ESR Dating Evidence on an Early Man's Dwelling at a Lower Palaeolithic Cave-Site in the Northern Caucasus

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The Caucasus is of special interest to researchers engaged in the study of early man, since this mountainous region's acknowledged to be one of the probable paths of man's penetration to Eastern Europe. Hence, his early appearance on the northern slope of the mountain range is still questionable because of the lack of undisturbed sites and reliable datings of archaeological finds. In the present paper the first comprehensive palaeodosimetric dating of cave-site deposits containing Early Palaeolithic lithic industries is reported. Eight terrestrial shell samples from recent excavations at the Triangular Cave (Northern Caucasus) were analysed by electron spin resonance (ESR) to produce a chronology for the most ancient Achshelian-bearing layer of the cave-site. The results suggest that the first (from the bottom) archaeological layer 7a is likely to be about 583,000 yr old, and the next, 5b, is some 705,000 yr old. These layers can be correlated with oxygen isotope stages 15 and 11, respectively. The estimates obtained imply that man presumably reached the Northern Caucasus at least as early as the beginning of stage 15, i.e., much earlier than generally recognized. The leaving of the cave by ancient man due to the development of glacial environment during the subsequent stage(s) can probably be linked with the penetration of man to the southern areas of the East European Plain.

INTRODUCTION

The problem exactly when and how the first inhabitants came to Eastern Europe has not yet been solved. Probably, there were several centres of settlement which sprang up at different times. The tribes migrated to Eastern Europe presumably from the southern (Caucasian), south-western and western directions. In all likelihood, the first inhabitants came to the Southern Caucasus from Western Asia. Evidence is derived from ancient dwelling places and characteristic archaeological material found there. Some researchers also assume that the Early Acheulian culture spread to the Northern Caucasus and the southern parts of the East European Plain, but the Early Palaeolithic chronology in these regions has remained virtually unknown.

Apparently, the reason is that most of the Palaeolithic monuments are represented by open-air sites. Archaeological evidence is derived from the surface of different relief elements. However, due to the evident reposition of the finds, they cannot serve as a reliable basis for stratigraphical conclusions or age determinations.

It seems that these Palaeolithic sites in the rock shelters could provide basic knowledge of the ancient population in the Caucasus. These sites which are normally in primary deposition, have a more reliable stratigraphical position and store valuable lithological, palaeontological and archaeological information. However, in dating the finds one will come across serious problems the age determination of a site on the basis of stone tool typology and manufacture technique is hardly possible; data on sites, especially under the specific natural conditions of the Caucasus, does not allow any categorical conclusions to be drawn; many archaeological sites are not datable by conventional dating methods at all since they are beyond the radio-carbon dating range and lack material which is suitable for other dating methods. That is the reason why the absolute age of the majority of pre-Wisianian Palaeolithic sites of the Caucasus is not yet established.

In the present work we are going to report on the first comprehensive palaeodosimetric study using ESR-spectroscopic analysis on terrestrial mollusc shells taken from the oldest cultural layers of a multi-level cave-site in the Northern Caucasus. The samples analysed come from excavations of the

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Triangular Cave which is currently acknowledged as the only site in Eastern Europe displaying finds of archaic pre-Monsterian industries (Golovanova, 1990, pers. comm.; Lysbin, 1993).

It is believed that as far back as the Early Achelian time the Greater Caucasus was an insurmountable obstacle in the path of ancient man's penetration from Western Asia to Eastern Europe. Therefore, the accurate dating of the oldest sediments from this cave would be of great importance, since it will essentially contribute to the understanding of cultural and environmental changes both in the Northern Caucasus and the Southern areas of the East-European Plain. On the other hand, the study of the relations of ancient man and his environment would yield valuable material for solving the problems concerning the climatic subdivisions in different regions of the continent.

