moderately bright grain (150-300cts/0.02s) and two bright grains (>300cts/0.02s) (Figure 5.11). Inset into each decay curve are the same five decay curves that have been normalised to the first data channel. Each decay curve is dominated by the ‘fast’ component (Bailey et al., 1997) and in most cases, after ~0.2 s, less than 5-15% of the OSL signal remains.

This is not the case for some grains, for example the yellow grain in Figure 5.11b (yellow) shows a much slower decay, with ~50% of the signal left after ~0.2 s of stimulation time. This also occurs in other samples, such as Figure 5.11e, where the red and grey grains exhibit slower decay rates than the other three grains.

5.4.5 Dose response curves

Dose response curves were constructed using the same five grains for each sample for which decay curves were shown in Figure 5.11. The dose response curves were fitted with a saturating exponential function plus additional linear term (Section 3.5.4.3). In all samples (Figure 5.12) the dose response curves show a similar shape up to ~25 Gy, from where the curves start to deviate. There is no relationship between inherent signal brightness and curve shape. Figure 5.12b and d, for example, show the dose response curves for bright grains in grey that continuously grow to higher doses. The dose response curves in Figure 5.12e and f, one of the dimmest grains for each of these three samples (green) exhibit continued growth when grains with greater inherent brightness’s show earlier saturation characteristics.

5.4.6 D\textsubscript{e} determination and results

The individual D\textsubscript{e} values for each sample (OSL 2, 4, 6-11) were plotted as radial plots following the rejection process (Figure 5.13). The data displayed within a radial plot is discussed in Section 3.4.5. OD values for OSL 2, 4, 6-11 are presented in Table 5.6. The number of grains presented in each radial plot is indicated in Table 5.5 and Figure 5.13a-h. The radial plots provide a visual representation of the distributions for OSL sample 2, 4, 6-11 to determine the precision and potential patterns of each population of grains.

OSL 2, 4, 6 and 8 (Figure 5.13a, b, c and e) all exhibit higher OD values than the other samples from PP5-6. A test was conducted on OSL 2, 4, 6 and 8 to determine if these values were the result of anomalous outlier grains. In each population from the above four samples,
Figure 5.11: Decay curves for 5 representative single grains from a) OSL 2, b) OSL 4, c) OSL 6, d) OSL 7, e) OSL 8, f) OSL 9, g) OSL 10, h) OSL 11. All grains were accepted for final $D_e$ determination. Grains are colour coded from dimmest to brightest (Green $\rightarrow$ Blue $\rightarrow$ Red $\rightarrow$ Yellow $\rightarrow$ Grey).
Figure 5.12: Dose response curves for the same 5 representative single grains for which decay curves were presented in Figure 5.12, from a) OSL 2, b) OSL 4, c) OSL 6, d) OSL 7, e) OSL 8, f) OSL 9, g) OSL 10, h) OSL 11. All grains were accepted for final $D_e$ determination. Grains are colour coded from dimmest to brightest (Green → Blue → Red → Yellow → Grey).

two of the grains grains that were identified as lying the furthest out from the weighted mean $D_e$ value were removed which resulted in a significant reduction in the OD values for each sample. For example, OSL 4 resulted in an OD of $\sim 30 \pm 5\%$, and weighted mean $D_e$ value of
44 ± 3 Gy and OSL 6 resulted in an OD value of 40 ± 5% with weighted mean $D_e$ of 57 ± 4 Gy. To test how much these changes affected the age of the samples, all four were recalculated and the ages were found to be within the error margins. This extra scatter across the four samples above can be explained by many inherent and external factors, including differences in beta dose rate received by individual grains (beta micro-dosimetry), bioturbation caused by natural, anthropogenic and biogenic processes, different responses of individual grains during measurement such as the preheat conditions or by contamination during sampling (Galbraith and Roberts, 2012). The most likely reason for these samples is a combination of beta micro-dosimetry and response to preheat conditions. In some layers such as Tiger and Leopard (Table 5.2), there is evidence of rip-up clasts which might represent older grains being mixed with younger grains. Chaotic orientations of these clasts in the CAFF probably indicate some mixing between layers during cutting and subsequent deposition of the fill.

The presence of the outlying grains warrants further investigation into why the OD values for these samples are so high. Analysis conducted on OSL samples collected from PP5-6 have shown quite low OD values of around 15-25% (Brown et al., 2009). In particular, it would be prudent to conduct further work on the outlying grains using normalised median absolute deviation (nMAD) and test the sensitivity of weighted mean ages based on the inclusion and exclusion of the outlying grains.

