Optical dating of Upper Palaeolithic deposits in the Altai Mountains, Siberia

K O'Gorman

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Abstract
Denisovans, Neanderthals and modern humans are hominin groups known to have occupied the Altai Mountains in the Late Pleistocene. The earliest Upper Palaeolithic (UP) in the Altai Mountains consists of two variants, the Initial Upper Palaeolithic (IUP) and Early Upper Palaeolithic (EUP). It is uncertain which of these hominin groups was responsible for the IUP and EUP.

There are two models to explain the emergence of the UP in the Altai Mountains: the local transition model and the chrono-stratigraphic model. The former argues for the regional development of the local Levallois-Mousterian Middle Palaeolithic (MP) variant (LMV) into the IUP and EUP, while the latter argues for modern humans migrating into the region, bringing with them UP technologies. The former also considers the IUP and EUP to be contemporaneous, while the latter considers the IUP and EUP to occur in succession, with the EUP overlying to IUP. The IUP displays MP and UP elements, while the EUP represents a fully developed UP technology. Denisovans, Neanderthals and modern humans have all been suggested as makers of the UP.

Optical dating, including optically stimulated luminescence (OSL) dating and postinfra-red infra-red stimulated luminescence (pIR-IRSL) dating, was conducted on UP stratigraphic sequences at four sites in the Altai Mountains: Denisova Cave, Anui-2, Anui-3 and Ust-Karakol-1 Trench 2.

Fifteen final ages and nine preliminary ages were obtained for these four sites. These ages were then compared to the existing radiocarbon (14C), palaeomagnetic excursion, and radiothermoluminescence (RTL) ages.

The ages produced in this project suggest that the IUP emerged around 62 ka, and the EUP emerged around 35 ka. This succession of the EUP overlying the IUP supports the chrono-cultural model. Further studies are required to determine if there was a hiatus between the LMV and the IUP and EUP, and to assign lithic assemblages to hominin groups based on reliable fossil associations.

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Optical dating of Upper Palaeolithic deposits in the Altai Mountains, Siberia

Kieran O’Gorman

A thesis presented in part fulfilment of the requirements of the honours degree of Bachelor of Science in the School of Earth and Environmental Sciences, Faculty of Science, Medicine and Health, University of Wollongong, 2016.
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.

Kieran O’Gorman

12 October, 2016
ABSTRACT

Denisovans, Neanderthals and modern humans are hominin groups known to have occupied the Altai Mountains in the Late Pleistocene. The earliest Upper Palaeolithic (UP) in the Altai Mountains consists of two variants, the Initial Upper Palaeolithic (IUP) and Early Upper Palaeolithic (EUP). It is uncertain which of these hominin groups was responsible for the IUP and EUP.

There are two models to explain the emergence of the UP in the Altai Mountains: the local transition model and the chrono-stratigraphic model. The former argues for the regional development of the local Levallois-Mousterian Middle Palaeolithic (MP) variant (LMV) into the IUP and EUP, while the latter argues for modern humans migrating into the region, bringing with them UP technologies. The former also considers the IUP and EUP to be contemporaneous, while the latter considers the IUP and EUP to occur in succession, with the EUP overlying to IUP. The IUP displays MP and UP elements, while the EUP represents a fully developed UP technology. Denisovans, Neanderthals and modern humans have all been suggested as makers of the UP.

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Chapter 1: Introduction
1.1. Introduction

Since 2010, the Altai Mountains have become a research hotspot due to the discovery of the enigmatic hominin group the Denisovans (Krause et al., 2010; Reich et al., 2010). This hominin group is known by its mitochondrial and nuclear genomes sequenced from a distal manual phalanx (Denisova 3) and two molars (Denisova 4 and Denisova 8) (Krause et al., 2010; Reich et al., 2010; Meyer et al., 2012; Sawyer et al., 2015). Neanderthals are also known to have been present in the Altai Mountains due to sequencing of the mitochondrial genome of Neanderthals from a humerus (Okladnikov 7), proximal pedal phalanx (Denisova 5), and bone fragment (Denisova 11) (Krause et al., 2007; Prüfer et al., 2014; Brown et al., 2016). Modern human fossil remains have not yet been discovered in the Altai Mountains; however, the complete left femoral diaphysis of a modern human, dating to around 45 ka, has been discovered at Ust-Ishim in western Siberia, around 1200 km to the north-west of the Altai Mountains (Fu et al., 2014). It is, thus, inferred that modern humans were also present in the Altai Mountains around 45 ka. The Altai Mountains are the only location on the globe where these three hominin groups are known to have been present during the Late Pleistocene.

There are Lower Palaeolithic (LP), Middle Palaeolithic (MP) and Upper Palaeolithic (UP) assemblages in the Altai Mountains, comprising many variants. The focus of this project is the UP, for which four variants have been described. Two variants have been described for the earliest UP occupation, the Initial Upper Palaeolithic (IUP) and the Early Upper Palaeolithic (EUP) (Zwyns, 2012), and two variants have been described for the later UP occupation, the Middle Upper Palaeolithic (MUP) and Final Upper Palaeolithic (FUP). These variants have not yet been assigned to specific hominin groups, but Denisovans, Neanderthals and modern humans have all been suggested as makers of the UP.

Two models have been proposed to explain the emergence of the UP in the Altai Mountains: the local transition model argues for a gradual development of the regional MP into the UP (Derevianko, 2011), while the chrono-cultural model argues for modern humans migrating into the region, bringing with them UP technologies (Zwyns, 2012).

In this project, optical dating was conducted on stratigraphic sequences from four UP sites in the Altai Mountains: Denisova Cave, Anui-2, Anui-3, and Ust-Karakol-1 Trench 2 (Figure 1.1). Chronologies were constructed for IUP, EUP, MUP and FUP assemblages from these sites. In this chapter, this project will be discussed in terms of its aims, significance, and structure.
1.2. Aims

This project had three aims, as follows:

- to construct chronologies for four UP sites in the Altai Mountains using optical dating techniques;
- to compare these chronologies to existing radiocarbon (\(^{14}\)C), palaeomagnetic excursion and radiothermoluminescence (RTL) ages; and
- to contribute to understandings of the emergence of the UP as a result of either a local transition from MP to UP, or migrations of modern human populations into the Altai Mountains.

The first aim focuses on producing data, the second aim focuses on comparing this data to existing data, and the third aim focuses on contributing data to the broader archaeological context.

Figure 1.1: The location of some of the archaeological sites in the Altai Mountains, including the sites investigated in this project: Denisova Cave, Anui-2, Anui-3 and Ust-Karakol-1 (after Zwyns et al., 2012).
1.3. Significance

The chronologies for the UP in the Altai Mountains are, at present, incomplete and, often, inconsistent. Many sites do not contain systematic chronologies, and where chronologies are present, the existing $^{14}$C, palaeomagnetic excursion and RTL ages are often in disagreement with one another. In this project, systematic optical dating was conducted on UP assemblages from multiple sites in the Altai Mountains. Optical chronologies were tested against existing independent $^{14}$C ages as a means of determining whether the optical dating chronologies were reliable and accurate. Operator variability in single-grain analysis was also tested for to ascertain reproducibility of chronologies using optical dating methods.

This project has contributed to determining if the IUP and EUP are contemporaneous, or if they occurred in succession, across multiple sites. By constructing reliable chronologies for the earliest UP occupation at multiple sites, future studies can construct chronologies for the MP to determine if there was a hiatus between the MP and UP, or if MP and UP assemblages represent continuous, evolving technologies. This project will, thus, contribute to lending support to either the local transition model (Derevianko, 2011) or chrono-cultural model (Zwyns, 2012).

If future studies are able to assign UP variants to specific hominin groups, then the chronologies constructed in this project will provide a timeframe for the occupation of the Altai Mountains for these hominin groups. This is significant because the three hominin groups known to have been present in the Altai Mountains in the Late Pleistocene – Denisovans, Neanderthals and modern humans – are known to have interbred with one another (Green et al., 2010; Reich et al., 2010; Reich et al., 2011; Skoglund & Jakobsson, 2011; Meyer et al., 2012; Sankararaman et al., 2012; Cooper & Stringer, 2013; Wall et al., 2013; Huerta-Sanchez et al., 2014; Prüfer et al., 2014; Vernot & Akey, 2014; Fu et al., 2015; Qin & Stoneking, 2015; Vernot & Akey, 2015; Kuhlwilm et al., 2016). If it is determined that there was a chronological overlap between the occupations of Denisovans, Neanderthals or modern humans in the Altai Mountains, then it could be suggested that the Altai Mountains was the location of interbreeding between two or more of these hominin groups. See the appendix for a detailed discussion of the evidence for interbreeding between these three hominin groups, and suggestions for the possible locations for interbreeding events.
1.4. Structure of the project

In this chapter, the aims and significance of this project have been discussed. In chapter 2, the background of the UP in the Altai Mountains will be summarised, including the two models used to explain its emergence, and the typological elements, technological elements, and possible makers of the IUP and EUP. The different measurement and analytical methods used to obtain optical ages in this project will be discussed in chapter 3. In chapters 4, 5 and 6, the sedimentological contexts, UP contexts and existing chronologies of the four sites investigated in this project will be discussed. These chapters will also present the optical dating results produced in this project, and compare the final ages to existing chronologies. In chapter 7, the final ages produced in this project will be discussed, and a timeframe for the UP variants in the Altai Mountains will be posited.
Chapter 2: Background
2.1. Introduction

The UP has been reported at many sites in the Altai Mountains, including the four sites investigated in this project: Denisova Cave, Anui-2, Anui-3 and Ust-Karakol-1 Trench 2. In this chapter, the two models to explain the emergence of the UP – the local transition model and the chrono-cultural model – will be discussed. Following the chrono-cultural model, the IUP and EUP will be discussed in terms of their typological elements, technological elements, and possible makers.

2.2. Local transition model

The local transitional model has been posited by Derevianko (2011) to explain the emergence of the UP in the Altai Mountains. Derevianko (2011) suggests that a hominin population settled in the Altai Mountains ~300 ka, bringing with it Mousterian MP technologies. This assignment is based on assemblages from the lowermost layers of Denisova Cave (Derevianko & Postnov, 2004; Shunkov, 2005; Derevianko, 2011). This Mousterian MP variant (MV) is said to have evolved into the local Levallois-Mousterian MP variant (LMV) observed at sites such as Kara-Bom, Denisova Cave, Anui-3, Ust-Karakol-1 Trench 2, Ust-Kanskaya Cave and Strashnaya Cave (Derevianko, 2011). The LMV is then said to have evolved into two UP variants, the IUP (the “Kara-Bom variant”) and the EUP (the “Ust-Karakol variant”) (Derevianko & Volkov, 2004). Following this model, the MP and UP occupations at the aforementioned sites are considered to be continuous; that is, there is no hiatus between the LMV and IUP or EUP assemblages (Derevianko, 2011). Derevianko (2011) has identified ‘transitional’ technologies that signal the evolution of the LMV into the IUP and EUP. The IUP and EUP are considered to be contemporaneous (Derevianko, 2011).

The local transitional model has its foundations in the multi-regional hypothesis (Thorne & Wolpoff, 1981; Wolpoff et al., 1984). The multi-regional hypothesis suggests that Homo sapiens consist of four sub-species that evolved in four regions independent of one another, including H. sapiens africanensis (Africa), H. sapiens neandertalensis (Europe), H. sapiens orientalensis (South-east Asia), and H. sapiens altaiensis (Siberia, Mongolia, Central Asia) (Derevianko, 2011). Following this model, the hominin group the Denisovans evolved into H. sapiens altaiensis in Siberia, Mongolia and Central Asia (Derevianko, 2011). This is not supported by genetic studies which indicate that the
Denisovans are a cousin group to modern humans, diverging around 600 ka (Meyer et al., 2012; Prüfer et al., 2014; Kuhlwilm et al., 2016); even so, Derevianko (2011) argues that there is no evidence consistent with modern human migrations out of Africa and, therefore, *Homo sapiens* must have evolved in multiple regions. The multi-regional hypothesis, as defined by Wolpoff et al. (1984) explains genetic findings that contradict the hypothesis as being influenced by local drifts and multidirectional gene flows.

### 2.3. Chrono-cultural model

The chrono-cultural model has been posited by Zwyns (2012) to explain the emergence of the UP in the Altai Mountains. Zwyns (2012) suggests that the UP did not emerge as a result of the evolution of the LMV, as suggested by Derevianko (2011), but rather as a result of modern human migrations. Zwyns (2012) also argues that the IUP and EUP are not contemporaneous; instead, the EUP succeeds the IUP. This argument is supported by the EUP being a more developed UP variant than the IUP (Zwyns, 2012). Zwyns (2012) assigned lithic assemblages from just one site (Ust-Karakol-1 Trench 2) to both the IUP and EUP; in this instance, the EUP overlies the IUP. Zwyns (2012) also made comparisons to lithic assemblages at Denisova Cave and other UP sites; however, final assignments were not made. For the chrono-cultural model to be better supported, consistent assignments of lithic assemblages, as well as reliable chronologies for stratigraphic sequences, are required.

The chrono-cultural model has its foundations in the out-of-Africa model. The out-of-Africa model posits that modern humans evolved in Africa and subsequently dispersed around the globe, following several routes (Zwyns, 2012). The ‘northern route’ crosses through Central Asia and passes into the Altai Mountains following the Tien Shan Mountains (Zwyns, 2012). Following this route, Goebel (1999, 2006) suggested that there was a fast dispersal of modern humans, based on similarities with lithic assemblages from the Near East.

Following the chrono-cultural model, Zwyns (2012) assigned lithic assemblages from three sites in this project (Anui-2, Anui-3 and Ust-Karakol-1 Trench 2) to either the IUP or EUP, and, as previously mentioned, made comparisons to lithic assemblages at Denisova Cave. These classifications for the IUP and EUP will be described in the following subsections.
2.3.1. The IUP

The IUP has been used to describe lithic assemblages from the Middle East (Marks & Ferring, 1988; Kuhn et al., 1999), Europe (Sinitsyn, 2003; Hoffecker, 2011), and southern Siberia (Zwyns, 2012; Rybin, 2014). Due to the breadth of the term, it does not represent a unified cultural phenomenon, but rather multiple cultural phenomena with similar features (Kuhn & Zwyns, 2014). It has been proposed that the IUP could represent multiple population dispersal events, diffusion of technological ideas across interconnected populations, or the convergence of technologies (Kuhn & Zwyns, 2014); therefore, the assignment of a lithic assemblage to the IUP can be rather arbitrary (Kuhn, 2003). The type site for the IUP in the Altai Mountains is Kara-Bom; it is, therefore, often referred to as the Kara-Bom variant (Zwyns, 2012). Lithic assemblages from Ust-Karakol-1 Trench 1 have also been assigned to the IUP, following the same classification (Zwyns, 2012). It has been suggested by Zwyns (2012) that lithic assemblages from Denisova Cave considered to contain ‘transitional’ technologies following the local transition model (Derevianko, 2011) are similar to IUP assemblages at Kara-Bom and Ust-Karakol-1 Trench 1 and, therefore, should be assigned to the IUP. In this project, these ‘transitional’ technologies will be considered as IUP to be consistent.

2.3.1.1. Typological elements

The IUP contains laminar and Levallois typological elements (Zwyns, 2012). Laminar typological elements are typical of UP assemblages, while Levallois typological elements are most often associated with MP assemblages (Zwyns, 2012). The combination of UP and MP typological elements has lead to IUP assemblages, as defined by Zwyns (2012), being interpreted as ‘transitional’ technologies (Derevianko, 2011).

The IUP is characterised by two types of laminar blanks: parallel and convergent blanks (Zwyns, 2012). The most typical tools are pointed blades displaying inverse proximal thinning (Figure 2.1; Zwyns, 2012). These blades are poorly standardised, but all display bilateral semi-steep and steep retouch aimed at producing points (Zwyns, 2012). This type of tool has not been found in any other MP or UP variants in the Altai Mountains and is, therefore, considered to be a “type fossil” for IUP assemblages (Rybin, 2000; Zwyns, 2012). The most typical Levallois typological elements of the IUP are elongated Levallois points displaying retouch on their proximal end (Figure 2.2; Zwyns, 2012). End-scrapers on blades, sickle-like blades, and bifacial tools are also typical of IUP
Ornaments have been discovered in association with the IUP, including ostrich egg-shell beads, and tubular bone beads with circular incised grooves (Figure 2.3; Derevianko & Rybin, 2003; Rybin, 2014).

2.3.1.2. Technological elements

The IUP technological tradition follows different reduction sequences for medium to massive blades, and small blades to bladelets (Zwyns, 2012). These two reduction sequences are summarised in Figure 2.4.

The medium to massive blade reduction was conducted on high quality raw materials, including large blocks of fine-grained metamorphic rocks (Zwyns, 2012). Minimal preparation was required because tabular blocks and pebbles were selected for reduction based on the suitability of their shapes (Zwyns, 2012). Flaking of these tabular blocks or pebbles occurred using a natural crest. The medium to massive blade reduction of cores followed a standardised method of sub-volumetric reduction (Zwyns, 2012). Many of the flat-faced

Figure 2.1: Pointed blade displaying inverse proximal thinning (after Derevianko et al., 1998; Zwyns, 2012).
cores produced in the medium to massive blade reduction sequence can be described as Levallois (Zwyns, 2012). The bidirectional reduction of cores involves a succession of short unidirectional sequences (Zwyns, 2012). From these cores, two types of blanks are detached: parallel and convergent blanks (Zwyns, 2012). Plain, faceted and dihedral platforms are typical of blanks produced using the medium to massive reduction sequence of the IUP (Zwyns, 2012). Partial faceting also occurs on some of the blanks, and extreme abrasion can also be observed on the external edges of platforms (Zwyns, 2012).

Figure 2.2: Massive retouched blade (after Derevianko et al., 1998; Zwyns, 2012).
The small blade to bladelet reduction was conducted on burin-core blanks selected among blades and laminar flakes produced by the medium to massive blade sub-volumetric reduction sequence (Zwyns, 2012). These burin-cores were reduced following a poorly standardised method of volumetric reduction (seven types of burin-cores are classified by Zwyns); however, the small blades and bladelets produced are standardised, reflecting the use of a range of methods to obtain similar tools (Zwyns, 2012). Most of these small blades and bladelets are detached from the intersection between the two surfaces of the burin-core blanks, and are either débordant, naturally backed or crested (Zwyns, 2012). There is often a triangular or trapezoidal section 10–40 mm in thickness (Zwyns, 2012). Beside the burin-core blanks, rare small truncated-facetted cores on blades are present in IUP assemblages; these cores are more similar to those produced following the medium to massive blade sub-volumetric reduction sequence (Zwyns, 2012).

2.3.1.3. Possible makers

Due to the absence of reliable hominin fossil associations in IUP assemblages, it is not clear who the makers of this UP variant were (Zwyns, 2012). UP technologies are almost always associated with modern humans (Bailey & Hublin, 2005; Wild et al., 2005; Anikovich et al., 2007; Bailey et al., 2009; Higham et al., 2011; Jones et al., 2015); however, at present there are no reliable modern human fossil associations with EUP assemblages (Viola et al., 2011). Neanderthals have also been found in association with UP technologies, and it is unclear if Denisovans were responsible for any lithic assemblages.
The IUP has been compared to the Châtelperronian technocomplex of southern France (Zwyns, 2012). The Châtelperronian also consists of MP and UP technologies, and reduction sequences are similar to that observed in IUP assemblages (Pelegrin, 1990; Boëda, 1995; Zwyns, 2012). Neanderthal remains have been found in association with the Châtelperronian technocomplex, suggesting that Neanderthals were responsible for this technocomplex (Hublin et al., 1996; d’Errico et al., 1998; Bailey & Hublin, 2006). It has been suggested that this is due to the acculturation of Neanderthals by modern humans (Hublin et al., 2012; Talamo et al., 2012; Higham et al., 2014). Pointed blades displaying
inverse proximal thinning, which are considered to be a “type fossil” for IUP assemblages (Rybin, 2000; Zwyns, 2012) are similar to blades present in the Lincombian-Ranisian-Jerzmanowician technocomplex of northwestern Europe (Flas, 2008; Zwyns, 2012). This technocomplex is also considered to have been developed as a result of the acculturation of Neanderthals by modern humans (Flas, 2011). Based on these comparisons, it seems plausible that Neanderthals developed the IUP from the LMV (if Neanderthals were, indeed, responsible for the LMV) due to acculturation by modern humans. To complicate this, another MP variant, known as the Sibiryachikha facies, is known to have been present in the Altai Mountains around the same time as the IUP (Derevianko & Markin, 2011; Derevianko et al., 2013; Derevianko & Markin, 2014). This variant is considered by both Derevianko (2011) and Zwyns (2012) to be a late, intrusive MP variant. The Sibiryachikha facies has been found in association with Neanderthal remains at Okladnikov and Chagyrskaya caves, suggesting that Neanderthals were the makers of this MP variant (Krause et al., 2007; Derevianko & Markin, 2011; Viola et al., 2011; Derevianko et al., 2013; Derevianko & Markin, 2014). For Neanderthals to also be responsible for the IUP, there would have been significant local variation in lithic technologies.

It is unclear if Denisovans were responsible for any lithic assemblages. Two Denisovan fossil remains, Denisova 3 and Denisova 4, have been found in association with ‘transitional’ or IUP assemblages in Denisova Cave; however, due to the presence of sediment mixing in the stratigraphic sequences at Denisova Cave, this association is considered unreliable (Krause et al., 2010; Reich et al., 2010; Viola et al., 2011; Meyer et al., 2012; Sawyer et al., 2015). It is, at present, uncertain if modern humans, Neanderthals or Denisovans were responsible for the IUP in the Altai Mountains.

2.3.2. The EUP

The EUP is often used interchangeably with the IUP to describe lithic assemblages in the Altai Mountains (Kuhn & Zwyns, 2014). Zwyns (2012) has defined the EUP using Ust-Karakol-1 (Trench 1 and Trench 2) as the type site; the EUP is, therefore, often referred to as the Ust-Karakol variant. Lithic assemblages from Anui-2 and Anui-3 have also been assigned to the EUP, following the same classification (Zwyns, 2012). It has been suggested by Zwyns (2012) that lithic assemblages from Denisova Cave should also be assigned to the EUP (Zwyns, 2012).
2.3.2.1. Typological elements

In contrast to the IUP, the EUP contains classic fully developed UP laminar technologies, absent of Levallois elements (Zwyns, 2012). The EUP involves the production of a range of tools, including blades, bladelets and microblades. Specific tools produced include Dufour bladelets and microblades, backed pieces, microblades displaying bilateral retouch, and retouched blades displaying oblique truncations (Figure 2.5; Zwyns, 2012). Small end-scrapers on laminar flakes or blades are also abundant (Figure 2.6; Zwyns, 2012).

![Figure 2.5: Burin (left), pointed bladelet (middle) and Dufour bladelet (right) (after Zwyns, 2012).](image)

2.3.2.2. Technological elements

The EUP technological tradition follows different reduction sequences for blades, and bladelets to microblades (Zwyns, 2012). These two reduction sequences are summarised in Figure 2.7.

