A CATHODOLUMINESCENCE/X-RAY MICROPROBE STUDY OF VARIOUS SEDIMENTS FROM INDIA AND ANTARCTICA

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Introduction:

This note reports the results of a simultaneous cathodoluminescence (CL)/x-ray microprobe study on a variety of sediments from India and Antarctica. This research was prompted by the increasing appreciation of the potential of TL for dating Quaternary sediments. Identification of the dominant luminescent mineral phase(s) in such samples, in conjunction with previous studies of the TL properties of various mineral groups, would be useful in gaining information on the process and mechanism of sun-bleaching, maximum bleachable TL levels, the role of retrapping (David, et al., 1981; Levy, 1982) and the dependence of bleaching on initial dose and UV/sunlamp dose-rate. In addition, knowledge of the luminescent mineral type is desirable for optimizing detector optics to select desirable phases of polymineralic samples. Previous mineralogic studies on loess by Weir, et al., 1978, have suggested that quartz and feldspars are the major minerals in this material.

Experimental Details:

Three types of sediments were studied in the present work:

1) Dune sands from Thar desert in India
2) Loess deposits in Kashmir
3) Dust trapped in glacial and Antarctic ice.

Fine grain discs of these samples were analyzed. The samples had been previously treated with HCl to eliminate post-depositional carbonates. In addition, the polymineralic, 90-105 μm grain fraction from several dune sands was studied. Most of the fine grain samples were coated with 200A of carbon for SEM studies. The coarse grains were coated with 100A of gold to eliminate luminescence caused by charging of the grains. The scanning electron microscope's cathodoluminescence system had an EMI 6255 photomultiplier tube with S-13 response and the x-ray system was an Ortec Si(Li) detection assembly. Individual bright luminescent grains were detected in the cathodoluminescence mode and were then microprobed by focussing and stabilizing the electron beam on each of these grains. Generally, the ten brightest grains on each fine grain disc were microprobed. The mineral types were assigned by identifying the major elements present in the x-ray spectra and the relevant details have been described earlier (Singhvi and Zimmerman, 1978). The results are given in Table 1. The frequency distribution of the occurrence of each mineral phase is given in Table 2.

Results:

1) In most cases, K-feldspar is the dominant CL mineral, which in view of its substantially high TL sensitivity is expected to dominate the polymineralic TL. In view of the broad similarities of glow curves of other sediments with those mentioned above, it appears logical to conclude that, in general, feldspar dominates sediment TL.

Table 1: Cathodoluminescence/x-ray microscope analysis of fine grain samples of glacier dust, loess and dune sands

Sample	K-feldspar	Plagio-clase	Quartz	Apatite	Zircon	Other
GLACIER DUST						
Nehnar						
NG-2	6	1	1	0	0	2
NG-9	3	0	6	0	0	0
NG-13	4	2	3	0	0	0
NG-16	0	1	3	4	0	1
NG-20	1	2	5	2	0	0
NG-29	3	4	1	1	0	0
NG-39(I)	3	2	3	1	0	0
NG-39(II)	4	0	5	0	0	0
NG-40	4	0	3	3	1	0
NG-48	2	0	8	0	0	0
NG-52	5	0	5	0	0	0
Changme-Khangpu						
CK-61-81	7	3	0	0	0	0
CK-61-81	7	3	0	0	0	0
CK-90-81	6	1	3	0	0	0
Zemu						
Z-26-I	5	0	5	0	0	0
Z-26-II	8	1	1	0	0	0
Z-27	6	2	1	1	0	0
Antarctica						
ANB-4	10	0	0	0	0	0
ANC-3	3	7	2	0	0	0
2-6A	2	0	1	0	0	7
NG-2	1	1	5	0	0	2
X	8	0	2	0	0	0
LOESS						
Dilpur						
TL-L-1	8	0	1	0	0	0
TL-L-1	2	0	0	0	0	0
TL-L-2	10	0	0	0	0	0
TL-1-3	7	2	1	0	0	0
TL-L-6	8	0	0	0	0	0
DUNE SAND						
Amarpura						
TL-S-7	6	3	0	0	0	0
TL-3-8	6	0	3	0	0	0
TL-5-9	10	0	0	0	0	0
Langhnaj						
TL-5-10	10	0	0	0	0	0
TL-5-11	2	0	0	0	0	0