5.4.7 Environmental dose rate determination

Total environmental dose rates are determined from the beta ($\beta$) and gamma ($\gamma$) and cosmic radiation external to the individual grains, along with a minor contribution of alpha ($\alpha$), radiation from the U and Th decay chain (Section 3.6) occurring inside sand sized quartz grains. For the calculations in this thesis, an $\alpha$ dose rate of 0.031 ± 0.011 Gy/ka was assumed for all samples. The $\beta$ dose rates were estimated using a GM-25-5 multi counter (Bøtter-Jensen and Mejdahl, 1988). An allowance was made for beta-dose rate attenuation due to the impact of moisture content and grain size in each sample (Aitken, 1985). See section 3.6.2 for further information. The measured moisture content for each sample is provided in Table 5.5. OSL 2, 4, 6, 9-11 were assigned an assumed long-term value of moisture content of 5 ± 2% at 2σ. For OSL 7 and 8 however, a long-term value of 10 ± 3% was used due to the increased organic material within the Panther StratLayer.

A grain size of 180-212 μm was used for all samples when making allowances for attenuation factors (Mejdahl, 1979). The beta dose rates for all samples are presented in
Figures 5.13: Individual $D_e$ and 1σ errors shown as radial plots for the 8 PP5-6 samples. Also shown are the number of grains and final $D_e$ values for age. a) PP5-6 OSL 2, b) PP5-6 OSL 4, c) OSL 6, d) OSL 7, e) OSL 8, f) OSL 9, g) OSL 10, h) OSL 11.
Table 5.5, and are also shown in Figure 5.14. The values range from 0.53 ± 0.03 Gy/ka (OSL 8 and 10) to 0.72 ± 0.03 Gy/ka (OSL 9). The spread of beta dose rates is quite small between all other samples (Figure 5.14).

Gamma dose rates were measured in the field for 1800s (30 min) per sample with an in-situ using two gamma spectrometers that utilized either a 1.5 or a 2-inch NaI (T) crystal. More detail on the technique used can be found in Section 3.6.3. Allowances were made for moisture content mentioned above. The gamma dose rates are listed in Table 5.5, with values ranging from 0.54 ± 0.02 Gy/ka (OSL 2) to 1.07 ± 0.05 Gy/ka (OSL 4). The gamma dose rates generally show a similar pattern to the beta dose rates (Figure 5.14b), however, OSL 2 exhibits a much lower gamma dose rate when compared to all other samples. Understanding why OSL 2 has such a low gamma dose rate warrants further investigation. One possibility is that the gamma spectrometer used was malfunctioning, as it is the only sample taken by the 1.5-inch spectrometer.

Cosmic dose rates were calculated using a site altitude (~25 m.a.s.l) and geomagnetic latitude (~ -33°), density (1.8 g/cm^3) and thickness of rock and sediment overburden (~8 m thick) (Prescott and Hutton, 1994). The calculated value used for all samples was 0.09 ± 0.01 Gy/ka (Table 5.5).

The total dose rate for all 5 samples were calculated by adding together the beta, gamma and cosmic dose rates from Table 5.5. These values ranged from 1.32 ± 0.04 Gy/ka (OSL 2) to 1.81 ± 0.06 Gy/ka (OSL 4). The total dose rate values are presented in Figure 5.14, showing a much lower total dose rate for OSL 2 (Figure 5.14c). The other samples are generally showing a similar trend to the beta and gamma plots.

5.4.8 PP5-6 OSL results

The final ages are presented in Table 5.5, along with the combination of dose rates from beta, gamma and total environmental dose rates, the representative D_e values with respective over-dispersion values for each of the PP5-6 samples.

The aim of applying OSL dating to PP5-6 was to determine the depositional sequence and extent of the CAFF feature, and its stratigraphic relationship within context of the upper StratAggs of the site. Three hypotheses were presented in Section 5.2.1 to interpret the stratigraphic relationship of the CAFF with the hearth structure (Mary) in the North Wall (Figure 5.4), the StratLayer Panther in the East Wall, and the already dated StratAggs BAS (<52 ± 3 ka) and OBS2 (63 ± 3 ka). Hypothesis 1 proposed that Panther was the upper most
section of the CAFF and that the CAFF had truncated BAS and OBS2 (Figure 5.6). To validate this, it requires that OSL 7-11 be younger than the already posited age of OBS2 which was
dated to 63 ± 3 ka (Table 5.1). This would make the CAFF younger then both BAS and OBS2 aggregates, and the hearth structure by default the younger still.