The blade reduction was conducted on local raw material, including metamorphic rock of varying quality deriving from the Anui River (Postnov et al., 2000). Blocks selected are thought to be smaller than those selected in IUP assemblages; this may indicate that blocks were selected for medium-size blade production (Zwyns, 2012). The reduction of cores is generally volumetric and semi-turning, unidirectional, displaying a single striking platform that is semi-circular (Zwyns, 2012). Bidirectional cores are rare, but have also been observed in EUP assemblages (Zwyns, 2012). Blade blanks are of medium size, and display parallel edges and unidirectional dorsal patterning (Zwyns, 2012). Regular blades display a diffuse bulb, macroscopic lip and traces of thin abrasion along the platform’s external edge (Zwyns, 2012), suggesting the use of soft hammer (Pelegrin, 1995).
The production for bladelets and microblades is poorly standardised, and is a distinguishing feature of EUP assemblages (Zwyns, 2012). The bladelet to microblade reduction is entirely different from the blade reduction described above. Medium-size blocks, small slabs and pebbles of fine-grained metamorphic rock and, occasionally, jasper-like material, were collected from the Anui River (Zwyns, 2012). Three main procedures for the bladelet and microblade reduction sequence is apparent. The first procedure, used in the production of bladelet blanks, involves unidirectional reduction of narrow-faced cores displaying a flaking surface on a narrow edge (Zwyns, 2012). The second procedure, used in the production of bladelet and microblade blanks, involves reduction of pebbles, thick slabs and, occasionally, cortical flakes from a plain striking platform by semi-turning removals (Zwyns, 2012). The third procedure, used in the production of bladelets and microblades, involves orientation changes in reduction, treating the core as a carinated form before being turned into a narrow-faced core (Zwyns, 2012).

2.3.2.3. Possible makers

Unlike the IUP, there is minimal uncertainty concerning the makers of the EUP, despite the absence of hominin fossil remains. In Europe, EUP assemblages have been clearly associated with modern humans (Bailey & Hublin, 2005; Bailey et al., 2009). Modern humans are known to have been present at Ust-Ishim in western Siberia, around 1200 km to the north-west of the Altai Mountains, around 45 ka (Fu et al., 2014), suggesting that they were also present in the Altai Mountains. There is no evidence for Neanderthals or Denisovans being responsible for the EUP.
The three main procedures used in the bladelet to microblade reduction sequence can be compared to many lithic assemblages in Europe. The first procedure is similar to that observed in Aurignacian (Normand et al., 2007), Proto-Aurignacian (Demidenko & Otte, 2001; Broglio et al., 2005), Early Ahmarian (Davidzon & Goring-Morris, 2003), and Baradostian (Tsanova et al., 2012) contexts. The second procedure is also similar to that observed in Aurignacian contexts (Williams, 2003; Le Brun-Ricalens, 2005). The third procedure is documented in many lithic assemblages in Aurignacian (Normand et al., 2007), Proto-Aurignacian (Demidenko & Otte, 2001; Broglio et al., 2005), and Baradostian (Tsanova et al., 2012) contexts. It, therefore, seems reasonable to assign the EUP to modern humans.

Figure 2.7: Reconstruction of the EUP reduction sequence for blades, and bladelets to microblades (after Zwyns, 2012).
2.4. Synopsis

In this chapter, the two models to explain the emergence of the UP – the local transition model and the chrono-cultural model – were discussed. Following the chrono-cultural model, the IUP and EUP were also discussed in terms of their typological elements, technological elements, and possible makers.
Chapter 3: Methods
3.1. Introduction

In this project, optical dating of mineral grains was conducted in the OSL laboratory at the University of Wollongong, New South Wales, Australia, to produce ages for four UP sites in the Altai Mountains, Siberia: Denisova Cave, Anui-2, Anui-3 and Ust-Karakol-1 Trench 2. Two optical dating techniques were utilised in this project to obtain ages for these sites, including optically stimulated luminescence (OSL) dating of single quartz grains, and post-infra-red infra-red stimulated luminescence (pIR-IRSL) dating of single potassium-feldspar grains and single aliquots of polymineral fine-grains.

3.2. Optical dating

Optical dating of sediments provides an estimate of the time elapsed since mineral grains, such as quartz and potassium-feldspar, were last exposed to sunlight; that is, the time elapsed since their last burial (Wintle & Huntley, 1979; Huntley, 1985; Feathers, 1996; Aitken, 1998; Duller, 2004; Jacobs & Roberts, 2007). Buried mineral grains receive radiation energy from the natural burial environment; this radiation energy is responsible for the movement of electrons or holes into traps that are defects in the crystal lattice of these mineral grains (Aitken & Valladas, 1992; Feathers, 1996; Duller, 2004; Jacobs & Roberts, 2007). Some of these traps are light-sensitive; therefore, upon stimulation by light, electrons and holes are released as luminescence (Huntley, 1985; Feathers, 1996; Duller, 2004; Jacobs & Roberts, 2007). The radiation energy received in the natural burial environment derives from alpha, beta and gamma radiation from naturally occurring radioactive minerals in the surrounding sediments, including uranium (U), thorium (Th), potassium (K), and rubidium (Rb), and their daughter products, as well as from cosmic-rays (Aitken, 1985).

The amount of luminescence emitted, known as the luminescence signal provides an estimate for the amount of energy stored by the mineral grains. The equivalent dose \( D_e \) can be estimated by comparing the amount of energy stored in nature since the last exposure to sunlight to known amounts received in controlled manner in the laboratory. The rate at which mineral grains were exposed to ionising radiation from environmental sources during its burial period can be measured in the field and laboratory to obtain an estimate of the environmental dose rate. An optical age is estimated using the luminescence age equation:
The ages of archaeological traces, including fossil remains and lithic tools, are inferred from the burial ages of the surrounding sediments (Feathers, 1996; Duller, 2004; Jacobs & Roberts, 2007; Wintle, 2008; Roberts et al., 2015).

3.3. Sample collection

Fifteen sediment samples were collected from three UP sites, three from Anui-2, five from Anui-3, and seven from Ust-Karakol-1, in 2012 by Professor Zenobia Jacobs and Professor Bert Roberts. One additional sediment sample was collected from the hillside near the site of Ust-Karakol-1 in 2016 by the author of this thesis and Professor Zenobia Jacobs.

Sediment samples were collected in plastic tubes with a 5 cm diameter. This method limits the amount of sediment exposed to sunlight. This is important because sediment samples used in determining the $D_e$ must be collected in dark conditions to prevent exposure of mineral grains to sunlight, a process known as bleaching, as this empties the light-sensitive electron traps (Wintle & Huntley, 1979; Huntley, 1985; Feathers, 1996; Aitken, 1998; Duller, 2004; Jacobs & Roberts, 2007; Wintle, 2008; Roberts et al., 2015). Additional sediment samples were also collected in sealed plastic bags for current water content determination as plastic tubes are not suitable for this (see Section 3.6.1).

Nine sediment samples were also collected from Denisova Cave, seven from the Main Chamber and two from the East Chamber in 2012 by Professor Zenobia Jacobs and Professor Bert Roberts. These samples were not prepared nor measured by the author of this thesis; instead, a comparative single-grain analysis was conducted to obtain $D_e$ values for two operators, and preliminary ages were obtained using preliminary dose rate estimates.

3.4. Sample preparation

Sample preparation followed two procedures: one for coarse-grains and one for fine-grains, as summarised in Figure 3.1. One to 2 cm of sediments from the ends of each of the plastic tubes were removed because these may have been exposed to sunlight. These
Wet sieving into >250, 250–90, and <90 µm in diameter grain-size fractions.

**Unprepared sample.**

Remove 1–2 cm of sediment from tube ends.

Wet sieving into >250, 250–90, and <90 µm in diameter grain-size fractions.

**Coarse-grains**

- HCl and H$_2$O$_2$ treatment of the 250–90 µm fraction to remove carbonates and organic matter.
- Wet sieving into 250–212, 212–180, 180–125 and 125–90 µm in diameter grain-size fractions.
- Density separations of quartz at 2.70–2.62 g/cm$^3$ and potassium-feldspar at >2.58 g/cm$^3$ using SPT.
- Etching of quartz with 40% HF for 40 minutes, and potassium-feldspar with 10% HF for 40 minutes.
- Dry sieving of grains due to decrease in diameter after HF etching.
- Loading of grains onto single-grain discs.

**Fine-grains**

- HCl and H$_2$O$_2$ treatment of the <90 µm fraction to remove carbonates and organic matter.
- Wet sieving into 90–63 and <63 µm in diameter grain-size fractions.
- Grain-size separation into 11–63 and <11 µm in diameter grain-size fractions using settling times.
- Grain-size separation into 4–11 and <4 µm in diameter grain-size fractions using settling times.
- Loading of aliquots by suspending 1 mg of grains in 1 mL of acetone in a glass tube.

**Figure 3.1:** Sample preparation for coarse-grain (left) and fine-grain (right) samples.
Sediments were used for measurements of the environmental dose rate, known as dosimetry (see Section 3.6.1). Sediments from the centre of each sample were wet sieved into >250, 250–90, and <90 µm in diameter fractions.

### 3.4.1. Coarse-grains

The 250–90 µm in diameter grain-size fraction of all samples was submerged in 10% hydrochloric acid (HCl) to remove carbonates, left in HCl for a few hours, and then washed with tap water four times to remove all traces of HCl. The same grain-size fraction was then submerged in 10% hydrogen peroxide solution (H$_2$O$_2$) to remove organic matter, left in H$_2$O$_2$ overnight, and then washed with tap water four times to remove all traces of H$_2$O$_2$. This grain-size fraction was then wet sieved once more, this time dividing the samples into 250–212, 212–180, 180–125 and 125–90 µm in diameter grain-size fractions.

The 212–180 and 125–90 µm in diameter grain-size fractions underwent density separations using sodium polytungstate (SPT) solutions diluted to densities of 2.70 g/cm$^3$ to separate out heavy minerals, 2.62 g/cm$^3$ to separate quartz from feldspars, and 2.58 g/cm$^3$ to separate sodium-feldspar from potassium-feldspar. Density separations were carried out by placing the sample and the SPT solution in a sealed tube in a centrifuge for 10 min.

The 212–180 and 125–90 µm in diameter quartz grain-size fractions were etched with 40% hydrofluoric acid (HF) for 40 min, and the 212–180 and 125–90 µm in diameter potassium-feldspar grain-size fractions were etched with 10% HF for 40 min. This was done to remove the outer ~0.02 mm of each grain, which is the sphere of influence of alpha particles; therefore, the alpha dose was removed and the alpha dose rate did not need to be determined. Once HF etching was complete, the fluoride crystals were removed using 40% HCl for 40 min. The samples were then dry sieved because the size of grains decreased during the HF etching process.

Individual grains were subsequently loaded onto aluminium single-grain discs for measurements of their OSL signals using the Risø TL/OSL instruments. Each disc is 9.7 mm in diameter and 1 mm in thickness, and holds 100 grains in a 10×10 square grid of chambers 300 µm deep and 300 µm in diameter. These chambers are spaced 600 µm apart to ensure cross-illumination is kept to a minimum (Bøtter-Jensen et al., 2000). Three larger holes are located on the outer edge of the disc in order to determine the orientation of the discs in the measurement chamber.
3.4.2. Fine-grains

The <90 µm in diameter grain-size fraction of UK OSL 7–13 was treated with HCl and H$_2$O$_2$ in the same manner as the coarse-grains to remove carbonates and organic matter. These fractions were then wet sieved to obtain the 90–63 and <63 µm in diameter grain-size fractions.

The 4–11 µm in diameter grain-size fraction was then separated from the 63–11 and <4 µm fractions using a settling procedure. Small amounts of sample were placed in an 8 cm deep column of water in a beaker, placed in an ultrasonic bath to ensure grains did not aggregate, and stirred to ensure grains did not settle. These beakers were then left for 12 min to allow the >11 µm in diameter grain-size fraction to settle on the bottom of the beaker, and the grains remaining in suspension (<11 µm) were placed in another beaker. The same procedure was followed again using the <11 µm in diameter grain-size fraction, but this time leaving the beakers for 1 h 36 min to allow the 4–11 µm in diameter grain-size fraction to settle. The grains that remained in suspension (<4 µm in diameter) were discarded. This procedure was repeated ~4 times until there were no more suspended grains. The settling times were determined using Stokes’ law, an equation that expresses the settling velocities of small spherical particles in a fluid, based on factors such as drag force, gravity force, viscosity of the fluid, radius of the particle, density of the particle, density of the fluid, and height of liquid.

Three mg of each dried sample was then suspended in 3 ml of acetone. One stainless steel disc 9.8 mm in diameter was placed in the bottom of a glass tube, and 1 ml of the acetone with suspended grains was pipetted onto the disc in a monolayer. Three discs were prepared for each sample for measurement of their IRSL signals using the Risø TL/OSL instruments.

3.5. $D_e$ determination

The $D_e$ of a sample is an estimate of the amount of energy stored by mineral grains in the burial environment. The $D_e$ is determined by stimulating mineral grains with a light source of a specific wavelength that will release the trapped holes and electrons in the form of light (luminescence), known as the luminescence signal. The light signal can be observed as an optical decay curve (Figure 3.2) The ‘natural’ signal is then compared to signals obtained after laboratory irradiations of known amounts of dose to build up a dose-
response curve (Huntley, 1985; Feathers, 1996; Aitken, 1998; Duller, 2004; Jacobs & Roberts, 2007). The natural signal is projected onto this curve; where it intersects with the dose-response curve, the $D_e$ can be estimated from interpolation onto the dose axis (see Fig 3.3) (Murray & Wintle, 2000). In this project, OSL dating was conducted on single quartz grains, and pIR-IRSL dating was conducted on single potassium-feldspar grains and polymineral fine-grains. For OSL dating, single quartz grains were stimulated using a green laser, and for pIR-IRSL dating, single potassium-feldspar grains were stimulated using an infrared (IR) laser, and single aliquots of polymineral fine-grains were stimulated using infrared emitting diodes (Huntley, 1985; Hütt et al., 1988; Bøtter-Jensen & Duller, 1992; Aitken, 1998; Jacobs & Roberts, 2007; Li & Li, 2011). The single aliquot regenerative-dose (SAR) protocol (Murray & Wintle, 2000) was used for single quartz grains, the multi-elevated-temperature post-IR IRSL (MET-pIRIR) protocol (Li & Li, 2011) was used for single potassium-feldspar grains, and the two-step pIRIR (pIRIR) protocol (Thomsen et al., 2008) was used for polymineral fine-grains.

### 3.5.1. Single-grain dating

Single-grain OSL and pIR-IRSL dating was used in this project in preference to aliquots because it has inherent benefits (Duller, 2008): it provides the ability to reject grains with aberrant luminescence behaviours prior to $D_e$ determination (see Section 3.5.7.1 for the single-grain rejection criteria used in this project), and to identify the

![Figure 3.2: Optical decay curve of a single quartz grain, displaying 5 channels of dark counts, followed by 90 channels of stimulation, and another 5 channels of dark counts. Each channel is recorded after 0.02 s of optical stimulation.](image)
possible impact of depositional and post-depositional processes on a sample (Roberts et al., 1999; Yoshida et al., 2000; Jacobs et al., 2006b; Jacobs & Roberts, 2007). The effects of depositional and post-depositional processes that may be identified using single-grains include the following: heterogenous or partial bleaching of grains prior to their last burial (Duller, 2000; Murray & Olley, 2002; Duller, 2008), differences in beta dose rates received by individual grains in heterogeneous sediments (Olley et al., 1997; Roberts et al., 1999; Duller, 2000), post-depositional sediment mixing (Jacobs et al., 2006a; David et al., 2007), and roof-spall contamination (Roberts et al., 1999; Bateman et al., 2003; Jacobs et al., 2011).

### 3.5.2. Selection of dating materials

Single quartz and potassium-feldspar grains, and polymineral fine-grains were used in attempts at $D_e$ determination for different samples in this project. The appropriate minerals were measured for each site based on several factors. Single quartz grains were the preferred dating material because the fast component of the quartz OSL signal is rapidly bleached (Wintle & Murray, 2006). Since this project focussed on dating the UP stratigraphic sequences, saturation of the OSL signal was not likely to be an issue. Despite
this, in some samples, problems were encountered using single quartz grains, so pIR-IRSL dating of single potassium-feldspar grains was conducted because potassium-feldspar pIR-IRSL signals are often brighter than quartz OSL signals (Li et al., 2007; Li & Li, 2011). Furthermore, in some samples, there was not an adequate amount of coarse-grain fractions for determination of $D_e$ for quartz or potassium-feldspar; therefore, pIR-IRSL dating of polymineral fine-grains was conducted to produce a $D_e$ value as a last resort, despite the known possible disadvantages of this technique (Rittenour, 2008).

**3.5.3. OSL and IRSL dating equipment**

In this project, $D_e$ measurements were carried out using an automated Risø TL/OSL reader. Single-grain discs and fine-grain aliquots were placed on a carousel with 48 positions, and this was placed in the measurement chamber of the Risø TL/OSL reader. The Risø TL/OSL reader is fitted with a heater plate to preheat grains and maintain elevated temperatures to eliminate the charge held in thermally unstable electron traps. The Risø TL/OSL reader is also fitted with a $^{90}\text{Sr}/^{90}\text{Y}$ beta irradiator to provide grains with regenerative doses for producing dose-response curves.

In conducting OSL dating, single quartz grains were stimulated using a 10 mW Nd:YVO$_4$ diode-pumped green laser (532 nm) with a power density of $\sim$50 W/cm$^2$ at 90% power. The beam focuses on a $\sim$10 $\mu$m in diameter location over each grain hole by using three lenses to steer the laser within the measurement chamber, and two mirrors which move orthogonally by two motor driven stages equipped with position encoders (Bøtter-Jensen et al., 2000). Since these grains were stimulated in green wavelengths, the OSL signals were recorded in ultraviolet (UV) wavelengths. The UV emissions were detected by an Electron Tubes Ltd. 9235QA photomultiplier tube, using two 3 mm in thickness Hoya U-340 optical filters.

In conducting pIR-IRSL dating, single potassium-feldspar grains were stimulated using an IR laser (830 nm) at 90% power, and single aliquots of polymineral fine-grains were stimulated using IR diodes (870 $\Delta$ 40 nm) at 90% power. Since these grains were stimulated in IR wavelengths, the pIR-IRSL signals were recorded in blue wavelengths. The blue emissions were detected by an Electron Tubes Ltd. 9235QA photomultiplier tube, using a blue filter package, including Schott BG-39 and Corning 7-59 filters.
3.5.4. SAR protocol

The SAR protocol developed by Murray and Wintle (2000) was used for OSL dating of single quartz grains in this project (Table 3.1). A key feature of the SAR protocol is that it monitors for sensitivity changes in quartz grains by measuring the OSL signal of a test dose of a constant dose after measuring each natural dose ($L_N$) and regenerative dose ($L_X$) signal, giving $T_N$ and $T_X$. The $L_N$ and $L_X$ signals are divided by their corresponding $T_N$ and $T_X$ signals to produce a sensitivity-corrected OSL signal ($L_N/T_N$ or $L_X/T_X$).

Prior to measuring OSL signals and test dose signals, grains were preheated to between 160°C and 300°C to eliminate the charge held in thermally unstable electron traps (Murray & Wintle, 2000). Stimulations were made at 125°C to prevent retrapping of electrons or holes in the trap corresponding to the 110°C TL peak (Murray & Wintle, 1998, 2000).

The natural OSL signals ($L_N$) and their test doses ($T_N$) were measured first. Grains were then given a series of known doses of radiation using a $^{90}$Sr/$^{90}$Y beta irradiator. Each of these ‘regenerative doses’ was used to construct a dose-response curve. The natural OSL signal was projected onto this dose-response curve, providing an estimate of the $D_e$ (Murray & Wintle, 2000). In this project, four regenerative doses of ~70, 140, 210 and 280 Gy were used to bracket the expected $D_e$ estimate.

In addition to these four regenerative doses, a zero dose was used to monitor for recuperation; that is, whether the dose-response curve passes through the origin. The dose-response curve may not pass through the origin due to a residual thermal signal that is not released by the preheat prior to measurement of $T_N$ or $T_X$, but is released by the preheat prior to measurement of $L_N$ or $L_X$ (Murray & Wintle, 2000). In addition to this, a repeat dose of ~70 Gy was also used to monitor for recycling; that is, whether the same OSL signal can be recorded from the same given dose (Murray & Wintle, 2003). Each grain was also stimulated with IR diodes and given the same repeat dose of ~70 Gy to monitor for feldspar inclusions. The depletion of the OSL signal after IR stimulation was used to obtain an IR depletion ratio (Duller, 2003). The recuperation ratio, recycling ratio and IR depletion ratio were used in the single-grain rejection criteria (3.5.7.1).

3.5.5. Post-IRIR protocol

The pIRIR protocol developed by Thomsen et al. (2008) was used for pIR-IRSL dating of single aliquots of polymineral fine-grains in this project (Table 3.2). This procedure is similar to SAR in that it uses test doses to produce sensitivity-corrected IRSL signals, zero
doses to monitor for recuperation, and repeat doses to monitor for recycling. Repeat doses were, however, not given to produce an IR depletion ratio as this tests for feldspar inclusions, and is, therefore, not appropriate when stimulating feldspars.

This protocol was used due to a phenomenon exhibited in feldspars known as anomalous fading (Wintle, 1973; Spooner, 1994; Huntley & Lamothe, 2001; Huntley & Lian, 2006). Anomalous fading refers to the leakage of electrons from traps that give rise to IRSL, and has been suggested to cause underestimation of the $D_e$ when IRSL measurements are made shortly after irradiation (Spooner, 1994; Huntley & Lamothe, 2001). The pIRIR protocol involves exposing grains to IR at a low stimulation temperature ($T_1$) to deplete these unstable traps before stimulating grains at a higher temperature ($T_2$) to record the IRSL signal, or pIR-IRSL signal, used for determining the equivalent dose (Thomsen et al., 2008). Grains were exposed to IR at 325°C for 40 s before further regenerative doses to remove all charge from traps.

3.5.6. MET-pIRIR protocol

The MET-pIRIR protocol developed by Li and Li (2011) was used for pIR-IRSL dating of single potassium-feldspar grains in this project (Table 3.3). This method is similar to the pIRIR method, but eliminates the fading component in feldspars using multiple IR stimulations by increasing the stimulation temperature in 50°C intervals. This provides a means to observe the effect of anomalous fading on an IR stimulation curve (Li et al., 2014).