Table 2: Frequency of mineral types

Sediment type	K-feldspars	Plagioclase	Quartz	Others
Glacier dust	0.45	0.14	0.29	0.06 apatite 0.06 misc.
Loess	0.90	0.05	0.05	---
Dune sand	0.85	0.075	0.075	---

2) Glacier dust samples were much brighter in CL than loess and dune sand samples. A possible explanation is a surfacial coating of iron compounds (e.g., haematite) on grains from dune sands and loess that may alter the transparency and luminescence efficiency of grains (Gardner and Pye, 1981).

3) Comparison of these data with glow curve shapes suggests that in general a broad glow peak at around 250°C is due to K-feldspars whereas a shoulder about 350°C is most likely due to Quartz. This is supported by glow curves of HF-etched samples that yield a glow peak at around 380°C. It is interesting to note that similar results have also been obtained by Debenham and Walton (1982) who were able to separate feldspar emission from quartz emission (see their Figs. 1 & 2) using filters (Schott and Jena-UG11, transmission peaking at 325 nm and Corning-5-58, transmission peaking at 410 nm).

4) The CL minerals in coarse grain fractions of dune sands are generally the same as those in the fine grain fractions, K-feldspar, quartz and plagioclase.

Conclusion:

This study has shown that K-feldspar generally dominates Indian and Antarctic sediment CL and, therefore, probably dominates the TL from these materials. This result suggests that more effort should be initiated to understand feldspar properties.

References:

M. David and C. M. Sunta. (1981) Ind. J. Pure & Appl. Physics 19, 1041.

P. W. Levy (1981) PACT 6, 224.

A. H. Weir, J. A. Catt and P. A. Madgett (1971) Geoderma 5, 131.

A. K. Singhvi and D. W. Zimmerman (1979) Archaeometry 21, 73.

R. Gardner and K. Pye (1981) Progress in Physical Geography, 5(4), 514.

N. C. Debenham and A. J. Walton (1983) Pact 9, (in press).

COMMENTS ON THE QUOTATION OF TL DATES

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Suggestions and comments on TL dates quotation have been made by authors after Aitken and Alldred (1972) and today publications such as "Ancient TL" (Sutton, 1981) or the "MASCA Journal" (Fleming, 1978) follow a derived format.

The basic features of this format are:

1. 1980 as a zero reference year
2. The use of two errors ($\pm$p, $\pm$ a) where p is the standard error on the average age of a set of sherds from the same context and a is the standard error for the context, absorbing all the various recognized sources of error, random and systematic.

We would like to point out a few problems in the light of radiocarbon dating experience. Both methods are linked because:

- intercomparisons will multiply with the growth of the TL method.

- TL is today one of the most important partners of radiocarbon in the range (7,000-40,000) for its calibration. (Dendrochronology is expected to reach the 11th millenium within the next years).

A consensus now exists for the presentation and calibration of radiocarbon dates (Klein et alii, 1982) and it is desirable that the juxtaposition of a ^{14}C and a TL date do not show any ambiquity. But it does! Actually BP means for ^{14}C that:

- the result has not been calibrated
- the zero reference year is 1950
- 5568 years half-life is taken for ^{14}C

So a ^{14}C and a TL date both given "BP" are absolutely not comparable. We suggest that this mention should be rejected in the TL field. Why not simply use "B.1980"?

"BC" and "AD" are very useful for historical archaeology but have no significance for geological events or civilizations without any relation with our calendar. Furthermore, the mention "B.1980" has the advantage to remind that the given number is more the result of a physical measurement than a date on the Gregorian calendar.