Hypothesis 2 proposed that Panther belongs to the bottom of BAS StratAgg, and that the cut event truncated OBS2 before Panther was deposited. This means, we expected OSL 9-11 on the East Wall (Figure 5.6) to be older then OSL 7 and 8 which were taken from Panther. OSL 6 is a lateral equivalent sample of the base of the fill on the North Wall (Figure 5.4), therefore, it is expected that OSL 6 is equivalent in age to OSL 10-11. This infers that the hearth structure (Mary) on the North Wall (Figure 5.5) is only younger than OBS2, but older than the BAS.

Eight OSL ages were obtained for PP5-6 to test these two hypotheses. OSL 4 has the youngest OSL age of 20.6 ± 2.3 ka (Table 5.5), which was taken from directly above the hearth structure Mary (Figure 5.4). OSL 6, 10 and 11 have the next youngest ages of 31.4 ± 2.7, 32.0
± 1.7 and 33.5 ± 2.2 ka respectively (Table 5.5). Interestingly, these 3 samples were taken from the fill part of the CAFF, towards its base, and are statistically indistinguishable from each other. OSL 9 (Figure 5.6) was believed to be part of the fill, however its OSL age of 47.2 ± 2.9 ka makes it the oldest OSL age obtained in this study of PP5-6 and similar to the previously youngest occupation deposits obtained from BBCSR. This places it between the older OBS2 and BAS and younger sediments from the CAFF. OSL 7 and 8 are the second oldest samples, with ages of 41.3 ± 3.1 and 43.4 ± 3 ka respectively, placing them stratigraphically above PP5-6 OSL 9, but older then PP5-6 OSL 6, 10 and 11.

This therefore confirms, that Lion and Panther do not belong to the CAFF feature, ruling out both proposed hypotheses (Section 5.2.1). This also places the CAFF within the late MIS-3, and potentially places Mary within between MIS3 and MIS2. Further investigation is required to develop a new hypothesis for the context of the CAFF, and revisit the hypothesis that the CAFF represents one event as this may not be the case.

5.5 Synopsis

This chapter contains a brief introduction to the archaeological site of PP5-6, including the sedimentological context of the CAFF being analysed within excavated squares 1-6, and the importance of untangling the complex nature of this CAFF. The existing chronological framework is supplied. Optical dating results including De determination, environmental dose rates and final optical ages are also presented, providing an enhanced data set for the MSA, particularly MIS 3. The implications of these findings will be thoroughly discussed in Chapter 6.
Table 5.5: Dose rate, Equivalent Dose and single-grain quartz optical ages for PP5-6 rock shelter. Samples are NOT presented in stratigraphic order.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Layer</th>
<th>Adj. Water Content (Measured WC) (%)&lt;sup&gt;1&lt;/sup&gt;</th>
<th>Dose Rate (Gy ka&lt;sup&gt;-1&lt;/sup&gt;)</th>
<th>Total Dose Rate (Gy ka&lt;sup&gt;-1&lt;/sup&gt;)&lt;sup&gt;5&lt;/sup&gt;</th>
<th>D&lt;sub&gt;e&lt;/sub&gt; (Gy)&lt;sup&gt;6&lt;/sup&gt;</th>
<th>No. Grains&lt;sup&gt;7&lt;/sup&gt;</th>
<th>σ&lt;sub&gt;d&lt;/sub&gt; (%)&lt;sup&gt;8&lt;/sup&gt;</th>
<th>OSL age (ka)</th>
</tr>
</thead>
<tbody>
<tr>
<td>OSL 2</td>
<td>Zebra</td>
<td>5 ± 2 (2)</td>
<td>0.66 ± 0.03</td>
<td>0.54 ± 0.02</td>
<td>0.09 ± 0.01</td>
<td>1.32 ± 0.04</td>
<td>45.0 ± 7.0</td>
<td>22</td>
</tr>
<tr>
<td>OSL 4</td>
<td>Zebra</td>
<td>5 ± 2 (2)</td>
<td>0.65 ± 0.03</td>
<td>1.07 ± 0.05</td>
<td>0.09 ± 0.01</td>
<td>1.81 ± 0.06</td>
<td>38.0 ± 4.0</td>
<td>42</td>
</tr>
<tr>
<td>OSL 6</td>
<td>Caitlyn</td>
<td>5 ± 2 (2)</td>
<td>0.57 ± 0.03</td>
<td>0.94 ± 0.04</td>
<td>0.09 ± 0.01</td>
<td>1.63 ± 0.05</td>
<td>51.0 ± 4.0</td>
<td>52</td>
</tr>
<tr>
<td>OSL 7</td>
<td>Panther</td>
<td>10 ± 3 (9)</td>
<td>0.60 ± 0.03</td>
<td>0.82 ± 0.04</td>
<td>0.09 ± 0.01</td>
<td>1.50 ± 0.05</td>
<td>62.0 ± 4.0</td>
<td>51</td>
</tr>
<tr>
<td>OSL 8</td>
<td>Panther</td>
<td>10 ± 3 (9)</td>
<td>0.53 ± 0.03</td>
<td>0.83 ± 0.04</td>
<td>0.09 ± 0.01</td>
<td>1.47 ± 0.05</td>
<td>64.0 ± 5.0</td>
<td>53</td>
</tr>
<tr>
<td>OSL 9</td>
<td>Lion</td>
<td>5 ± 2 (3)</td>
<td>0.72 ± 0.03</td>
<td>0.88 ± 0.04</td>
<td>0.09 ± 0.01</td>
<td>1.71 ± 0.05</td>
<td>81.0 ± 4.0</td>
<td>48</td>
</tr>
<tr>
<td>OSL 10</td>
<td>Leopard</td>
<td>5 ± 2 (2)</td>
<td>0.53 ± 0.03</td>
<td>0.85 ± 0.04</td>
<td>0.09 ± 0.01</td>
<td>1.49 ± 0.05</td>
<td>47.9 ± 1.7</td>
<td>81</td>
</tr>
<tr>
<td>OSL 11</td>
<td>Tiger</td>
<td>5 ± 2 (3)</td>
<td>0.62 ± 0.03</td>
<td>0.93 ± 0.04</td>
<td>0.09 ± 0.01</td>
<td>1.67 ± 0.05</td>
<td>56.0 ± 3.0</td>
<td>68</td>
</tr>
</tbody>
</table>

