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RECENT ADVANCES IN QUATERNARY

GEOCHRONOLOGY

by

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ABSTRACT

Significant progress has occurred in quaternary geochronology. These include both (i) improvements in Fission Track and Thermoluminescence dating, as well as (ii) new technologies for short-lived (i.e. with half lives  $< 10^6$  yrs) radionuclide measurements as with the  $^{14}\text{C}$  or uranium series disequilibrium dating, and finally (iii) the emergence of entirely new dating approaches as the Electron Spin Resonance Method. The aim of this paper is to review these progresses and the new areas they open to geochronology for the past-miocene times.

RESUMO

Avanços importantes têm ocorrido em geocronologia do quaternário. Tais avanços incluem:

- (i) melhoramentos nas datações por traços de fissão e por termoluminescência, bem como
- (ii) novas tecnologias para para medidas de vida curta (i.e. com meia vida  $< 10^6$ ) tais como a da datação por  $^{14}\text{C}$  e pelo desequilíbrio da série do Urânio, e finalmente
- (iii) o surgimento de uma forma completamente nova de datação - o método de Ressonância Paramagnética Eletrônica.

O objetivo deste trabalho é apresentar uma revisão desses avanços bem como as novas áreas que se abrem à geocronologia para o período do post-mioceno.

## 1. INTRODUCTION

It is only since about thirty years that radioactivity has been successfully applied to the absolute dating (i.e. dating expressed in years, rather than in periods as conventionally used in stratigraphy) of geological events. More than 20 methods (see f.i. Faure 1977) based on natural radioactivity and isotope measurements allow now to decipher with increasing details and precision the past history of the earth, meteorites and the moon. One period for which absolute dating is extremely important is the quaternary. This period, which extends over the past few millions of years, has in effect been a time of wide climatic variations, with alternances from cold, - glacial - , and warmer periods, humid and dry climates, as well as of important biological changes. It is the geological period where the emergence of man occurred while significant changes of vertebrate fauna and flora occurred. The precise timing of all these events would be as fundamental for the prediction of future climatic evolution as for a better understanding of the possible influence of environmental changes on the biosphere.

Before about 1980, the geochronologic tools for the quaternary were unfortunately very limited:  $^{14}\text{C}$  dating was widely applied to biologic materials as well as to inorganic carbonates and waters, up to  $\sim 40000$  yr ago (and exceptionally 70000 yr, using isotopic enrichment techniques); the methods based on uranium and thorium disequilibrium of decay series were useful for a somewhat more extended time range, between  $\sim 10000$  and 300000 yr. The fission-track and K-Ar methods for volcanic rocks could in general be used for recent volcanic rocks for times only slightly under  $10^6$  yr to  $10^5$  yr, and this with the utmost cautions and a limited degree of reliability, according to the dated materials (see Hay, 1980). Magneto-stratigraphy, once combined with one or more of the above methods proved to be a useful support for defining periods of deposition of volcano-sedimentary stratigraphic series.

Among the new isotopic methods of dating the quaternary, it is worthwhile to cite dendrochronology and varve dating, known since long, and effective in the limited time range from the present time to respectively about 8000 yrs and 12000 yrs ago. The chemical methods once proposed for biological materials and based on the measurement of fluor and nitrogen in bones, or the racemisation rate of amino-acids failed to prove reliable and are now mainly discarded (Fleming, 1979; Von Endt, 1979). Variations of environmental conditions in effect do modify in a very complex way the rate of the chemical reactions involved. As a consequence, it appears extremely difficult to make predictions on the rate of these reactions in natural environments from the much simpler conditions realised in laboratory simulation experiments. For similar reasons, dating using the hydration rate of obsidian requires extreme cautions and in any case do require some calibration with an otherwise dated sample.