This later remark introduces our second comment on the TL dates quotation: we think that calculating the mean age of a set of sherds "taken in the same context" is an intrusion of the physicist in the archeological field. This proceeding comes to presuppose the contemporarity of the different samples and the physicists are not competent to deal with this question. In fact, this is never done with ^{14}C dates but by date-users which take their own responsibilities.

When the TL samples have evidently the same age (samples collected in, or under, a lava flow example), the mean age and p might be calculated and notified in the datation comments.

Endly, we fear that the date-users will find the double quotation quite heavy and that they will re-publish them in their own publications (i.e. archaelogical, historical...) with only one of the two with a human tendancy to choose the one which serves best their demonstration.

So, why not publish every single TL result with its own overall error and leave the averaging to date-users when they think they are allowed to?

Maybe it is untimely to think of a complete unification within the different dating techniques but now that the dating methods and laboratories are multiplying it is worthwhile to think of erasing the causes of errors and confusions. Our last proposal is to initiate a discussion about this problem now and conclude it at the next TL Specialist Seminar in Worms.

REFERENCES

Aitken M.J., Alldred J.C., 1972, Archaeometry 14, 257
Fleming S.J., 1978, MASCA Journal 1, 12-13
Sutton S.R., 1981, Ancient TL, 15, 6
Klein J., Lerman J.C., Damon P.E., Ralph E.K., 1982, Radiocarbon 24, n° 2, 103-105

The next issue of Ancient TL has some space still available for contributions. Research notes and additional comments on thermoluminescence date quotations are particularly of interest. Contributions should be sent to the editor before December 1, 1983, to insure inclusion in the upcoming issue.

LETTERS TO THE EDITOR

Comments on "Universidad Nacional de Ingenieria (Peru) Ancient TL Dates - 1983" (Ancient TL, v.1., n.1, 1983)

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I have had the opportunity of seeing your "Ancient TL", Vol.1, No.1 (January, 1983) and I have found information about a site in Huarmey which I excavated years ago. This information is not correct (p.9, c, Sample UNI-TL-4).

1) The correct situation of the site is 10°02´45"S,78°10´21"W.

2) The site is not Chavin de Huantar. The exact name is Los Gavilanes (PV35-1).

3) Fragments of the same sample delivered to the U.N.I. laboratories in Lima, have been treated by Laboratoire de Cristallographie et de Physique Cristalline of the Faculty of Sciences of the University of Bordeaux in France, and the dating was checked two times for security. The result was 4800 ± 500 years BP (BOR 20).

4) For control, samples of charcoal from the same strata was processed by the C14 method, and the date obtained was 4140 ± 160 (GX-5076). With Damons et al. calibration (I) the date become 4730 ± 90 years BP. This coincides with thermoluminescence dating.
The report of Rouanet (1976) quoted by López Carranza, et al., is only a thesis. But in any case the data are correct.

5) The archaeological context of the site is in complete agreement with those datings.

6) The results of my research in Huarmey, informing of the thermoluminescence dating was published in a preliminary report in Nature (II) and in a final report in my last book (III). In this book the reader can not only find the datings (p.73-75) but also a lengthy discussion on this topic (p.275-276; 280).

"References"

(I) Damons, P.E.; Ferguson, C.W.; Long, A.: Wallick, E.I. (1974) "Dendrochronologic calibration of the radiocarbon time scale" American Antiquity Vol. 30, No 2, part 1, April. Washington. pp.350-366.

(II) Grobman, Alexander and Bonavia Duccio. (1978) "Pre-ceramic maize on the north-central coast of Peru" Nature, Vol. 276, No. 5686, 23 November. London. pp. 386-387.

(III) Bonavia, Duccio. 1982
Preceramico peruano. Los Gavilanes. Mar,desierto y oasis en la historia del hombre.
Corporación Financiera de Desarrollo S. A. Instituto Arqueoloógico Alemán. Lima.