![Figure 3.4: IR stimulation curve (after Li et al., 2014).](image-url)
<table>
<thead>
<tr>
<th>Step</th>
<th>Treatment</th>
<th>Observed</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Natural and regenerative doses (excluding the second repeat dose).</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>Preheat at temperature between 160 and 300°C for 10 s.</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>OSL measurement for 2 s at 125°C.</td>
<td>L&lt;sub&gt;N&lt;/sub&gt; or L&lt;sub&gt;X&lt;/sub&gt;</td>
</tr>
<tr>
<td>4</td>
<td>Test dose.</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>Preheat at temperature between 160 and 300°C for 5 s.</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>OSL measurement for 2 s at 125°C.</td>
<td>T&lt;sub&gt;N&lt;/sub&gt; or T&lt;sub&gt;X&lt;/sub&gt;</td>
</tr>
<tr>
<td>7</td>
<td>Repeat steps 1–6 for natural and regenerative doses (excluding the second repeat dose).</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 steps 2–6.</td>
<td></td>
</tr>
</tbody>
</table>

**Table 3.1:** The SAR protocol used in the measurement of single quartz grains and single aliquots of quartz in this project.
<table>
<thead>
<tr>
<th>Step</th>
<th>Treatment</th>
<th>Observed</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Natural, regenerative, zero and repeat doses.</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>Preheat at temperature between 250 and 320°C for 10 to 60 s.</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>IRSL measurement for 200 s at T1.</td>
<td>$L_{N(T1)}$ or $L_{X(T1)}$</td>
</tr>
<tr>
<td>4</td>
<td>IRSL measurement for 200 s at T2.</td>
<td>$L_{N(T2)}$ or $L_{X(T2)}$</td>
</tr>
<tr>
<td>5</td>
<td>Test dose.</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>Preheat at temperature between 250 and 320°C for 10 to 60 s.</td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>IRSL measurement for 200 s at T1.</td>
<td>$T_{N(T1)}$ or $T_{X(T1)}$</td>
</tr>
<tr>
<td>8</td>
<td>IRSL measurement for 200 s at T2.</td>
<td>$T_{N(T2)}$ or $T_{X(T2)}$</td>
</tr>
<tr>
<td>9</td>
<td>IR bleaching for 40 s at 325°C.</td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>Repeat steps 1–9.</td>
<td></td>
</tr>
</tbody>
</table>

*Table 3.2:* The pIRIR protocol used in the measurement of single aliquots of polymineral fine-grains in this project.
<table>
<thead>
<tr>
<th>Step</th>
<th>Treatment</th>
<th>Observed</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Natural, regenerative, zero and repeat doses.</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>Preheat at temperature between 250 and 320°C for 10 to 60 s.</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>IRSL measurement for 100 s at 50°C.</td>
<td>( L_{N(50)} ) or ( L_{X(50)} )</td>
</tr>
<tr>
<td>4</td>
<td>IRSL measurement for 100 s at 100°C.</td>
<td>( L_{N(100)} ) or ( L_{X(100)} )</td>
</tr>
<tr>
<td>5</td>
<td>IRSL measurement for 100 s at 150°C.</td>
<td>( L_{N(150)} ) or ( L_{X(150)} )</td>
</tr>
<tr>
<td>6</td>
<td>IRSL measurement for 100 s at 200°C.</td>
<td>( L_{N(200)} ) or ( L_{X(200)} )</td>
</tr>
<tr>
<td>7</td>
<td>IRSL measurement for 100 s at 275°C.</td>
<td>( L_{N(275)} ) or ( L_{X(275)} )</td>
</tr>
<tr>
<td>8</td>
<td>Test dose.</td>
<td></td>
</tr>
<tr>
<td>9</td>
<td>Preheat at temperature between 250 and 320°C for 10 to 60 s.</td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>IRSL measurement for 100 s at 50°C.</td>
<td>( T_{N(50)} ) or ( T_{X(50)} )</td>
</tr>
<tr>
<td>11</td>
<td>IRSL measurement for 100 s at 100°C.</td>
<td>( T_{N(100)} ) or ( T_{X(100)} )</td>
</tr>
<tr>
<td>12</td>
<td>IRSL measurement for 100 s at 150°C.</td>
<td>( T_{N(150)} ) or ( T_{X(150)} )</td>
</tr>
<tr>
<td>13</td>
<td>IRSL measurement for 100 s at 200°C.</td>
<td>( T_{N(200)} ) or ( T_{X(200)} )</td>
</tr>
<tr>
<td>14</td>
<td>IRSL measurement for 100 s at 275°C.</td>
<td>( T_{N(275)} ) or ( T_{X(275)} )</td>
</tr>
<tr>
<td>15</td>
<td>IR bleaching for 100 s at 325°C.</td>
<td></td>
</tr>
<tr>
<td>16</td>
<td>Repeat steps 1–15.</td>
<td></td>
</tr>
</tbody>
</table>

**Table 3.3:** The MET-pIRIR protocol used in the measurement of single potassium-feldspar grains and single aliquots of polymineral fine-grains in this project.
3.5.7. Single-grain analysis

Single-grain data requires significant analysis to produce a final $D_e$ value for age determination. Single grains are required to pass a set of rejection criteria, need to be graphically displayed in an appropriate manner such as a radial plot, have overdispersion (OD) values calculated, and have a model chosen.

3.5.7.1. Single-grain rejection criteria

Individual mineral grains, even those from the same sample, display a range of physical behaviours; therefore, many grains may not produce accurate $D_e$ values (Jacobs et al., 2006b). Rejection criteria were applied to individual grains to remove aberrant grains from each sample following the criteria proposed by Jacobs et al. (2006b).

There were five initial rejection criteria used in this project for OSL dating of single quartz grains, plus some additional rejections made by an operator. Grains were rejected if at least one of the following criteria could not be passed:

1. the $T_N$ signal was less than 3 times the background signal;
2. the error associated with the $T_N$ signal was >20%;
3. the recycling ratio was >2$\sigma$ from unity;
4. the IR depletion ratio was >2$\sigma$ from unity; or
5. the ratio of $(L_0/T_X)/(L_N/T_N)$ was >10%.

There were three initial rejection criteria used in this project for pIR-IRSL dating of single potassium-feldspar grains, plus some additional rejections made by an operator. Grains were rejected if at least one of the following criteria could not be passed:

1. the $T_N$ signal was less than 3$\sigma$ of the background signal;
2. the recycling ratio was >2$\sigma$ from unity;
3. The recuperation of $L_0/T_0$ was >5% of $L_N/T_N$.

The additional rejections made by an operator were on basis of $L_N/T_N$ ratios not intercepting with dose-response curves due to saturation or not being able to fit a saturating exponential curve.

3.5.7.2. Radial plots and OD values

The $D_e$ values and associated standard error values of all accepted single grains were presented as radial plots to help identify $D_e$ distribution patterns (Galbraith, 1988, 1990). OD values – the amount of scatter in single-grain $D_e$ distributions after measurement
uncertainties are accounted for – were also calculated for each D<sub>e</sub> distribution to quantify the scatter (Galbraith et al., 1999).

Radial plots display the overall patterning and precision of D<sub>e</sub> values for individual grains in D<sub>e</sub> distributions (Galbraith, 1988, 1990). The D<sub>e</sub> value for each grain is read by extending a straight line from the origin of the standardised estimate axis, through the data point, and onto the radial axis on the right: the point of interception of the line with the radial axis is the D<sub>e</sub> value. The uncertainty in the D<sub>e</sub> value for each grain is read by extending a straight, vertical line from the data point onto the x-axis: the point of interception of the line with the x-axis is the relative error as a percentage of the associated D<sub>e</sub> value. Two straight lines are extended from the ±2 standardised estimate axis to the weighted mean value on the radial axis on the right: if at least 95% of the D<sub>e</sub> values for individual grains fall between these two lines, then measurement errors alone are able to explain the scatter (Jacobs & Roberts, 2007).

OD values were obtained as part of the central age model (CAM; see Section 3.5.7.3) to reflect the amount of scatter in single-grain D<sub>e</sub> distributions. The higher the OD values, the more likely some depositional or post-depositional processes have impacted the D<sub>e</sub> distributions. Utilising radial plots and OD values, four different types of D<sub>e</sub> distributions have been observed, including the following:

1. Single component D<sub>e</sub> distributions (Figure 3.5a): these indicate that grains were well-bleached prior to their last burial, were not affected by any post-depositional processes, and received similar beta dose rates in a homogeneous sediment (e.g., Olley et al., 2006).
2. Scattered D<sub>e</sub> distributions (Figure 3.5b): these indicate that grains were well-bleached prior to their last burial, but received different beta dose rates in a heterogeneous sediment (e.g., Jacobs et al., 2008).
3. Partially bleached D<sub>e</sub> distributions (Figure 3.5c): these indicate that some grains were partially bleached prior to burial and, therefore, retained a residual dose (e.g., Jankowski et al., 2016).
4. Mixed D<sub>e</sub> distributions (Figure 3.5d): these indicate that grains were well-bleached prior to their last burial, but post-depositional sediment mixing has occurred as a result of bioturbation or other processes, mixing sediments from two or more different layers into discrete D<sub>e</sub> components (e.g., Jacobs et al., 2008). Bioturbation can also result in D<sub>e</sub> distributions of a continuous broad range of D<sub>e</sub>
values that can not be easily resolved and for which final $D_e$ values are difficult to calculate.

3.5.7.3. Central age model (CAM)

For single-grain measurements, the CAM is the simplest and best model for samples that do not appear to have undergone depositional or post-depositional processes that cause scattering of grains and high OD values. This model assumes that the $D_e$ values for individual grains are centred around a weighted mean $D_e$ value, and the OD values for distributions are incorporated into the uncertainty estimate for the $D_e$ value (Galbraith et al., 1999).

![Figure 3.5](image_url)

**Figure 3.5**: Radial plots of the $D_e$ values for single quartz grains from archaeological sites, displaying some of the different types of $D_e$ distributions that have been observed, including a) a single component $D_e$ distribution from Lake Mungo, Australia (Olley et al., 2006), b) a scattered $D_e$ distribution from Sibudu, South Africa (Jacobs et al., 2008), c) a partially bleached $D_e$ distribution from Kudjal Yolgah Cave, Australia (Jankowski et al., 2016) and d) a mixed $D_e$ distribution from Sibudu, South Africa (Jacobs et al., 2008).
3.5.7.4. Finite mixture model (FMM)

The FMM was developed for fission track dating by Galbraith and Green (1990), and adapted for OSL and pIR-IRSL dating by Roberts et al. (2000). It can be applied to single-grain $D_e$ distributions that display two or more discrete $D_e$ components that may be the result of one of the following: differences in beta dose rates received by individual grains in heterogeneous sediments (Olley et al., 1997; Roberts et al., 1999; Duller, 2000), post-depositional sediment mixing (Jacobs et al., 2006a; David et al., 2007), or roof-spall contamination (Roberts et al., 1999). The FMM fits more than one component, determines the likelihood of each grain belonging to a specific component, and the $D_e$ values associated with each of these components using the CAM. The optimum OD value for the $D_e$ distribution is determined using the Bayes Information Criterion (BIC) and maximum log likelihood (llik).

In this project, OD values were systematically varied by one percentage point until the BIC recorded its lowest value and started to increase with increasing OD, while llik remained stable. This was the OD used for the chosen number of components. Each single-grain distribution was fitted to 2, 3 and, in most cases, 4 components. The $D_e$ value for the component incorporating the largest proportion of grains was used in determination of the final age as this was assumed to be the portion of the sample unaffected by the aforementioned depositional and post-depositional processes.

3.6. Dose rate determination

The total environmental dose rate of a sediment sample represents the sum of the alpha, beta and gamma radiation released by the radioactive decay of parent radionuclides $^{238}$U, $^{235}$U, $^{232}$Th, $^{40}$K and $^{87}$Rb, as well as the daughter products in the U and Th decay chains, and a contribution from cosmic-rays (Aitken, 1985). These different forms of radiation have different spheres of influence: alpha particles travel up to 0.02 mm, beta particles travel 2–3 mm, gamma rays travel up to 300 mm, and cosmic-rays can penetrate sediments many tens of metres (Aitken, 1985).

Since alpha particles have such a small sphere of influence, the external alpha contribution for coarse-grains was removed through HF etching during sample preparation. Fine-grains are too small to undergo HF etching, so thick-source alpha counting (TSAC) was conducted to directly determine the alpha dose rates received by the
fine-grain samples. The beta dose rates were determined using a GM-25-5 beta counter, the gamma dose rates were determined using in situ gamma spectrometry, and the cosmic-ray dose rates were calculated based on the geomagnetic latitude, altitude, sediment density and depth of the samples. TSAC and GM-25-5 beta counting were also used to determine laboratory gamma dose rates, and were compared to the dose rates derived from in situ gamma spectrometry. Small internal dose rates for quartz grains were also accounted for.

3.6.1. Dosimetry sample preparation and water content determination

Sediments from the 1–2 cm in thickness plastic tube ends were dried in a 100°C oven, and weighed before and after to determine their water content. Sediments collected in sealed plastic bags were also dried in a 100°C oven, and weighed before and after to determine their water content. Sediments collected in sealed plastic bags tend to provide a more accurate measure of water content as these bags retain moisture better. Sediments from tube ends, on the other hand, may provide an underestimate of water content due to evaporation, as well as absorption by the newspaper placed in the ends of plastic tubes to limit exposure of the sediments to sunlight. The samples used in this project were collected in 2012, but only opened in 2016, allowing plenty of time to dry out. Table 3.4 shows a comparison between the water contents determined for sealed plastic bags and tube ends for all 15 samples. Tube ends underestimated the water content of the samples relative to sealed plastic bags by ~10–90%; therefore, the water contents determined from the sealed plastic bags were used in dose rate determination for all samples.

For laboratory dosimetry measurements including TSAC and GM-25-5 beta counting, tube ends were used instead of sealed plastic bags because the tube ends were more closely associated with the measured sample. Sediments from the tube ends were crushed into a fine powder using a ball mill, and left for at least 3 weeks prior to measurements to allow Radon (Rn) isotopes, predominantly the longer lived isotope $^{222}$Rn, and their daughter nuclides to equilibrate.

3.6.2. Fine-grain alpha dose rates

The alpha dose rates received by the fine-grains were directly measured using a Daybreak-538 thick-source alpha counter. TSAC measures the combined contribution of all the alpha particles from the U and Th decay chains (Aitken, 1985). For each
<table>
<thead>
<tr>
<th>Sample name</th>
<th>Water content (%)</th>
<th>Ratio (tube ends/bags)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Tube ends (%)</td>
<td>Sealed plastic bags (%)</td>
</tr>
<tr>
<td>ANUI 2 OSL 3</td>
<td>7.72</td>
<td>23.82</td>
</tr>
<tr>
<td>ANUI 2 OSL 2</td>
<td>4.13</td>
<td>12.70</td>
</tr>
<tr>
<td>ANUI 2 OSL 1</td>
<td>9.97</td>
<td>23.12</td>
</tr>
<tr>
<td>ANUI 3 OSL 11</td>
<td>6.96</td>
<td>7.77</td>
</tr>
<tr>
<td>ANUI 3 OSL 10</td>
<td>5.30</td>
<td>10.53</td>
</tr>
<tr>
<td>ANUI 3 OSL 9</td>
<td>5.62</td>
<td>12.47</td>
</tr>
<tr>
<td>ANUI 3 OSL 8</td>
<td>8.33</td>
<td>19.73</td>
</tr>
<tr>
<td>ANUI 3 OSL 7</td>
<td>3.56</td>
<td>14.91</td>
</tr>
<tr>
<td>UK OSL 13</td>
<td>3.38</td>
<td>16.26</td>
</tr>
<tr>
<td>UK OSL 12</td>
<td>3.81</td>
<td>15.47</td>
</tr>
<tr>
<td>UK OSL 11</td>
<td>7.04</td>
<td>25.29</td>
</tr>
<tr>
<td>UK OSL 10</td>
<td>4.43</td>
<td>21.59</td>
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<tr>
<td>UK OSL 9</td>
<td>15.85</td>
<td>24.08</td>
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<tr>
<td>UK OSL 8</td>
<td>4.51</td>
<td>23.47</td>
</tr>
<tr>
<td>UK OSL 7</td>
<td>2.61</td>
<td>23.39</td>
</tr>
</tbody>
</table>

Table 3.4: Water contents for samples ANUI 2 OSL 1–3, ANUI 3 OSL 7–11, and UK OSL 7–13 obtained from tube ends and sealed plastic bags, expressed as a percentage of the dry weight. The ratio of these two sub-samples for determining water content is expressed with the water contents derived from the tube ends as the numerator, and the water contents derived from the sealed plastic bags as the denominator.
measurement, a zinc sulphide (ZnS) phosphor screen 14.70 cm² in diameter was placed in
a sample holder, and covered with a >1 mm thick layer of powdered sample. The surface
of the sample was made flat to ensure a similar surface area for each sample was
measured, and the holder was left ‘unsealed’ to allow Rn to escape from the sample
because the accumulation of Rn in a sealed holder can cause substantial over-counting
(Aitken, 1985). The sample holder was placed within the alpha counter above a
photomultiplier tube (PMT). Emitted alpha particles from the sample hit the ZnS phosphor
screen, producing scintillations, resulting in the emission of photons which are detected by
the photocathode of the PMT. The total count of alpha particles emitted per unit area of
the ZnS phosphor screen over time provides an estimate of the alpha dose rate of each
fine-grain sample. In this project, samples were measured until at least 2000 counts were
recorded to produce an accurate estimate of the alpha dose rate.

Prior to measurement of samples, two ZnS phosphor screens were placed in the holder
face to face and measured in the alpha counter for a period of ~24 h to measure the
background count rate. The background count rate for each ZnS phosphor screen was,
therefore, half the measured background count rate. This background count rate was
subtracted from the measured count rates for each sample to produce background-
corrected count rates.

The alpha dose rate was derived from the count rate using the dose rate conversion
factors provided by Guérin et al. (2011). The alpha dose rate was corrected for water
content following Aitken (1985), alpha attenuation following Bell (1980), and alpha

The uncertainties in the alpha dose rate using TSAC included a 2% uncertainty for the
dose rate conversion factors of Guérin et al. (2011), a 20% uncertainty for the alpha
efficiency factors of Adamiec and Aitken (1998), and a 25% relative uncertainty in water
content.

3.6.3. Beta dose rates

GM-25-5 beta counting, an emission counting method described by Bøtter-Jensen and
Mejdahl (1988), was conducted to obtain a direct estimate of the beta dose rate for each
sample. The GM-25-5 beta counter measures the beta emissions derived from U, Th and K
in the sample. It consists of five GM detectors and a common guard counter, surrounded
by lead shielding to minimise interference from cosmic-rays. Plastic pots with a ~25 mm
diameter were used to contain samples: these pots were filled to the brim, covered with cling film, and presented to the GM detectors in a uniform manner.

Three sub-samples containing equal quantities of powdered sample were measured for each sample. One loess sample, Nussi, was used as a standard as this sample has a known radionuclide content and beta dose rate of $1.5399 \pm 0.02766$ Gy/ka estimated using high-resolution gamma spectrometry measurements (Kalchgruber, 2002). One magnesium oxide (MgO) sample was also used to measure the background dose rate as MgO is non-radioactive. The three sub-samples, Nussi and MgO were measured for 24 hours for each sample. Each time a beta particle was emitted, the GM detectors recorded a pulse. If a pulse was recorded by the GM detector and the guard counter at the same time, it was rejected as background interference. Using a background, MgO, that is subtracted from the measured sample counts provides a means to produce a background-corrected measurement. Using a standard, Nussi, means that changes in instrument behaviour are accounted for.

The beta dose rate was determined using the following formula:

$$\text{Beta dose rate} = \frac{\bar{y} - z}{x - z} \times c_1 \times c_2 \times c_3 \times c_4$$

where $\bar{y}$ is the average of the three sub-sample counts, $z$ is the background dose rate, $x$ is the measured Nussi beta dose rate, $c_1$ is the known beta dose rate for Nussi of 1.5399 Gy/ka, $c_2$ is the dose rate conversion factors of Guérin et al. (2011), $c_3$ is a correction for grain-size attenuation, and $c_4$ is a correction for water content (Jacobs & Roberts, 2015).

The uncertainties in the beta dose rate using GM-25-5 beta counting were calculated following the equations presented in Jacobs and Roberts (2015), and include the variance between the three sub-samples, a 1.8% measurement error ($\pm 0.02766$ Gy/ka) for Nussi using the dose rate estimates determined by Kalchgruber (2002) and dose rate conversion factors of Guérin et al. (2011), a 2% uncertainty for the dose rate conversion factors of Guérin et al. (2011), a 2% uncertainty for the grain-size attenuation of beta particles (Murray & Olley, 2002), and a 25% relative uncertainty in water content.

### 3.6.4. Gamma dose rates

The gamma dose rates for each sample were obtained using two methods: *in situ* gamma spectrometry to derive a field gamma dose rate, and TSAC and GM-25-5 beta counting to derive a laboratory gamma dose rate.
In situ gamma spectrometry was conducted as it encompasses the total 300 mm sphere of influence of gamma rays, accounting for inhomogeneities in the sediments (Aitken, 1985). In situ gamma spectrometry measurements were made in the holes remaining after the removal of the sample. These measurements were conducted using an Ortec Digidart portable gamma spectrometer with a 5 cm sodium iodide (NaI) detector (580 v) entirely inserted into each of the holes. The detector was calibrated using the “threshold” calibration technique, following Mercier and Falguères (2007). Each sample was counted for 1800 s to determine the total field gamma dose rate.

The uncertainties in the gamma dose rate using in situ gamma spectrometry include a measurement error of 2.2% based on a large number of repeat estimates, a 3% uncertainty in the calibration ratio, and a 25% relative uncertainty in water content.

Laboratory gamma dose rates were also determined using a combination of TSAC and GM-25-5 beta counting. The U and Th count rate was determined using TSAC, following the method described in Section 3.6.2. The “pairs” method was used to derive the gamma dose rate from this count rate (Aitken, 1985). The emission of two alpha particles in fast succession, known as a “pair”, is caused by the decay of $^{220}$Rn into $^{216}$Po in the $^{232}$Th series, as both are alpha emitters, and the latter has a half-life of 0.145 s (Aitken, 1985). This provides a measure for the Th activity in the sample. Using the total number of counts and the total number of “pairs”, the U activity in the sample can be calculated (Aitken, 1985). Using the total number of counts derived from TSAC and the total number of counts derived from GM-25-5 beta counting, the K activity in the sample can be determined by subtraction. The U, Th and K activities from each sample were then converted to gamma dose rates using the conversion factors of Guérin et al. (2011).

The uncertainties in the gamma dose rate using a combination of TSAC and GM-25-5 beta counting include an error based on counting statistics, a 2% uncertainty for the dose rate conversion factors of Guérin et al. (2011), and a 25% relative uncertainty in water content.

3.6.5. Cosmic-ray dose rates

The cosmic-ray dose rate is often quite small in comparison to the beta and gamma dose rates; however, in order to obtain an accurate total dose rate, an estimate of the cosmic-ray dose rate is required (Aitken, 1985). The cosmic-ray dose rate was calculated following the equation presented in Prescott and Hutton (1994). The geomagnetic latitude
was calculated based on the latitude and longitude of each site, the altitude was estimated at around the same as Denisova Cave at ~670 m (Kuzmin, 2004), the sediment density was estimated based on the density of sandy soil (1.8 g/cm³), and the depth of each sample was measured at the time of sampling. Since Anui-2, Anui-3 and Ust-Karakol-1 Trench 2 are all open-air sites, there was no need to account for rocky overburden or the angular distribution of cosmic-rays such as at cave sites.