Since about 5 years, important progresses were made in sever-

al directions, offering new possibilities in quaternary geochronology. They include new developments of already existing methods, as for the Fission Track (FT) and Thermoluminescence (TL) dating methods; the re-n<sup>o</sup>wing of other methods by the use of new technologies for measuring ra<sup>o</sup>dioactive decays, as with the "γ-X" way of uranium series disequilibrium dating, or accelerator dating for cosmogenic isotopes as <sup>14</sup>C; finally, completely new ways of dating were proposed as with the Electron Spin Resonance method. It is the aim of this paper to briefly review these advances and make some suggestions as to some of their possible appli-cations.

## 2. FISSION TRACK DATING

Although this method was proposed as early as 1963 by PRICE and WALKER it has for years not known a wide development, principally because of problems linked to the partial track stability in rocks (Fleischer et al., 1975). This method is in effect based on the reten-tion by natural minerals and glasses of the radiation damage left by the two recoil fission fragments produced by the spontaneous fission of <sup>238</sup>U. The development of age correction methods (Storzer and Wagner, 1969) and later of model ages with the plateau-methods (Storzer and Poupeau, 1973; Poupeau et al. 1980; Poupeau and Ceylan 1982) allowed to overcome these early difficulties (see review papers by Poupeau 1981a and espe-cially 1981b).

It was in effect shown that the use of a stepwise heating treatment (more or less equivalent to those used in <sup>39</sup>Ar-<sup>40</sup>Ar dating meth-od, Faure 1977) made possible to obtain significant FT ages. Applica-tions of such methods may allow now a fine study of cooling /erosion rates of old basement rocks (see companion paper in this congress by Poupeau 1982 and Baksi and Poupeau, 1982).

As far as the quaternary period is concerned, it was shown by Storzer and Poupeau (1973) that glassy lava flows (obsidiens ) and glasses produced on earth by meteorite impact (impactites, tektites) could be dated with a precision of better than 5%. Naeser et coll. (1980) in a study of volcanic ashes (tephras) from the Western United States, discovered that, for glass shards of ~ 100 μm granulometry the isochro-nal plateau method of Storzer and Poupeau could not give satisfying re-sults because of tephra glass shards hydration following their deposi-tion. We have shown (Carpena et al., 1980; Poupeau 1981b) that using an other type of plateau method ("isothermal" plateau method) significant FT ages could be obtained on glassy tephra material, as well as on mas-sive glassy volcanic rocks.

Obsidiens can be dated with FT from ~ 10<sup>4</sup>yr ago and glass shards from tephra from ~ 5x10<sup>5</sup>yr. The glassy crust of oceanic pillow la-vas is extremely difficult to date for samples of age < 10<sup>5</sup>yr due to their very low uranium content (down to 0.1ppm) (Poupeau 1979; Lalou et al., 1978). Using minerals rich in uranium as zircons or apatites, vol-canic rocks of ages respectively > 0.5x10<sup>6</sup> yrs (Carbonnel and Poupeau, 1969) and > 10<sup>6</sup> yrs can be dated. All these evaluations are for materi-als with "typical" uranium contents. They are subject to wide varia-tions according eventual deviations from these averages.

## 3. THERMOLUMINESCENCE

Thermoluminescence (TL), although proposed as a dating meth-od in 1953, was put into work only at the end of the sixties, with man-made materials: ceramics.

The principle of TL dating is rather simple. The radioisoto-pes present in any rock produce ionising radiations which free elec-trons from their parent atoms. The majority of these electrons do re-combine immediately but a small fraction of these remain trapped in im-perfections of mineral lattices. Some of these traps are "deep" enough, so that the electrons cannot escape through thermal agitation. In the deepest traps of interest to TL dating, a temperature of ~ 300°C to 600°C is needed to empty totally the electrons.

If therefore we do have one material with no trapped elec-trons at a starting time ( which could be either the time of cooling fol-lowing extrusion of a volcanic rock, the formation of a calcareous mate

rial as a stalagmite or the firing of a ceramic), the accumulation of electrons in traps will progress linearly with time. This proportionality is due to the fact that natural radioactivity delivers ionising radiations at a constant rate with time.

