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ELECTRON SPIN RESONANCE (ESR) SIGNAL INTENSITY OF QUARTZ E_1' CENTRE AND ITS POTENTIAL USE IN FLUVIAL SEDIMENTS PROVENANCE TRACING

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Abstract: To assess if the heat-treated E_1' centre in quartz is suitable for fluvial sediment provenance tracing, eight granitic samples from sediments and their source rocks with distinct geological ages and origins were collected for electron spin resonance (ESR) measurements. The results show that the maximum heat-treated E_1' signal intensity of quartz in the fluvial sediments occurs upon heating at about 300°C, as previously observed in aeolian quartz. The heat-treated E_1' signal intensity of quartz in sediment samples also shows significant correspondence to those of their source rocks as well as their formation age. We propose that the heat-treated E_1' centre of sedimentary quartz, combined with other lithological discrimination parameters, could serve as an effective indicator for fluvial sediment provenance tracing.

Keywords: fluvial sediment; quartz; electron spin resonance (ESR); the heat-treated E_1' centre; provenance tracing

1. INTRODUCTION

The E_1' centre is one of the best characterized paramagnetic lattice defects in crystalline quartz, which is an unpaired electron at an oxygen vacancy. The currently accepted model of E_1' centre is an unpaired electron at a single silicon sp^3 orbital oriented along a bond direction into the vacancy where the silicon relaxes toward the vacancy, while the other silicon on the opposite side with

a positive charge relaxes away from the basal plane of its three oxygen neighbours (Rudra and Fowler, 1987). Previous studies suggested that the E_1' centre is not suitable for electron spin resonance (ESR) dating and dosimetry (Zhao *et al.*, 1991; Jin *et al.*, 1991, 1993; Falgueres *et al.*, 1994; Toyoda and Hattori, 2000; Toyoda *et al.*, 2005). However, more recent studies indicated that the ESR signal intensity of heat-treated E_1' centre, after gamma-ray irradiation to 2.5 kGy followed by heating at 300°C for 15 min, was correlated to the age of quartz (Ono *et al.*, 1998; Toyoda and Hattori, 2000; Toyoda and Naruse, 2002; Sun *et al.*, 2007). Thus, on the basis of the correlation between the quartz ESR intensity and its source rock age, the heat-treated E_1' centre could be used to trace

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sediment provenance (Attanasio *et al.*, 2000; Nagashima *et al.*, 2007, 2011; Sun *et al.*, 2008, 2013; Toyoda, 2011; Yamamoto *et al.*, 2013; Shimada *et al.*, 2013, 2016; Tissoux *et al.*, 2015; Toyoda *et al.*, 2016; Satio *et al.*, 2017). Previous studies have mainly focused on the heat-treated E_1' centre of aeolian sediment, while studies on fluvial and lacustrine sediments are limited. It is still not clear whether the heat-treated E_1' centre is useful for fluvial-lacustrine sediment provenance because of the doubt that the quartz signal intensity in such sediments might have been modified at the time of weathering, therefore not corresponding to that of source rock. In this study, the heating effect on E_1' centre of quartz extracted from fluvial sediments and their source rocks is studied to examine the relationships. The potential use of the heat-treated E_1' centre of quartz in the fluvial sediment provenance tracing is also discussed.

2. SAMPLING AND METHOD

Geological settings

The study area located on the south-eastern Tibetan Plateau with high erosion and deposition rate is particularly suitable for studies designed towards a better understanding modern source to sink transport process (Wei *et al.*, 2017). Sampling sites consist of four small river drainages, Mian'ning River, Kang'ding River, Pan'zhuhua River, Diao'tengzi River, as shown in Fig. 1. Each drainage is dominated by single granitic source rocks, and source rocks are S type granites. The ages of the source rocks of Mian'ning River, Kang'ding River, Pan'zhuhua River and Diao'tengzi River are 134 Ma, 227 Ma, 783 Ma and 821 Ma, respectively (data derived from China Intrusive Rock Isotope Age Atlas by K-Ar dating method, Ma and Zhou, 2002).

