Thesis abstracts

Thesis title: Optically stimulated luminescence of quartz: methodological developments and dating applications to Upper Pleistocene sequences from North-western France.

Author: Elise Folz
Address: LSCE, Avenue de la Terrasse, 91198, Gif sur Yvette, France
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Supervised by: Dr Hélène Valladas
Examined by: Prof. Michel Lamothe and Dr Didier Miallier
Awarded by: Paris 7 University, France.

The first part of this work deals with the setting up of quartz OSL in our laboratory (Laboratoire des Sciences du Climat et de l'Environnement, Gif-sur-Yvette) and with the development of experimental procedures for the determination of the radiation dose accumulated in these minerals (paleodose).

A specific protocol based on the regeneration of the luminescence signal and requiring only a few milligrams of sample (single-aliquot) for each determination is suggested. It is demonstrated that different regenerative doses, bracketing the paleodose, are needed. In order to allow for sensitivity changes due to successive measurements, the use of a master curve, fitted to the experimental data corresponding to one of these doses is proposed. Experiments were done on five laboratory bleached and dosed quartz samples and the effects of the choice of the main regenerative dose and preheat temperature were tested. The measured ED was found to be in excellent agreement with the known value: for the five samples considered in this study, the largest deviation of the mean ED (30 determinations) from the expected value was 1.3%. This single-aliquot protocol was also tested on the Australian WIDG8 sample, and the estimated ED is consistent with previously published ones.

The single-aliquot approach is particularly valuable in providing information on sediment heterogeneity and the protocol outlined above has been applied to the study of

- Alluvial sediments from a Late Glacial prehistoric site (Le Closeau, Hauts-de Seine, France) where a comparison of OSL and ^{14}C ages, supporting geological and archaeological evidence, is possible. Despite tightly and symmetrically distributed EDs, OSL ages are overestimated by about 40%. We have reviewed potential problems in luminescence dating procedures, such as partial bleaching at deposition or disequilibrium in the radioactive decay chains, but our measurements, so far limited to the coarse grain quartz fraction, failed to identify the reason of the observed discrepancy. Other theories regarding sediment deposition and evolution will have to be examined in the future.

- Three aeolian dunes from the Northern coast of Massif Armoricain, where radiometric dates are scarce, mainly because of poor conservation of organic matter. Measurements on the Paleolithic site named Le Rozel (Manche) confirm the attribution of the lithic remains to the Middle Paleolithic blade industry, as proposed by D. Cliquet and B. Van Vliet-Lanoë. We studied Upper Pleniglacial loessic sediments from Port-Racine (Manche) and Sables-d'Or-les-Pins (Côte d'Armor), where a comparison with the ages obtained on the same samples by M. Frechen (Cheltenham University) using TL and IRSL multiple-aliquot on fine grains is undertaken. The results show that, at about the Last Glacial Maximum (ca. 20-22 ka), this area experienced rapid sediment accumulation, at the rate of 1 – 1.5 m/ka. For all samples but one from Port-Racine, we noticed ED distributions much larger than those obtained while testing the protocol, on the same bleached and dosed samples, revealing a complex sedimentation process.

Thesis title: Thermoluminescence dating of granitic quartz.

Author: Han Zhiyong
Address: Radioisotope Unit, Pokfulam Road, Hong Kong
Date: June 1999
Examined by: E.J. Rhodes (Oxford University) and J.K.C. Leung (HK University)
University of: Hong Kong

A new geochronometer for granite is discovered from measurements of quartz thermoluminescence (TL) sensitivities to ionizing irradiation. The TL sensitivities show age dependence for samples from 15 to 3306 million years. A new physical model is introduced to explain the age dependence. In this, the TL sensitivity is interpreted as a measure of trap populations. There are two kinds of traps in quartz, namely thermally sensitive and radiation sensitive. In ambient temperature, the lifetimes of the traps are at least four orders of magnitude longer than those of corresponding trapped electrons.

Three dating techniques are established. The additive alpha dose technique based on the observations that radiation sensitive traps at temperatures around 400°C can only be created by alpha radiation in nature, and the population of such traps was close to zero immediately after crystallization. The age of the sample can be determined by dividing the total alpha dose by annual alpha dose. The other two techniques are based on empirical equations established from granite samples of known age; one is from the radiation sensitive traps and able to date granites older than 100 million years. Another is from the thermally sensitive traps and can be applied for granites younger than 400 million years.

Combination of these dating techniques provides a quick and economical way of estimation of cooling ages for granites, and may also be applied to date quartz from other origins. The features of the TL sensitivity can also have important implications for the evolution of the earth system, such as thermal history.

Thesis title: Archaeomaterial dating with thermally and optically stimulated luminescence: TL and OSL of silicates or carbonates.

Author: Emmanuel Vartanian
Address: CRPAA, Esplanade des Antilles, Domaine Universitaire, F.33405, Talence cedex
Date: Dec. 1999
Supervised by: Max Schvoerer
University: Bordeaux, France

From a long experience in thermoluminescence (TL) dating (method, developed since the end of the sixties in the university of Bordeaux), we have explored new ways and researches in TL and OSL (Optically Stimulated Luminescence) dating.

With thermoluminescence we have searched for answers for un-solved chronological problems, as well as a solution for the datability of calcium carbonates either coming from caves, or heated in the past or exposed to the light.

Among the results obtained, we can point to datings for the neolithisation of Italy of the south around 6000 ± 200 BC (Matera - Trasano), for the eneolithic occupation of the Sorrento's peninsula (Piano di Sorrento - La Trinita), around 2400 ± 250 BC, and for the Mochica culture of the north coast of Peru (Tomb of the Priest, from Sipán) connected with the 8th century of our era and not with the 3rd, previously considered from an unique C14 data. Otherwise, we are able to block the structural modification of calcium carbonates under heating (decarbonation) using CO_2 atmosphere during TL studies, so it is possible to consider their dating. Results obtained on fired stones from Combe-Saunière (Dordogne) are positive and encouraging.

We have also explored the possibility to use OSL for archaeological material dating, from the intercomparison between OSL and TL results obtained on the same samples (ceramics, terra cotta...). This study has required the development of an original apparatus allowing the selection of the stimulation wavelength in the visible range. Experiments carried on crystals extracted from archaeological baked earths on the one hand, and on natural or synthesised quartz on the other hand, led us to have some thought about changes in point defects distribution occuring during bleaching, irradiation and heating. For quartz, from identification of luminogen centers and traps involved in TL or OSL, it was possible to precise the role played by the charge compensator due to their different mobility: alkali ions (Li^+, Na^+,...) on the one hand and hydrogen (H^+, i.e. OH^-) on the other hand; it was also possible to modelize some phenomena observed in OSL, introducing irreversible modifications in radiative recombination centres population due to specific thermodynamic conditions.

Finally, an original OSL dating procedure has been totally defined. It is based on the direct comparison between OSL intensities measured on natural irradiated crystals and on the same crystals bleached then laboratory irradiated. Results obtained with this protocol are accurate and encouraging; furthermore they show that taking into account pre-dose effects is necessary.