3.6.6. Internal dose rates

Since U and Th are often present in quartz in very small amounts (Aitken, 1998), an internal dose rate of 0.030 ± 0.011 Gy/ka was included in the total dose rate for all quartz samples. No ages were produced for any potassium-feldspar samples; therefore, the internal dose rates for potassium-feldspar did not need to be accounted for. Fine-grains are too small to have an internal dose rate, so no dose rate was accounted for in fine-grain samples.

3.7. Synopsis

In this chapter, I have discussed the different measurement and analytical methods used in this project to produce the final age estimates for Anui-2, Anui-3 and Ust-Karakol-1 Trench 2, and preliminary age estimates for Denisova Cave. This included a description of the sample preparation and D_e measurement methods for both quartz and potassium-feldspar coarse-grains and polymineral fine-grains. The rejection of single grains based on objective criteria, and their graphical display as radial plots were described to help establish which age model to use. The two age models used in this project – the CAM and FMM – were explained. I have also described the different components – alpha, beta, gamma, cosmic-ray and internal – that make up the total environmental dose rate, and the methods used to determine the individual dose rate components and their errors. Together, the D_e estimates and dose rate estimates so obtained were used to calculate individual age estimates for each of the samples. The results will be presented in chapters 4, 5 and 6.
Chapter 4: Denisova Cave
4.1. Introduction

The site of Denisova Cave (51°23’48” N, 84°40’35” E) is located near the Anui River in the Anui River valley, around 670 m above sea level (Figure 1.1; Kuzmin, 2004; Zwyns, 2012). This site consists of three chambers – the Main, East and South chambers – and an entrance area (Figure 4.1). The Main, East and South chambers of Denisova Cave contain UP artefacts in layers 11 and 9 (Derevianko et al., 1998, 2001, 2003, 2011; Rybin, 2014); some $^{14}$C ages have been obtained for these layers (Krause et al., 2010; Reich et al., 2010). In this chapter, these Main and East chambers will be discussed in terms of their sedimentological contexts, UP contexts, hominin contexts, and existing chronologies. The optical dating results produced in this project for these two chambers will also be presented, including OSL $D_e$ measurements, operator comparisons, and preliminary ages.

4.2. Sedimentological context

The stratigraphic sequences of the Main and East chambers of Denisova Cave have been divided into many layers, including ‘transitional’ layer 11 and UP layer 9 (Figure 4.2). In the Main chamber, layer 11 is divided into four sub-layers (11.1–11.4). It consists of a series of lenticular-shaped loam sediments, displaying various colours from dark grey (sub-layer 11.4) to brown (sub-layer 11.3) and red-brown (sub-layer 11.2) (Derevianko et al., 1998). In Figure 4.2, sub-layer 11.1 is not visible because layers and sub-layers are not always visible in different excavation profiles. Sub-layer 11.4 directly overlies the latest

Figure 4.1: Planform map of Denisova Cave, displaying the location of the entrance area and three chambers, and the excavation history of the site (after Leonov et al., 2014).
MP layer, layer 12. This layer displays horizontal bedding and it is clearly discernible from layer 12; some bioturbation, however, can be observed macroscopically. Layer 10 is 1 cm in thickness, and contains no artefacts. This layer does, however, contain organic material, and ferriferous and manganese formations. Layer 10 may represent a hiatus in sedimentation. Layer 9 consists of ~50 cm in thickness, pale yellow loess-like loam sediments, and includes roots, fine detritus and phosphate accumulations. Ice-lensing and bioturbation can also be observed in the sediments. Layer 9 is unconformably overlain by Holocene deposits; the contact is horizontal and clearly discernible (Derevianko et al., 2001).

In the East chamber, layer 11 is also divided into four sub-layers (11.1–11.4). Similar to the Main chamber, layer 11 consists of a series of loam sediments, displaying various colours from a pale yellow (sub-layer 11.4), reddish brown (sub-layer 11.3), yellowish

Figure 4.2: Stratigraphic sequences of the Main (left) and East (right) chambers of Denisova Cave. The stippled lines display the demarcated sub-layers in Layer 11 in both chambers, and the overlying layer 9.
brown (sub-layer 11.2) to pale whitish brown (sub-layer 11.1) (personal observation and Kozlikin, pers.comm.). Layer 11 appears to be intact in the middle-to-left of the excavation profile, but bioturbation in the form of animal burrows is prevalent toward the right. It is also apparent that sediment cracks may have formed along the concave-shaped rock walls, causing sediment mixing. Layer 11 is unconformably overlain by layer 9 in the East Chamber. Layer 9 is heavily bioturbated in the East chamber and is separated from the overlying Holocene layers by Layer 8, a bright orange-coloured layer that contains no artefacts.

Little is known about the pollen and faunal records of the East chamber; analyses are ongoing. Palynological studies of the stratigraphic sequence of the Main chamber suggest that layer 11 is associated with a damp, cold phase due to the abundance of dark coniferous taxa, including spruce and Siberian pine (Malaeva, 1995). Layer 9 has been associated with a dry phase due to the abundance of grass species, indicating the spread of steppes (Malaeva, 1995; Derevianko et al., 2001). In the Main chamber, the large mammal assemblages are dominated by steppe inhabitants, regardless of layer. The only discernible difference is a gradual decline in woodland and forest-steppe large mammals, starting in layer 12, with no evidence for woodland species in Layer 9, but significant increases in rock-dwelling species in layers 11 and 9 (Derevianko et al., 2001). Small mammal assemblages follow the same trend, with a reduction in woodland species in favour of mountain-steppe species, starting in layer 12 (Shunkov & Agadjanian, 2000; Agadjanian & Serdyuk, 2005).

4.3. UP context

The artefact assemblages from layer 11 in the Main chamber have been interpreted by Derevianko et al. (1998, 2001) and Derevianko (2011) as a ‘transitional’ technology that illustrates the gradual transition from the MP toward the initial stages of the UP, following the local transition model (see Section 2.2). This is due to there being an equal proportion of MP and UP artefacts present in layer 11 (Derevianko et al., 2001; Zwyns, 2012). More recently, Rybin (2014) has described layer 11 as belonging to the IUP. This is supported by Zwyns’ (2012) suggestion that UP-like lithic artefacts found in layer 11 in the Main chamber are most similar to the IUP artefacts at Ust-Karakol-1 Trench 1. Reduction sequences display parallel reductions, and diagnostic elements include grattoirs, burins,
borers, retouched blades and backed microblades. Fragments of bifacial leaf-points and carinated end-scrapers are also present. Similar technological and typological elements were observed in the East chamber (Zwyns, 2012). Significantly, layer 11 in both the Main and East chambers is associated with a rich collection of bone artefacts and ornaments (Figure 4.3). Bone artefacts include needles and awls (Derevianko et al., 2001, 2003) and ornaments include perforated animal teeth of fox, deer and bison, tubular bird bones with circular grooves, bead preforms and beads, including some made using ostrich egg-shells (Derevianko et al., 2001; Rybin, 2014).

Layer 9 in the Main chamber has been associated with the EUP (Zwyns, 2012). The main lithic artefact feature is a significant increase in the occurrence of blades and microblades (Derevianko et al., 2003). Bone artefacts and ornaments have also been excavated from these layers and include eyed-needles and a perforator, a perforated deer tooth and mammoth tusk; however, these are much less abundant in layer 9 than layer 11. Similar types of bone artefacts and ornaments have also been found in the upper sub-layers of layer 11 in the East chamber.
4.4. Hominin context

Two hominin fossil remains are associated with layer 11 in Denisova Cave (Figure 4.4). This includes *Denisova 3*, a distal manual phalanx (Krause et al., 2010; Reich et al., 2010; Viola et al., 2011; Meyer et al., 2012) found in layer 11.2 in the East chamber, and *Denisova 4*, a molar (Reich et al., 2010; Viola et al., 2011; Sawyer et al., 2015) found in layer 11.1 in the South chamber. Both fossils have been identified as belonging to the hominin group the Denisovans (Krause et al., 2010; Reich et al., 2010; Meyer et al., 2012; Sawyer et al., 2015).

![Figure 4.4: Hominin fossil remains from layer 11 in Denisova Cave, including *Denisova 3* (left) and *Denisova 4* (right) (after Reich et al., 2010; Gibbons, 2012).](image)

4.5. Existing chronologies

Ten radiocarbon ages have been reported for layer 11 in the Main, East and South chambers of Denisova Cave (Table 4.1; Krause et al., 2010; Reich et al., 2010; Zwyns, 2012). Those that were not infinite ages were calibrated using the IntCal13 radiocarbon calibration curve (Reimer et al., 2013). Eight of these ages were produced from bone collagen, some from samples displaying human modifications such as cut marks. The age for layer 11 in the South chamber of 32.4 – 34.1 ka cal BP was produced from charcoal. The range of ages indicates that there are younger elements mixed in with older elements displaying finite ages.
<table>
<thead>
<tr>
<th>Site</th>
<th>Layer</th>
<th>Uncalibrated $^{14}$C age (ka BP)</th>
<th>Calibrated $^{14}$C age (ka cal BP) at 95% C.I.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Main Chamber</td>
<td>11</td>
<td>$&gt;37.2$</td>
<td>–</td>
</tr>
<tr>
<td>East Chamber</td>
<td>11</td>
<td>$15.7 \pm 0.1$</td>
<td>$18.8 - 19.2$</td>
</tr>
<tr>
<td></td>
<td>11.2</td>
<td>$23.2 \pm 0.1$</td>
<td>$27.2 - 27.7$</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$30.1 \pm 0.2$</td>
<td>$33.8 - 34.6$</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$&gt;50.0$</td>
<td>–</td>
</tr>
<tr>
<td></td>
<td>11.3</td>
<td>$&gt;50.0$</td>
<td>–</td>
</tr>
<tr>
<td>South Chamber</td>
<td>11</td>
<td>$29.2 \pm 0.4$</td>
<td>$32.4 - 34.1$</td>
</tr>
<tr>
<td></td>
<td>11.2</td>
<td>$&gt;50.0$</td>
<td>–</td>
</tr>
</tbody>
</table>

Table 4.1: The existing published chronologies for UP layers at Denisova Cave (after Krause et al., 2010; Reich et al., 2010; Zwyns, 2012).
4.6. Optical dating results

In this project, preliminary ages were produced for nine samples from Denisova Cave, seven from the Main chamber and two from the East chamber. The seven samples from the Main chamber corresponded to layer 9 (DCM-1; DCM-2), sub-layer 11.2 (DCM-4), sub-layer 11.3 (DCM-5), and sub-layer 11.4 (DCM-3; DCM-6; DCM-7), and the two samples from the East chamber corresponded to sub-layer 11.1 (DCE-3) and sub-layer 11.2 (DCE-4). OSL $D_e$ and dosimetry measurements were not conducted in this project; instead, OSL single quartz grain data and preliminary dose rates were provided by Professor Zenobia Jacobs. The author of this thesis analysed OSL single quartz grain data to compare to analyses conducted by Professor Zenobia Jacobs. Preliminary ages were produced using the $D_e$ values deriving from the analyses conducted by both operators.

4.6.1. Single-grain rejection and analysis

Single quartz grains were rejected following the rejection criteria outlined in Section 3.5.8.1, the results of which are presented in Table 4.2. The operator rejections in Table 4.2 are those conducted by the operator KO and account for 0.9% of measured grains. These grains were rejected on the basis of $L_N/T_N$ ratios not intercepting with dose-response curves due to saturation or not being able to fit a saturating exponential curve. The operator KO accepted just 77.8% of the number of grains accepted by the operator ZJ.

4.6.2. Single-grain $D_e$ distributions

The nine samples from Denisova Cave display two very different $D_e$ distributions (Figure 4.5). DCM-1, DCM-5, DCM-6 and DCM-7 display distributions that are well-bleached, but with some outlier $D_e$ values, both higher and lower. Most grains are scattered around a mean value. To obtain a $D_e$ value for age determination, the CAM was used; the grey bands in Figure 4.5a–b, g and k–n are centred on this value.

DCM-2, DCM-4, DCM-3, DCE-3 and DCE-4 display at least two or three discrete dose components with one of these components always the dominant component. This can happen when younger grains are included in the main sediment matrix by burrowing animals; such burrows are ubiquitous in the sediments and can be observed macroscopically. To obtain a $D_e$ value for age determination, the FMM was used; the grey bands and lines in Figure 4.5c–f, i–j and o–r are centred on this value.
<table>
<thead>
<tr>
<th>Sample name</th>
<th>Measured grains</th>
<th>Criterion 1</th>
<th>Criterion 2</th>
<th>Criterion 3</th>
<th>Criterion 4</th>
<th>Criterion 5</th>
<th>Operator rejections</th>
<th>Σ rejected grains</th>
<th>Σ accepted grains</th>
</tr>
</thead>
<tbody>
<tr>
<td>DCM-1</td>
<td>4000</td>
<td>3252</td>
<td>488</td>
<td>41</td>
<td>50</td>
<td>59</td>
<td>37</td>
<td>3927</td>
<td>73</td>
</tr>
<tr>
<td>DCM-2</td>
<td>4000</td>
<td>3229</td>
<td>530</td>
<td>40</td>
<td>40</td>
<td>46</td>
<td>48</td>
<td>3933</td>
<td>67</td>
</tr>
<tr>
<td>DCM-4</td>
<td>3500</td>
<td>2926</td>
<td>393</td>
<td>24</td>
<td>43</td>
<td>34</td>
<td>22</td>
<td>3442</td>
<td>58</td>
</tr>
<tr>
<td>DCM-5</td>
<td>4000</td>
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<td>454</td>
<td>58</td>
<td>73</td>
<td>74</td>
<td>28</td>
<td>3932</td>
<td>68</td>
</tr>
<tr>
<td>DCM-3</td>
<td>4000</td>
<td>3362</td>
<td>421</td>
<td>41</td>
<td>51</td>
<td>29</td>
<td>39</td>
<td>3943</td>
<td>57</td>
</tr>
<tr>
<td>DCM-6</td>
<td>4000</td>
<td>3283</td>
<td>454</td>
<td>60</td>
<td>50</td>
<td>52</td>
<td>25</td>
<td>3924</td>
<td>76</td>
</tr>
<tr>
<td>DCM-7</td>
<td>4000</td>
<td>3207</td>
<td>471</td>
<td>82</td>
<td>139</td>
<td>36</td>
<td>28</td>
<td>3963</td>
<td>37</td>
</tr>
<tr>
<td>DCE-3</td>
<td>2000</td>
<td>1574</td>
<td>259</td>
<td>42</td>
<td>23</td>
<td>22</td>
<td>15</td>
<td>1935</td>
<td>65</td>
</tr>
<tr>
<td>DCE-4</td>
<td>2000</td>
<td>1530</td>
<td>285</td>
<td>35</td>
<td>53</td>
<td>15</td>
<td>33</td>
<td>1951</td>
<td>49</td>
</tr>
</tbody>
</table>

| Total       | 31500         | 25608      | 3755       | 423        | 522        | 367        | 275               | 30950           | 550             |

Table 4.2: The number of grains measured, rejected and accepted for DCM-1–7 and DCE-3–4 based on 5 rejection criteria: 1) $T_N$ signal <3×BG; 2) $T_N$ error >20%; 3) recycling ratio >2σ from unity; 4) IR depletion ratio >2σ from unity; and 5) ratio of (L0/TX)/(LN/TN) >10%, as well as additional rejections by the operator KO.
a) DCM-1 | KO
N = 73
OD = 38.4 ± 4.0
De = 68.6 ± 2.8

b) DCM-1 | ZJ
N = 92
OD = 40.9 ± 3.8
De = 71.7 ± 3.4

c) DCM-2 | KO
N = 67
OD = 47.1 ± 4.8
De = 65.4 ± 4.3

d) DCM-2 | ZJ
N = 93
OD = 47.1 ± 4.1
De = 69.4 ± 4.3

e) DCM-4 | KO
N = 58
OD = 32.4 ± 4.1
De = 51.5 ± 1.7

f) DCM-4 | ZJ
N = 69
OD = 35.4 ± 3.9
De = 52.3 ± 1.6

g) DCM-5 | KO
N = 68
OD = 27.3 ± 3.3
De = 72.2 ± 2.3

h) DCM-5 | ZJ
N = 82
OD = 33.7 ± 3.4
De = 72.9 ± 2.3
i) DCM-3 | KO
N = 57
OD = 27.8 ± 4.0
D_e = 90.7 ± 3.8

j) DCM-3 | ZJ
N = 78
OD = 29.1 ± 3.3
D_e = 93.9 ± 3.2

k) DCM-6 | KO
N = 76
OD = 29.7 ± 3.3
D_e = 69.3 ± 2.4

l) DCM-6 | ZJ
N = 93
OD = 30.1 ± 2.9
D_e = 68.7 ± 2.5

m) DCM-7 | KO
N = 37
OD = 26.8 ± 4.3
D_e = 66.5 ± 3.5

n) DCM-7 | ZJ
N = 63
OD = 46.6 ± 4.9
D_e = 61.4 ± 4.0

o) DCE-3 | KO
N = 65
OD = 58.6 ± 5.8
D_e = 43.5 ± 4.0

p) DCE-3 | ZJ
N = 70
OD = 59.7 ± 5.5
D_e = 49.3 ± 7.4
4.6.3. Operator comparisons

The operators KO and ZJ produced $D_e$ distributions that are similar (Figure 4.5). In the cases of DCM-5 and DCE-4, a different number of components were chosen by each operator (Figure 4.3g–h and q–r), but for the seven other samples, the same number of components were chosen. The $D_e$ values (Figure 4.6; Table 4.3) for these samples are in agreement at 1$\sigma$, with the exception of DCE-4. The two $D_e$ values for DCE-4 are not in agreement at 2$\sigma$ with a ratio of 0.36 ± 0.03. KO’s analysis produced a $D_e$ value of 31.6 ± 2.5, while ZJ’s analysis produced a $D_e$ value of 88.4 ± 3.0. This is due to there being two discrete components with a similar number of grains. The analyses conducted by the two operators resulted in different components being dominant. This could be interpreted in two different ways: substantial sediment mixing between two different layers has occurred, as suggested by Reich et al. (2010), or during sediment sampling, a different layer was passed through as it is difficult to distinguish between layers in dark conditions. Sub-layer 11.2 should be re-sampled to determine which component derives from the true sub-layer 11.2.

4.6.4. Preliminary ages

Preliminary total dose rates, as provided by Professor Zenobia Jacobs, are presented together with the $D_e$ values and final OSL ages for operators KO and ZJ in Table 4.4. Seven ages were obtained for the Main chamber of Denisova Cave. Ages of 62.0 ± 4.0 (DCM-3), 52.8 ± 3.1 (DCM-6) and 58.8 ± 4.3 ka (DCM-7) were obtained for sub-layer 11.4 by operator KO. This is the earliest IUP sub-layer present in the Main chamber. These
<table>
<thead>
<tr>
<th>Sample name</th>
<th>N</th>
<th>D₀ (Gy)</th>
<th>OD (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>KO</td>
<td>ZJ</td>
<td>KO</td>
</tr>
<tr>
<td>DCM-1</td>
<td>73</td>
<td>92</td>
<td>68.6 ± 2.8</td>
</tr>
<tr>
<td>DCM-2</td>
<td>67</td>
<td>93</td>
<td>65.4 ± 3.3</td>
</tr>
<tr>
<td>DCM-4</td>
<td>58</td>
<td>69</td>
<td>51.5 ± 1.7</td>
</tr>
<tr>
<td>DCM-5</td>
<td>68</td>
<td>82</td>
<td>72.2 ± 2.3</td>
</tr>
<tr>
<td>DCM-3</td>
<td>57</td>
<td>78</td>
<td>90.7 ± 3.8</td>
</tr>
<tr>
<td>DCM-6</td>
<td>76</td>
<td>93</td>
<td>69.3 ± 2.4</td>
</tr>
<tr>
<td>DCM-7</td>
<td>37</td>
<td>63</td>
<td>66.5 ± 3.5</td>
</tr>
<tr>
<td>DCE-3</td>
<td>65</td>
<td>70</td>
<td>43.5 ± 4.0</td>
</tr>
<tr>
<td>DCE-4</td>
<td>49</td>
<td>67</td>
<td>31.6 ± 2.5</td>
</tr>
</tbody>
</table>

Table 4.3: Number of grains (N), D₀ values, and OD values for samples DCM-1–7 and DCE-3–4 for operators KO and ZJ, as well as ratios for D₀ and OD values with KO’s values as the numerator and ZJ’s values as the denominator.
<table>
<thead>
<tr>
<th>Sample name</th>
<th>Total dose rate (Gy/ka)</th>
<th>(D_e) (Gy)</th>
<th>OSL age (ka)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DCM-1</td>
<td>2.31 ± 0.11</td>
<td>68.6 ± 2.8</td>
<td>71.7 ± 3.4</td>
</tr>
<tr>
<td>(Layer 9.1)</td>
<td></td>
<td>29.7 ± 1.9</td>
<td>31.1 ± 2.2</td>
</tr>
<tr>
<td>DCM-2</td>
<td>2.06 ± 0.10</td>
<td>65.4 ± 3.3</td>
<td>69.4 ± 4.3</td>
</tr>
<tr>
<td>(Layer 9.2)</td>
<td></td>
<td>31.8 ± 2.3</td>
<td>33.7 ± 2.7</td>
</tr>
<tr>
<td>DCM-4</td>
<td>1.40 ± 0.06</td>
<td>51.5 ± 1.7</td>
<td>52.3 ± 1.6</td>
</tr>
<tr>
<td>(Layer 11.2)</td>
<td></td>
<td>36.7 ± 2.2</td>
<td>37.3 ± 2.2</td>
</tr>
<tr>
<td>DCM-5</td>
<td>1.69 ± 0.08</td>
<td>72.2 ± 2.3</td>
<td>72.9 ± 2.3</td>
</tr>
<tr>
<td>(Layer 11.3)</td>
<td></td>
<td>42.7 ± 2.5</td>
<td>43.1 ± 2.5</td>
</tr>
<tr>
<td>DCM-3</td>
<td>1.46 ± 0.07</td>
<td>90.7 ± 3.8</td>
<td>93.9 ± 3.2</td>
</tr>
<tr>
<td>(Layer 11.4)</td>
<td></td>
<td>62.0 ± 4.0</td>
<td>64.1 ± 3.8</td>
</tr>
<tr>
<td>DCM-6</td>
<td>1.31 ± 0.06</td>
<td>69.3 ± 2.4</td>
<td>68.7 ± 2.5</td>
</tr>
<tr>
<td>(Layer 11.4)</td>
<td></td>
<td>52.8 ± 3.1</td>
<td>52.4 ± 3.2</td>
</tr>
<tr>
<td>DCM-7</td>
<td>1.13 ± 0.05</td>
<td>66.5 ± 3.5</td>
<td>61.4 ± 4.0</td>
</tr>
<tr>
<td>(Layer 11.4)</td>
<td></td>
<td>58.8 ± 4.3</td>
<td>54.3 ± 4.4</td>
</tr>
<tr>
<td>DCE-3</td>
<td>1.00 ± 0.04</td>
<td>43.5 ± 4.0</td>
<td>49.3 ± 7.4</td>
</tr>
<tr>
<td>(Layer 11.1)</td>
<td></td>
<td>43.3 ± 4.4</td>
<td>49.2 ± 7.7</td>
</tr>
<tr>
<td>DCE-4</td>
<td>1.10 ± 0.05</td>
<td>31.6 ± 2.5</td>
<td>88.4 ± 3.0</td>
</tr>
<tr>
<td>(Layer 11.2)</td>
<td></td>
<td>28.6 ± 2.6</td>
<td>80.0 ± 4.7</td>
</tr>
</tbody>
</table>

Table 4.4: Total dose rates, \(D_e\) values and preliminary OSL ages for samples DCM-1–7 and DCE-3–4 for operators KO and ZJ.
Ages are consistent with those obtained by operator ZJ. Ages of 42.7 ± 2.5 (DCM-5) and 36.7 ± 2.2 ka (DCM-4) were obtained for sub-layers 11.3 and 11.2, respectively. These sub-layers are also associated with the IUP, and the obtained ages are consistent with those obtained by operator ZJ. Ages of 31.8 ± 2.3 (DCM-2) and 29.7 ± 1.9 ka (DCM-1) were obtained for sub-layers 9.2 and 9.1, respectively. These sub-layers are associated with the EUP, and the obtained ages are consistent with those obtained by operator ZJ. These seven ages are consistent with the single finite $^{14}$C age obtained for layer 11 of >37.2 ka (Table 4.1).