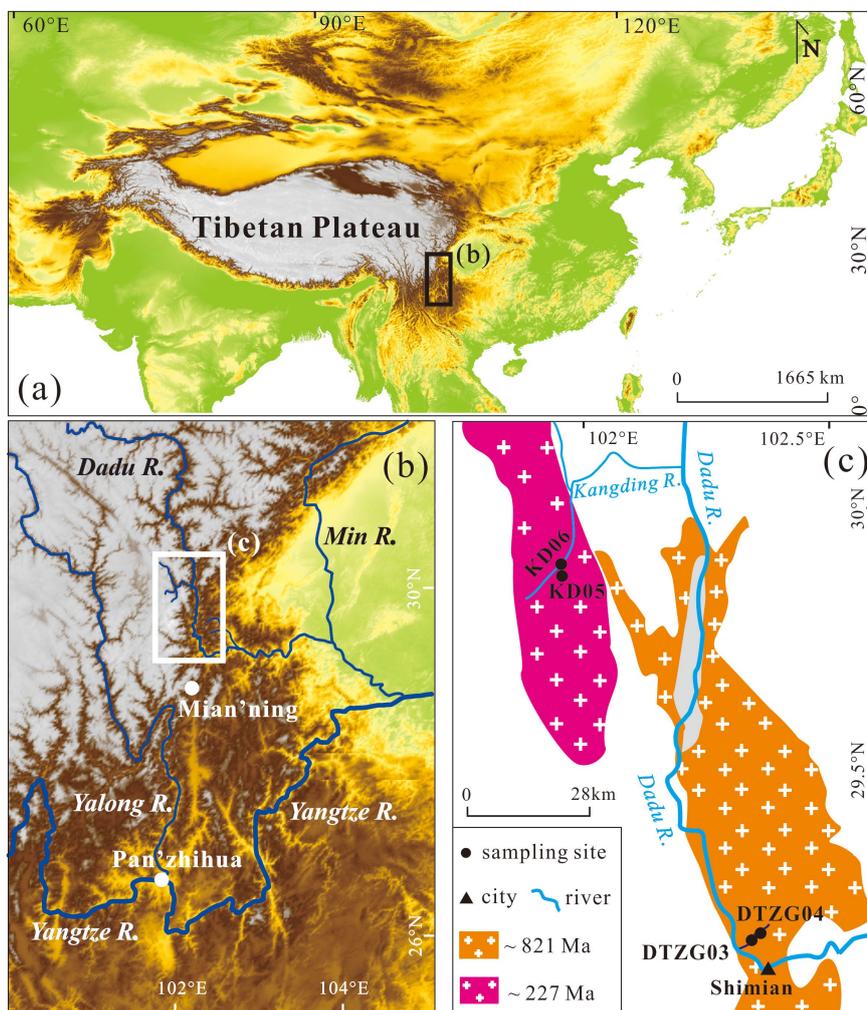


Fig. 1. Map showing study area and geological setting of sampling sites. (a) and (b) show study area located in the eastern Tibetan Plateau. (c) show geological setting and sampling site of Kang'ding River and Diao'tengzi River. The Pan'zhuhua River and the Mian'ning River are marked as two black circles on 1-(b). The source rock age data are from China Intrusive Rock Isotope Age Atlas by K-Ar dating method.

Sampling

Previous studies showed that the source rock age could make a significant effect on the signal intensity of the heat-treated E_1' centre in quartz (Toyoda and Hattori, 2000; Toyoda and Naruse, 2002). To better understand the heating effect on diverse quartz of known origins, we collected four surface fluvial sediments, three source rocks and one late Holocene sediment from the four different small river systems draining on the SE Tibetan Plateau. All modern samples were taken from river banks, and sampling depth is from the surface to 1 cm down; the historical sample was taken from a short core down to 0.8 m deep, far away from river surface; source rock samples were directly collected from granites outcrops with a prospecting hammer. More sample details can be found in **Table 1**. In addition, in order to examine the quartz thermal activation diversity, two granitic samples are collected from different river drainage (Pan'zhihua River and Diao'tengzi River) with different formation ages for the heating effect experiments.