Author's Reply

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1. Prof. Bonavia is right in the correction of the exact geographical situation of the excavation site.

2. "Chavin de Huantar (?)" in our paper is a designation of the predominant Peruvian culture in the time 90 BC (our datation). I understood that in this place in the paper should appear a culture designation. The reference of the site PV35-1 is given also by us.

3. Our datation 90 BC was communicated to Professor Bonavia before publication. I showed him the first manuscript of our paper and he was unopposed to its publication. He assured me that the sample which we dated was not the same sample BOR 24 dated at Bordeaux. The last datation, 4979 years, was communicated in the second version of our paper but was dropped in the final version for reasons of space.

4. We believe that the Bordeaux datation (1976), communicated to us by Professor Bonavia, is right and our datation (1979) is also right, because it comes from a different sample (in the hypothesis which we assumed). The annual doses of both samples are almost the same but the difference in age comes from a factor 2 in the accumulated dose. We feel reasonably confident of the general correctness of our procedures. Dates from this laboratory agree in most cases with results from other labs obtained by different methods.

SOME RECENT BIBLIOGRAPHY *

W. Wang (1982) Ultrathin TLD system for beta dose determination in thermoluminescent dating. Kexue Tongbao, 27, 1139-1141.

H. Valladas (1983) Estimation de la temperature de chauffe de silex prehistoriques par leur thermoluminescence. C. R. Acad. Sc. Paris, 296, Serie II, 993-996.

For burnt flints, the thermoluminescence sensitivity to radiation of the 380°C peak increases with the heating temperature. Thus a flint that has been heated at 500°C in the laboratory emits, for a given dose, twice as much thermoluminescence as the same flint after heating at 400°C. This property makes it possible to propose a procedure to give an approximation of the temperatures reached in the past by prehistoric flints. This method has been applied to various Mousterian flints from Southern France and shows that they have been heated between 400 and 600°C.

S. Charalambous and F. Hasan (1983) Regenerated thermoluminescence. Phys. Letters A, 95, 259-262. A regenerated, non-radiation induced TL was observed. Results versus dose, storage time, storage temperature, maximum first heating, are presented. For the phenomenon a theoretical model is given, based on the

* Abstracts from the quoted papers are given here to inform readers on contents.

formation of a peculiar composite defect from migrated defects and trapped electrons.

S. V. Moharil and S. P. Kathuria (1983) On the general order kinetics in thermoluminescence. J. Phys. D, 16, 425-429.

In view of the recent debate on the order of kinetics in LiF TLD-100, the physical significance of the term is discussed. It is shown that the general order equation of May and Partridge is only empirical, and Antonov-Romanovski's equation is more appropriate to describe the non-integral order of kinetics. A method is proposed to obtain a parameter in this equation which is physically more relevant than the order of kinetics.

A. F. Skinner (1983) Overestimate of stalagmitic calcite ESR dates due to laboratory heating. Nature, 304, 152-154.

In the use of ESR for dating stalagmitic calcite from palaeolithic caves it has recently been reported that to obtain reliable ages the samples should be heated before measurement, for example, for 24 h at 170-190°C. Specifically this procedure has been used by Yokoyama et al. for samples from the lowest stalagmitic floor in Caune de l'Arago, the site of the Tautavel man in southern France, and an age of ~700 kyr obtained. However, the results presented here indicate that straight-line extrapolation involved in finding the age is not valid if this heating procedure is used and that its use leads to a substantial overestimate of the age, by nearly a factor of two in the application mentioned.

N. C. Debenham (1983) Reliability of thermoluminescence dating of stalagmitic calcite. Nature, 304, 154-156.

The dating of stalagmitic calcite at the early hominid site of Caune de l'Arago, France, by the ESR technique of Yokoyama, et al., has yielded ages for the underlying floor of $\sim 7\times10^5$ yr. These are in serious disagreement with the chronology obtained by thermoluminescence (TL), which indicates a date of around 3×10^5 yr. The discrepancy has been attributed to instability of the TL 280°C dating signal, for which a mean life of 2×10^5 yr was proposed. Here the arguments on which this claim is based are criticized, and evidence is presented that corrections to TL age measurements incurred by signal fading are, even for the oldest stalagmites from the site, probably <15%. The conflict between the TL and ESR chronologies at Caune de l'Arago is explicable in terms of the ESR results reported by Skinner.