<sup>1</sup> Water contents were adjusted from laboratory measured readings to better estimate both current and past levels of water content within the site.

<sup>2</sup> Beta dose rates were determined using beta counting as outlined in section 3.#. The value is a mean ± 1σ standard error.

<sup>3</sup> Gamma dose rates were determined in field using in-situ gamma spectrometry.

<sup>4</sup> Cosmic dose rates were determined using geomagnetic latitude, thickness of rock/sediment overburden and height above sea level.

<sup>5</sup> Total dose values calculated through addition of beta, gamma and cosmic dose rates.

<sup>6</sup> Mean D<sub>e</sub> values were determined using the central age model (CAM) created by Galbraith et al. (1999). Errors ± 1σ include laboratory uncertainties from beta-dose calibration.

<sup>7</sup> Total number of grains measured from those grains with acceptable D<sub>e</sub> values after rejection criteria are applied.

<sup>8</sup> Over-dispersion values consider uncertainties in measurement, and are a measurement of the spread of D<sub>e</sub> values within a given sample population.
Chapter 6 – Discussion and conclusions
Chapter 6 – Discussion and conclusions

6.1 Introduction

Single-grain OSL dating was used to obtain optical ages for 13 sediment samples at two unrelated MSA archaeological sites in South Africa (five from MRS and eight from PP5-6) to determine the sedimentary context of the two complex stratigraphic features outlined in Sections 4.2 (MRS) and 5.2 (PP5-6). Sediment analysis was conducted on seven samples from MRS, using grain size and geochemical analysis techniques, to help untangle site formation processes. Data obtained through both techniques provide significant insight into MSA site occupation during MIS 3 by AMH. This Chapter will discuss the optical chronologies for both study sites, the sediment analysis results for MRS and the importance of these results for understanding site formation processes through the formation of complex sedimentary features and the impact for understanding AMH occupations during MIS 3. Finally, recommendations are provided for bolstering evidence at both sites.

6.2 Optical dating at MRS and PP5-6

Single-grain OSL dating was strategically conducted on two stratigraphically complex features at MRS and PP5-6. The results were used to determine the sedimentological contexts of the features with respect to investigations into the site formation processes and MSA archaeology of AMH during the little understood period of MIS 3. At MRS, 5 OSL samples (Figure 6.1) were taken from StratAgg and StratLayers (Table 4.1) surrounding and within the complex feature. When initially excavated, the feature resembled a wedge of sediment that had been deposited after erosional truncation of the underlying StratAggs, or perhaps had originally continued horizontally as its own stratigraphic layers, later being truncated by the overlying pit in Figure 6.1.