Dating with TL consists in the measure of the number of trapped electrons. In TL dating, one gives enough energy by heating (usually at a rate of the order of  $10^{\circ}\text{C}/\text{sec}$ ) to the electrons to force them out of their traps; once release of their traps, some electrons will recombine with a luminescent center while emitting some light. The quantity of light (as measured with a photomultiplier tube) emitted at high temperature is a function of the accumulation time. Combining TL and radioactivity measurements (the latter to calculate the effect of radioactivity upon the trapping probability of electron per unit of time), one can deduce a TL age (for a general presentation of TL dating, see Aitken 1974).

The range of time accessible to TL dating is essentially limited by saturation of the electron traps. There is in effect only a finite number of lattice defects where ionised electrons can be trapped. As a result, and according both to the dated mineral lattice properties and the local level of radioactivity, TL dating is essentially limited to the last  $0.05 \times 10^6$  yrs to  $\sim 10 \times 10^6$  yrs (see below).

Up to the late seventies, TL dating was almost exclusively limited to ceramics and, as a consequence, to archaeological applications. Recent progresses were made in several directions and geological application of TL dating is now possible in a wide variety of contexts. A detail review of many of the data given below can be found in Wintle (1980).

Burnt Stones. Burnt stones can be found as the result of intentional firing (archaeological context: stones from fireplaces or constructed hearths), accidental firing (forest fires, burning of ancient settlements or monuments) or volcanism (baked soils beneath lava flows). It has been shown since about 1975 that different types of burned stones in these contexts could be dated by TL. They include granites and sandstones (Poupeau et al. 1976), flints (Goksu et al. 1974; Danon et al., 1980) from archaeological fire-places as well as baked sediments beneath hearths (Huxtable and Aitken, 1977) or underlying lava flows (Sutton 1978; Gillot et al., 1979).

The dating range of burnt stone materials seems to extend, from a survey of the still limited literature on the subject, from  $\approx 200$  yr to  $0.5 - 1 \times 10^5$  yr for quartz from granites, quartzites and sandstones as well as filonian quartz; from  $\sim 1000$  yr to  $50000 - 500000$  yr for various types of flints and cherts. Potash Feldspars from burnt stones proved also to be convenient for TL dating (Mejdahl, 1972).

Volcanic Lavas. Although volcanic lavas can be TL-dated by the underlying material they baked, they also can be themselves dated. We have shown that, using an unconventionally "high" TL temperature analysis, volcanic plagioclases do have a peak at  $\sim 600^{\circ}\text{C}$ , where electrons are stable enough for dating (Valladas et al., 1977, 1978; Gillot et al. 1979). Another approach is to take advantage of the presence of burnt quartzitic xenoliths in volcanic lava flows (Gillot et al. 1979). Others have shown that obsidian glass could be used for TL dating (Bechtel et al., 1979).

Volcanic plagioclases seem to be quite interesting material for TL dating, as they would allow to cover a wide age spectrum, from  $\sim 10^3$  yr to  $10^6$  yr, thus filling the gap between  $^{14}\text{C}$ , and FT and K-Ar datings. Only preliminary data were obtained on obsidian and the possibilities of TL dating here are still largely unknown.

Calcite. Calcite formed within the quaternary in a variety of environments, as cave deposits (stalagmites and stalagmites, travertines), lacustrine deposits (calcareous concretions, calcretes), products of biological activity (stromatolithes, etc...) or chemically deposited marine carbonates.

TL dating of calcite is still in its infancy. Its use was for long prevented by a variety of methodological difficulties (the main one being triboluminescence), which now seem in many cases mastered. It

seems that calcite will soon be largely used as a datable material for all the quaternary period.