Two ages were obtained for the East chamber of Denisova Cave. Ages of 28.6 ± 2.6 (DCE-4) and 43.3 ± 4.4 ka (DCE-3) were obtained for sub-layers 11.2 and 11.1, respectively. These sub-layers are associated with the IUP. The latter age is consistent with the age obtained by operator ZJ, but the former age is inconsistent. The operator ZJ obtained an age of 80.0 ± 4.7 for sub-layer 11.2. This is due to there being two discrete components present in the $D_e$ distribution with a similar number of grains. The age obtained by the operator ZJ is more consistent with the other preliminary ages obtained in this project and one finite $^{14}$C age of >50 ka, but the age obtained by the operator KO is closer in age to two $^{14}$C ages of 27.2 – 27.7 and 33.8 – 34.6 ka cal BP. Further work needs to be done to understand this result.

Figure 4.6: The $D_e$ values for KO (red) and ZJ (blue) for samples DCM-1–7 and DCE-3–4. Each value is presented with its 1σ standard error.
4.7. Synopsis

In this chapter, the sedimentological context, UP context, hominin context, and existing chronologies for Denisova Cave have been discussed. The optical dating results produced in this project for the Main and East chambers were presented, including OSL D_e measurements, operator comparisons, and preliminary ages. These preliminary ages were then compared to the existing chronologies. The implications for the preliminary ages presented in this chapter will be discussed in Chapter 7.
Chapter 5: Anui-2 and Anui-3
5.1. Introduction

The archaeological sites of Anui-2 and Anui-3 are open-air sites located near the Anui River. Anui-2 is located around 70 m downslope of Denisova Cave (51°23’48’’ N, 84°40’35’’ E), while Anui-3 (51°23’35’’ N, 84°40’49’’ E) is located a little further upriver, around 1.3 km from the confluence of the Anui River and the Karakol River (Figure 1.1; Derevianko et al., 2000; Derevianko et al., 2003; Zwyns, 2012). Anui-2 contains UP artefacts in layers 13.2–8 (Derevianko et al., 2003; Zwyns, 2012); a number of $^{14}$C ages have been obtained for these layers (Orlova, 1995; Derevianko et al., 1998). Anui-3 contains UP artefacts in layers 12–2 (Derevianko et al., 2000; Zwyns, 2012); only a single RTL age is known from this site (Derevianko & Shunkov, 2002). In this chapter, these two sites will be discussed in terms of their sedimentological contexts, UP contexts, and existing chronologies. The optical dating results produced in this project for these two sites will also be presented, including OSL $D_e$ measurements, dosimetry measurements, and final ages.

5.2. Sedimentological context

The stratigraphic sequence of Anui-2 has been divided into 15 layers, including UP layers 13–8 (Figure 5.1; Derevianko et al., 2003; Zwyns, 2012). The beginning of the UP occupation, sub-layer 13.2, consists of medium-brown homogeneous sandy loam sediments with thin gravels (Zwyns, 2012). Sub-layer 13.1 consists of darker laminated sediments with traces of humus and charcoal, and scattered gravels (Zwyns, 2012). Layers 12–8 consist of medium-brown sandy loam sediments, sub-angular gravels, some laminated structures, and charcoal (Zwyns, 2012). Layer 11, in particular, contains a large amount of charcoal. The preservation of ash lenses indicates that there has been minimal sediment mixing (Derevianko et al., 2003); however, layers 11–8 suggest the influence of some erosional processes (Zwyns, 2012). Palynological studies suggest that there were two main alternating flora spectra during the UP occupation: layers 13, 9 and 8 consist of birch and pine-tree pollens, indicating dry conditions, and layers 12, 11 and 10 consist of dark coniferous taxa, indicating damp conditions (Malaeva, 1995; Derevianko et al., 1998; Derevianko et al., 2003).

The stratigraphic sequence of Anui-3 has been divided into 21 layers, including UP layers 12–2 (Figure 5.2; Derevianko et al., 2000; Derevianko & Shunkov, 2002; Zwyns,
Layers 12–4 consist of a succession of medium-brown and grey sandy loam sediments, displaying bands of sub-angular gravels in layers 9, 7, 6 and 5. Layers 3 and 2 consist of loess-like sediments with traces of laminated structures (Zwyns, 2012). Faunal remains suggest that there were two main climatic phases during the UP occupation at Anui-3: layers 12–5 contain an abundance of mole and zokor remains, as well as some vole, forest vole and pika remains, indicating warm climate conditions, and layers 4–2 contain a poor assemblage of faunal remains, indicating cold climate conditions (Agadjanian & Shunkov, 1999).

5.3. UP context

Layers 13–8 of Anui-2 yielded UP artefacts, including developed blade, bladelet and microblade technologies. These artefacts occur in 12 occupation horizons separated by sterile horizons, indicating intermittent occupations (Zwyns, 2012). Taphonomic analyses have not yet been conducted on these assemblages; however, the artefacts from layers 13–11 are similar in terms of their typological characteristics (Derevianko et al., 2003; Zwyns,
2012). The raw material of these artefacts is local, consisting, for the most part, of nodules or slabs of metamorphic rocks collected from the Anui River (Postnov et al., 2000; Derevianko et al., 2004). Blade reduction sequences are variable, but the most common involve parallel reduction from prismatic and narrow-faced cores (Zwyns, 2012). Microblade production occurs from wedged-shaped, narrow-face and prismatic cores, and all microblade cores display parallel microblade removals on their flaking surface (Figure 5.3; Zwyns, 2012). Gravettian micro-points, micro-end-scarpers, retouched microblades and angle-borers are typical microblade tools (Derevianko et al., 2003; Zwyns, 2012). Blade cores are present in small numbers, displaying a minor occurrence of bidirectional reduction on volumetric and flat-faced cores (Zwyns, 2012). Retouched blades include end-scarpers, burins and side-scarpers (Derevianko et al., 2003; Zwyns, 2012).

These artefacts from layers 13–11 reflect a predominance of UP features, including developed blade, bladelet and microblade technologies obtained by means of direct percussion, and are, therefore, considered by Zwyns (2012) to belong to the EUP (Figure 5.1). Layers 10–8 are, as yet, not assigned to a specific UP variant, but are assumed to be either MUP or FUP.

Anui-3 has yielded UP artefacts in layers 12, 11, 9, 4, 3 and 2, including blade, bladelet and microblade technologies; however, the assemblage is rather small, making it difficult to interpret (Derevianko et al., 2000; Zwyns, 2012). Zwyns (2012) conducted taphonomic analyses on artefacts from layer 12. The raw material for layer 12 is local, consisting of metamorphic and sedimentary pebbles collected from the Anui River (Kulik & Shunkov, 2000; Zwyns, 2012).

Figure 5.2: The stratigraphic sequence of the south face of Anui-3, showing the layers assigned to the MP, UP and EUP (after Derevianko et al., 2000; Derevianko & Shunkov, 2002; Zwyns, 2012).
Reduction sequences involve parallel reduction from prismatic cores (Figure 5.4; Zwyns, 2012), and the artefacts are described as carinated end-scrapers, retouched blades with asymmetrical points, and backed microblades (Derevianko & Shunkov, 2002). Retouched microblades are also present, and reflect variable retouch types, indicating poor standardisation of retouching procedures (Zwyns, 2012). This assemblage displays evidence for the use of soft hammer, as well as unidirectional cores, and artefacts display a predominance of UP features; therefore, Zwyns (2012) has assigned layer 12 to the EUP (Figure 5.2). Layer 13 immediately underlies layer 12, and has been assigned to the MP (Derevianko & Shunkov, 2002). Following the local transitional model, Rybin (2014) has assigned layers 12 and 11 to belong to the IUP based on the presence of blanks with ventral trimming of their transverse distal edges, oblique points, backed points on bladelets and backed bladelets, and stemmed blades. Derevianko and Shunkov (2002) have assigned layer 12, 11 and 9 to the IUP, also following the local transition model. Layers 4, 3 and 2 have not yet been assigned to a specific variant, but are assumed to be either MUP or FUP.

Figure 5.3: Microblade core from layer 11 at Anui-2, displaying several reduction phases, a typical characteristic of microblade cores from this site (after Zwyns, 2012).
5.4. Existing chronologies

Nine $^{14}$C ages have been reported for six UP layers and sub-layers from Anui-2 – 13.2, 13.1, 12, 11, 10.2 and 10.1 – ranging from 24.4 to 35.0 ka cal BP (Table 5.1; Orlova, 1995; Derevianko et al., 1998). Eight of these ages were produced from charcoal, and the 28.8 – 35.0 ka cal BP age for sub-layer 13.2 was produced from humates. These ages were calibrated using the IntCal13 radiocarbon calibration curve (Reimer et al., 2013), and are consistent with the stratigraphic sequence.

No $^{14}$C ages have been reported for Anui-3; however, there is a single RTL age for layer 12 of 54 ± 13 ka (Table 5.1; Figure 5.2; Derevianko & Shunkov, 2002). Stratigraphic comparisons between Anui-3 and Ust-Karakol-1 Trench 2 have been made by Derevianko and Shunkov (2002), resulting in inferred ages for the UP layers of Anui-3; these comparisons will be discussed in Section 7.3.
<table>
<thead>
<tr>
<th>Site</th>
<th>Layer</th>
<th>Uncalibrated $^{14}$C age (ka BP)</th>
<th>Calibrated $^{14}$C age (ka cal BP) at 95% C.I.</th>
<th>RTL age (ka)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Anui-2</td>
<td>10.1</td>
<td>21.3 ± 0.4</td>
<td>24.4 – 26.4</td>
<td></td>
</tr>
<tr>
<td></td>
<td>10.2</td>
<td>21.5 ± 0.6</td>
<td>24.4 – 27.1</td>
<td></td>
</tr>
<tr>
<td></td>
<td>11</td>
<td>23.4 ± 1.5</td>
<td>24.5 – 30.8</td>
<td></td>
</tr>
<tr>
<td></td>
<td>12</td>
<td>20.4 ± 0.3, 22.6 ± 0.1, 24.2 ± 0.4</td>
<td>23.8 – 25.3, 26.5 – 27.3, 27.6 – 29.1</td>
<td></td>
</tr>
<tr>
<td></td>
<td>13.1</td>
<td>27.1 ± 0.8</td>
<td>29.5 – 33.0</td>
<td></td>
</tr>
<tr>
<td></td>
<td>13.2</td>
<td>26.8 ± 0.3, 27.9 ± 1.6</td>
<td>30.5 – 31.3, 28.8 – 35.0</td>
<td></td>
</tr>
<tr>
<td>Anui-3</td>
<td>12</td>
<td></td>
<td></td>
<td>54 ± 13</td>
</tr>
</tbody>
</table>

Table 5.1: The existing published chronologies for UP layers at Anui-2 and Anui-3 (after Orlova, 1995; Derevianko et al., 1998; Derevianko & Shunkov, 2002).
5.5. Optical dating results

In this project, OSL dating of single quartz grains was conducted for three samples from Anui-2 and five samples from Anui-3. The three samples from Anui-2 corresponded to sub-layer 13.2 (ANUI 2 OSL 1), sub-layer 13.1 (ANUI 2 OSL 2) and layer 11 (ANUI 2 OSL 3) (Figure 5.5), and the five samples from Anui-3 corresponded to layer 12 (ANUI 3 OSL 7), layer 11 (ANUI 3 OSL 8), layer 10 (ANUI 3 OSL 9), layer 8 (ANUI 3 OSL 10) and layer 4 (ANUI 3 OSL 11) (Figure 5.6).

5.5.1. OSL D_e measurements

The SAR protocol was used for OSL dating of single quartz grains. Dose recovery tests were not conducted for Anui-2 and Anui-3; instead, it was assumed that the same preheat temperature combinations as those used for Denisova Cave were appropriate because these sites are in close proximity to Denisova Cave. A preheat temperature of 260°C for 10 s was used prior to measurement of L_N and L_X, and a preheat temperature of 160°C for 5 s was used prior to measurement of T_N and T_X. A total of 13,000 quartz grains were measured for these eight samples.

5.5.2. Single-grain rejection and analysis

Single quartz grains were rejected following the rejection criteria outlined in Section 3.5.8.1, the results of which are presented in Table 5.2. The most grains (71.8% from Anui-2 and 79.1% from Anui-3) were rejected on the basis of criterion 1; that is, the sensitivity of their T_N signals. The operator KO rejected 2.8% of grains from Anui-2 and 1.0% of grains from Anui-3 on the basis of L_N/T_N ratios not intercepting with dose-response curves due to saturation or not being able to fit a saturating exponential curve. Samples from higher in the stratigraphic sequences (ANUI 2 OSL 3, ANUI 3 OSL 10 and ANUI 3 OSL 11) yielded fewer quartz coarse-grains than those lower in the stratigraphic sequence, even after both the 125–90 and 212–180 μm in diameter grain-size fractions were measured.

5.5.3. Optical decay curves

Optical decay curves were produced for all measured grains. Figure 5.7 displays optical decay curves measured after a test dose of ~12 Gy for 10 quartz grains from two samples, ANUI 2 OSL 1 and ANUI 3 OSL 7: these are representative of quartz grains from all eight samples. For each sample, three bright grains, four moderately bright grains, and three dim
Figure 5.5: The location of samples ANUI 2 OSL 1–3 in the stratigraphic sequence at Anui-2.
Figure 5.6: The location of samples ANUI 3 OSL 7–11 in the stratigraphic sequence at Anui-3.
<table>
<thead>
<tr>
<th>Sample name</th>
<th># measured grains</th>
<th>Criterion 1</th>
<th>Criterion 2</th>
<th>Criterion 3</th>
<th>Criterion 4</th>
<th>Criterion 5</th>
<th>Operator rejections</th>
<th>Σ rejected grains</th>
<th>Σ accepted grains</th>
</tr>
</thead>
<tbody>
<tr>
<td>ANUI 2 OSL 3</td>
<td>200</td>
<td>151</td>
<td>32</td>
<td>1</td>
<td>6</td>
<td>2</td>
<td>4</td>
<td>196</td>
<td>4</td>
</tr>
<tr>
<td>ANUI 2 OSL 2</td>
<td>1500</td>
<td>1050</td>
<td>256</td>
<td>29</td>
<td>29</td>
<td>28</td>
<td>37</td>
<td>1429</td>
<td>71</td>
</tr>
<tr>
<td>ANUI 2 OSL 1</td>
<td>1500</td>
<td>1098</td>
<td>191</td>
<td>42</td>
<td>26</td>
<td>27</td>
<td>47</td>
<td>1431</td>
<td>69</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>3200</strong></td>
<td><strong>2299</strong></td>
<td><strong>479</strong></td>
<td><strong>72</strong></td>
<td><strong>61</strong></td>
<td><strong>57</strong></td>
<td><strong>88</strong></td>
<td><strong>3056</strong></td>
<td><strong>144</strong></td>
</tr>
<tr>
<td></td>
<td></td>
<td><strong>71.8%</strong></td>
<td><strong>15.0%</strong></td>
<td><strong>2.3%</strong></td>
<td><strong>1.9%</strong></td>
<td><strong>1.8%</strong></td>
<td><strong>2.8%</strong></td>
<td><strong>95.5%</strong></td>
<td><strong>4.5%</strong></td>
</tr>
<tr>
<td>ANUI 3 OSL 11</td>
<td>1000</td>
<td>800</td>
<td>136</td>
<td>12</td>
<td>14</td>
<td>13</td>
<td>8</td>
<td>983</td>
<td>17</td>
</tr>
<tr>
<td>ANUI 3 OSL 10</td>
<td>800</td>
<td>621</td>
<td>125</td>
<td>6</td>
<td>9</td>
<td>24</td>
<td>8</td>
<td>793</td>
<td>7</td>
</tr>
<tr>
<td>ANUI 3 OSL 9</td>
<td>2000</td>
<td>1552</td>
<td>296</td>
<td>27</td>
<td>33</td>
<td>30</td>
<td>17</td>
<td>1955</td>
<td>45</td>
</tr>
<tr>
<td>ANUI 3 OSL 8</td>
<td>2500</td>
<td>1980</td>
<td>345</td>
<td>31</td>
<td>32</td>
<td>27</td>
<td>27</td>
<td>2442</td>
<td>58</td>
</tr>
<tr>
<td>ANUI 3 OSL 7</td>
<td>3500</td>
<td>2797</td>
<td>483</td>
<td>37</td>
<td>20</td>
<td>51</td>
<td>39</td>
<td>3427</td>
<td>73</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>9800</strong></td>
<td><strong>7750</strong></td>
<td><strong>1385</strong></td>
<td><strong>113</strong></td>
<td><strong>108</strong></td>
<td><strong>145</strong></td>
<td><strong>97</strong></td>
<td><strong>9600</strong></td>
<td><strong>200</strong></td>
</tr>
<tr>
<td></td>
<td></td>
<td><strong>79.1%</strong></td>
<td><strong>14.1%</strong></td>
<td><strong>1.2%</strong></td>
<td><strong>1.1%</strong></td>
<td><strong>1.5%</strong></td>
<td><strong>1.0%</strong></td>
<td><strong>97.9%</strong></td>
<td><strong>2.1%</strong></td>
</tr>
</tbody>
</table>

Table 5.2: The number of grains measured, rejected and accepted for ANUI 2 OSL 1–3 and ANUI 3 OSL 7–11 based on 5 rejection criteria: 1) $T_N$ signal <3×BG; 2) $T_N$ error >20%; 3) recycling ratio >2σ from unity; 4) IR depletion ratio >2σ from unity; and 5) ratio of $(L_0/T_X)/(L_N/T_N) >10\%$, as well as additional rejections by the operator KO. The 125–90 µm in diameter grain-size fraction was measured for each sample, and for samples ANUI 2 OSL 1, ANUI 3 OSL 10 and ANUI 3 OSL 11, the 212–180 µm in diameter grain-size fraction was also measured. The above values are a combination of the two grain-size fractions.
grains were selected. The insets in Figure 5.7 display the normalised OSL signals for each of these grains; it can be seen that the shapes of the optical decay curves are very similar between grains and that <5% of the OSL signal remains after ~0.3 s of optical stimulation. The shapes of the optical decay curves suggest that the OSL signals are dominated by the ‘fast’ component in quartz (Bailey et al., 1997).

Figure 5.7: Optical decay curves and normalised optical decay curves (inset) for 10 single quartz grains from a) ANUI 2 OSL 1, and b) ANUI 3 OSL 7. The x-axis displays the stimulation time in seconds, and the y-axis displays the measured OSL signal as counts per channel recorded after each 0.02 seconds. The first five channels and the last five channels were dark counts.
5.5.4. Dose-response curves

Dose-response curves were constructed for each measured grain using regenerative doses of ~72, 144, 216 and 288 Gy. Figure 5.8 displays dose-response curves for four quartz grains from two samples, ANUI 2 OSL 1 and ANUI 3 OSL 7. The dose-response curves display a wide range of shapes as a result of different saturation characteristics. Some grains saturate more easily than others, showing little to no growth with an increase in dose, whereas others continue to grow with an increase in dose.

5.5.5. Single-grain $D_e$ distributions

The three samples from Anui-2 display two very different $D_e$ distributions (Figure 5.9a–c). ANUI 2 OSL 1 and OSL 2 display distributions that are well-bleached, but with some outlier $D_e$ values, both higher and lower. Most grains are scattered around a mean

![Dose-response curves for 4 quartz grains from a) ANUI 2 OSL 1, and b) ANUI 3 OSL 7. The x-axis displays the dose (Gy), and the y-axis displays the sensitivity-corrected OSL signal.](image-url)

Figure 5.8: Dose-response curves for 4 quartz grains from a) ANUI 2 OSL 1, and b) ANUI 3 OSL 7. The x-axis displays the dose (Gy), and the y-axis displays the sensitivity-corrected OSL signal.
Figure 5.9: Radial plots, numbers of grains, OD values and $D_e$ values for samples a) ANUI 2 OSL 1, b) ANUI 2 OSL 2, c) ANUI 2 OSL 3, d) ANUI 3 OSL 7, e) ANUI 3 OSL 8, f) ANUI 3 OSL 9, g) ANUI 3 OSL 10, and h) ANUI 3 OSL 11.
To obtain a $D_e$ value for age determination, the CAM was used; the grey bands in Figure 5.9a and b are centred on this value. ANUI 2 OSL 3 displays a distribution with significant scatter, and there are too few values for a reliable interpretation to be made. To obtain an estimate of the $D_e$ value, the CAM was used; the grey band in Figure 5.9c is centred on this value.

The five samples from Anui-3 also display two very different $D_e$ distributions. ANUI 3 OSL 7, OSL 8 and OSL 9 display at least two or three discrete dose components with one of these components always the dominant component. This can happen when younger grains are included in the main sediment matrix by burrowing animals. The presence of mole, zokor, vole, forest vole and pika remains in these layers supports this interpretation. To obtain an estimate of the $D_e$ value, the FMM was used; the grey bands and lines in Figure 5.9d–f are centred on the values of the discrete dose components. ANUI 3 OSL 10 and ANUI 3 OSL 11 display distributions with significant scatter, and there are too few values for a reliable interpretation to be made; although, the distribution of ANUI 3 OSL 11 suggests that if more grains were measured and accepted, it may have 2 or more discrete dose components. To obtain an estimate of the $D_e$ value, the CAM was used; the grey bands in Figure 5.9g and h are centred on this value.

