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SCIENCE DIRECT
Quaternary International 144 (2006) 61–67

ESR dating of glacial tills and glaciations in the Urumqi River headwaters, Tianshan Mountains, China

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Available online 7 October 2005

Abstract

Electron spin resonance (ESR) dating of the Shangwangfeng, the Xiawangfeng, and the Gaowangfeng tills in the headwaters of the Urumqi River was carried out using Ge centers in quartz grains. The Shangwangfeng till is dated at 35 ± 3.5 ka BP. Three dates from the lower portion of the Xiawangfeng till are 171.1 ± 17, 176 ± 18, and 184.7 ± 18 ka BP, respectively, and the age of the Gaowangfeng till is 459.7 ± 46 ka BP. Considering the available ages (i.e. ^{14}C, TL and ESR) and the principles of geomorphology and stratigraphy, the Shangwangfeng till is determined to be deposited in marine isotopic stage 2–3 (MIS2–3). The upper part of the Xiawangfeng till was formed in MIS4 and the lower part was deposited in MIS6. The Gaowangfang till is the oldest at the head of the Urumqi River, corresponding to MIS12. The age of the Gaowangfang till also demonstrates that the Tianshan Mountains lay at a suitable altitude for a glacial climate at that time, when the glaciers on this segment of the mountain began to develop.

1. Introduction

In recent decades, methods utilizing radiation-defects in quartz (i.e. electron spin resonance (ESR), thermoluminescence (TL), and optically stimulated luminescence (OSL) dating techniques) have developed rapidly and are well suited to determine the ages of terrestrial sediments. ESR has advantages compared to the others: a wider dating scope (from several thousand to several million years); ubiquitous datable materials (fossils, oceanic and terrestrial deposits, meteorites, etc); small samples required (less than 1 g quartz for some special samples) and simple preparation. It is also a nondestructive method, and the samples may be reused for other purposes. An ESR age can be derived from the following formula:

$$\mathrm{TD} = \int_0^t D(t)\,\mathrm{d}t,$$

where TD is the total dose the sample has accumulated over time and D is the annual dose rate that is generated by the radioactive elements (U, Th, and K_2O) in the sample (internal dose rate) and its surroundings (external dose rate) as well as cosmic rays. TD is determined by the additive artificial dose method, and the annual dose is derived from the concentration of the radioactive elements (U, Th, and K_2O) in the sample and its surroundings.

ESR dating has been applied in geology (Hennig and Grün, 1983; Grün, 1989; Ikeya, 1993; Rick, 1997). Dating of unconsolidated sediment by ESR was proposed by Yokoyama et al. (1985). Tanaka et al. (1986) chose Ge centers to date sun-bleached sediment and obtained reasonable geological results. Grün (1991) proposed that Ge centers should be used in future ESR dating studies. Schwarcz (1994) suggested that glacial till was suitable for future ESR studies. Many scholars (Kuang et al., 1997; Shi et al., 2000; Wu et al., 2001; Yi et al., 2001; Zhou et al., 2002b) have applied the ESR technique to investigate glacial tills, and obtained

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1040-6182/$ - see front matter © 2005 Published by Elsevier Ltd.
doi:10.1016/j.quaint.2005.05.013

reliable results, indicating that these quantitative experimental techniques have replaced a conventional qualitative descriptive base in the studies of Quaternary glaciations at present, and without doubt will promote further progress.

Quaternary glacial landforms and moraines at the headwater area of the Urumqi River in the Tianshan Mountains have been studied extensively since the 1960s (e.g. Shi and Su, 1965; Yang and Chiu, 1965; Cui, 1981a, b; Cui and Xiong, 1989; Li et al., 1981; Wang, 1981; Ma, 1984; Feng and Qin, 1984; Qin et al., 1984; Li, 1995; Yi et al., 1998, 2001), but controversial conclusions about these tills are still in existence, especially for the Xiawangfeng till. These controversies restrict understanding of the paleo-climate in this region. In this paper, we present our further investigation results from the Shangwangfeng, the Xiawangfeng, and the Gaowangfeng tills at the headwaters of the Urumqi River and try to present a concept about the Quaternary glaciations in this area.