Quartz separation

The 100 ~ 200 μm size fraction of eight samples were separated by wet sieving method, as suggested by Liu *et al.* (2015), in the State Key Laboratory of Earthquake Dynamics, Institute of Geology, China Earthquake Administration (IGCEA), Beijing. The sample pretreatment protocol employed was as described by Liu *et al.* (2010). Each sample was treated with 30% H_2O_2 for 24 hours to remove organic material and then washed and cleaned with distilled water. These samples were treated with 10% HCl for 24 hours to remove carbonates and then washed and cleaned with distilled water. Heavy minerals were removed by heavy liquid. Magnetic minerals were removed by mechanical, magnetic selection. The remaining part was etched by 40% HF for 40 minutes to remove feldspars and rinsed with distilled water to neutralize the acid, and then dried at low temperature. After that, the XRD examination was performed to show the quartz content of the samples around 97.19 ~ 99.9%. In order to

eliminate the error caused by single aliquot data, each pure quartz sample was divided into two sub-samples, which were then divided into aliquots for later irradiation, heating experiment and ESR measurements.

Gamma-ray dose irradiation

Aliquots of all samples were irradiated with 2.5 kGy by the Gamma-ray dose irradiation (Toyoda *et al.*, 2016), using the ^{60}Co facility at Peking University, with a dose rate of 42.25 Gy/min.

Heating experiment

Different aliquots of irradiated quartz were prepared for different heating temperature treatments, including 0, 50, 115, 165, 210, 255, 275, 290, 300, 310, 330, 350, 400, 500, 600, and 700°C, respectively. Each aliquot was heated for 15 minutes in order to convert the oxygen vacancies into E_1' centre of quartz (Toyoda and Hattori, 2000). The designated temperatures were first reached in the muffle furnace, and then the samples were quickly put into it for heating 15 minutes. The heating experiment was carried out at the ^{14}C dating laboratory at State Key Laboratory of Earthquake Dynamics, Institution of Geology, China Earthquake Administration (IGCEA), Beijing.

ESR signal intensity measurements

The ESR signal intensity of the heat-treated E_1' centre in quartz was measured with a Bruker EMX spectrometer at State Key Laboratory of Earthquake Dynamics, Institution of Geology, China Earthquake Administration. The measurements were performed at room temperature with a microwave power of 0.01 mW, modulation frequency of 100 kHz and modulation amplitude of 1.00 G, seen in **Fig. 2** (Toyoda and Hattori, 2000; Toyoda *et al.*, 2000). Each aliquot was measured three times to estimate the angular dependence after rotating by 120°. The quartz heat-treated E_1' centre magnitude was calculated as the average value of the aliquots signal intensities provided by the two sub-samples. The ESR signal intensities of all samples were normalized by sample weight.

Table 1. Sample information used in this study.

No.	Drainage	Sample name	Sample type	Latitude (°N)	Longitude (°E)	Rock age and dating method used
1	Mian'ning River	MN05	modern river sediments	28°31'30"	102°03'42"	134 Ma (kb)
2		MN06	bedrocks	28°31'30"	102°03'42"	134 Ma (kb)
3	Kang'ding River	KD05	bedrocks	29°54'34"	101°57'24"	227 Ma (kb)
4		KD06	modern river sediments	29°59'42"	101°56'50"	227 Ma (kb)
5	Pan'zhihua River	PZH01	bedrocks	26°25'21"	101°44'00"	783 Ma (uz)
6		PZH03	modern river sediments	26°25'28"	101°44'48"	783 Ma (uz)
7	Diao'tengzi River	DTZG03	historical sediments	29°16'01"	102°18'26"	821 Ma (kw)
8		DTZG04	modern river sediments	29°16'01"	102°18'26"	821 Ma (kw)

All samples were collected within the river drainage of single granitic lithology. Rock age: formation age of quartz-containing rock, kb is K-Ar dating of biotite, uz is U-Pb dating of zircon, kw is K-Ar dating of whole rock. The quartz origin ages are compiled from the Geological Map of the Isotope Dating of Intrusive Rocks in China (Ma and Zhou, 2002).