Sediments. This is one of the great potentials for TL applications to quaternary geology. It is based on the fact that fine materials wind-transported are practically emptied, during their time of flight, of their geological TL, under the action of sunlight. Proposed by Wintle and Huntley in 1980 for oceanic sediments, this method seems to give satisfying results within the last  $2 \times 10^5$  yr. More recently, Wintle (1981) has shown that another type of wind-deposited material, the continental deposits known as loess, could also be TL dated. Results of loess dating from southern England gave age ranges of  $\sim 15000 - 30000$  yr, in an apparent good agreement with expected values.

The potentials of TL dating for loess and oceanic sediments, and more generally for wind-transported sediments where geological TL was bleached by sunlight, seem very promising.

Fossil Sand Dunes. Based on the same principle as sediment dating: i.e. emptying of geological TL of quartz sands crystals by exposure under the sunlight while residing at the surface of dunes, this method, first explored by Singhvi et al. (1982) seem also of great potential. At present, only dunes of a few thousands of years old have been dated by this way, giving results in reasonable agreement with  $^{14}$  datings.

Bone Dating. Considered impossible for a long time, because of the presence of organic compounds giving a parasite source of light (by chemiluminescence) at elevated temperatures during TL analysis, bone dating might become soon possible. New purification techniques, on one hand, might insure a nearly complete elimination of organic products, allowing to measure TL at temperatures  $> 300^\circ\text{C}$ ; the use of a phototransfer technique on the other hand might permit to eliminate any non-radiogenic emitted light by working at low ( $\sim 150^\circ\text{C}$ ) temperature.

#### 4. URANIUM AND THORIUM DECAY SERIES DISEQUILIBRIUM METHODS

The presence, in the decay series of  $^{238}\text{U}$  and  $^{235}\text{U}$ , of relatively long-lived radio-isotopes (with half-lives  $> 10^3$  yr) has been the basis of several so-called U-series disequilibrium methods of dating.

In effect, under certain circumstances, uranium alone will fix in various types of materials (bones, calcareous deposits) and then progressively build up its decay series up to an equilibrium value, where all daughter isotopes will present the same activity (i.e. number of radioactive decay per unit of time). The measure of the relative activity of one daughter isotope and parent isotope is a measure of the degree of disequilibrium of the part of the decay series to which they belong, therefore of the time elapsed since deposition of the parent isotope. (At equilibrium, any such activity ratio within the  $^{235}\text{U}$  or  $^{238}\text{U}$  decay series is equal to 1).

Progresses in solid state detectors (production of High Purity Ge (HP-Ge) crystals) allow now to replace the previous technique of  $\alpha$ -counting for U-series disequilibrium dating by a direct " $\gamma$ -X" counting method. The latter is so called as the HP-Ge crystal for the analysis of the  $\gamma$  radiations emitted by a sample is very sensitive and with a very low background down to photon energies of  $\sim 10\text{KeV}$ . In other terms, this new type of detector extends the possible analysis of  $\gamma$  radioactivity down to photons with energies within the X-rays range, well below  $100\text{KeV}$ . (The preceding generation of detectors using Germanium crystals doped with lithium (GeLi) were operative mostly above  $100\text{KeV}$ ). This is especially interesting for the direct measurement of isotopes like  $^{230}\text{Th}$  ( $E_\gamma = 67.7\text{keV}$ ),  $^{210}\text{Pb}$  ( $E_\gamma = 46.5\text{keV}$ ) and  $^{238}\text{U}$ , from its descendent  $^{234}\text{Th}$  ( $E_\gamma = 63.3; 92.3$  and  $92.8\text{keV}$ ).

The great advantages of  $\gamma$ -X counting over  $\alpha$ -counting are twofold: (i) they suppress any chemical treatment, -extremely long and delicate in the  $\alpha$ -method-, as samples are simply powdered for counting in standard, simple geometrical conditions; (ii) if needed, the  $\gamma$ -X method can be non-destructive. This latter possibility is of a paramount importance for the dating of very rare and precious samples (e.g. human

fossil bones. In this case, an internal calibration of the counter detection efficiency must be made from a radioisotope-doped casting of the sample, counted in identical geometrical conditions.