2. Geological setting

The head of the Urumqi River is located on the north side of Mt. Kelawucheng (43°7′N, 86°49′E) in the central Tianshan Mountains. The heights of mountain ridges are 4100–4300 m a.s.l., with the highest peak being Tianger (4486 m a.s.l.). Modern snowlines at 4000–4100 m result in development of glaciers here, including cirque glaciers, hanging glaciers, and small valley glaciers. The No. 1 Glacier is a small valley glacier. Some tongues extend more than 300 m below the snowline, with the terminals at 3650–3700 m. There are 124 glaciers around the Daxigou River, the key research area and the primary tributary of the Urumqi River, with a total area of 35.7 km^2, accounting for 77.6% of the whole Urumqi River's drainage basin. There are five sets of Quaternary glacial tills in the Daxigou valley (Fig. 1). The glacial landforms are very conspicuous from the No. 1 Glacier to the station of the Wangfeng road maintenance squad. Three end moraines are present 250, 460, and 530 m from the terminus of the glacier No. 1. The surface of the sediments is little weathered and striated erratics can be found. There is neither soil developed on these moraines nor vegetation covering them except moss and other pioneering plants. The lithology is schist, gneiss, granitic diorite, and greenstone. According to the lichenological dating, the moraines were deposited 1538 ± 20, 1777 ± 20, and 1871 ± 20 a AD, respectively (Chen, 1989). AMS ^{14}C ages are 390 ± 211 and 420 ± 150 a BP (Yi et al., 1998), indicating that they are glacial tills of the Little Ice Age. The second set of tills, found around the Tianshan Weather Station and the Glacier Observatory, about 1 km downwards from the tills of Little Ice Age, and terminating at about 3500 m a.s.l., is the Shanbei Group. A thin soil layer with grasses has developed. The lithology is similar to that of the Little Ice Age moraines. ^{14}C dates from the Shanbei Group till are 5680 ± 150, 4080 ± 150, and 3949 ± 141 a BP (Zheng and Zhang, 1983), and AMS ^{14}C ages are 1860 ± 110 a BP (inner layer of carbonate coating) and 6560 ± 150 a BP (outer layer of carbonate coating) (Yi et al., 1998), illustrating that they formed along the Neoglaciation.

The Shangwangfeng, the Xiawangfeng, and the Gaowangfeng tills were deposited during the different periods of the Pleistocene. The Shangwangfeng till and the Xiawangfeng till are distributed in the lower U-shaped valley, from the Luobudaogou's to the Wangfeng road maintenance squad's station. The Shangwangfeng till terminates at about 3000 m a.s.l., with a thin gray soil layer underlain by a 0.5 m loess layer. There is an overthrust unconformity between the Shangwangfeng and the Xiawangfeng tills, which is convincing evidence to demonstrate that they are formed at different glaciation periods. The Xiawangfeng till is present in the 3.5 km long U-shaped valley around the Wangfeng road maintenance squad and its terminus is at about 2900 m a.s.l. Around the house of the

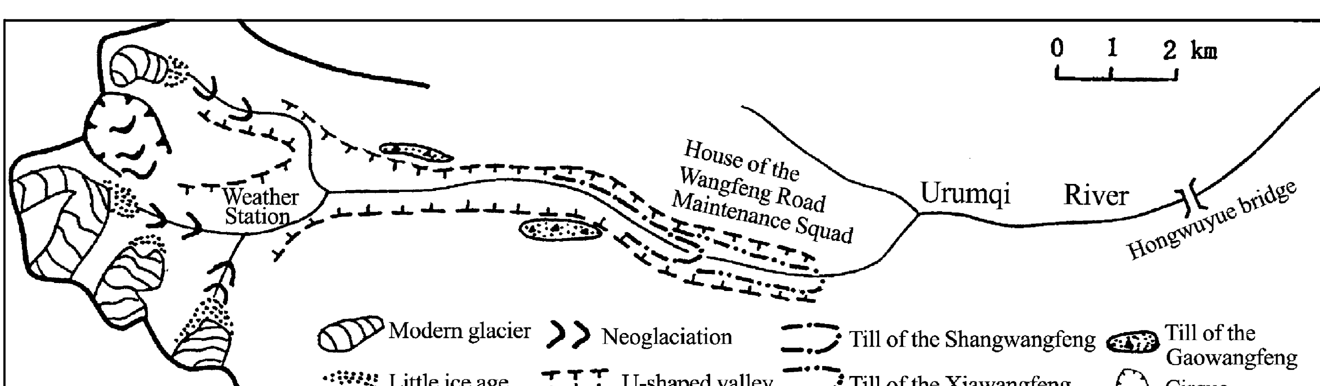

Fig. 1. Distribution of Quaternary glacial features in the Urumqi Valley, Tianshan Mountains.