It’s important to note that while Table 5.2 presents the number of grains obtained for both the 125–90 and 212–180 μm in diameter grain-size fractions, just the 125–90 μm in diameter grain-size fraction is presented in Figure 5.9, as this was the grain-size fraction used in age determination.

### 5.5.6. Dosimetry measurements

Total dose rates were calculated for each of the eight samples from Anui-2 and Anui-3. Beta dose rates were measured directly using the GM-25-5 beta counter, the field gamma dose rate was measured directly using in situ gamma spectrometry, the cosmic-ray dose rate was calculated using the equation provided in Prescott and Hutton (1988), and an internal dose rate of $0.030 \pm 0.011$ Gy/ka was assumed. Beta, gamma and cosmic-ray dose rates were corrected for water content, and the beta dose rate was corrected for a grain-size of 100 μm in diameter because individual grains of 90–125 μm in diameter were measured for $D_e$ determination. The individual dose rate values for each sample are provided in Table 5.3 together with their measured water contents and total environmental dose rates.
<table>
<thead>
<tr>
<th>Sample name</th>
<th>Grain size (µm)</th>
<th>Water content (%)</th>
<th>Beta dose rate (Gy/ka)</th>
<th>Gamma dose rate (Gy/ka)</th>
<th>Cosmic-ray dose rate (Gy/ka)</th>
<th>Total dose rate (Gy/ka)</th>
</tr>
</thead>
<tbody>
<tr>
<td>ANUI 2 OSL 3</td>
<td>125–90</td>
<td>24 ± 6</td>
<td>1.36 ± 0.09</td>
<td>0.78 ± 0.05</td>
<td>0.11 ± 0.02</td>
<td>2.28 ± 0.11</td>
</tr>
<tr>
<td>ANUI 2 OSL 2</td>
<td>125–90</td>
<td>13 ± 3</td>
<td>1.43 ± 0.07</td>
<td>0.83 ± 0.04</td>
<td>0.11 ± 0.02</td>
<td>2.40 ± 0.09</td>
</tr>
<tr>
<td>ANUI 2 OSL 1</td>
<td>125–90</td>
<td>23 ± 6</td>
<td>1.36 ± 0.09</td>
<td>0.91 ± 0.06</td>
<td>0.10 ± 0.02</td>
<td>2.40 ± 0.11</td>
</tr>
<tr>
<td>ANUI 3 OSL 11</td>
<td>125–90</td>
<td>8 ± 2</td>
<td>1.57 ± 0.07</td>
<td>0.89 ± 0.04</td>
<td>0.20 ± 0.03</td>
<td>2.69 ± 0.08</td>
</tr>
<tr>
<td>ANUI 3 OSL 10</td>
<td>125–90</td>
<td>11 ± 3</td>
<td>1.65 ± 0.08</td>
<td>0.73 ± 0.03</td>
<td>0.18 ± 0.03</td>
<td>2.59 ± 0.09</td>
</tr>
<tr>
<td>ANUI 3 OSL 9</td>
<td>125–90</td>
<td>13 ± 3</td>
<td>1.50 ± 0.08</td>
<td>0.90 ± 0.04</td>
<td>0.18 ± 0.03</td>
<td>2.61 ± 0.09</td>
</tr>
<tr>
<td>ANUI 3 OSL 8</td>
<td>125–90</td>
<td>20 ± 5</td>
<td>1.55 ± 0.09</td>
<td>1.00 ± 0.06</td>
<td>0.16 ± 0.02</td>
<td>2.74 ± 0.11</td>
</tr>
<tr>
<td>ANUI 3 OSL 7</td>
<td>125–90</td>
<td>15 ± 4</td>
<td>1.53 ± 0.08</td>
<td>1.01 ± 0.05</td>
<td>0.17 ± 0.03</td>
<td>2.73 ± 0.10</td>
</tr>
</tbody>
</table>

Table 5.3: Total environmental dose rates for ANUI 2 OSL 1–3 and ANUI 3 OSL 7–11, using GM-25-5 beta counting for the beta dose rate, in situ gamma spectrometry for the gamma dose rate, the cosmic-ray dose rate equation for the cosmic-ray dose rate, and an internal dose rate of 0.030 ± 0.011 Gy/ka, corrected for water contents as measured in this project.
The water contents for samples ranged between 8 ± 2% and 24 ± 6%; the sediments at Anui-2 are mostly wetter than those at Anui-3. The beta dose rates range between 1.36 ± 0.09 and 1.43 ± 0.07 Gy/ka for samples from Anui-2, and 1.50 ± 0.08 and 1.65 ± 0.08 Gy/ka for samples from Anui-3. The gamma dose rates range between 0.78 ± 0.05 and 0.91 ± 0.06 Gy/ka for samples from Anui-2, and 0.73 ± 0.03 and 1.01 ± 0.05 Gy/ka for samples from Anui-3. At both sites, there is an increase in gamma dose rate with an increase in depth, with the exception of ANUI 3 OSL 10. This may reflect differences in the amount of clay versus gravel present in the sediment. A comparison between the field gamma dose rate and a laboratory gamma dose rate is provided in Table 5.4. The field gamma dose rates are both higher and lower than their respective laboratory gamma dose rates, suggesting that the differences are not systematic due to the methods used, but perhaps reflect real differences in sediment composition within the ~300 mm gamma sphere of influence. The total dose rates are lower at Anui-2 than at Anui-3 because the cosmic-ray dose rates are lower; this is because samples were collected from at least twice the depth at Anui-2 than at Anui-3, and the sediments at Anui-2 are mostly wetter.

5.5.7. Final ages

The total environmental dose rates are presented together with the $D_e$ values and final OSL ages for each sample in Table 5.5. Three ages were obtained for Anui-2. Ages of 32.6 ± 2.4 (ANUI 2 OSL 1) and 32.3 ± 2.5 ka (ANUI 2 OSL 2) were obtained for samples from sub-layers 13.2 and 13.1, respectively. Layer 13 is the earliest layer associated with the EUP at Anui-2. Both of these ages are consistent with the $^{14}C$ ages obtained for the these two sub-layers (see Table 5.1). The $D_e$ distribution for ANUI 2 OSL 3 from layer 11 displayed a scattered distribution containing few grains from which a reliable estimate of the $D_e$ for age determination could not be obtained, resulting in an age of 49.7 ± 19.0 ka; this age is inaccurate, imprecise and inconsistent with the $^{14}C$ age for this layer. Further work needs to be done to understand this result.

Five ages were obtained for Anui-3. The age for layer 12, which is, according to Zwyns (2012), the earliest layer associated with the EUP at Anui-3 was 29.1 ± 2.0 ka (ANUI 3 OSL 7). This is consistent with ages obtained for the EUP at Anui-2, but inconsistent with the RTL age of 54 ± 13 ka, albeit within 2$\sigma$ of this age. OSL ages for the overlying layers decrease from 25.1 ± 1.8 ka (ANUI 3 OSL 8) in layer 11 to 23.7 ± 1.5 ka (ANUI 3 OSL 9) in layer 10 to 19.4 ± 5.2 ka (ANUI 3 OSL 10) in layer 8 to 12.9 ± 6.1 ka (ANUI 3 OSL 11)
<table>
<thead>
<tr>
<th>Sample name</th>
<th>Field gamma dose rate (Gy/ka)</th>
<th>Laboratory gamma dose rate (Gy/ka)</th>
<th>Ratio (Field/Laboratory)</th>
</tr>
</thead>
<tbody>
<tr>
<td>ANUI 2 OSL 3</td>
<td>0.78 ± 0.05</td>
<td>0.78 ± 0.06</td>
<td>1.00 ± 0.10</td>
</tr>
<tr>
<td>ANUI 2 OSL 2</td>
<td>0.83 ± 0.04</td>
<td>0.81 ± 0.05</td>
<td>1.03 ± 0.08</td>
</tr>
<tr>
<td>ANUI 2 OSL 1</td>
<td>0.91 ± 0.06</td>
<td>0.79 ± 0.05</td>
<td>1.15 ± 0.11</td>
</tr>
<tr>
<td>ANUI 3 OSL 11</td>
<td>0.89 ± 0.04</td>
<td>0.94 ± 0.05</td>
<td>0.95 ± 0.07</td>
</tr>
<tr>
<td>ANUI 3 OSL 10</td>
<td>0.73 ± 0.03</td>
<td>0.99 ± 0.06</td>
<td>0.73 ± 0.06</td>
</tr>
<tr>
<td>ANUI 3 OSL 9</td>
<td>0.90 ± 0.04</td>
<td>0.90 ± 0.05</td>
<td>0.99 ± 0.08</td>
</tr>
<tr>
<td>ANUI 3 OSL 8</td>
<td>1.00 ± 0.06</td>
<td>0.89 ± 0.06</td>
<td>1.13 ± 0.10</td>
</tr>
<tr>
<td>ANUI 3 OSL 7</td>
<td>1.01 ± 0.05</td>
<td>0.93 ± 0.05</td>
<td>1.09 ± 0.08</td>
</tr>
</tbody>
</table>

Table 5.4: Comparison between the field gamma dose rate obtained using in situ gamma spectrometry and the laboratory gamma dose rate obtained using TSAC and GM-25-5 beta counting for ANUI 2 OSL 1–3 and ANUI 3 OSL 7–11, and the ratio of these two measurements with the field gamma dose rate as the numerator and the laboratory gamma dose rate as the denominator.
<table>
<thead>
<tr>
<th>Sample name</th>
<th>Total dose rate (Gy/ka)</th>
<th>$D_e$ value (Gy)</th>
<th>OSL age (ka)</th>
</tr>
</thead>
<tbody>
<tr>
<td>ANUI 2 OSL 3 (Layer 11)</td>
<td>2.28 ± 0.11</td>
<td>113.4 ± 43.1</td>
<td>49.7 ± 19.0</td>
</tr>
<tr>
<td>ANUI 2 OSL 2 (Layer 13.1)</td>
<td>2.40 ± 0.09</td>
<td>77.4 ± 5.1</td>
<td>32.3 ± 2.5</td>
</tr>
<tr>
<td>ANUI 2 OSL 1 (Layer 13.2)</td>
<td>2.40 ± 0.11</td>
<td>78.2 ± 4.3</td>
<td>32.6 ± 2.4</td>
</tr>
<tr>
<td>ANUI 3 OSL 11 (Layer 4)</td>
<td>2.69 ± 0.08</td>
<td>34.6 ± 16.4</td>
<td>12.9 ± 6.1</td>
</tr>
<tr>
<td>ANUI 3 OSL 10 (Layer 8)</td>
<td>2.59 ± 0.09</td>
<td>50.2 ± 20.3</td>
<td>19.4 ± 5.2</td>
</tr>
<tr>
<td>ANUI 3 OSL 9 (Layer 10)</td>
<td>2.61 ± 0.09</td>
<td>61.7 ± 3.0</td>
<td>23.7 ± 1.5</td>
</tr>
<tr>
<td>ANUI 3 OSL 8 (Layer 11)</td>
<td>2.74 ± 0.11</td>
<td>68.7 ± 3.9</td>
<td>25.1 ± 1.8</td>
</tr>
<tr>
<td>ANUI 3 OSL 7 (Layer 12)</td>
<td>2.73 ± 0.10</td>
<td>79.4 ± 4.2</td>
<td>29.1 ± 2.0</td>
</tr>
</tbody>
</table>

Table 5.5: Total dose rates, $D_e$ values and final OSL ages for ANUI 2 OSL 1–3 and ANUI 3 OSL 7–11.
in layer 4. The latter two ages are imprecise due to there not being enough grains to obtain a reliable \( D_\varepsilon \) estimate. No \(^{14}\text{C}\) ages were reported for this section with which these results could be compared.

5.6. Synopsis

In this chapter, the UP sites of Anui-2 and Anui-3 have been discussed in terms of their sedimentological contexts, UP contexts, and existing chronologies. The results of optical dating conducted in this project were presented, including OSL \( D_\varepsilon \) measurements, dosimetry measurements, and final ages. These ages were then compared to the existing chronologies. The implications for the ages presented in this chapter will be discussed in Chapter 7.
Chapter 6: Ust-Karakol-1 Trench 2
6.1. Introduction

The archaeological site of Ust-Karakol-1 (51°22′50″ N, 84°41′20″ E) is an open-air site located 2 km southeast of Denisova Cave, at the confluence of the Anui and Karakol rivers (Figure 1.1; Derevianko et al., 2003). Trench 2 of this site contains UP artefacts in layers 11–2 (Zwyns, 2012); ¹⁴C ages, palaeomagnetic excursion ages, and an RTL age were obtained for these layers (Derevianko et al., 1998; Derevianko et al., 2003; Derevianko & Rybin, 2003). In this chapter, this site will be discussed in terms of its sedimentological context, UP context, and existing chronologies. The optical dating results produced in this project for this site will also be presented, including several dose recovery tests, pIR-IRSL Dₜ measurements, dosimetry measurements, and final ages.

6.2. Sedimentological context

The UP stratigraphic sequence of Ust-Karakol-1 Trench 2 has been divided into 21 layers including UP layers 11–2 (Figure 6.1; Derevianko et al., 2003; Zwyns, 2012). Layers 11–9 consist of dark-brown sandy loam sediments, orange mottles, and sub-horizontal laminations. Layers 8–6 also consist of dark-brown sandy loam sediments, and contain some mole-rat burrows. Layer 5 consists of light-brown sandy loam sediments, and contains abundant mole-rat burrows. Layers 4–2 consist of loose loess-like sediments, displaying abundant mole-rat burrows, as well as vertical cracks (Zwyns, 2012).
Palynological studies suggest that there were two main alternating flora spectra during the UP occupation at Ust-Karakol-1 Trench 2: the first consists of birch and pine-tree pollens, suggesting dry conditions, and the second consists of taiga forest taxa, suggesting cool, humid conditions (Derevianko et al., 2003). The most abundant faunal remains in the UP layers are zokor (Agadjanian & Serdyuk, 2005). The faunal assemblage, however, is rather poor, providing limited insights into climate conditions. The presence of taphonomic issues including bioturbation and frost cracks may have caused sediment mixing; therefore, small-scale vertical artefact movement in the UP layers may have occurred, complicating chronologies and stratigraphic associations (Slavinsky, 2007; Zwyns, 2012).

6.3. UP context

Ust-Karakol-1 Trench 2 has yielded UP artefacts in layers 11–8 and 5–2. At present, only artefacts from layers 11–8 have been fully documented, and these layers have been documented as a single unit due to uncertainties caused by sediment mixing (Zwyns, 2012). Layers 11–8 contain blade, bladelet and microblade technologies, produced from metamorphic rocks derived from the Anui and Karakol rivers (Zwyns, 2012). Blade cores display unidirectional and bidirectional reduction sequences (Figure 6.2). Uni-directional sequences, however, occur more often and, therefore, there are more unidirectional blanks present (Zwyns, 2012). Despite the presence of large blade technologies, the most representative artefacts of Ust-Karakol-1 Trench 2 are those associated with the production of microblade blanks, including microblade cores, blanks and retouched blanks, similar to that observed at Anui-2 and Anui-3 (Zwyns, 2012). The production of microblade blanks uses direct percussion, and does not display evidence of pressure flaking (Zwyns, 2012). There are Levallois elements present in layer 11, but these may be due to small-scale vertical artefact movement from MP layers caused by the taphonomic issues discussed above (Slavinsky, 2007; Zwyns, 2012). Based on the presence of developed microblade technologies, and concluding that Levallois elements are a result of taphonomic issues, layers 11–8 have been assigned to the EUP (Zwyns, 2012).

Trench 1 of Ust-Karakol-1, located just 5 m west of Trench 2 (Slavinsky, 2007) complicates the assignment of the aforementioned layers to the EUP. Occupation horizons 5.4 and 5.5 in Trench 1 are thought to correspond to layers 11 and 12 in Trench 2, respectively (Zwyns, 2012). While layer 11 in Trench 2 has been assigned to the EUP and
layer 12 is culturally sterile, occupation horizons 5.4 and 5.5 have been assigned to the IUP (Slavinsky, 2007; Zwyns, 2012; Rybin, 2014). If the Levallois elements present in layer 11 of Trench 2 are not due to small-scale vertical artefact movement from MP layers (layers 13–19), layer 11 may belong to the IUP. Producing reliable chronologies for Ust-Karakol-1 Trench 2 may help to assign layer 11 to either the EUP or IUP based on comparisons with other sites containing either EUP or IUP technologies. Layer 5 has been assigned to the MUP and layers 4–2 have been assigned to the FUP (Derevianko & Shunkov, 2002).

6.4. Existing chronologies

Twelve $^{14}$C ages have been reported for five UP layers from Ust-Karakol-1 Trench 2 – 10, 9, 5, 3 and 2 – ranging from 29.8 to 44.4 ka cal BP (Table 6.1; Derevianko et al., 1998; Derevianko & Rybin, 2003). Nine of the ages from layers 10, 9, 5 and 3 were obtained from charcoal, one age from layer 5 (30.6 – 31.4 ka cal BP) was obtained from humates, and the two ages from layer 2 were obtained from bone. These ages were calibrated using the IntCal13 radiocarbon calibration curve (Reimer et al., 2013). In some cases, these $^{14}$C

![Blade core from layers 11–8 at Ust-Karakol-1 Trench 2, displaying the negatives of blanks, and testifying to the production of convergent blanks (after Zwyns, 2012).](image)

Figure 6.2: Blade core from layers 11–8 at Ust-Karakol-1 Trench 2, displaying the negatives of blanks, and testifying to the production of convergent blanks (after Zwyns, 2012).
<table>
<thead>
<tr>
<th>Layer</th>
<th>RTL age (ka)</th>
<th>Palaeomagnetic excursion age (ka)</th>
<th>Uncalibrated $^{14}$C age (ka BP)</th>
<th>Calibrated $^{14}$C age (ka cal BP) at 95% C.I.</th>
</tr>
</thead>
<tbody>
<tr>
<td>2</td>
<td></td>
<td>11 – 13</td>
<td>28.7 ± 0.9, 31.4 ± 1.2</td>
<td>31.1 – 34.2, 33.3 – 38.4</td>
</tr>
<tr>
<td>3</td>
<td></td>
<td></td>
<td>31.3 ± 1.3, 31.4 ± 1.2</td>
<td>32.7 – 38.6, 33.3 – 38.4</td>
</tr>
<tr>
<td>5</td>
<td></td>
<td>25 – 30</td>
<td>26.3 ± 0.3, 26.9 ± 0.3, 27.0 ± 0.4, 30.5 ± 2.0</td>
<td>29.8 – 31.0, 30.6 – 31.4, 30.2 – 31.8, 30.6 – 39.1</td>
</tr>
<tr>
<td>9</td>
<td>50 ± 12</td>
<td></td>
<td>29.7 ± 0.4, 29.9 ± 0.4, 33.4 ± 1.3</td>
<td>33.1 – 34.5, 33.4 – 34.6, 34.8 – 40.4</td>
</tr>
<tr>
<td>10</td>
<td></td>
<td></td>
<td>35.1 ± 2.9</td>
<td>33.7 – 44.4</td>
</tr>
<tr>
<td>11</td>
<td></td>
<td>42 – 44</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table 6.1: The existing published chronologies for UP layers at Ust-Karakol-1 Trench 2 (after Derevianko et al., 1998; Derevianko et al., 2003; Derevianko & Rybin, 2003).
ages are not consistent with the stratigraphic sequence. Three palaeomagnetic excursion ages have also been reported for UP layers at Ust-Karakol-1 Trench 2, including ages of 42 – 44 ka for layer 11, 25 – 30 ka for layer 5, and 11 – 13 ka for layer 2 (Table 6.1; Derevianko et al., 2003). One RTL age of 50 ± 12 has also been reported for layer 9 (Table 6.1; Derevianko et al., 1998).

6.5. Optical dating results

In this project, optical dating was conducted for seven samples from Ust-Karakol-1 Trench 2. These seven samples correspond to layer 11 (UK OSL 7), layer 10 (UK OSL 8), layer 9 (UK OSL 9), layer 7 (UK OSL 10), layer 6 (UK OSL 11), layer 5 (UK OSL 12) and layer 4 (UK OSL 13) (Figure 6.3). Three approaches were taken to obtain \( D_e \) values for these samples. The first approach was OSL dating of single quartz grains. Dose recovery tests were conducted to determine the most appropriate preheat temperature combinations. The second approach was pIR-IRSL dating of single potassium-feldspar grains. Dose recovery tests were conducted to determine the most appropriate preheat temperature combinations. The third approach was pIR-IRSL dating of single aliquots of polymineral fine-grains. Dose recovery tests were conducted to determine the most appropriate preheat temperature combinations. \( D_e \) values for final age determination were obtained using this third approach.

6.5.1. OSL quartz dose recovery tests

Dose recovery tests were conducted on single aliquots of quartz grains. The aim of using single aliquots was to test a number of preheat temperature combinations to determine the most appropriate preheat temperature combination prior to applying this treatment to single quartz grains. Coarse grains of quartz were rare in UP layers, so dose recovery tests were conducted on quartz from MP layers, samples UK OSL 1 and UK OSL 4. Dose recovery tests were conducted using the SAR protocol. Each aliquot was bleached in sunlight and given a laboratory dose of ~86 Gy. Three different preheat temperature combinations were tested, including the following: 1) preheat for \( L_N \) or \( L_X \) at 180°C for 10 s, and preheat for \( T_N \) or \( T_X \) at 180°C for 5 s, 2) preheat for \( L_N \) or \( L_X \) at 240°C for 10 s, and preheat for \( T_N \) or \( T_X \) at 160°C for 5 s, and 3) preheat for \( L_N \) or \( L_X \) at 260°C for 10 s, and preheat for \( T_N \) or \( T_X \) at 160°C for 5 s.
Figure 6.3: The location of samples UK OSL 7–13 in the stratigraphic sequence at Ust-Karakol-1 Trench 2.
Four aliquots were measured for each preheat temperature combination and sample. Figure 6.4 displays the weighted mean measured dose values obtained for each of these preheat temperature combinations, using the CAM. The two samples (UK OSL 1 and UK OSL 4) behaved differently in response to the different preheat temperature combinations: UK OSL 4 recovered the dose best for combination 1 (see above), while UK OSL 1 appeared saturated for combination 1 and, therefore, a weighted mean measured dose value could not be obtained for this preheat temperature combination. For UK OSL 4, the weighted mean measured doses for combinations 2 and 3 underestimated the given dose. In contrast, UK OSL 1 showed a lot of scatter between aliquots for combinations 2 and 3, resulting in weighted mean measured dose values that were imprecise, but consistent with the given dose.

Due to variability in the behaviours of quartz grains between samples, the limited amount of sample available for dose recovery tests of single quartz grains and measurement of the natural OSL signal, and the weak sensitivity of the OSL signal, it was decided to attempt to use pIR-IRSL dating of single potassium-feldspar grains instead of OSL dating of single quartz grains. Potassium-feldspar pIR-IRSL signals are often

![Figure 6.4: Dose recovery test for single aliquots of quartz grains for two samples from Ust-Karakol-1 Trench 2: UK OSL 1 (blue) and UK OSL 4 (red). The three different preheat temperature combinations, including 1) preheat for LN or LX at 180°C for 10 s, and preheat for TN or TX at 180°C for 5 s, 2) preheat for LN or LX at 240°C for 10 s, and preheat for TN or TX at 160°C for 5 s, and 3) preheat for LN or LX at 260°C for 10 s, and preheat for TN or TX at 160°C for 5 s, are represented on the x-axis. The weighted mean measured dose is represented on the y-axis, and the blue dotted line represents the given dose.](image-url)
brighter than quartz OSL signals (Li et al., 2007; Li & Li, 2011), so it was hopeful that a larger number of grains would be yielding pIR-IRSL signals suitable for $D_e$ determination.

