# Recuperated optically stimulated luminescence dating of fine-grained quartz in Paleolithic site of Bonneval "La Jouanniere", Eure-et-Loir, France

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#### Abstract

Quartz extracted from samples of Paleolithic site Bonneval (France), using recuperated OSL signal, was dated by multiple-aliquot additive-dose protocol. Employing ages obtained ( $100 \sim 450$  ka), correlating with the marine oxygen isotope record (OIS) and European stratigraphy, we attempt to interpret the paleoclimate and paleoenvironmental for hominid.

Keywords: Paleolithic site, Recuperated OSL, paleoclimate and paleoenvironment.

#### Introduction

The site "La Jouanniere" is located by the city of Bonneval, between highway 142 and highroad 10, in Eure-et-loir, the centre of France.

Bonneval is situated in the upper Loir valley, connecting two geological areas: the lacustrine alluvium and the Cretaceous chalk of Perch region. Since Quaternary loess has been deposited on the surface of this kinetic area.

At the end of 19th century and beginning of 20th century, due to quarrying loess for building by local farmers, many flakes and handaxes cut by flint were discovered. And then Bonneval is presence known for of series bifaces (Moustériens and Acheuléens). Nowadays, many of them are preserved in Bonneval museum. In 2005, impelling by a project building industry sector in the south-west of the city, the site Bonneval, "La jouanniere" was discovered by archeological field survey before building.

Conducted by Mister J. Despriee, a pit of 6 meters depth was dug by respecting intermediate landings of a meter. Excavation was operated at three times between 2005 and 2006. 17 handaxes, 5 scrapers, 28 cores and 341 flakes were founded in the Loess-palaeosol stratigraphy within the profile and were studied by Nelly Connet.

# The Paleolithic industry in loess-palaeosol stratigraphy

Regarding paleolithic industries, in Paris Basin, north France, paleolithic sites within stratigraphic sequences are very rare. Discovered in these sites nearby "la jouannière" located at Saint-Firmin-des-Prés (Loir-et-Cher), around 40 km downstream in the Loir river valley, the cutting methods are more varied: Levallois, Discoïd, and pyramidal. Forwards, in the north of the Paris Basin, the Villers-Adam site in Vald'Oise and the Bettencourt site in Somme which has similar stratigraphies as "la Jouannière", however their Levallois industries are very different from "la Jouannière" (Desprie, 2005).

In excavation of Bonneval, archaeologist discovered four series paleolithic industry horizons filling in the loess-palaeosol stratigraphy: At the surface of the sit there are a few pieces among which one stripy removals core cutting. The first level is 170 cm depth, characterized by numerous Levallois cutting with Levallois points and Levallois flakes, some



Fig.1. Map showing location of sites discussed in the text.

scrapers, and many flat triangular or heart-shaped handaxes. Between 310 and 330 cm depth is the second archaeological level including two sectors, were excavated cutting masses which are made of numerous flakes. The flakes series obtained during the "chaîne opétratoire" are homogeneous. The frequent cutting method used is the unifacial or bifacial Discoïd Method.

Around 380 cm is the third level: two handaxes with red-brown patina and cortex residue were found in their original depositional situation. The fourth level is about 410 cm: a thick sole-shaped handaxe, with a greenish patina, frosty removals and shiny surfaces was founded with flint flakes (Desprie, 2005).

#### **Dating method**

#### Sample collection and preparation

Eight samples of loess or palaeosol were taken either as blocks or in metal tubes for dating and were noted down. Samples were collected from freshly cleaned sections, as  $10 \times 10$  cm

blocks or in metal tubes hammered into the section face from bottom to top. BVLl-1, BVL-2, BVL-3 and BVL-4 are blocks, being put into big black and waterproof bags and BVL-5, BVL-6, BVL-7 and BVL-8 were hammered into metal tubes with both side blocked by tight and waterproof adhesive tape.

The sunlight-exposed outer surface of blocks and the ends of tube samples were removed in the laboratory under subdued orange light.

Unexposed loess or palaeosol was prepared for equivalent dose (ED) determination while exposed material was retained for water content and radioisotope content analyses. Carbonates and organic matter were removed from sample by treatment with 3N HCl and 3N H<sub>2</sub>O<sub>2</sub>, respectively. Quartz was separate from the resulting material by immersion in H<sub>2</sub>SiF<sub>6</sub> for up to a week, with a subsequent HCl wash to remove fluorite precipitates. Grains were dried in the oven at 45 °C and then mounted on aluminium discs using silicone oil.