THE DESCRIPTION OF THE CAVE

The Triangular Cave is a karst cavity formed on the Baraash limestone plateau. The cave was discovered by L. Golovanova and V. Dromichev in 1986. It is situated in the basin of the Urup River, 42°34' N, 41°12' E, at an elevation of 1510 m above sea level on the northern slope of the Greater Caucasus. Till the discovery of the cave only single Monsterian and Achelian artefacts had been found there at elevations less than 1000 m a.s.l. (Lysbin, 1989).

The excavations conducted in this cave since 1986 revealed the presence of a long sequence of palaeontologically and archaeologically rich deposits. The stratigraphic sequence of the cave reaches a cumulative depth of 4.5 m, and is characterized by a series of units labelled (from top to bottom) 1 to 8 (Fig. 1).

Fig. 1. A simplified stratigraphic section through the Triangular Cave site (after Dromichev, 1990, pers. comm.) Terrestrial mollusc shells for ESR-analysis were taken from Achelian-bearing layers 7a and 5b.
According to faunistic, lithological and archaeological data layer 1 is ascribed to the Late Holocene (the Middle Ages) (Doronichev, 1991); layer 2 which is related to the Holocene times is archaeologically sterile. Layers 3a and 3b (with redeposited Achaeolian artefacts) are ascribed to the Weichselian glaciation. Between units 3b and 4a there occurs a hiatus in the sedimentation. Layers 4a-c, 5a-c and lenses β and R are associated with the Mindel glaciation, layers 6, 7a and 7b – with the Günz-Mindel times. Layer 8 is the bottom of the cave. The lithic materials from layers 4a-b, 5a-b and 7a are classified as Early Achaeolian; layers 6a and 7b are archaeologically sterile.

The Achaeolian fauna of the cave was found to be common to the Tiraspol faunal assemblages of mammals (Baryshnikov, 1991). The presence of the Tiraspol fauna was interpreted as one representing warm climate in the vicinity of the cave (ibid).

In the lower part of the deposits numerous terrestrial mollusc shells were identified: *Chondrina crenata* (Kryn.), *Chondrina tridens* (Mull.), *Impressa pupoides* (Kryn.), *Monacha capensis* (Lindh.), *Pseudochondrina tuberifera* (O. Bitt.), *Quadriplicata aggera aggera* (O. Bitt.), *Sphenarium deliosum* (Bitt.).

**PALEODOSIMETRIC STUDY**

Up to the present a great number of papers have dealt with the investigations of marine mollusca remains (see e. g. Ikeya and Ohmura, 1981; Radke et al., 1985; Katzenberger et al., 1988; Skinner, 1989; Molodkov, 1986, 1988, 1989). There are only scanty data available on palaeodosimetric and radioscopical properties of non-marine mollusc shells, and even these are mainly the results of earlier studies on fresh-water gastropods (Molodkov et al., 1988; Molodkov, 1988; Ongelas and Molodkov, 1993). No data have been reported concerning the dating of terrestrial mollusc shells. The present work is the first effort to use these shells as dating material. Besides, it is the first attempt to date a Lower Palaeolithic site on the basis of ESR analysis of mollusc skeletal remains taken from Achaeolian-bearing layers.

Terrestrial mollusc shell fossils were collected in the cave during the excavations in 1990. Eight shell samples were selected for study.

The comparative study of marine and terrestrial shell ESR-absorption spectra, a comparison of the experimental absorption curves with a calculated Lorentz line of an individual component of the ESR spectrum (Fig. 2) (Molodkov, 1993) and also a series of other experiments (e. g. Molodkov, 1988, 1989) have shown, that paramagnetic centres with the g-factor at 2.0012 and the relevant signal are the most reliable for the quantitative analysis and dating of terrestrial mollusc shells. This signal is usually not detected in the multi-component differential spectra of the shells due to interference of signals from other centres, including those of non-radiation origin. However, it can be separated (Molodkov, 1988, 1993) and used for measuring centre concentration and establishing time of burial.

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**Fig. 2.** A calculated Lorentz line (g=2.0012) and the experimental ESR-absorption curve at g = 2.0012, separated in the ESR-spectrum of the terrestrial mollusc shells from the Triangular Cave.
Fig. 3. Arrhenius plot in $\tau$ vs $1/T$ of the $2.0012$ centre decay gives a life-time of $7 \times 10^8$ yr at $5^\circ$C for shells from Triangular Cave.