6.5.2. Post-IR IRSL potassium-feldspar dose recovery tests

Dose recovery tests were conducted on single potassium-feldspar grains. Prior to these tests, sample preparation had not been completed on the samples collected from the UP samples, so potassium-feldspar grains from MP samples (UK OSL 2, UK OSL 4 and UK OSL 5) were used instead. These samples had been prepared earlier. Dose recovery tests were conducted using the MET-pIRIR protocol. Grains were bleached in a solar simulator for 4 hours, and given a laboratory dose of ~200 Gy. Two preheat temperature combinations were tested: 1) preheat for $L_N$ or $L_X$ and $T_N$ or $T_X$ at 320°C for 60 s, and 2) preheat for $L_N$ or $L_X$ and $T_N$ or $T_X$ at 300°C for 10 s. Single grains were rejected following the rejection criteria presented in Section 3.5.7.1. Figure 6.5 displays the measured to given dose ratios for the application of combination 1 (see above) to UK OSL 4 and UK OSL 5, and combination 2 to UK OSL 4 and UK OSL 2. The measured to given dose ratios for both samples for which combination 1 was applied shows an underestimation of the dose of ~15–20%. The measured to given dose ratio for UK OSL 4 when combination 2 was applied (0.96 ± 0.03) is consistent with unity at 2$\sigma$ for stimulation temperatures 200 and 275°C. The measured to given dose ratio for UK OSL 2 when combination 2 was applied (0.90 ± 0.02), however, show an underestimation of ~10% at 275°C, but appears to recover the dose better at 200°C. Further dose recovery tests are required to determine the suitability of preheat temperature combinations for the UP layers at Ust-Karakol-1 Trench 2. Due to small amounts of coarse potassium-feldspar grains in all samples, this was not possible. Due to these limitations, it was decided to attempt polymineral fine-grains as a last resort.

6.5.3. Post-IR IRSL polymineral fine-grain dose recovery tests

Dose recovery tests were conducted on polymineral fine-grain aliquots for one sample from Ust-Karakol-1 Trench 2 (UK OSL 13). The MET-pIRIR protocol that was used for single potassium-feldspar grains was also attempted for polymineral fine-grains, because it is the pIR-IRSL signal emitted by fine-grain feldspars that is of interest. Aliquots were bleached in a solar simulator for 4 hours, and given a laboratory dose of ~119 Gy. The preheat temperature combination of 300°C for 10 s that was suitable for single potassium-
Figure 6.5: Dose recovery tests on single potassium-feldspar grains of samples UK OSL 2, UK OSL 4 and UK OSL 5 using two preheat temperature combinations: 1) preheat for LN or Lx and TN or Tx at 320°C for 60 s, and 2) preheat for LN or Lx and TN or Tx at 300°C for 10 s. The measured to given dose ratios of accepted grains are displayed in a, c, e and g. The grey band represents the given dose. The weighted mean dose ratios of all grains at each IR stimulation temperature (50, 100, 150, 200 and 275°C) are shown as stimulation temperature curves in b, d, f and h. The blue dotted line represents the given dose.
feldspar grains from sample UK OSL 4 was applied to the polymineral fine-grain aliquots. This preheat temperature combination produced optical decay curves that were dim and dominated by an isothermal signal between 0–50 s of stimulation time (Figure 6.6).

To minimise this isothermal signal, the pIRIR protocol was used instead. The aim was to maximise the relative proportion of the pIR-IRSL signal compared to the isothermal signal. Two different stimulation temperature combinations were tested: 1) 100°C for the first step, and 275°C for the second step, and 2) 150°C for the first step, and 275°C for the second step. Prior to each stimulation, aliquots were preheated at 300°C for 100 s. The pIR-IRSL signal was brighter for combination 1 than combination 2 (Figure 6.7). It is well-known that there is still a residual component present. This was determined by measuring the pIRIR signal following the same approach as above for both stimulation temperature combinations, but without giving aliquots a known dose prior to measurement. Dose-response curves were constructed by giving two regenerative doses of ~12 and 36 Gy. The measured residual signals were 13.6 ± 0.7 Gy for combination 1 and

![Figure 6.6: Optical decay curves for two polymineral fine-grain aliquots from UK OSL 13 obtained at a stimulation temperature of 275°C using the MET-pIRIR protocol, and a preheat temperature combination of 300°C for 10 s prior to measurement of L_X and T_X. The x-axis displays the stimulation time in seconds, and the y-axis displays the measured pIR-IRSL signal as counts per channel recorded after each second. The first 50 channels were dark counts. The inset graph shows the same information, but with the signals normalised to the first second of IR stimulation.](image-url)
19.5 ± 1.3 Gy for combination 2. These were subtracted from the measured doses, giving the residual-corrected weighted mean measured doses presented in Figure 6.8. Each of the residual-corrected weighted mean measured doses for both stimulation temperature combinations were in agreement with unity at 1σ. Since stimulation temperature combination 1 produced a brighter pIR-IRSL signal, this was used for \( D_e \) determination for all samples.

### 6.5.4. Post-IR IRSL \( D_e \) measurements

Post-IR IRSL dating of polymineral fine-grain aliquots was conducted for seven samples from Ust-Karakol-1 Trench 2. These seven samples correspond to layer 11 (UK OSL 7), layer 10 (UK OSL 8), layer 9 (UK OSL 9), layer 7 (UK OSL 10), layer 6 (UK OSL 11), layer 5 (UK OSL 12) and layer 4 (UK OSL 13) (Figure 6.3). Three aliquots (each containing many thousands of grains) were measured for each sample, and the residual dose of 13.6 ± 0.7 Gy determined during dose recovery tests was subtracted from

![Optical decay curves for two polymineral fine-grain aliquots from UK OSL 13 obtained at a stimulation temperature of 275°C using the pIRIR protocol. The pIR-IRSL signal for one aliquot (blue) was stimulated at 100°C prior to stimulation at 275°C, while the pIR-IRSL signal for the other aliquot (red) was stimulated at 150°C prior to stimulation at 275°C. The x-axis displays the stimulation time in seconds, and the y-axis displays the measured pIR-IRSL signal as counts per channel recorded after each second. The first 50 channels were dark counts. The inset graph shows the same information, but with the signals normalised to the first second of IR stimulation.](image)

**Figure 6.7:** Optical decay curves for two polymineral fine-grain aliquots from UK OSL 13 obtained at a stimulation temperature of 275°C using the pIRIR protocol. The pIR-IRSL signal for one aliquot (blue) was stimulated at 100°C prior to stimulation at 275°C, while the pIR-IRSL signal for the other aliquot (red) was stimulated at 150°C prior to stimulation at 275°C. The x-axis displays the stimulation time in seconds, and the y-axis displays the measured pIR-IRSL signal as counts per channel recorded after each second. The first 50 channels were dark counts. The inset graph shows the same information, but with the signals normalised to the first second of IR stimulation.
the $D_e$ values, giving residual-corrected $D_e$ values presented for each aliquot in Figure 6.9. The $D_e$ values for each sample were determined using the CAM. The final $D_e$ values, together with their OD values are provided in Table 6.2. Two samples displayed some OD despite the large averaging effect of fine-grain aliquots. This should be kept in mind during the interpretation of the ages and comparisons to existing chronologies.

6.5.5. Partial bleaching test

Fine-grains are more susceptible to partial bleaching than coarse-grains due to a number of possible factors, including being transported at a faster rate through fluvial systems, and being transported as aggregates that hinder solar bleaching (Rittenour, 2008). A partial bleaching test was, therefore, conducted to determine whether the fine-grains at Ust-Karakol-1 Trench 2 were partially bleached, leading to potential overestimates of $D_e$ values and, thus, ages. UK OSL 14 was collected from the surface of the hillside near Ust-Karakol-1 Trench 2. The upper 5 cm of this sample was removed due to an abundance of plant roots. The sample comprised fine-grains of 5–15 cm depth, or an average depth of ~10 cm. The $D_e$ value for this sample was determined following the same procedure as
<table>
<thead>
<tr>
<th>Sample name</th>
<th>$D_e$ value (Gy)</th>
<th>OD value (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>UK OSL 13</td>
<td>98.5 ± 2.1</td>
<td>0.0 ± 0.0</td>
</tr>
<tr>
<td>UK OSL 12</td>
<td>104.0 ± 2.3</td>
<td>0.0 ± 0.0</td>
</tr>
<tr>
<td>UK OSL 11</td>
<td>119.1 ± 2.8</td>
<td>0.0 ± 0.0</td>
</tr>
<tr>
<td>UK OSL 10</td>
<td>133.1 ± 13.0</td>
<td>16.4 ± 7.1</td>
</tr>
<tr>
<td>UK OSL 9</td>
<td>129.8 ± 2.7</td>
<td>0.0 ± 0.0</td>
</tr>
<tr>
<td>UK OSL 8</td>
<td>136.3 ± 7.0</td>
<td>7.9 ± 4.1</td>
</tr>
<tr>
<td>UK OSL 7</td>
<td>212.9 ± 6.0</td>
<td>0.0 ± 0.0</td>
</tr>
</tbody>
</table>

Table 6.2: $D_e$ values and OD values for samples UK OSL 7–13.
UK OSL 7–13, but for just two aliquots. The residual dose of $13.6 \pm 0.7$ Gy determined during dose recovery tests was subtracted from the $D_e$ values, giving a residual-corrected $D_e$ value for this sample of $34.7 \pm 1.4$ Gy. The $D_e$ values for each sample were determined using the CAM. The sample displayed some OD ($3 \pm 5\%$) despite the large averaging effect of fine-grain aliquots. These results will be discussed in Section 6.5.9.

6.5.6. Optical decay curves

Optical decay curves were produced for all measured aliquots. Figure 6.10 displays optical decay curves measured after a test dose of $\sim60$ Gy for three polymineral fine-grain aliquots from UK OSL 7. These are highly reproducible, and are representative of the optical decay curves for each aliquot from all seven samples. The optical decay curves display an isothermal signal for the first 50 seconds prior to IR stimulation.

6.5.7. Dose-response curves

Dose-response curves were constructed for each measured aliquot using regenerative doses of $\sim60$, 120, 240 and 360 Gy. Figure 6.11 displays dose-response curves for three polymineral fine-grain aliquots from UK OSL 7. The dose-response curves for each aliquot from each sample were highly reproducible due to the large averaging effect of fine-grains.
6.5.8. Dosimetry measurements

Total dose rates were calculated for each of the seven samples from Ust-Karakol-1 Trench 2. Alpha dose rates were measured directly using the alpha counter, beta dose rates were measured directly using the GM-25-5 beta counter, the field gamma dose rate was measured directly using in situ gamma spectrometry, and the cosmic-ray dose rate was calculated using the equation provided in Prescott and Hutton (1988). Beta, gamma and cosmic-ray dose rates were corrected for water content. The individual dose rate values for each sample are provided in Table 6.3 together with their measured water contents and total environmental dose rates.

The water contents for samples ranged between $16 \pm 4\%$ and $24 \pm 6\%$. The alpha dose rates range between $0.74 \pm 0.02$ and $0.91 \pm 0.02$ Gy/ka, the beta dose rates range between $1.55 \pm 0.10$ and $1.79 \pm 0.10$ Gy/ka, and the gamma dose rates range between $0.83 \pm 0.05$ and $1.03 \pm 0.05$ Gy/ka. A comparison between the field gamma dose rate and a laboratory gamma dose rate is provided in Table 6.4. The field gamma dose rates are both higher and lower than their respective laboratory gamma dose rates, suggesting that the differences

![Figure 6.10: Optical decay curves for three polymineral fine-grain aliquots from UK OSL 7 obtained at a stimulation temperature of 275°C using the pIRIR protocol. The x-axis displays the stimulation time in seconds, and the y-axis displays the measured pIR-IRSL signal as counts per channel recorded after each second. The first 50 channels were dark counts. The inset graph shows the same information, but with the signals normalised to the first second of IR stimulation.](image-url)
are not systematic due to the methods used, but perhaps reflect real differences in sediment composition within the ~300 mm gamma sphere of influence.

6.5.9. Final ages

The total environmental dose rates are presented together with the $D_s$ values and final pIR-IRSL ages for each sample in Table 6.5. Seven ages were obtained for Ust-Karakol-1 Trench 2. Ages of 64.2 ± 3.1 (UK OSL 7), 40.7 ± 2.7 (UK OSL 8) and 35.8 ± 1.6 ka (UK OSL 9) were obtained from samples from layers 11, 10 and 9, respectively. These three ages are in stratigraphic order. Layer 11 is the earliest layer associated with the EUP at Ust-Karakol-1 Trench 2. The age obtained for layer 11 is not consistent with the palaeomagnetic excursion age of 42 – 44 ka for this layer (see Table 6.1). The ages obtained for layer 10 and 9 are consistent with the $^{14}$C ages obtained for the these two layers. The age for layer 9 is inconsistent with the RTL age of 50 ± 12 ka, albeit within 2σ of this age. The difference in age between layers 11 and 10 may indicate a significant hiatus in sediment deposition. Layer 8 is said to be the latest layer associated with the EUP. This layer was too thin to sample, so the age obtained for layer 9 of 35.8 ± 1.6 ka is the best indication for the latest EUP occupation at Ust-Karakol-1 Trench 2.

Ages of 37.6 ± 4.0 (UK OSL 10), 34.2 ± 1.7 (UK OSL 11), 26.8 ± 1.1 (UK OSL 12) and 27.6 ± 1.2 ka (UK OSL 13) were obtained from samples from layers 7, 6, 5 and 4, respectively. These ages are also in stratigraphic order. Layers 7 and 6 contain no artefacts,

Figure 6.11: Dose-response curve for one polymineral fine-grain aliquot from UK OSL 7. The $x$-axis displays the dose (Gy), and the $y$-axis displays the sensitivity-corrected pIR-IRSL signal.
<table>
<thead>
<tr>
<th>Sample name</th>
<th>Water content (%)</th>
<th>Alpha dose rate (Gy/ka)</th>
<th>Beta dose rate (Gy/ka)</th>
<th>Gamma dose rate (Gy/ka)</th>
<th>Cosmic-ray dose rate (Gy/ka)</th>
<th>Total dose rate (Gy/ka)</th>
</tr>
</thead>
<tbody>
<tr>
<td>UK OSL 13</td>
<td>16 ± 4</td>
<td>0.79 ± 0.02</td>
<td>1.62 ± 0.09</td>
<td>1.00 ± 0.05</td>
<td>0.17 ± 0.03</td>
<td>3.58 ± 0.11</td>
</tr>
<tr>
<td>UK OSL 12</td>
<td>16 ± 4</td>
<td>0.91 ± 0.02</td>
<td>1.79 ± 0.10</td>
<td>1.03 ± 0.05</td>
<td>0.16 ± 0.02</td>
<td>3.89 ± 0.12</td>
</tr>
<tr>
<td>UK OSL 11</td>
<td>25 ± 6</td>
<td>0.77 ± 0.02</td>
<td>1.58 ± 0.11</td>
<td>0.99 ± 0.07</td>
<td>0.14 ± 0.02</td>
<td>3.48 ± 0.13</td>
</tr>
<tr>
<td>UK OSL 10</td>
<td>22 ± 5</td>
<td>0.80 ± 0.02</td>
<td>1.65 ± 0.11</td>
<td>0.95 ± 0.06</td>
<td>0.14 ± 0.02</td>
<td>3.54 ± 0.12</td>
</tr>
<tr>
<td>UK OSL 9</td>
<td>24 ± 6</td>
<td>0.84 ± 0.02</td>
<td>1.67 ± 0.11</td>
<td>0.96 ± 0.06</td>
<td>0.16 ± 0.02</td>
<td>3.63 ± 0.13</td>
</tr>
<tr>
<td>UK OSL 8</td>
<td>24 ± 6</td>
<td>0.74 ± 0.02</td>
<td>1.55 ± 0.10</td>
<td>0.91 ± 0.06</td>
<td>0.15 ± 0.02</td>
<td>3.35 ± 0.12</td>
</tr>
<tr>
<td>UK OSL 7</td>
<td>23 ± 6</td>
<td>0.80 ± 0.02</td>
<td>1.55 ± 0.10</td>
<td>0.83 ± 0.05</td>
<td>0.15 ± 0.02</td>
<td>3.31 ± 0.12</td>
</tr>
</tbody>
</table>

**Table 6.3**: Total environmental dose rates for UK OSL 7–13 using TSAC for the alpha dose rate, GM-25-5 beta counting for the beta dose rate, *in situ* gamma spectrometry for the gamma dose rate, and the cosmic-ray dose rate equation for the cosmic-ray dose rate, corrected for water contents as measured in this project.
<table>
<thead>
<tr>
<th>Sample name</th>
<th>Field gamma dose rate (Gy/ka)</th>
<th>Laboratory gamma dose rate (Gy/ka)</th>
<th>Ratio (Field/Laboratory)</th>
</tr>
</thead>
<tbody>
<tr>
<td>UK OSL 13</td>
<td>1.00 ± 0.05</td>
<td>0.84 ± 0.05</td>
<td>1.12 ± 0.10</td>
</tr>
<tr>
<td>UK OSL 12</td>
<td>1.03 ± 0.05</td>
<td>0.99 ± 0.06</td>
<td>1.04 ± 0.09</td>
</tr>
<tr>
<td>UK OSL 11</td>
<td>0.99 ± 0.07</td>
<td>0.87 ± 0.07</td>
<td>1.14 ± 0.12</td>
</tr>
<tr>
<td>UK OSL 10</td>
<td>0.95 ± 0.06</td>
<td>0.87 ± 0.06</td>
<td>1.09 ± 0.10</td>
</tr>
<tr>
<td>UK OSL 9</td>
<td>0.96 ± 0.06</td>
<td>0.90 ± 0.07</td>
<td>1.07 ± 0.10</td>
</tr>
<tr>
<td>UK OSL 8</td>
<td>0.91 ± 0.06</td>
<td>0.82 ± 0.06</td>
<td>1.12 ± 0.11</td>
</tr>
<tr>
<td>UK OSL 7</td>
<td>0.83 ± 0.05</td>
<td>0.87 ± 0.07</td>
<td>0.95 ± 0.09</td>
</tr>
</tbody>
</table>

Table 6.4: Comparison between the field gamma dose rate obtained using *in situ* gamma spectrometry and the laboratory gamma dose rate obtained using TSAC and GM-25-5 beta counting for UK OSL 7–13, and the ratio of these two measurements with the field gamma dose rate as the numerator and the laboratory gamma dose rate as the denominator.
<table>
<thead>
<tr>
<th>Sample name</th>
<th>Total dose rate (Gy/ka)</th>
<th>$D_e$ value (Gy)</th>
<th>pIR-IRSL age (ka)</th>
</tr>
</thead>
<tbody>
<tr>
<td>UK OSL 13 (Layer 4)</td>
<td>3.58 ± 0.11</td>
<td>98.5 ± 2.1</td>
<td>27.6 ± 1.2</td>
</tr>
<tr>
<td>UK OSL 12 (Layer 5)</td>
<td>3.89 ± 0.12</td>
<td>104.0 ± 2.3</td>
<td>26.8 ± 1.1</td>
</tr>
<tr>
<td>UK OSL 11 (Layer 6)</td>
<td>3.48 ± 0.13</td>
<td>119.1 ± 2.8</td>
<td>34.2 ± 1.7</td>
</tr>
<tr>
<td>UK OSL 10 (Layer 7)</td>
<td>3.54 ± 0.12</td>
<td>133.1 ± 13.0</td>
<td>37.6 ± 4.0</td>
</tr>
<tr>
<td>UK OSL 9 (Layer 9)</td>
<td>3.63 ± 0.13</td>
<td>129.8 ± 2.7</td>
<td>35.8 ± 1.6</td>
</tr>
<tr>
<td>UK OSL 8 (Layer 10)</td>
<td>3.35 ± 0.12</td>
<td>136.3 ± 7.0</td>
<td>40.7 ± 2.7</td>
</tr>
<tr>
<td>UK OSL 7 (Layer 11)</td>
<td>3.31 ± 0.12</td>
<td>212.9 ± 6.0</td>
<td>64.2 ± 3.1</td>
</tr>
</tbody>
</table>

Table 6.5: Total environmental dose rates, $D_e$ values and final pIR-IRSL ages for UK OSL 7–13.
and no chronologies have been reported for these layer with which these results can be compared. Layer 5 is said to be associated with the MUP. The age obtained for this layer is younger than the $^{14}$C ages for this layer, three of which were produced from charcoal, and one of which was produced from humates, but is consistent with the palaeomagnetic excursion age for this layer. Layer 4 is said to be associated with the FUP. There are no chronologies reported for this layer with which these results can be compared; however, this age is younger than the $^{14}$C ages for the overlying layers 3 and 2.

The partial bleaching test conducted in this project (see Section 6.5.5) produced a residual-corrected $D_e$ value for UK OSL 14 of $34.7 \pm 1.4$ Gy from $\sim 10$ cm beneath the surface of the hillslope near Ust-Karakol-1 Trench 2. This suggests that partial bleaching occurred at Ust-Karakol-1 Trench 2, causing ages to be overestimated. The agreement of pIR-IRSL ages with $^{14}$C ages for layers 10 and 9, and the pIR-IRSL ages that are younger than $^{14}$C ages for layers 5 and 4, however, suggest that partial bleaching did not occur at Ust-Karakol-1 Trench 2. It can be concluded that the average depth of the sample of $\sim 10$ cm due to the abundance of plant roots is not ideal for this test. The sediments at $\sim 10$ cm depth may, in fact, have been fully bleached prior to deposition, and have just received an environmental dose of $\sim 35$ Gy since that time. The $D_e$ value obtained from this test was, therefore, not subtracted from the $D_e$ values obtained for UK OSL 7–13.