Wangfeng road maintenance squad, a moraine platform has a total thickness of about 110 m, covered by a brown soil layer and grasses. The lithologies of the Shangwangfeng and the Xiawangfeng tills are similar, with the main ingredients of granite, gneiss, and granitic diorite. The Gaowangfeng till has no sedimentation relationships with the Shangwangfeng and the Xiawangfeng tills. The original shape of the landform was destroyed, and only about 10 m in thickness was preserved in the upper U-shaped valley, about 200–300 m above the river bed. Therefore, the distribution elevation of the Gaowangfeng till is about 3400 m a.s.l. Taupe soil developed on it, covered with alpine meadow. The main lithological ingredients of the Gaowangfeng till are granite, schist, gneiss, granitic diorite, gabbro, and quartzite.

The samples for dating were collected from artificial and natural sections of the Shangwangfeng, Xiawangfeng, and Gaowangfeng tills (Figs. 2 and 3). Samples were stored in the opaque bags and sent to the

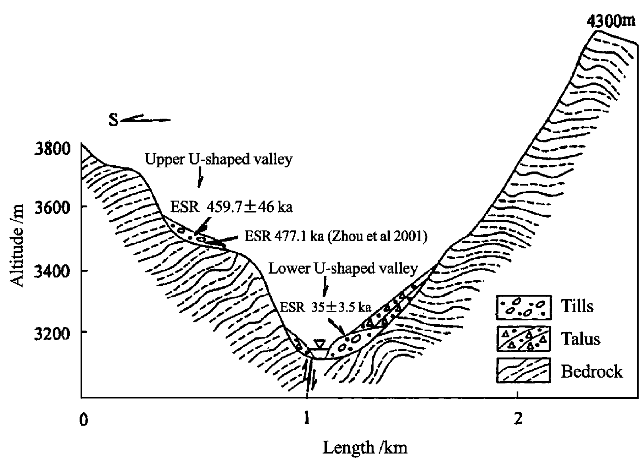

Fig. 2. Cross section near Gaowangfeng at the head waters of the Urumqi Valley.

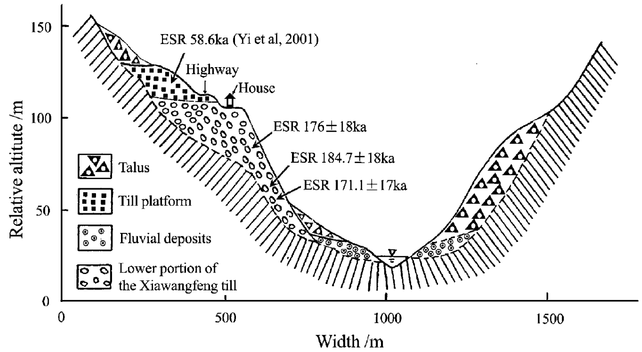

Fig. 3. Xiawangfeng section near the Wangfeng road maintenance squad's house and ESR ages.

laboratory, in order to avoid direct illumination by sunlight in the field.

3. Analytical procedures and results

3.1. Sample preparation

The preparation was carried out in the chronology laboratory, Department of Geography, Lanzhou University. The preparation conditions were not as strict as those required for TL and OSL because the collected samples could be dealt with in a lit laboratory (Schwarcz, 1994) and all samples were treated under natural light conditions. The following procedures were: (1) Division of each sample into two. One function was dried in a constant temperature apparatus at 105 °C to measure the water content and then ground in an agate mortar and sieved through a <0.1 mm screen. The disposed samples were used to determine the concentrations of radioactive elements (U, Th, K_2O, etc). (2) The other fractions were washed in water several times to remove unbound clay and silt and then sieved on screens to isolate the 125–250 μm fraction, which were treated with H_2O_2, to remove the organic materials. The grains were soaked in 6 mol/l hydrochloric acid (HCl) for 2 weeks, in order to remove the calcium carbonate and other substances that could be dissolved. These grain fractions were then subjected to ultrasonic washing to remove very fine-grained materials that were bound to the surface of grains. (3) The samples were soaked in concentrated hydrofluoric acid (HF) for 2 h to etch the outer 10 μm surface layer, which is commonly damaged by α rays. This treatment also helps to concentrate the quartz grains by dissolving impurities. (4) The samples were thoroughly washed and dried in a constant temperature apparatus at 40 °C. Magnetic minerals were removed using a magnetic separator at 1.0~2.0 A. Samples were divided into 9 aliquots (about 250 mg each) and irradiated with an artificial ^{60}Co-source with different doses at the Institute of High Energy Physics, Chinese Academy of Sciences. The doses were monitored with alanine/ESR dosimeters.