At PP5-6, eight OSL samples were taken from the CAFF to determine the extent of the erosional truncation and its impact on surrounding StratAggs. Of interest, was the impact of the cut event on StratAggs BAS and OBS2 (Figure 6.2), and the ages for the subsequent filling of this cut with sediment. The small hearth structure, Mary (Table 5.2), overlying the fill on the North wall (Figure 6.3) had the potential to be the youngest evidence of human occupation at PP5-6, depending on the returned ages from the OSL chronology.
6.3 Chronology of MRS

Figure 6.1 provides the five ages generated from OSL dating of sediments conducted as part of this study. In Section 4.2.1 two competing hypotheses were presented to explain the complex sedimentary relationship between the two StratLayers VDGS and CWGS that appeared as an unresolved wedge of sediment, and the StratAggs RGBS, LGS, LRS and DGS. The five OSL ages presented in Section 4.4.7 were instrumental in determining when each of these sedimentary units were deposited, and enhancing understanding of the site formation processes that occurred at MRS.

Hypothesis 2 from Section 4.2.1 proved to be the most likely of the two hypotheses following age analysis with OSL dating, due to the age differences between VDGS (22.4 ± 1.0 ka) and LGS (40.8 ± 1.8 ka), and CWGS (34.1 ± 1.4 ka) and LGS (49.4 ± 2.0 ka) represented in Figure 6.1. Interestingly, it was originally thought that StratLayers VDGS and CWGS were lateral continuations of LRS and LGS after visual interrogation of the stratigraphic units was conducted in-situ.

![Figure 6.1: OSL ages with respect to StratAggs and StratLayers they were collected from. The OSL sample numbers are inset within the yellow circles, and OSL 5-6 were translated horizontally onto the East Face from the South Face for ease of comparison.](image)
It is easy to see how this assumption could have been made when looking at the sediment and sedimentary relationships contained within these units (Figure 6.1). The apparent nature of the dipping contact between LRS and DGS on the northern side of the excavation appeared to directly translate into the sedimentary contact between CWGS and DGS. The difference in optical ages between these two sediment packages prove the impossibility of such an assumption, and is more consistent with the archaeology of the layers. This highlights the relevance of conducting additional quantitative analysis to enhance understandings of stratigraphic relationships in the field.

6.4 MRS site formation

The primary inputs of sediment at MRS during the MSA were anthropogenic entrainment and deposition through aeolian processes. Because of this, the sedimentary profile is quite shallow (~1.5-2 m) when compared to PP5-6 (~14 m). A natural sediment trap exists at the front of the rock shelter, consisting of large stacked boulders. The trap serves as a barrier that has allowed sediment to accumulate against it, toward the front of the rock shelter. The stratigraphy appears to be dipping slightly toward the front of the rock shelter where it abuts the natural barrier. This relationship was observed in the field (Mackay, 2017 unpublished) and is visible in Figure 6.1 with the contacts between LGS, LRS and DGS on the left side of the photograph. The stratigraphy appears to dip significantly toward the rear of the rock shelter, visible with the stratigraphic contacts between VDGS, DGS and the sediment underlying DGS on the right-hand side of the photograph (Figure 6.1). The sediment grain size analysis conducted in in Section 4.2.2 displays a pattern of coarsening sediment toward the front of the site, and fining toward the back (Figure 4.6; Table 4.3). These results, coupled with the OSL ages generated in this project provide encouraging insight into how the sediment was deposited from the front to the rear of the rock shelter. Erosional scouring of fine grain sediment fractions from the front, subsequent aeolian entrainment and deposition toward the rear of the cave could explain this difference in grain size prevalence between the front and back. Additionally, the narrowing of the shelter toward the rear would most certainly have prevented human activity toward the rear of the rock shelter. This would limit anthropogenic entrainment of sediment toward the rear, resulting in the dipping stratigraphy, due to lower sediment accumulation rates. Sediment could roll down these dipping layers, and it is expected if this is the case, that the sedimentary units thicken toward the rear of the shelter, and are likely thinning toward the centre due to anthropogenic compaction. Eventually, the sediment would have levelled out.
toward the top of the profile. Figure 6.2 provides a diagrammatic representation of this hypothesis.

**Figure 6.2:** Schematic reconstruction of proposed sedimentary deposition at MRS from this study. Lensoidal thickening of sedimentary deposits toward the rear, thinning in the centre and dipping stratigraphy toward the natural sedimentary trap at the front of the rock shelter. Profile of roof and current floor accurately transcribed from GIS data provided by Dr. Alex Mackay.