A. V. Sankaran, K. S. V. Nambi and C. M. Sunta (1983) Progress of thermoluminescence research of geological materials. Proceedings of the Indian National Science Academy, 49A, 18-112.

The phenomenon of thermoluminescence (TL) in some minerals has been observed for many centuries, but its systematic and scientific evaluation started only during the fifties of the present century. When its potential applications in the field of geochronology was recognized, detailed basic studies regarding the build up, stability and several other facets of thermoluminescence were taken up. Over the past few decades, many applications of this phenomenon to probe geological problems and processes were attempted. Among these can be mentioned the studies on sediments, stratigraphy, ore-prospecting, palaeoclimates and meteorites. This paper reviews the observations made regarding thermoluminescence in a number of minerals and also its application in some of the important branches in geology. Pertinent areas in the context of Indian geology is also furnished at the end, along with the methodology in field collection and laboratory practice.

S. R. Sutton and G. Crozaz (1983) Thermoluminescence and nuclear particle tracks: Evidence of a brief transit time for ALHA-81005. Geophys. Res. Let., Vol.10, No.9, 809-812.

Thermoluminescence and nuclear particle track measurements were made on the Antarctic meteorite ALHA-81005. No nuclear particle tracks were found in lithic fragments indicating that the clast material never resided at the very surface of the parent body. The unusually low natural thermoluminescence of this material is interpreted as being due to a combination of anomalous fading and thermal decay. The thermal decay could be due to very long terrestrial age or heating either during atmospheric entry, in a near sun orbit or during a parent body impact event. Impact heating is considered the more likely of these possibilities for this meteorite. If the impact heating interpretation is correct the thermoluminescence data constrains the space exposure time of the object to be less than 2,500 years. Such a brief Earth transit time is consistent with a lunar origin for this meteorite.

G. Poupeau (1983) Recent advances in quaternary geochronology. Anais do XXXII Congresso brasileiro de Geologia, Salvador, Bahia, Vol.4, 1442-1450.

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 $\leq 10^6$ yrs) radionuclide measurements as with the ^{14}C or uranium series desequilibrium dating, and finally (iii) the emergence of entirely new dating approaches as the Electron Spin Resonnance Method. The aim of this paper is to review these progresses and the new areas they open to geochronology for the past-miocene times.

Two papers presented at the 1st International Symposium "Archeologic Africaine et Sciences de la Nature Appliquees a l'Archeologie" 26-30 Septembre, 1983, Bordeaux, France:

M. Schvoerer, F. Bechtel and P. Guibert, "Datation par Thermoluminescence de Céramiques Neolithiques Sahariennes Provenant d'Hassi Movillah et de Ti-N-Hanakaten (Algerie)."

P. Guibert, M. Schvoerer and F. Bachtel, "Datation par Gamma-thermoluminescence: Recherche Théorique et Nouveaux Resultats Expérimentaux."

Presentation at X^{éme} Congrés International d'Etudes des Civilixations Précolombiennes des Petites Antilles, Fort de France, Martinique, 25-30, Juillet, 1983:

M. Schvoerer, P. Guibert, F. Bechtel, M. Mattioni, and J. Evin. " Des Hommes en Martinique Vingt Siecles avant christophe colomb? ou, Contribution à la résolution, grâce à une nouvelle méthode de datation, la GAMMA-THERMOLUMNESCENCE (-TL, d'un probleème chronologique au Lorrain-Fond Brûlé (Martinique).

Extended abstracts of the preceding three papers may be obtained from:
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