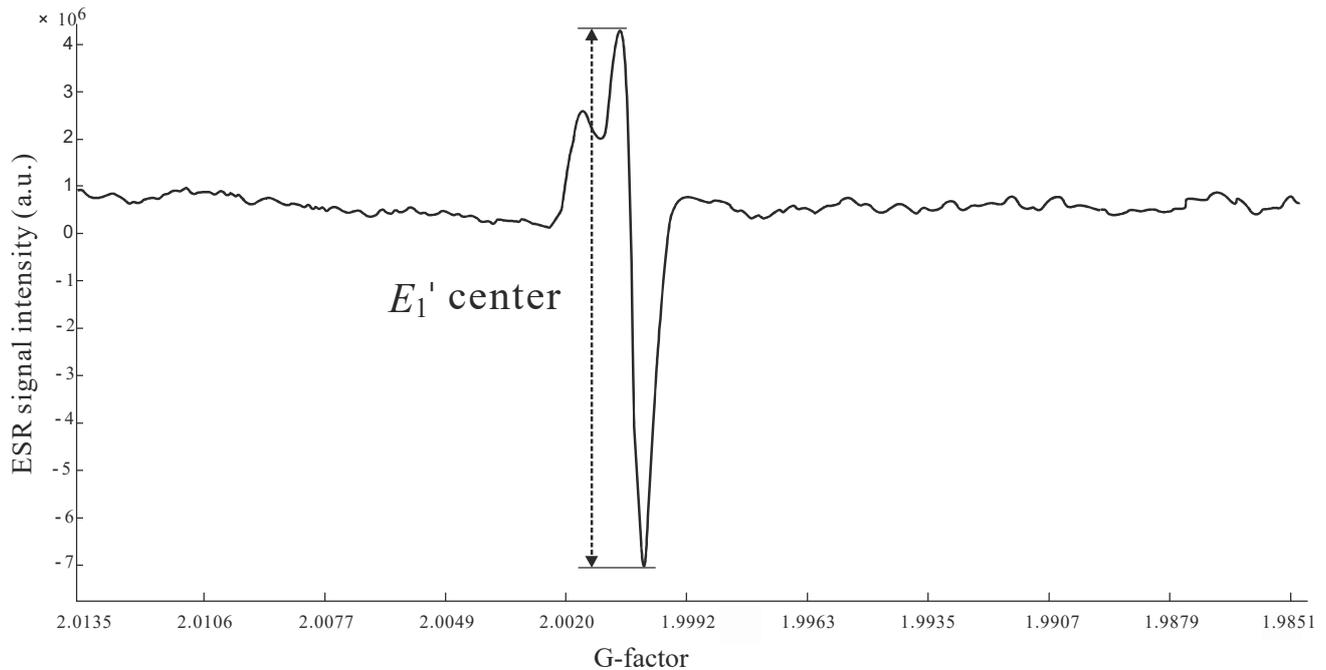


Fig. 2. Typical E_1' centre ESR spectrum of quartz in this study.

3. RESULTS AND DISCUSSIONS

Heating effect on maximum quartz heat-treated E_1' centre

Weeks and Nelson (1960) first reported the intensity increase of the E_1' centre as a result of heating. Effects of heating on the ESR signal intensity in granitic quartz were investigated by Toyoda and Schwarcz (1997). Their experiment results showed that the E_1' signal intensity increased up to 300°C and then decreased until the signal disappeared at 500°C. Hence, they proposed that the E_1' centre could reach maximum value while heating at about 300°C and named it the heat-treated E_1' centre. Following the above experiments, Diao (2003) found that most of the core sediments had the maximum heat-treated E_1' signal intensity at 300°C while a few samples had it at 350°C. He attributed the quartz thermal activation difference to the variously original source rocks. Thus, to detect if the maximum heat-treated E_1' signal intensity of different samples would change at different heating temperature, we selected two samples (PZH03 and DTZG03) collected from different river drainage had them heated from 0°C to 700°C.