One first account of this new technology for U-series dating was given by Yokoyama and Nguyen (1980). It is expected that through the considerable gain of time it provides, the U-series dating can develop to a much larger number of applications than its predecessor  $\alpha$  method.

## 5. ACCELERATOR DATING

Any radioactive cosmogenic isotope produced by nuclear interaction between the cosmic rays and the atoms of the terrestrial atmosphere at high altitude may potentially give rise to a dating method. At present, the only cosmogenic isotope widely involved in terrestrial geochronology is  $^{14}\text{C}$ , produced by slow neutron capture on atmospheric  $^{14}\text{N}$ . Others, as  $^{10}\text{Be}$  or  $^{26}\text{Al}$ , were only occasionally used because of their low activity in terrestrial materials, barely above background counting levels.

In effect previous to the eighties all geochronologic methods using cosmogenic radio isotopes were based on radioactivity measurements of these isotopes. New possibilities were open when in 1977 was proposed a completely new detection principle for these isotopes: High Energy Mass Spectrometry, using nuclear physics machines as particle accelerators of the electrostatic type, as Tandem Van de Graaf, or cyclotrons. With these machines, isotopic ratios of the order of  $10^{-12}$  to  $10^{-16}$  can be measured with a precision sufficient for many geochronologic purposes (Conventional mass spectrometer cannot resolve isotopic ratios of that extremely low values because of contamination problems only, see f.i. Anbar 1978 for  $^{14}\text{C}$ . These measurements are possible only because of the high energy given to the isotopes in the accelerating tubes (see review paper by Litherland, 1980).

The isotopes of interest considered as prime targets for this new geochronologic field are  $^{14}\text{C}$  (half-life  $T_{1/2} = 5730\text{yr}$ );  $^{10}\text{Be}$  ( $T_{1/2} = 1.5 \times 10^6\text{yr}$ ),  $^{26}\text{Al}$  ( $T_{1/2} = 716000\text{yr}$ ),  $^{36}\text{Cl}$  ( $T_{1/2} = 308000\text{yr}$ ),  $^{32}\text{S}$  ( $T_{1/2} = 400\text{yr}$ ) etc. If one considers that accelerator geochronology might extend over about 10 radioactive periods, it is a time spectrum comprising the last 10 millions of years which could open to new geochronologic analysis. One of the advantages of these methods over conventional approaches, when they were possible, is that they require much less material. For instance,  $^{14}\text{C}$  dating with an accelerator would not require more than 10 mg of carbon whatever its age from the present time to  $\sim 10^5\text{yr}$ , whereas at least a few grams are required for a conventional, radioactive measurement up to  $\approx 30000 - 40000\text{yr}$ , and up to 120g for dating with isotope enrichment at  $\sim 70000\text{yrs}$ . Another advantage of accelerator methods is in the time needed for one measurement, which usually counts in hours, rather than days for radioactive methods.

## 6. ELECTRON SPIN RESONANCE

Electron Spin Resonance dating is based on the same basic phenomena as TL dating, i.e. the presence of trapped electrons in a lattice structure. The difference between ESR and TL is in the method used to measure the number of trapped electrons. In the ESR method, the electrons are excited within a magnetic field in the presence of a flux of microwaves. The measure of the microwave absorption at the external magnetic field resonance value provides a measure of the number of trapped electrons. One major advantage of this method over TL is that it does not need any heating of the material to be dated, which makes it a preferred method for the dating of bones. On the other hand TL is more sensitive and in any case must be employed preferentially to ESR when parasite signals due to impurities (especially large amount of  $\text{Mn}^{2+}$ ) prevent the reading of the radioactive signal in the ESR spectrum. In many instances, although both methods can be used independently, TL and ESR appear as complementary and their combined use when possible on the same sample permits useful internal controls on the behavior of this electron-defect based geochronometer.