Fig. 4. A comparison of ESR signals of terrestrial mollusc shells from the Triangular Cave. Solid line: natural samples; dotted line: shells irradiated to a dose of $5$ kGy (a) or heated for $12$ h at $100^\circ$C (b). The spectra were recorded with different amplification factors.

Fig. 5. A theoretical plot of the $2 = 2.0012$ ESR signal of shells vs gamma-dose, $D_g$. The circles are experimental values of the $g = 2.0012$ signal intensity $I$ vs $D$. 70
Kinetic studies have shown that the life-time of the centres is extremely high in the shells from the cave and makes up about 3 x 10^6 yr at 5 °C (Fig. 3). In principle, it would be possible to cover the whole Acheulian with such shells without making a long-term fading correction (Molodkov, 1989).

Irradiation at a dose of 3 kGy and heating at 180 °C for 12 h have revealed the similarity between life-times of the spectral components in the region of interest as well as the similarity of dose dependencies of the signals up to the saturation dose (Fig. 4).

Sensitivity to irradiation in shells from the cave deposits makes up about 12 x 10^4 Gy, i.e. it is approximately twice as high as for marine shells from the Arctic region (Molodkov, 1989).

The signal vs. dose dependence of the shell substance in the range of the doses applied was well described by an exponential function and reached the level of 0.95 from saturation value Ip at about 2.5 kGy (Fig. 5). It secures the correct determination of the accumulated palaeodose, Pe, and hence, the reliable dating of the shells.

Research into the shells from the Triangular Cave showed that the mineralogical composition, even of the oldest shells, is represented by aragonite. It means that the original structure of the skeletal substance was preserved. This fact is of great consequence, because the loss of accumulated age information due to inversion of the initial crystalline structure can be eliminated.

ESR-DATING

The principles of the ESR-dating of shells have been described elsewhere (see e.g. Molodkov, 1986, 1988, 1989, 1993). Briefly, ESR-dating is based on the direct measurement of the amount of radiation-induced paramagnetic centres (radiation damage) in shell substance. These centres are created by natural radiation resulting from radioactivity in the shell itself, and in the environment (embedding and cosmic matrix). The concentration of stable CO3²⁻ type carbonate paramagnetic centres in mollusc shell substance is determined from ESR signal intensity. The signal is absent in modern shells, but increases in intensity as a function of the total radiation dose absorbed by the shell over the time of burial.

The dose rate of natural radiation is determined by an analysis of the natural radioactivity of the shell and its immediate environment, while the absorbed dose is determined by ESR.

The details of sample preparation were basically the same as described in earlier works (e.g. Molodkov, 1986). To determine the unknown value of accumulated palaeodose, fraction samples of shells cleaned, etched, powdered and sieved to 75–400 µm were divided into 8 aliquots 100–200 mg each, and then irradiated from 96 to 1,000 Gy using a Co source delivering 1.6–2.6 x 10^7 Gy s⁻¹. The maximum artificial dosage was 5 kGy. For the reconstruction of the growth curve outside the additive dose section, a logarithmic transformation of dose vs. ESR intensity was made.

Typical spectra of the shells from the Triangular Cave recorded by a microwave spectrometer of 1 mW and a modulation amplitude of 0.25 x 10⁻¹⁰ T are shown in Fig. 4. To separate the analytical line at g = 2.0012 the overmodulation (OM) method was used according to Molodkov (1988, 1993). Dosimetric reading was performed with a power of 2 mW and a modulation of 1 mT.

U concentration in the shells was measured by neutron activation analysis. The external dose rate was deduced from the gamma-spectrometric analysis of the embedding matrix (Molodkov, 1986) using the conversion factors of Nambu and Aitken (1986) and taking into account shell geometry and water content in the sediments.