Fig. 3 shows that the E_1' signal intensity has slight variation below 200°C, followed by a fast increase and a decrease in the temperature range of 200 ~ 500°C, and disappeared above 500°C. This result is similar to previous works of Toyoda and Schwarcz (1997) and Diao (2003). However, it is worth noting that the PZH03 reaches the maximum heat-treated E_1' centre at 300°C while that of the DTZG03 at 330°C and the error caused

by a difference in the heating process is excluded. The similar physical and chemical pretreatment process of samples could not lead to the disparity of the results of the two samples. Thus, the source rock formation history diversity and crystallization differentiation may be the factors which cause the different results. Nevertheless, this needs further investigation.

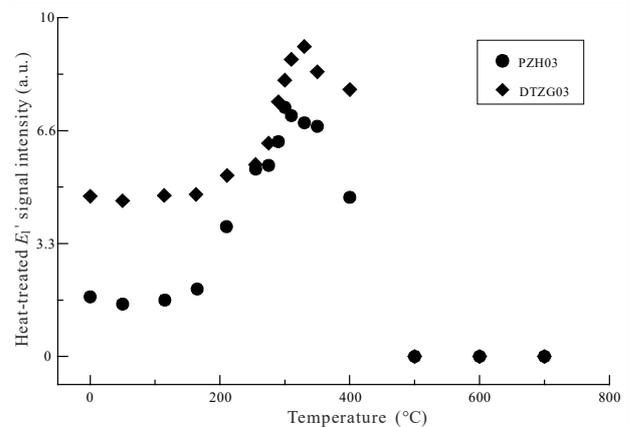


Fig. 3. The heat-treated E_1' signal intensity response to heating temperatures. The similar changes upon heating indicate that the heat-treated E_1' signal intensity of quartz extracted from fluvial sediment can reach the maximum value at about 300°C for 15 min. However, the maximum heat-treated E_1' signal intensity varies in quartz with different formation ages. For example, PZH03 reaches the maximum value at 300°C while DTZG03 at 330°C. Above about 400°C, the E_1' signal intensity of PZH03 and DTZG03 are zero.

The results of stepwise heating experiments of the two samples demonstrate that the heat-treated E_1' centre of quartz extracted from fluvial sediments will also reach the maximum value at about 300°C. This result is similar to the heating effect on the quartz extracted from granitic source rocks (Toyoda and Schwarcz, 1997) and marine sediments (Diao, 2003). These results indicate that the heat-treated E_1' centre of quartz with variously storing environments can reach the maximum value at about 300°C.

Relationship of ESR signal intensity of quartz between sediment and its source rock

One necessary condition for the quartz heat-treated E_1' centre used as a sediment provenance indicator is its stability after a long erosion-transportation-deposition cycle from source to sink. Thus, the quartz heat-treated E_1' signal intensity correlation between sediment and its source rock is evaluated. Fig. 4 shows that the quartz heat-treated E_1' signal intensity in sediment is similar to

those of its source rock in the same drainage system, such as MN05 and MN06, 16KD05 and 16KDO6, 16PZH01 and 16PZH03, 16DTZG03 and 16DTZG04. To better understand the relationship between sediment and its source rock, their numerical difference was calculated using the equation:

$$|A-B| / [(A+B)/2] \times 100\% \quad (3.1)$$

where A is the quartz heat-treated E_1' signal intensity in sediment whilst B is the quartz heat-treated E_1' signal intensity in its source rock, Table 2.

Correlation between the quartz heat-treated E_1' centre and its source rock age

Odom and Rink (1989) suggested that for the correlation relationship between quartz natural E_1' signal intensity and its formation age in the range of 0.1 Ga ~ 1.4 Ga. Toyoda and Hattori (2000) found that the heat-treated E_1' signal intensity correlates with the age of the source granite in the range of 10 Ma ~ 1 Ga (Toyoda *et al.*, 2016).