3.2. Determination and results

The irradiated samples were kept untouched for more than 2 months. Ge centers were chosen to date signals, measured with an ECS106ESR spectrometer manufactured by Bruker (Germany), with the following measurement conditions: room temperature, X-band, microwave power: 2 mW; modulation amplitude: 0.1 mT; the central magnetic field: 348 mT; sweep width: 5 mT; change time: 5.12 mS; time constant: 40.96 mS; amplification: 1×10^5. The typical ESR spectra of the quartz samples are shown in Fig. 4.

3.2.1. TD determinations

Least-squares analysis was used to fit the data points on the basis of the different artificial irradiation doses and the corresponding signal intensities and linear fit was chosen here. The curve was then extrapolated to zero and to obtain the total dose (TD) (Fig. 5).

3.2.2. D determinations

The annual dose is composed of internal doses and external doses:

$$D_{in} = D_{in}\alpha + D_{in}\beta + D_{in}\gamma,$$

$$D_{ex} = D_{ex}\alpha + D_{ex}\beta + D_{ex}\gamma + D_{com},$$

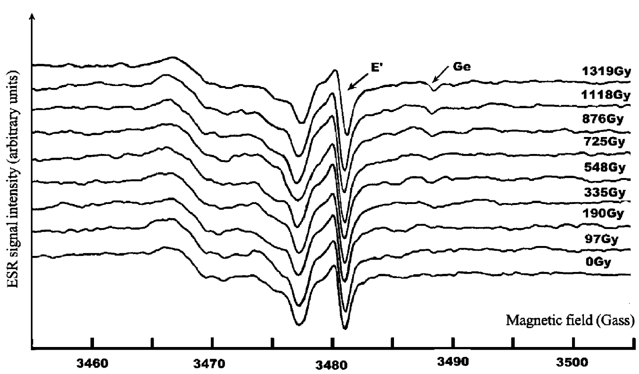

Fig. 4. Typical ESR spectra at room temperature.

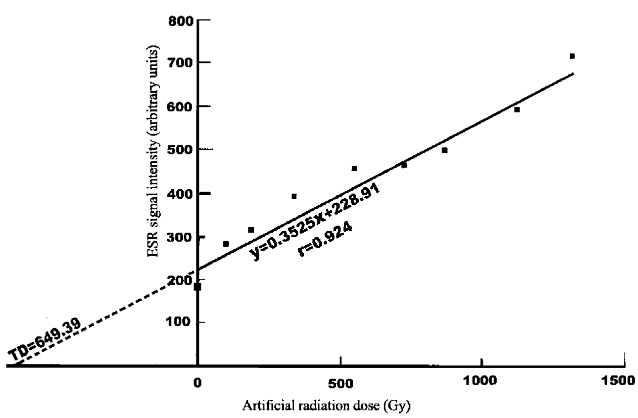

Fig. 5. Best-fit line between artificial radiation doses and ESR signal intensity of Xiawangfeng till.

$$\begin{aligned} D_{ex} &= D_{ex} + D_{in} \\ &= (D_{ex} + D_{in})\alpha + (D_{ex} + D_{in})\beta \\ &\quad + (D_{ex} + D_{in})\gamma + D_{com}, \end{aligned}$$

where D_{in} is the internal dose, D_{ex} is the external dose, and $(D_{ex} + D_{in})\alpha$, $(D_{ex} + D_{in})\beta$, $(D_{ex} + D_{in})\gamma$, and D_{com} are the contributions of α-rays, β-rays, γ-rays, and cosmic-rays, respectively.

The concentrations of radioactive elements, U, Th, and K_2O, were determined by laser fluorescence, colorimetric spectrophotometry, and atomic absorption, respectively. The annual dose was estimated with these radioactive elements. The purity of the quartz grains was high and the concentration of radioactive elements in quartz was below the range of measurement. The internal dose contributions were not taken into account in estimating the annual dose. More than 10 μm of the surficial layers were etched by HF. Therefore, the contribution of α-rays was ignored. The cosmic rays were controlled by longitude, latitude, and elevation, and the contributions lessened with sample depth. In addition, as all samples were collected from the artificial and natural sections several meters below the top, the contribution of cosmic rays can also be neglected. The poorly sorted and porous glacial till results in quick diffusion of the radioactive gases (e.g., radon). Thus, the formula can be simplified as

$$D = D_{ex}\beta + D_{ex}\gamma.$$

All the parameters and the results are listed in Table 1.