The method appeared in 1975, with an article by IKEYA, who further demonstrated its possibilities in a series of short papers. One summary on this early work is given in IKEYA (1978). Improvements in the evaluations of the annual radioactive dose rates to the dated minerals were later proposed by Yokoyama et al. (1980).

ERS dating has been tested up to now on calcite, bones-where the phase giving the dating signal is a calcium phosphate, apatite-, and quartz. For calcite it seems to apply in the range from  $\sim 10^3$  yr to  $\sim 10^7$  yr. This includes stalagmites and stalagmites (IKEYA 1975, 1978; YOKOYAMA et al. 1981a) as well as marine shells (IKEYA and OHMURA, 1981). Bones seem to be datable for times up to several millions of years (IKEYA, 1978; YOKOYAMA et al., 1981b). Investigations of cherts, flints, quartz and other minerals is just beginning. Quartz and gypsum seem promising, while the potentialities of cherts and flints seem to be largely variable according to wide structural and impurity content variations.

## 7. CONCLUSIONS AND PERSPECTIVES

Very exciting developments have taken place in quaternary geochronology along the past few years. New types of instrumentation, new methods appeared, new datable materials were discovered, new types of applications proposed. Instrumental problems are still far from being all resolved, especially in accelerator dating (see f.i. Bertier et al., 1980; Litherland, 1980; Hedges, 1981). The thermal stability of fission tracks and of electrons in their traps has still to be better known from a theoretical point of view. Further studies on anomalous fading in a number of new TL/ESR datable materials (bones, shells, etc.) will be welcome. The precision and accuracy of some of these methods is not yet precisely known for all types of materials and actual sampling site environmental conditions, etc...

It remains however that where we had a narrow spectrum of dating method the perspective has now enlarged considerably. Multiple datings of a simple rock or event appears possible and certainly our knowledge of the quaternary chronology will soon improve considerably.

As far as geology is concerned, important developments are expected in a number of directions, among which the following.

Palaeoclimatology. The prevision of the near future of climates on earth lays largely on the knowledge of past quaternary climates as a basis for the search of the responsible parameters for changes. Palaeoclimatologists need long and continuous, well dated series of stratigraphic records of past climatic variations as recorded in marine and lacustrine cores, or ice cores from glaciers and inland seas (e.g. Antarctica). Accelerator geochronology, with its very small required sample size and various isotopes available for dating, could provide the thousands of measures needed for this work. Climatic changes are also recorded in continental lakes by the deposition of calcrete layers, in caves by travertine layers, stalagmites and stalagmites, and here TL and ESR could prove a valid approach. Dry periods could be dated using quartz from fossil dunes, etc...

Quaternary Geoid Deformation and Sea Level Changes. It is well known that, in addition to the major wide scale sea level changes provoked by glaciation - deglaciation alternances in the recent quaternary, beach levels may also have changed as a response of geotectonic pulses. The dating of these pulses, previously possible with  $^{14}\text{C}$  and U-series disequilibrium dating, can now be extended over all the quaternary applying TL or ESR dating either shells, or carbonate formations of reef type, as well as TL dating of marine sediments. Correlations with other tectonic features up to miocene times would be possible in several instances providing a basis for the study of recent plate deformability.

Neotectonics. Neotectonics as part of applied geology for the implantation of dams, nuclear plants, etc... has widely developed recently. Dating faults supposedly still active is in this context of the highest importance. Dating recent fault displacements with  $^{14}\text{C}$  accelerator dating was suggested several years ago. TL and ESR datings of faults is

a potential not yet explored. High stress in fault zone may cause either heating or recrystallisation of quartz for instance.

Many other suggestions are possible and have been done. They include, in addition to other possible geological applications, such diverse fields as archaeology, palaeoanthropology and paleosolar physics. Certainly, we will see in the next years a considerable improvements in the timing of the quaternary period and a better understanding of phenomena still poorly correlated in time

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