There is significant evidence for sediment mixing at Ust-Karakol-1 Trench 2. Slavinsky (2007) and Zwyns (2012) observed sediment mixing caused by bioturbation and frost cracks. Mole-rat burrows can be observed macroscopically. This may explain the inconsistencies between the $^{14}$C, palaeomagnetic excursion and RTL ages for this site. This sediment mixing may also have affected the pIR-IRSL ages obtained in this project. Two samples (UK OSL 8 and UK OSL 10) displayed some OD, indicating potential mixing. The age of $64.2 \pm 3.1$ ka obtained for layer 11 is older than expected for the EUP, and layer 11 is associated with some Levallois elements deriving from MP layers. This may indicate that the age obtained for layer 11 is an overestimate due to the input of sediments from an older MP layer. Future studies should persevere with single-grain optical dating at Ust-Karakol-1 Trench 2, using the dose recovery tests for quartz (see Section 6.5.1) and potassium-feldspar (see Section 6.5.2) presented in this chapter as a starting point. This approach may be able to help untangle the chronologies at Ust-Karakol-1 Trench 2.
6.6. Synopsis

In this chapter, the UP site of Ust-Karakol-1 Trench 2 has been discussed in terms of its sedimentological context, UP context, and existing chronologies. The results of optical dating conducted in this project were presented, including dose recovery tests for quartz, potassium-feldspar and polymineral fine-grains, pIR-IRSL $D_e$ measurements, dosimetry measurements, and final ages. These ages were then compared to the existing chronologies. The implications for the ages presented in this chapter will be discussed in the following chapter.
Chapter 7: Discussion
7.1. Introduction

In this project, optical dating was conducted on samples collected from UP layers at Denisova Cave, Anui-2, Anui-3, and Ust-Karakol-1 Trench 2. In this chapter, the following will be discussed: the reliability of the optical dating methods used in this project, the optical chronologies for the four project sites, the emergence of the UP in the Altai Mountains, the possible makers of the UP, and directions future studies may take. Conclusions will be made in relation to the aims stated in Chapter 1.

7.2. Optical dating

In this project, systematic optical dating was conducted on UP assemblages from Denisova Cave, Anui-2, Anui-3, and Ust-Karakol-1 Trench 2. Optical chronologies were tested against existing independent $^{14}$C ages as a means of determining whether the optical dating chronologies were reliable and accurate. For the site of Anui-2, the two reliable OSL ages obtained for sub-layers 13.2 and 13.1 were in close agreement with the $^{14}$C ages for these sub-layers. The preservation of ash lenses at Anui-2 indicates that there has been minimal sediment mixing (Derevianko et al., 2003); this is supported by the single-grain $D_e$ distributions for these sub-layers (see Section 5.5.5). This site should, therefore, be considered a good indicator that the OSL ages obtained in this project were reliable and accurate.

Operator variability in single-grain analysis was also tested for to ascertain reproducibility of chronologies using OSL dating methods. Two operators (KO and ZJ) conducted single-grain analyses for the nine samples from Denisova Cave. Eight of the nine $D_e$ values obtained were in agreement at 1$\sigma$ (Table 4.3), suggesting that chronologies were reproducible using OSL dating methods.

Dose recovery tests were conducted for samples from Ust-Karakol-1 Trench 2. These tests included OSL dating of quartz grains, pIR-IRSL dating of potassium-feldspar grains, and pIR-IRSL dating of polymineral fine-grains. The results of these tests can be used as a starting point for further studies to conduct single-grain optical dating of sediments at Ust-Karakol-1 Trench 2.
7.3. Optical chronologies

In this project, 24 optical ages were produced for four UP sites in the Altai Mountains. These ages, their associated layers or sub-layers, and their technological assignment, are presented in Figure 7.1. The earliest ages for the IUP come from sub-layer 11.4 in the Main chamber of Denisova Cave. The oldest of these is around 62 ka. In the Main chamber, the IUP continues until at least around 37 ka (sub-layer 11.2). In the East chamber of Denisova Cave, an age of around 43 ka was obtained for sub-layer 11.1, consistent with layer 11 in the Main chamber. Sub-layer 11.2 yielded an age of around 29 ka, but this is in contradiction to the age obtained by the operator ZJ of around 80 ka; therefore, this layer either represents the latest IUP occupation dated in this project, or the earliest.

The earliest age for the EUP comes from layer 11 at Ust-Karakol-1 Trench 2. This age was obtained using polymineral fine-grains. Since this age is so much older than other EUP ages, it should be treated as a maximum age for the EUP. There are three possible scenarios to explain this age. The first scenario is that sediment mixing has occurred between layer 11 and underlying MP layers, causing an overestimation of the $D_e$ value. Since single-grain optical dating was not conducted, this mixing could not be identified. This scenario reinforces the assignment of layer 11 to the EUP by explaining the presence of Levallois elements in this layer through sediment mixing (Zwyns, 2012). The second scenario is that sediment mixing occurred between layer 11 and overlying UP layers, causing an underestimation of the $D_e$ value. In this scenario, layer 11 belongs to the MP, and the presence of laminar elements in the assemblage is due to the downward vertical movement of artefacts from UP layers. This is not supported by any archaeological interpretations. The third scenario is that the age is not influenced by sediment mixing, and the presence of Levallois or laminar elements in the assemblage is not due to sediment mixing. In this scenario, layer 11 should be assigned to the IUP due to the presence of both Levallois and laminar technological elements. This is supported by Slavinsky’s (2007), Zwyns’ (2012) and Rybin’s (2014) assignment of the corresponding occupational horizon 5.4 at Ust-Karakol-1 Trench 1 to the IUP. In the third scenario, the age produced is consistent with the earliest ages obtained for the IUP in the Main chamber of Denisova Cave. Ages of around 35 and 40 ka were obtained for layers 10 and 9, respectively, at Ust-Karakol-1 Trench 2, and these ages are in agreement with $^{14}$C ages for these layers. Despite this, these ages should be treated with caution due to sediment mixing observed at
Figure 7.1: The 24 optical ages produced in this project for samples from the Main (DCM) and East (DCE) chambers of Denisova Cave, Anui-2, Anui-3, and Ust-Karakol-1 Trench 2. Age is displayed on the x-axis, and layers or sub-layers are displayed on the y-axis; ages are presented in stratigraphic order from top to bottom for each site. The coloured circles indicate the technological assignment of a layer or sub-layer, as displayed in the legend.
The earliest reliable ages for the EUP come from sub-layer 13.2 and 13.1 at Anui-2. Ages of around 32 ka were obtained for these sub-layers, in agreement with the $^{14}$C ages for these layers. Ages of between 29–32 ka were obtained from sub-layers 9.2 and 9.1 in the Main chamber of Denisova Cave, and layer 12 from Anui-3. These five ages were single-grain OSL ages. It can be said then that the EUP emerged in the Altai Mountains at least around 32 ka, and possibly quite a bit earlier.

Derevianko and Shunkov (2002) compared the stratigraphic sequences of Anui-3 and Ust-Karakol-1 Trench 2, comparing layers 12–10 at Anui-3 to layers 11–9 at Ust-Karakol-1 Trench 2. The ages of 29.1 ± 2.0, 25.1 ± 1.8 and 23.7 ± 1.5 ka obtained for layers 12, 11 and 10 at Anui-3, respectively, are in disagreement with the ages of 64.2 ± 3.1, 40.7 ± 2.7 and 35.8 ± 2.7 ka obtained for layers 11, 10 and 9 at Ust-Karakol-1 Trench 2, respectively. This suggests that the stratigraphic sequences at these two sites are not comparable. The original Anui-3 trench for which Derevianko and Shunkov (2002) compared to Ust-Karakol-1 Trench 2 was different to that sampled in this project. The original trench was located further up-hill, and layers were a lot thicker and easier to distinguish. Due to the thinner layers and possible sediment mixing at the trench sampled in this project, it is uncertain if the ages produced for Anui-3 are true or not.

7.4. Emergence of the UP

The optical ages obtained in this project suggest, for the most part, that the IUP and EUP occurred in succession, following Zwyns’ (2012) chrono-cultural model. This is represented in Figure 7.2. Ages that were not assigned to either the IUP or EUP were excluded. The ages obtained for layer 11 at Ust-Karakol-1 Trench 2 and sub-layer 11.2 in the East chamber of Denisova Cave were excluded due to the issues regarding their interpretation (see Section 7.3). The age obtained for layer 11 at Anui-2 was also excluded because it is imprecise and inconsistent with the stratigraphic sequence. The earliest age obtained for the IUP in this project is around 62 ka for sub-layer 11.4 in the Main chamber of Denisova Cave. The EUP appears to have succeeded the IUP around 35 ka, and continued until around 25 ka based on reliable $^{14}$C ages from Anui-2. The only evidence that the IUP and EUP were contemporaneous comes from ages that are considered to be unreliable.
Figure 7.2: The 13 reliable optical ages produced in this project for samples from the Main (DCM) and East (DCE) chambers of Denisova Cave, Anui-2, Anui-3, and Ust-Karakol-1 Trench 2 assigned to the IUP or EUP. Age is displayed on the x-axis, and layers or sub-layers are displayed on the y-axis; ages are presented in stratigraphic order from top to bottom for each site. The coloured circles indicate the technological assignment of a layer or sub-layer, as displayed in the legend, and the open circles indicate those ages omitted on the basis of not being assigned to the IUP or EUP, or not being reliable.
7.5. Possible makers of the UP

Denisovans, Neanderthals and modern humans are all known to have been present in, or near, the Altai Mountains during the Late Pleistocene. It is known that modern humans were present in western Siberia around 45 ka (Fu et al., 2014), and modern humans are attributed to lithic assemblages similar to the EUP in Europe and the Middle East. If the EUP emerged 35 ka, then it seems reasonable to assume that modern humans were responsible for the EUP, as well as the following MUP and FUP. There is no evidence, as yet, for modern humans being present in, or near, the Altai Mountains around 62 ka, making it more difficult to determine the makers of the IUP.

Denisova 3, a Denisovan distal manual phalanx, was found in sub-layer 11.2 in the East chamber of Denisova Cave (Krause et al., 2010; Reich et al., 2010; Meyer et al., 2012) in association with IUP artefacts. If this Denisovan fossil remain is determined to be in its original context, the age produced for this layer by operator KO of around 29 ka would indicate a chronological overlap between Denisovans and modern humans in the Altai Mountains, assuming modern humans were responsible for the EUP. The Altai Mountains could, therefore, be the location of interbreeding between Denisovans and modern humans (Huerta-Sanchez et al., 2014; Qin & Stoneking, 2015). The age produced for this layer by operator ZJ of around 80 ka appears more consistent with the stratigraphic sequence. There is no evidence for modern humans being present at Denisova Cave around 80 ka; therefore, it seems more logical to associate the artefacts discovered in sub-layer 11.2 with Denisovans.

7.6. Future studies

To obtain reliable ages for the stratigraphic sequence at Ust-Karakol-1 Trench 2, single-grain optical dating should be conducted. In 2016, Professor Bert Roberts, Professor Zenobia Jacobs and the author of this thesis collected two sediment samples from a new trench (Trench 3) at Ust-Karakol-1. UK16 OSL 1 was collected from layer 11, and UK16 OSL 2 was collected from layer 8. Two sample tubes were collected for each sample in the hope that a larger number of grains would be yielding OSL or pIR-IRSL signals suitable for $D_e$ determination. Some progress was made in dose recovery tests in this project, so building upon these tests, ages may be able to be produced for these layers. Ages for these
two layers will bracket the EUP occupation at Ust–Karakol-1, and help to unravel the chronologies for this site.

In regard to the thinner layers and possible sediment mixing at the Anui-3 trench sampled in this project, one layer from the original Anui-3 trench should be sampled to determine if the ages obtained in this project are true or not.

Future studies should construct chronologies for the MP at Denisova Cave, Anui-3 and Ust-Karakol-1 Trench 2 to determine if there was a hiatus between the MP and UP in the Altai Mountains. If there was found to be a hiatus, this would suggest that the MP did not evolve into the UP, as suggested by the local transition model (Derevianko, 2011), but instead lend further support to Zwyns’ (2012) chrono-cultural model, which posits that the UP is a result of modern human migrations.

7.7. Conclusions

This project had three aims, as follows:

- to construct chronologies for four UP sites in the Altai Mountains using optical dating techniques;
- to compare these chronologies to existing $^{14}$C, palaeomagnetic excursion and RTL ages; and
- to contribute to understandings of the emergence of the UP as a result of either a local transition from MP to UP, or migrations of modern human populations into the Altai Mountains.

The first of these aims was completed successfully. Twenty-four optical ages were obtained for four UP sites in the Altai Mountains: Denisova Cave, Anui-2, Anui-3 and Ust-Karakol-1 Trench 2.

These 24 optical ages were then compared to existing chronologies for these sites, including $^{14}$C, palaeomagnetic excursion and RTL ages, successfully completing the second of these aims.

This project also contributed to understandings of the emergence of the UP. It has been suggested that the IUP and EUP were not contemporaneous, that the IUP emerged at least around 62 ka, and that the EUP emerged around 35 ka. These ages provide support for Zwyns’ (2012) chrono-cultural model.
References


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Appendix
A.1. Introduction

There is substantial evidence for interbreeding between Denisovans, Neanderthals and modern humans. This evidence comes from the genomes of Denisovans, Neanderthals and modern humans, as well as present-day humans. In the following sections, the evidence regarding the interbreeding between these three hominin groups will be discussed, including interbreeding between Denisovans and Neanderthals, Denisovans and modern humans, and Neanderthals and modern humans.

A.2. Denisovans – Neanderthals

Evidence for interbreeding between Denisovans and Neanderthals comes from the presence of Neanderthal admixture in the Denisovan genome (Prüfer et al., 2014). Prüfer et al. (2014) found that at least 0.5% of the Denisovan genome derives from Neanderthal admixture (Figure A.1), and that this admixture occurred with a Neanderthal population more closely related to Denisova 5, a proximal pedal phalanx belonging to a Neanderthal discovered in the Altai Mountains, than to other Neanderthals found in Eurasia. This suggests that interbreeding between Neanderthals and modern humans may have occurred in, or near, the Altai Mountains.

![Figure A.1: Late Pleistocene landscape of hominin interbreeding. Red arrows indicate admixture events, and the direction of gene flows, between Denisovans, Neanderthals, and modern human populations. The associated value is an estimate of the proportion of admixture present in the hominin genome. The dotted line indicates uncertainty as to the origins of Denisovan admixture in Eastern Eurasians and North Americans (after Prüfer et al., 2014; Qin & Stoneking, 2015; Kuhlwilm et al., 2016).](image-url)
A.3. Denisovans – modern humans

Evidence for interbreeding between Denisovans and modern humans comes from the presence of Denisovan admixture in present-day modern human populations (Reich et al., 2010; Reich et al., 2011; Skoglund & Jakobsson, 2011; Meyer et al., 2012; Huerta-Sanchez et al., 2014; Prüfer et al., 2014; Qin & Stoneking, 2015). It has been documented that Oceanian populations contain a significant amount of Denisovan admixture, with the most recent estimate being around 3.5% (Figure A.1; Reich et al., 2010; Meyer et al., 2012; Qin & Stoneking, 2015); however, these are not the only populations to contain Denisovan admixture. Many Eastern Eurasian and North American populations also display a small proportion of Denisovan admixture in their genomes, with the most recent estimate being around 0.15% (Figure A.1; Skoglund & Jakobsson, 2011; Prüfer et al., 2014; Qin & Stoneking, 2015). Denisovan admixture in these Eastern Eurasian and North American populations reflects the same admixture event as that in Australian, New Guinean and Mamanwan populations as it derives from the common ancestor of Australians, New Guineans and Mamanwans (Reich et al., 2011; Qin & Stoneking, 2015). Denisovan admixture in Oceanians other than Australians, New Guineans and Mamanwans occurred as recent admixture with New Guineans rather than the common ancestor of Australians, New Guineans and Mamanwans (Reich et al., 2011; Qin & Stoneking, 2015).

There are two possible scenarios to explain the above findings. The first scenario is that after Denisovan admixture occurred in the ancestral population of Australians, New Guineans and Mamanwans (Reich et al., 2011; Qin & Stoneking, 2015), but before these populations split, there would have been a back-migration from island Southeast Asia to mainland Asia, contributing Denisovan genes to the ancestral populations of Eastern Eurasians and North Americans (Figure A.2; Qin & Stoneking, 2015). Reich et al. (2011) and Cooper and Stringer (2013) support this, suggesting that Denisovan admixture occurred in island Southeast Asia. This scenario is also supported by the absence of Denisovan admixture in the Ust-Ishim modern human discovered in western Siberia dated to around 45 ka (Fu et al., 2014). If this scenario is correct, this means that Denisovans were distributed from Siberia to the tropics, giving them a wider ecological and geographical distribution than any other hominin group excluding, of course, modern humans (Reich et al., 2011); however, the genetic diversity of Denisovans was much less than that of present-day humans (Meyer et al., 2012; Prüfer et al., 2014; Sawyer et al.,
2015), suggesting that Denisovans had a smaller geographical range. This low genetic diversity could be explained by the Denisovans having a small population size distributing with limited time for genetic diversity to increase (Meyer et al., 2012). In addition to this, the nuclear DNA sequences of Denisovans are more diverse than those among Neanderthals, so if Neanderthals were able to distribute themselves across much of Eurasia, it seems plausible that Denisovans populated a wide geographical range (Reich et al., 2010; Prüfer et al., 2014; Sawyer et al., 2015). The divergence between the introgressing Denisovan genome and the genome of Denisova 3, a distal manual phalanx belonging to a Denisovan, occurred 276–403 ka, suggesting that the Denisovan population was larger, more diverse and more subdivided than Neanderthal populations; therefore, Denisovans may have populated a wide geographical range (Prüfer et al., 2014). The issue with this scenario is that there is no evidence, as yet, of a back-migration from Oceania to mainland Asia (Qin & Stoneking, 2015).

The second scenario is that the ancestral population of Eastern Eurasians, North Americans, Australians, New Guineans and Mamanwans interbred with Denisovans. Australian, New Guinean and Mamanwan populations split from Eastern Eurasian and

**Figure A.2:** Scenario 1 for interbreeding between Denisovans and modern humans, where AU = Australians, NG = New Guineans, MN = Mamanwans, EE = Eastern Eurasians, and NA = North Americans. This scenario follows two steps: 1) interbreeding between Australians, New Guineans and Mamanwans, and Denisovans occurred somewhere in island Southeast Asia, and 2) there was a back migration from island Southeast Asia to mainland Asia, contributing Denisovan genes to Eastern Eurasians and North Americans (after Qin & Stoneking, 2015).
North American populations, and subsequent modern human migrations into the Eastern Eurasian and North American populations diluted the Denisovan genes, explaining the lower proportion of Denisovan admixture in these populations compared to Australian, New Guinean and Mamanwan populations (Figure A.3; Qin & Stoneking, 2015). This scenario means that Denisovan admixture did not have to occur in island Southeast Asia as suggested by Reich et al. (2011) and Cooper and Stringer (2013); therefore, interbreeding may have occurred nearer to the Altai Mountains, supporting the narrow geographical range suggested by the low genetic diversity present in the Denisovan genome (Meyer et al., 2012; Sawyer et al., 2015). Genetic studies have found that adaptations to high altitude conditions present in Tibetans derives from interbreeding with archaic Denisovan-like populations: the Tibetan haplotype responsible for these adaptations is much closer to the Denisovan haplotype than other modern human haplotypes, and appears more divergent from other modern human haplotypes than the Denisovan haplotype (Huerta-Sanchez et al., 2014). These adaptations involve better physiological responses to low oxygen levels by limiting increases in blood haemoglobin levels, thereby reducing the risk of cardiac events (Huerta-Sanchez et al., 2014). This haplotype is absent in Oceanian populations.
despite these populations displaying evidence of a significant amount of Denisovan admixture (Reich et al., 2010; Reich et al., 2011; Meyer et al., 2012; Prüfer et al., 2014; Qin & Stoneking, 2015), but is present in some Han Chinese individuals (Huerta-Sanchez et al., 2014); therefore, Huerta-Sánchez et al. (2014) suggests that significant Denisovan admixture occurred nearer to Tibet to explain the presence of this haplotype in Tibetans and some Han Chinese individuals, and the absence of this haplotype in Oceanians. This suggests that interbreeding between Neanderthals and modern humans may have occurred in, or near, the Altai Mountains.

A.4. Neanderthals – modern humans

Like Denisovans, evidence for interbreeding between Neanderthals and modern humans comes from the presence of Neanderthal admixture in present-day modern human populations (Green et al., 2010; Reich et al., 2010; Prüfer et al., 2014). Neanderthal admixture is present in all non-African populations, with one recent estimate being 1.5–2.1% (Figure A.1; Prüfer et al., 2014); however, the proportion of Neanderthal admixture in non-African populations varies, with the proportion of Neanderthal admixture in Europeans being much less than in East Asians (Meyer et al., 2012; Wall et al., 2013; Prüfer et al., 2014; Vernot & Akey, 2014, 2015). This is due to a particular Neanderthal haplotype being more frequent in East Asians, leaving East Asians with, on average, 17.5% more introgressed Neanderthal sequence than Europeans (Vernot & Akey, 2015). This is consistent with there having been two pulses of Neanderthal introgression into East Asian populations, whereas there was only one in European populations, disproving the scenario suggested by Meyer et al. (2012) that European Neanderthal admixture was diluted by a modern human population absent of Neanderthal admixture (Wall et al., 2013; Vernot & Akey, 2014, 2015). These pulses of Neanderthal introgression may have occurred over a long period of time, and during that period, the ancestors of Europeans may have diverged from the ancestors of East Asians, leading to there being more Neanderthal introgression present in East Asians (Wall et al., 2013). It is uncertain whether this Neanderthal introgression occurred in the ancestral population of Europeans and East Asians, or if introgression into these two populations occurred independent of one another (Sankararaman et al., 2012). The Ust-Ishim modern human, dated to around 45 ka, contains $2.3 \pm 0.3\%$ Neanderthal admixture in its genome; therefore, the first pulse of
interbreeding between modern humans and Neanderthals had occurred prior to 45 ka, perhaps around 50–60 ka (Fu et al., 2014). Measurement of the extent of linkage disequilibrium in present-day Europeans suggests that this admixture occurred 47–65 ka (Sankararaman et al., 2012), in agreement with the findings of Fu et al. (2014). Despite this, a modern human male from Peștera cu Oase, Romania, dated to 37–42 ka contains ~7.3% Neanderthal admixture, and had a Neanderthal ancestor less than 200 years prior to the time that he was living, suggesting that some Neanderthal admixture may have occurred in modern human populations at a later time (Fu et al., 2015). The Peștera cu Oase individual belonged to a population that did not contribute much, or not at all, to later Europeans, so this is not reflected in genetic analyses of present-day humans (Sankararaman et al., 2012; Fu et al., 2014; Fu et al., 2015). The introgressing Neanderthal genome is more closely related to a Neanderthal from the Caucasus than to Denisova 5 or Neanderthals from Croatia; therefore, it is suggested that interbreeding between Neanderthals and modern humans may have occurred near the Caucasus at a time when Neanderthal populations had separated from one another, estimated to be 77–114 ka (Prüfer et al., 2014).

Further evidence for interbreeding between Neanderthals and modern humans comes from the presence of modern human admixture in the Neanderthal genome (Kuhlwilm et al., 2016). Modern human admixture of 0.1–2.1% has been discovered in the genome of Denisova 5 (Figure A.1; Kuhlwilm et al., 2016). This admixture occurred prior to the split between the ancestors of Denisova 5 and other Neanderthals 68–167 ka, and prior to the split between Africans and non-Africans around 200 ka (Gronau et al., 2011; Kuhlwilm et al., 2016), suggesting that perhaps this admixture occurred in Africa or, at least, near Africa.
References (appendix)