4. Discussion and conclusion

ESR dating of sediments is based on the assumption that paramagnetic centers in quartz grains are bleached when they are heated, or collided, or exposed to the sunlight, or re-crystallized, or the other geological agents acted on them. After deposition of sediments, the paramagnetic centers are formed again by radiation emitted from natural radioactive elements and cosmic rays, and then it changes over time. Previous studies (Tanaka et al., 1986; Buhay et al., 1988; Ye et al., 1993, 1998) indicated that Ge centers in quartz grains were sensitive to sunlight or grinding, and these mechanisms could bleach them. Tanaka et al. (1986) suggested that

Table 1
Results of ESR dating and the correlated parameters

Samples	Water (%)	U (10^{-6})	Th (10^{-6})	K_2O (%)	TD (Gy)	Age (ka)
SWF	5.01	2.07	12.2	3.03	140.5	35±3.5
XWF-1	4.73	2.18	10.3	2.77	649.4	176±18
XWF-2	1.9	2.58	12.9	2.58	727.6	184.7±18
XWF-3	7.83	2.45	13.6	2.91	682.6	171.1±17
GWF	7.79	1.99	13.3	3.2	1884.8	459.7±46

the ESR signal at a g-value of 1.997 (which is thought to be associated with the Ge center) disappeared completely after only 7 h of sunlight irradiation. Subsequently, Buhay et al. (1988) and Ye et al. (1993) pointed out that a Ge center could bleach within a few hours exposed to ultraviolet (UV) lamp or sunlight. Ye et al. (1998) showed that grinding could bleach the Ge centers. According to the theory of glacier movement, the materials the glaciers carried could be exposed. During glacier movement, the subglacial and englacial materials are subject to grinding, and most fine sediment formed in this way. Accordingly, the Ge centers in the quartz grains were bleached before the sediments were deposited, and tills meet the dating prerequisite.

Quaternary glacial landforms and moraines at the headwaters of the Urumqi River have been studied extensively. Former scholars have used different dating techniques to compare the dating results of the investigated glacial tills and interrelated sediments (all the results are listed in Table 2). It is believed that the isotope record of the marine sediments can be used to refer to the changes of the climate and ice volumes on the land. Accordingly, previous scholars have compared their results in the study area with the record of marine isotopic stages (MIS) (Wang 1981; Yi et al., 2001; Zhou et al., 2001, 2002a). Although many dating techniques have been applied to date glacial tills, the reliability of the results presented here still needs to be discussed in detail.

The Shangwangfeng till and the Xiawangfeng till were distributed in the lower U-shaped valley of the study area, with the convincing evidence of an overthurst unconformity between them to distinguish the two sets of tills. As the Shangwangfeng till is looser than the Xiawangfeng, it was deposited later. The Xiawangfeng till around the Wangfeng road station can be divided into two parts, upper and lower. The field investigations confirmed that the cementation of the lower part was harder than the upper one, illustrating that the Xiawangfeng tills were deposited in different periods. The Gaowangfeng till in a higher position of the upper U-shaped valley demonstrates that a much earlier glaciation occurred before the formation of the lower U-shaped valley and they were the oldest tills in this area. The wide ESR dating ranges from young to old, without a reversal, reveals these tills were deposited at different glaciations on the basis of the geological setting.

The available ^{14}C date (14 920 ± 750 a BP) (Wang, 1981) and AMS ^{14}C dates (19 000–23 000 a BP) (Yi et al., 1998) of the Shangwangfeng till suggested that it was formed in the later period of the Last Glaciation, corresponding to MIS2. However, our measured age (35 ± 3.5 ka BP) is earlier than previous ^{14}C and AMS ^{14}C ages, showing that the Shangwangfeng till is correlated to MIS3. Discussion is required as to whether our result is accurate or if the Shangwangfeng till was actually deposited during this period. According to the marine isotopic record and corresponding climatic characteristics, MIS3 can be divided into three substages (a, b, and c). Current studies suggest that MIS3, especially of MIS3b (44–54 ka BP), was a cold and wet

Table 2
Different dating techniques and the ages of tills and interrelated sediments