Fig. 2. Photo of excavation site and sampling levels.

#### Instrument and measurement

All measurements were performed using a TL/OSL DA-15 RisØ reader equipped with a Y-Sr90 sour light emitting diodes (LEDs). luminescence measurements were made at 125°C. Luminescence emissions were detected by an EMI 9635Q photomultiplier tube through 7.5mm thick U-340 filters. Irradiations were done with a Cs-137 gamma-ray source delivering 1.48 Gy/min (Valladas, 1978). All luminescence measurements sequence was made with a TL/OSL RisØ Sequence Editor and all mathematical fitting was carried out using Anatol 1.5 software (written by N. Mercier).

### Dating protocol and Equivalent dose curve



Fig. 3. Equivalent dose curve of BVL3 as a function of laboratory doses: natural, natural +  $d_1$ , natural + $d_2$ , natural + $d_3$ .

Recuperated OSL signal had been mentioned by Aitken (1998), while a main protocol to obtain recuperated OSL signal was outlined by Wang and Winlte (2006), they used regenerateddose protocol. In our dating, we employed multiple-aliquot additive-dose protocol (Mercier et al., 1992), using an unattached Cs-137 gamma-ray source delivering 1.48 Gy/ min to irradiate four different parts of the natural sample: natural + 0, natural +  $d_1$ , natural + $d_2$ , natural  $+d_3$  (2d<sub>1</sub>= d<sub>2</sub> 3d<sub>1</sub> = d<sub>3</sub>). After obtaining thermal-transferred OSL signals and basictransferred OSL intensity and investigation of sensitivity changes during measurements and also during inter-aliquot normalization (Murray and Wintle, 2000), we are able to corrected recuperated intensity. Corrected recuperated OSL was then calculated as: Corrected Re-OSL =  $[L_{TTOSL}/T_{TTOSL}] - [L_{BTOSL}/T_{BTOSL}]$  (Wang et al., 2006). The corrected recuperated OSL of the four parts then used to match with additional dose and then the growth curves were built.

Using linear equation fitting, stretched red dashed line crossing with abscissa, we are able to extrapolate the palaeodose in minus abscissa.

### Age calculations

The external dose rate of samples from loesspalaeosol level within archaeological site was measured using a portable multichannel analyzer. Internal dose-rate of each sample was calculated

Sample	U (ppm)	Th (ppm)	K (ppm)	Moisture Contain (%)	Cosmic dose (Gy/ka)	External dose (Gy/ka	Annual dose (Gy/ka)	Paleodose (Gy/ka)	Age (ka)
BVL1	3.50±0.10	13.40±0.10	0.84±0.01	17%	0.18703	0.900	2.347±0.065	$1062 \pm 28.3$	453±40
BVL2	3.68±0.13	13.90±0.10	0.77±0.01	12%	0.16648	0.960	2.359±0.063	937.2±28.7	397±35
BVL3	3.31±0.20	13.76±0.15	$0.60 \pm 0.01$	18%	0.15789	0.940	2.261±0.068	845.0±17.6	374±30
BVL4	3.31±0.12	13.80±0.10	0.89±0.01	18%	0.14791	0.932	2.426±0.065	661.9±34.0	273±30
BVL5	4.05±0.09	13.60±0.30	1.23±0.02	15%	0.13870	1.105	2.941±0.065	427.0±8.00	147±12
BVL6	$2.80{\pm}0.20$	14.20±0.02	1.39±0.02	15%	0.13019	1.144	2.910±0.067	428.5±16.5	147±13
BVL7	3.50±0.20	13.40±0.02	1.25±0.02	12%	0.11095	1.083	2.799±0.065	313.2±13.3	111±10
BVL8	4.25±0.22	13.51±0.24	1.23±0.02	15%	0.10208	1.154	3.010±0.068	285.9±13.8	95±90

**Tab. 1.** Annual dose rates, ED values and ages for the OSL dated samples. U, Th and K contents were measured by by Jean-Louis Reyss. Water content was measured for 8 samples. The external dose rate was measured using a portable multichannel analyzer. The cosmic dose rate corresponds to a burial depth of each sample.

from the isotopic contents (U-238, Th-232, and K-40) determined using gamma spectrometric measurements done by Jean-Louis Reyss (LSCE). Counting in all the factors: U, Th content and percentage of K, water content, cosmic doses depending on the depths.

According to the equation: Age (years) = ED / (The external dose rate + the internal dose rate) —Data uncertainties (Aitken,1998). Using Anatal-1.5 procedure, we obtained a series of age estimate of the sediments sequence in the fig.