The cosmic dose rate was calculated following Prescott and Stephane (1982), also according to the burial half-depth of the sediment and the thickness of the rock projecting over this site. It is estimated to be about 50 μGy a⁻¹.

RESULTS AND OBSERVATIONS

Table 1 presents the results obtained from the samples from the cave-site. Errors in individual dates amount to ca. 10%. The mean ages based on dating syntgenic samples are 368 ± 25 and 393 ± 27 kyr BP for layers 7a and 5b, respectively. The ages obtained for different species agree well within each layer and increase with depth. The deviations from the mean ages are rather small in both cases. It demonstrates that the actual inexactness in ESR estimates may be much less than it first appears from error determinations which are derived from the assessment of all known sources of error.

The uranium content in the shells seems to be highly uniform, ranging from 0.38 to 0.53 ppm, indicating that little or no post-depositional U enrichment has occurred. The internal dose rate is much less than the external one. It suggests a negligible inexactness which is connected with the unknown diagenetic history of shell substance.

Due to the extraordinary high thermal stability of the centres at g = 2.0012 in the shells studied...
(≈3 × 10⁵ yr), the dating of the embedded deposits was performed without taking into account the long-term fading of the absorbed palaeodose.

The age of culture-bearing layer 7а has been determined by six shell samples. On the average it makes up 583 ± 25 kyr BP. The finds of subfossil in the layer suggest forestation in the vicinity of the cave-site and a warm climate (Darynyshkin, 1991). The data obtained allows to link the first cultural layer of the cave with oxygen isotope stage 15 and to correlate it with the time of soil formation in the Chinese and East-European loess-palaeosol sequences (Fig. 6). It can also be correlated with a corresponding high-sea-level event fixed by previous USR studies of marine deposits on the Arctic islands (Molodkow et al., 1992; Makeev et al., 1992). Our dating results suggest that man made his first appearance somewhere in the early part of stage 15, i.e. soon after the global climatic amelioration and the subsequent decay of the mountain ice domes in the Caucasus.

The age of the next culture-bearing layer, 5b, is 393 ± 27 kyr BP. According to palaeoecological data the layer could have accumulated under warm climatic conditions (Darynyshkin, 1991) (see Fig. 6) or during the initial phase of cooling in the final period of an interglacial event in that area (Doronichev, 1992, pers. comm.). These observations and dating results are in agreement with the data on the Arctic islands, where deposits of the regressive phase of Polar basin seas were dated by ESR at 385 ± 37 kyr BP (Molodkow et al., 1987).

Therefore, the archaeologically sterile layer 6, sandwiched between cultural layers 7а and 5b, may be indicative of the time when man had to leave the cave due to a remarkable cooling of the climate, during which the altitude of the snowline considerably fell down to 800-1000 m. Probably, it made man move down to valleys and foothills. At the same time regressive phases of the Mediterranean–Black Sea system resulted in the drainage of the Don–Drut river-strait that opened the way for man's settlement in the Southern areas of the East-European Plain (Fig. 7). The subsequent climatic amelioration during the interglacial period promoted the further penetration of man deep into the plain. The aforementioned has been confirmed by the finds of stone-arts at the sites of the Lower Don River terraces (Mikhalitsova and Kuchenchich) and the Azov sea coast (Gerashinokva) (Fig. 7), deposited together with fossils of the Tiranopolian mammal fauna and Black Sea molluscs, both related to the Early-Middle and the Middle-Middle Pleistocene.

CONCLUSION

The present work is the first effort to use a variety of species of terrestrial gastropods as dating material for Lower Palaeolithic archaeology. The results obtained for Achaeolian-bearing deposits of the Triangular Cave are interesting from two aspects. On the one hand, the study shows that:

1. the signal at g = 2,0012 ± 0,0001 (linewidth = 1.25 × 10⁻⁴ T at the microwave power of 100 mW) seems to be the only signal suitable for the concentration analysis and dating of terrestrial molluscan shells;
2. the life-time of the relevant centre is about 3 × 10⁵ yr at 5°C; it allows to cover the whole Achaeolian with such shell material even without making correction for long-term fading;
3. the lifetimes of the other radiation-induced components of the spectrum seem to have the same order of magnitude that favours the quantification of the 2,0012 centre concentration;