Sampling sites	Dating techniques	Results and sources	Glaciations or MIS
Between the No.1 glacier and glacier observatory	Lichenology	1871 ± 20 a AD; 1777 ± 20 a AD; 1538 ± 20 a ± AD Chen (1989)	Little Ice Age
	AMS ^{14}C	390 ± 211 a BP and 420 ± 150 a BP Yi et al., 1998	
Glacier observatory, meteorological station and shanbei group	AMS ^{14}C	6560 ± 150 a BP (inner layer of coating); 1860 ± 110 a BP (out layer of coating) Yi et al., 1998	Neoglaciation
	^{14}C	5680 ± 150 a BP 4080 ± 150 a BP (Shanbei Group I) and 3949 ± 141 a BP (Shanbei Group II) Zheng and Zhang (1983)	
Shangwangfeng	^{14}C	9170 ± 400 a BP (loess above the till), 14920 ± 750 a BP Wang (1981)	MIS2-3
	AMS ^{14}C	19010 ± 450 a BP, 23080 ± 510 a BP Yi et al., 1998	
	ESR	27.6 ka BP and 37.4 ka BP Yi et al., 2001	
		35 ± 3.5 ka BP	
Xiawangfeng	TL	37.7 ± 2.6 ka BP (fluvial sand at the Xiawangfeng tills' bottom) Li (1995)	MIS2
	ESR	54.6 ka BP, 56.6 ka BP, 58.6 ka BP, 72.6 ka BP, 40.1 ka BP (fluvial sand) Yi et al., 2001	MIS4
		176 ± 18 ka BP, 184.7 ± 18 ka BP, 171.1 ± 17 ka BP	MIS6
Gaowangfeng	ESR	477.1 ka BP Zhou et al., 2001	MIS12
		459.7 ± 46 ka BP	

period, which caused glacial advance (Shi and Yao, 2002). The investigations carried out on the south slope of Himalayas indicated that the extent of glaciation during MIS3 is greater than the general Last Glacial Maximum (LGM) (Owen et al., 2002a, b; Zech et al., 2003; Kamp Jr. et al., 2004). Two other ESR ages of the Shangwangfeng till determined by Yi et al. (2001) showed that the upper till's age is 37.4 ka BP, which is coincident with our result, and the underlying one is 27.6 ka BP. Thus, our measured age of the Shangwangfeng till should be believable. It is reasonable to infer that the Shangwangfeng tills were deposited in MIS2–3 based on the above ages. The great debate about the Xiawangfeng till has not been fully solved. Wang (1981) inferred it was formed in MIS6, but Li (1995) thought it was deposited in a later period of the Last Glaciation. Yi et al. (2001) concluded that it was deposited in MIS4 on the basis of ESR ages (50–70 ka BP). We found that the Xiawangfeng group could be divided into an upper and lower portion. The lower portion was poorly preserved, most of it was destroyed, and only a section around the station of the Wangfeng road maintenance squad was well preserved. Three samples were collected from this section and the dating results were consistent. Two circumstantial pieces of evidence confirm the believability of the ages: Zhou et al. (2002a) determined the ESR age of the second outwash terrace around Erying in the Houxia wide valley is 125.6 ± 13 ka BP and the TL age of the bottom of the loess which covers the outwash terrace is 90.0 ± 7.5 ka BP. The ESR age of the third outwash terrace which developed at the exit of the Urumqi River Valley is 114.4 ± 11 ka BP. These outwash terraces and their ages show the presence of MIS6 glaciation. Thus, the upper Xiawangfeng till should have been deposited in the early period of Last Glaciation, corresponding to MIS4, and the lower portion around the station of the Wangfeng road maintenance squad was formed in MIS6.

The Gaowangfeng till was distributed in the upper U-shaped valley, about 150–200 m above the lower valley and 200–300 m above the river. The position of the till means an older age of its formation. The other available ESR age is 477.1 ka BP dated by Yi Chaolu (Zhou et al., 2001). These two results are consistent, indicating that the Gaowangfeng till was formed in MIS12.

Acknowledgements

The authors would like to thank Jiao Keqin, Yang Huian, Xu Liubing, and Cui Jianxin for their fieldwork assistance, Diao Shaobo who helped to date the samples in the Institute of Marine Geology, the Ministry of Geology and Mineral Resources, Qingdao, and the anonymous scholars who reviewed and revised the manuscript. This work was supported by the Hundred-Talents (2002-43) Programs, Chinese Academy of Sciences, and by the National Natural Science Foundation of China (Grant nos. 40371013, 90202013, 90302006), and the Knowledge-Innovation (2004102).

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