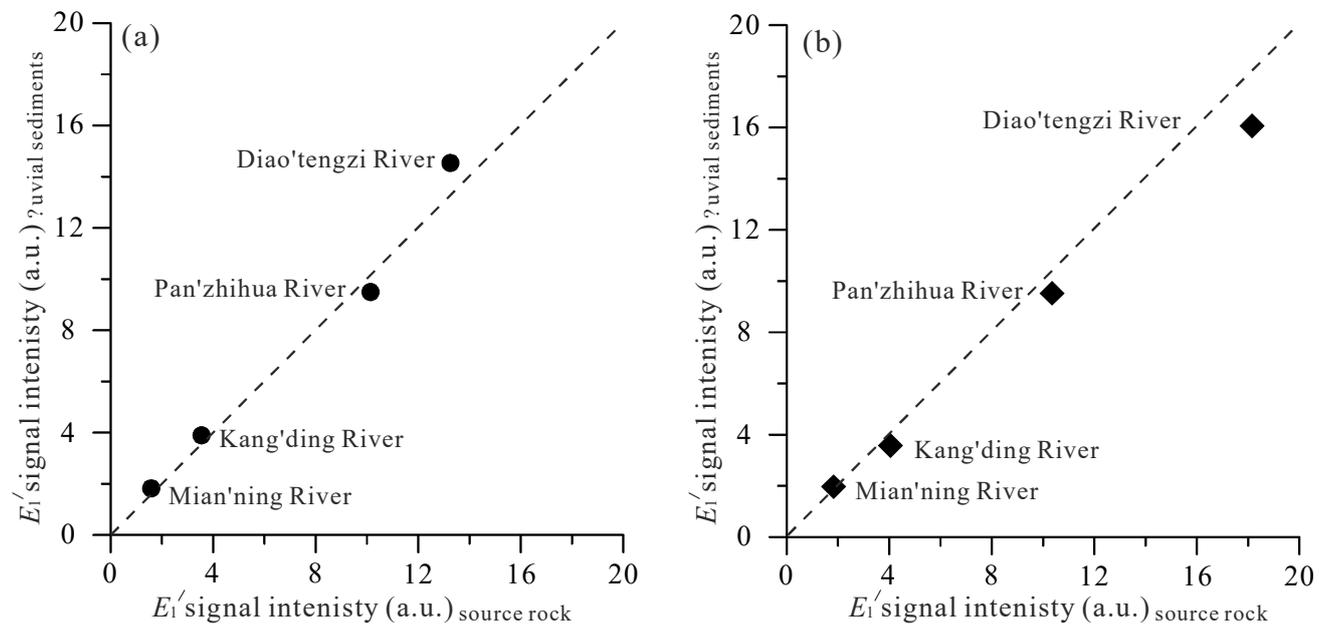


Fig. 4. Correlation between heat-treated E_1' signal intensity of quartz in source rock and its fluvial sediments heated at 300°C (a) and 330°C (b).

Table 2. Difference of quartz heat-treated E_1' signal intensity between sediment and its source rock.

drainage	heating temperature (°C)	heat-treated E_1' signal intensity		A-B	(A+B)/2	difference value (%)
		sediment	source rock			
Mian'ning River	300	1.06	1.22	0.16	1.14	14
	330	1.06	1.21	0.15	1.135	13
Kang'ding River	300	2.60	2.36	0.24	2.48	10
	330	2.61	2.36	0.25	2.49	10
Pan'zhi'hua River	300	6.75	6.31	0.44	6.53	7
	330	6.74	6.30	0.44	6.52	7
Diao'teng'zi River	300	8.82	9.64	0.82	9.23	9
	330	11.92	10.58	1.34	11.25	12