### 6.5 Chronology and site formation processes of PP5-6

Figures 5.4 (northern wall of Sq. 1 and Sq. 2) and 5.5 (eastern wall of Sq. 3 and Sq. 4) provide the eight OSL ages generated from the second site studied in this project, PP5-6. In Section 5.2.1 two competing hypotheses were proposed to explain the complex stratigraphic relationship between the CAFF and the already documented *StratAggs* BAS and OBS2. Whilst in the field, the mechanism for how this complex sedimentary feature was created was simply explained, as smaller scale water runoff from the existing dripline of the rock shelter during excavations had resulted in surface water flow similar to what likely caused the CAFF. Understanding the lateral and vertical extent of this feature proved far more problematic. The eight OSL ages were crucial for untangling these complex sedimentary relationships, and proving that both hypotheses presented in Section 5.2.1 inadequately described the relationships.
Analysis of OSL 6, 10 and 11 produced three ages of 31.4 ± 2.7 ka, 32.0 ± 1.7 ka and 33.5 ± 2.2 ka that were consistent with each other within error. This was expected, as lateral continuity was hypothesised in the field between these 3 samples. OSL 9 produced an age of 47.2 ± 2.9 ka, which was unexpected, as field observations seemed to indicate that StratLayer Lion (Figure 6.4) was part of the same fill deposit that also contained OSL 6, 10 and 11. The two hypotheses failed to account for Lion not being part of the fill, and the OSL ages show that Lion is in-fact much older (~15 kyr) than the other layers in the fill, Tiger and Leopard.

Figure 6.3: OSL ages with respect arbitrary StratLayers located on the northern wall of Sq. 1 & Sq. 2. The OSL sample numbers are contained in the yellow circles for ease of reference.

Another hypothesis is proposed here for how this complex relationship might be explained. This 3rd hypothesis proffers the idea that the CAFF either does not extend far enough to the east laterally, to encompass the sediment contained within Panther and/or Lion (Figure 6.4) or that it does not extend vertically to the point where it encompasses Panther and/or Lion. Determining whether this is the case is difficult, as the excavated area at PP5-6 that contains the CAFF inadequately displays the full context of the CAFF in relation to Panther, Lion, BAS and OBS2. In Table 5.2, it was mentioned that Lion was a possible continuation of Hyena (Figure 6.4) due to the similarities in the sediment observed in the field. To determine the validity of this theory, sampling and OSL analysis of Hyena may provide insight into the possible relationship of Lion and Hyena, and show that the vertical extent of the CAFF did not extend past the sedimentary contact between Leopard/Tiger and Hyena. Alternatively,
extending the excavation of Sq. 4 into Sq. 3 (Figure 5.8) progressively back toward the eastern wall of Sq. 3, which contains Lion, may reveal more information to confirm where the CAFF terminates. More detail on how this can be achieved is provided in PP5-6 recommendations in Section 6.9.

**Figure 6.4:** OSL ages with respect arbitrary *StratLayers* located on the eastern wall of Sq. 3 & Sq. 4. The OSL sample numbers are contained in the yellow circles for ease of reference.

### 6.6 Complex nature of site formation processes

Analysis conducted on two different complex stratigraphic features MRS and PP5-6 has highlighted the importance of interrogating the sedimentary record at archaeological sites to better understand site formation processes. Undertaking this analysis can provide greater understanding of the stratigraphic relationships between depositional and post depositional processes, and how they relate to the other archaeological layers at a site. It is already believed that this type of sedimentary analysis is not conducted enough at archaeological sites (Gladfelter, 1977; Goldberg and Sherwood, 2006; Goldberg and Berna, 2010). However, it can provide valuable insight into human occupation of sites such as those studied in this project.

### 6.7 Strengthening evidence for AMH occupation of rock shelters during MIS 3

Prior to the research conducted in this project, limited quantitative evidence of human inhabitancy during MIS 3 had been carried out at MRS and PP5-6. Evidence from artefacts excavated at MRS suggested that humans were living at the site during MIS 3 (Table 4.1), and the ages presented in Chapter 4 (Table 4.8) support these conclusions. The quantitative ages
provide tangible evidence of occupation of MIS 3 sites away from the coast during a period where successively rapid glacial and interglacial periods (Figure 2.4) probably made for a relatively unstable living environment. However, MRS’s location next to a possible perennial water source may have been the key to its occupation during MIS3 (Mackay, 2016, 2017 unpublished).

Similarly, at PP5-6, much of the existing evidence prior to this project, suggests that limited occupation of PP5-6 occurred during MIS 3. Age analysis conducted in StratAgg RBSR (Table 5.1) provided the only chronological evidence of sedimentation at PP5-6 during MIS 3, at ~ 51 ± 2 ka. The presence of the hearth structure, Mary, deposited on the fill above OSL 6 and below OSL 4 (Figure 6.3) provides some of the most compelling evidence for human occupation of PP5-6 during late MIS 3 or early MIS 2, with ages of OSL 6 and OSL 4 suggesting that Mary was deposited between ~31.4 ± 2.7 ka and ~ 20.6 ± 2.3 ka. This is important, as it provides evidence of occupation at PP5-6 much later then has previously been discovered.