### Correlating loess-palaeosol stratigraphy with the marine oxygen isotope record

In Europe, loess-palaeosol sequences have been intensively studied during the past century (Kukla, 1977). Especially, focusing in the Holecene and Upper Pleistocene stratigraphy has provided excellent high-resolution terrestrial archives climate signal. of Owing to improvements in numerical age determinations, for example  $C_{14}$  and luminescence dating methods. more reliable time-based а paleoclimate reconstruction of the and paleoenvironmental change has become available for the loess record in Europe. In recent years much discussion and controversy has surrounded problems of correlation and dating in the European Middle Pleistocene stratigraphy. The difficulties, as recognised by many Europe authors, are due to the fragmentary occurrence of most sediment series and hiatuses of unknown duration (Vandenberghe, 2000), as the recent findings in Chinese loess record (Stevens et al., 2006; Lu et al., 2006). Anyway, no sufficient independent age control is the mainly problem both in European and Chinese loess; and, the Chinese loess is probably "no hiatus" at orbital time scale (Liu, 1985; Lu et al., 2004)

In this paper, we used absolute ages in combination with characteristic paleolithic industries to link the well-established marine stratigraphy and the European land stratigraphy.

In Fig.2, it is obvious the first layer of Paleolithic industry is in the same level as BVL2, then the age of first Paleolithic industry stratum is around  $111\pm10$  ka ,credible corresponding almost exactly with OIS stage 5 and "Eemian" period in European stratigraphy. The second stratum of Paleolithic remains include two sectors: the upper one within same horizon as BVL5 is round  $273\pm26$  ka and the lower one should be about  $313\pm28$  ka, according to mass accumulation rates between BVL5 and BVL6.

Such an age extension is much more precise in comparison with time scale derived from OIS stage 9 and European "Landos" interglacial stratigraphy. While the third and fourth Paleolithic industry level, consulting mass accumulation rates between BVL6 and BVL7, should be between  $379 \pm 32$  ka and  $386 \pm 32$  ka which have good correspondences with OIS stage 11 and "Holsteinian" interglacial in European stratigraphy. Therefore, come into a meaningful phenomenon it shows striking similarity that all levels containing Paleolithic remains and hominid activities are entirely within stratums of interglacial period.

### Conclusions

The multiple-aliquot additive-dose protocol of recuperated OSL method gives an ideal framework of the chronology from 100ka to 450ka in the archaeological site of Bonneval, where  $C^{14}$  and classical OSL dating methods can not touch. Bonneval have a widely chronology and several paleolithic industries levels. It seems



**Fig. 4.** Distribution of different period paleolithic industries, compared with the marine oxygen isotope record(Shackleton, 1990).

the chronology and morphologic character of paleolithic industries match very well.

The age of the paleolithic industries is from middle paleolithic industries to lower paleolithic industries. Many big tools, such as "Handaxe", have been considered as product in glacial stage

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by many archaeologists, while in our result, it is evident "Handaxe" and biface belong to interglacial period, furthermore, all the paleolithic industries levels were during interglacial period, none of exception.

#### Summary

The recuperated optically stimulated luminescence (Re-OSL) signal separated from thermally transferred OSL signal may be used to estimate the equivalent doses for fine-grained quartz, whereas the basic transferred OSL signal, another part of thermally transferred, does not provide information on the sedimentary age.

Using a new multiple-aliquot additive-dose protocol, the recuperated OSL dating method was applied. Equivalent doses were determined with this multiple-aliquot additive-dose protocol for 8 sequential samples from the Paleolithic site of Bonneval "La jouanniere", Eure-et-Loir, France. Ages obtained for samples are from ~100 ka to ~450 ka. The recuperated OSL dating method, therefore, can be used to date old sediments which traditional OSL method and C<sup>14</sup> can not measure. This recuperated OSL dating method can go more beyond the Middle Paleolithic and probably cover a large part of the Lower Paleolithic. Correlating with the marine oxygen isotope record (OIS) and standarded European stratigraphy, we attempt to interpret the paleoclimate and paleoenvironmental for hominid. It is evident "Handaxe" and biface belong to interglacial period, furthermore, all the paleolithic industries levels  $(111\pm10 \text{ ka};$  $273 \pm 26$  ka;  $313 \pm 28$  ka;  $379 \pm 32$  ka;  $386 \pm 32$ ka;) were during interglacial period, none of exception.

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