<table>
<thead>
<tr>
<th>Layer</th>
<th>Sample No.</th>
<th>Environmental concentrations of radioisotopes</th>
<th>U-content in shells</th>
<th>Thickness of shells</th>
<th>Removed</th>
<th>Dose rate (µGy a⁻¹)</th>
<th>Palaeo- dose Dp (kGy)</th>
<th>CSR- dose T (ka)</th>
</tr>
</thead>
<tbody>
<tr>
<td>7а-1</td>
<td>161-062</td>
<td>0.91</td>
<td>3.55 ± 0.61</td>
<td>51</td>
<td>270</td>
<td>20</td>
<td>910 ± 98</td>
<td>720 ± 570</td>
</tr>
<tr>
<td>7а-2</td>
<td>162-062</td>
<td>1.07</td>
<td>2.31 ± 0.43</td>
<td>43</td>
<td>280</td>
<td>20</td>
<td>796 ± 171</td>
<td>610 ± 600</td>
</tr>
<tr>
<td>7а-3</td>
<td>163-062</td>
<td>0.91</td>
<td>3.55 ± 0.61</td>
<td>61</td>
<td>250</td>
<td>20</td>
<td>1024 ± 147</td>
<td>745 ± 610</td>
</tr>
<tr>
<td>7а-4</td>
<td>164-062</td>
<td>1.07</td>
<td>2.31 ± 0.43</td>
<td>42</td>
<td>250</td>
<td>20</td>
<td>807 ± 170</td>
<td>560 ± 545</td>
</tr>
<tr>
<td>7а-5</td>
<td>166-062</td>
<td>1.07</td>
<td>2.31 ± 0.43</td>
<td>31</td>
<td>180</td>
<td>20</td>
<td>836 ± 114</td>
<td>565 ± 565</td>
</tr>
<tr>
<td>7а-6</td>
<td>167-062</td>
<td>0.65</td>
<td>3.36 ± 0.51</td>
<td>36</td>
<td>250</td>
<td>20</td>
<td>886 ± 144</td>
<td>660 ± 610</td>
</tr>
</tbody>
</table>

Mean age for layer 7а: 383 ± 25 kyr

| 5б   | 171-062     | 1.43                                        | 4.52 ± 0.76         | 53               | 150     | 20                | 1420 ± 198       | 700 ± 420     |
| 5б   | 172-062     | 1.43                                        | 4.52 ± 0.76         | 43               | 250     | 20                | 1300 ± 152       | 570 ± 365     |

Mean age for layer 5б: 393 ± 27 kyr
Fig. 6: A comparison of ESR-chronologies for the Triangular Cave (triangles) and raised marine deposits (circles) with the main Pleistocene events. Correlation chart after Molodkov, 1990, unpubl., 1992.
4. The sensitivity of the shells to irradiation is about $12 \times 10^4 \text{ Gy}^\text{2}$.

5. The dose-effect curve of the 2.9012 signal is well described by an exponential function and reaches the level of 0.95 $D_{\text{lim}}$ at about 2.5 kGy.

On the other hand, ESR results show that ancient man came to the Northern Caucasus no later than 583 ± 25 kyr B.P. i.e. at least close to the beginning of oxygen isotope stage 15. For the second time the cave was occupied about 393,000 years ago, apparently during stage 11. Thus, the occupations of the cave during stages 15 and 11 were probably coeval with the global ameliorations of the climate in those periods. The absence of any sign of man's dwelling in the cave during stages 14–12 may be indicative of unfavourable living conditions in the vicinity of the cave due to a cooling of the climate in that region. This time interval can probably be linked with the penetration of ancient man into the southern areas of the East-European Plain.

Finally, the first attempt to date shell material from an ancient cave-site by ESR demonstrates the possibilities of the new method in studying Palaeolithic cultures.

References