**Figure 6.5:** Compilation of all OSL ages and associated age errors for MRS and PP5-6 showing distributions that dominate the MIS 3. MRS 6 and OSL 4 show the only ages not representing MIS 3.

The new OSL dates obtained for MIS provide a framework, particularly at MRS, in which existing archaeological, cultural and behavioural data can be interrogated with potential
to provide evidence for and against the competing behavioural models presented in Section 2.2.

6.8 Recommendations for further research at MRS

Further work is required at MRS to provide a more robust chronological sequence that will bolster understanding for the site formation processes and results that suggested Hypothesis 2 was the most likely scenario for the deposition of the sedimentary sequence at the site. To achieve this, it is suggested the following additional work be conducted at MRS:

1. The first recommendation is that the excavation pit be pushed further back into the rock shelter. Excavating further toward the back of the cave will allow confirmation or falsification that all sedimentary layers dip and thicken toward the back of the rock shelter as proposed in Section 6.4. This will also support the idea that human occupation of the cave occurred close to the front because of the narrowing profile toward the back of the shelter.

2. The second recommendation is that further targeted sampling for OSL dating should be conducted around and within the complex sedimentary feature studied in this thesis, to enhance the evidence for how and when it was deposited. Figures 6.6 and 6.7 provides the recommended sampling locations to achieve this.

3. The last recommendation is that detailed micromorphological analysis be conducted with the layers that comprise the sedimentary feature, and around it to determine at the microscopic level if there are any characteristics within the sediment that will help better inform depositional trends of the feature. This may also help untangle the complex relationship between the large pit, RGBS, LGS, LRS, VDGS and CWGS (Figure 5.5b), and explain why LRS and LGS seem to not continue further back into the rock shelter.
Figure 6.6: Five proposed OSL sample locations (P1-5) in the East Wall at MRS to improve fidelity of stratigraphic context interpretations, shown in relation to existing chronology.

Figure 6.7: Six proposed OSL sample locations (P6-11) in the South Wall at MRS to improve fidelity of stratigraphic context interpretations, shown in relation to existing chronology.

6.9 Recommendations for further research at PP5-6

In the broader scale, the depositional mechanism of the complex sedimentary feature at PP5-6 was easily defined. However, understanding the complex relationship between BAS and
OBS2, and the CAFF feature and the extent of the CAFF proved to be more difficult. The following recommendations will allow for improved understanding of these relationships:

1. Further excavation is required at PP5-6 to reveal the full extent of the CAFF. It is proposed that Sq. 3 be excavated laterally toward the east to determine where the CAFF feature stops (purple arrows in Figure 6.8). This will help determine whether the additional hypothesis in Section 6.5 adequately describes the context between StratLayers Lion, Hyena, Tiger and Leopard (Figure 6.4). Additionally, Sq. 4 (trench) where OSL10 and OSL11 were taken from (Figure 6.8) needs to be excavated vertically down to find the contact between the CAFF and the underlying stratigraphy, and laterally to the west to determine the western extent of the CAFF (green arrows Figure 6.8). Sq. 2 needs to undergo further excavation to provide a connective link between the west and east walls of the excavation area.

2. Further OSL analysis is required to determine the chronological context for the fill contained within the CAFF, and its relationship between BAS, OBS2, Lion and Panther. By excavating the trench laterally to the east (toward Panther and Lion), additional OSL sampling can be conducted closer to these layers to determine how far the fill extends east. Failing this, sampling of Hyena (Figure 6.4) will possibly provide the link between Leopard/Tiger and Lion/Panther. If Hyena is a part of Lion, it may provide the upper contact of the fill feature StratLayers Lion and Leopard.

3. Re-analyse more single quartz grains for all PP5-6 OSL samples to provide more robust grain populations to derive $D_c$ values from. The problematic dose recovery test results need to be further investigated. It is also suggested that the low gamma dose rate obtained from OSL 2 needs to be re-measured.

4. The final recommendation for PP5-6 is that detailed sediment analysis be conducted using micromorphology to determine the nature of the CAFF, and provide insight into micro-stratigraphic relationships between the above-mentioned layers.
**Figure 6.8:** Proposed excavation of Sq. 2, 3 and 4 to provide better resolution data on the stratigraphic contact between the CAFF and the surrounding stratigraphy.

### 6.10 Conclusions

This project had 5 primary aims:

- Describe and identify complex stratigraphic features in the field using geological techniques.
- Formulate and test hypotheses for how these features formed, and their context within the site they occupy.
- Determine ages of sediment samples using single-grain OSL dating to constrain when these features occurred.
- Provide a more robust chronology for human occupation of rock shelters during MIS 3.
- Integrate age and field data to augment existing understanding of site formation processes.
The first aim was completed successfully, with detailed field analysis at PP5-6 and a similar virtual approach at MRS. The second aim was also successfully completed, with 4 total hypotheses being tested across both sites. For MRS, Hypotheses 2 in Section 4.2.1 was the most likely hypothesis explaining the stratigraphic context of the complex stratigraphic feature. At PP5-6, both hypotheses were proven to be incorrect; indicating that more work needs to be conducted at the site. The third aim was partially achieved. Although the OSL samples analysed provided constraining ages for the complex features at both sites, further analysis and sampling is required as per the recommendations in Sections 6.9 and 6.10 to provide a complete chronological framework for both stratigraphic features. The third aim was instrumental however, in the success of the fourth aim. This project successfully provided a more robust chronology for two MSA sites during the MIS 3 period, at two very different geological and environmental locations. The final aim was also successful, as an augmented understanding of complex sedimentary features with respect to site formation processes was achieved.
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Appendix
Appendix 1

This appendix displays the full process used for single grain sample preparation. The OSL sediment samples collected from both sites underwent a series of steps to get them ready for analysis:

1. Primary wet sieving – involving fractions >212, 212-180, 180-125, 125-90 μm in diameter.
2. Sample drying and storage - >212, 180-125, 125-90 μm diameter fractions were dried in a 50°C oven and stored in plastic sample bags, as well as double black light proof bagging for potential usage later.
3. Chemical treatment – 212-180 μm diameter fraction is the primary fraction used for analysis, and undergoes two steps of treatment before it can undergo density separation.
   a. Hydrochloric Acid (HCl) – The fraction is treated with 32% HCl to remove any carbonate material from the sample. This is stirred and left for 24 hrs, and then rinsed 3 times under tap water to remove the HCl from the sample.
   b. Hydrogen Peroxide (H₂O₂) – The fraction is treated with 10% H₂O₂ to remove organic material. This sample is stirred and then left overnight, when it is rinsed 3 times with tap water to remove any excess H₂O₂ from the sample.
   c. The sample is then dried in the 50°C oven to prepare it for the next step.
4. Density separation – The sample fraction then undergoes two steps of density separation using sodium polytungstate (SPT) in a separation funnel to separate the quartz and feldspar from heavy minerals, and then to separate the quartz from feldspar. The two steps are explained below:
   a. 2.70 g/cm³ SPT – This step is undertaken to separate the denser heavy minerals from the quartz and feldspar grains. The heavy minerals will settle out first, as they are > 2.70 g/cm³ in density. The remaining quartz and feldspar float.
   b. 2.62 g/cm³ SPT – This step is undertaken to separate the quartz and feldspar grains from each other. The denser quartz grains sink to the bottom as they have a density >2.62 g/cm³ and the less dense feldspars float.
   c. The feldspars are dried and bagged, then stored for later.
5. Hydrofluoric Acid (HF) - The quartz grains collected from step 4 are then etched using 40% HF acid for ~40 min. This results in the removal of the outer ~0.02 mm of each grain, negating the alpha particle sphere of influence for each grain. This removes the alpha dose, and negates the need for alpha dose to be calculated for each individual grain (Jacobs et al. 2006; Roberts et al. 2015).

6. Dry Sieving – The quartz grains are then dry sieved again using >212, 212-180 and <180 μm to ensure that no grains that were reduced in size have migrated into smaller fractions, and to ensure that no >212 μm fractions are still in the sample.

7. Disc loading – This involves loading individual quartz grains into aluminium single grain discs. These discs provide the platform for analysis in the Risø TL/OSL instruments. The discs are 9.7 mm in diameter and 1 mm thick, holding a total of 100 grains in a 10 x 10 pattern. The individual chambers are 300 μm deep and 300 μm in diameter. The chamber holes are spaced at 600 μm to ensure minimal cross-illumination during analysis (Bøtter-Jensen et al. 2000). To prevent multiple grains from being analysed, each chamber is carefully checked using a microscope, and a fine bristled paint brush is used to remove doubled up grains.

Appendix 2

This appendix shows figures of the OSL samples collected from PP5-6 on the North walls of Square 1 and 2, and East wall of Square 3. The OSL samples have their field collection numbers assigned, where 571364 = OSL 1 through to 571374 = OSL 11.

Appendix Figure 1: OSL samples from the North wall of Sq 1 & 2. Sample labels are explained above.
Appendix Figure 2: OSL samples collected from East wall from layers Panther and Lion.