Based on above studies, Toyoda *et al.* (2016) proposed that since the quartz heat-treated E_1' centre is correlated with the age of quartz, it can be an effective indicator of age in source rocks. Therefore, the heat-treated E_1' centre of quartz have been considered as a new powerful tool for provenance study (Ono *et al.*, 1998; Attanasio *et al.*, 2000; Duttine *et al.*, 2002; Toyoda and Naruse, 2002; Crook *et al.*, 2002; Nagashima *et al.*, 2007, 2011; Sun *et al.*, 2007, 2008, 2013; Toyoda, 2011; Yamamoto *et al.*, 2013; Shimada *et al.*, 2013; Tissoux *et al.*, 2015; Toyoda *et al.*, 2016; Shimada *et al.*, 2016; Satio *et al.*, 2017). In order to detect the correlation between the heat-treated E_1' signal intensity of fluvial sedimentary quartz and its source rock age, we compared the ESR signal intensities for the fluvial sediments and source rocks when heated at 300°C and 330°C, respectively (Fig. 5).

Our results indicate that the quartz heat-treated E_1' signal intensity is proportional to its formation age. The younger quartz with lower heat-treated E_1' signal intensity occupies the lower-left corner of Fig. 5a and Fig. 5b, whereas quartz extracted from older source rocks or sediments show higher heat-treated E_1' signal intensity at the top right corner of Fig. 5a and Fig. 5b. To better understand the correlation between the quartz heat-treated E_1'

signal intensity and its formation age, we made a statistical analysis. The regression equations of heating at 300°C is $y = 0.0099x - 0.0379$ while that of heating at 330°C is $y = 0.0116x - 0.363$. The regression coefficient is $R^2 = 0.93$ and $R^2 = 0.84$, both showing clear linear relationship. The result is consistent with the previous works performed by Toyoda and Hattori (2000). Thus, we confirm that the quartz extracted from fluvial sediments and their source rocks also show significant correlation between its heat-treated E_1' signal intensity at about 300°C and its formation age.

Heat-treated E_1' centre in quartz as a provenance tracer

Previous studies showed that the possible sources and transport path of aeolian dust brought to North Asia, Chinese deserts and Chinese Loess Plateau are well defined with heat-treated E_1' centre signal intensity, together with the quartz crystallinity index (Sun *et al.*, 2007, 2008, 2013; Toyoda *et al.*, 2016). Fig. 5 shows that both heat-treated E_1' centre signal intensity at the two heating temperatures (300°C and 330°C) exhibit small variation within each river drainage but vary significantly among the four river drainages. The similar minor differences between the source rock and its fluvial sediment within each river drainage demonstrate that the heat-treated E_1' signal intensity can directly reflect the formation age of the quartz and would not be affected by the storage environment (stored in source rocks or sediment). Furthermore, the quartz heat-treated E_1' signal intensity of sediment almost equals that of its source rocks, which also imply that the quartz heat-treated E_1' centre would be a stable provenance tracer during the transportation from source to sink. Besides, the differences in the heat-treated E_1' signal intensity are apparent among the four river drainages, no matter when the quartz on heating at 300°C or 330°C. For example, the heat-treated E_1' signal intensities of PZH and DTZG are relatively higher compared to that of the MN and KD. Fig. 5a shows that PZH and DTZG with relatively high values (6.8 ~ 9.1 a.u.) while MN and KD with lower values (1.0 ~ 2.5 a.u.). Fig. 5b shows that PZH and DTZG with relatively high values (6.6 ~ 9.6 a.u.), whereas MN and KD with lower values (1.0 ~ 2.2 a.u.). The ESR signal intensity difference among the four river drainages demonstrate that the heat-treated E_1' centre can vary significantly depending on the formation age of the quartz. Thus, the quartz heat-treated E_1' centre marked with the characteristics that remain stable during transportation from source to sink and have variations depending on its source rock age.

Unlike previous provenance tracers that can directly indicate the source of the sediment, the heat-treated E_1' centre can only represent the quartz formation age, part of the source rock formations. In this case, it is unable to discriminate source rocks of different lithologies with the same quartz formation age. However, the quartz nature E_1' centre, which varies significantly in quartz of igneous,

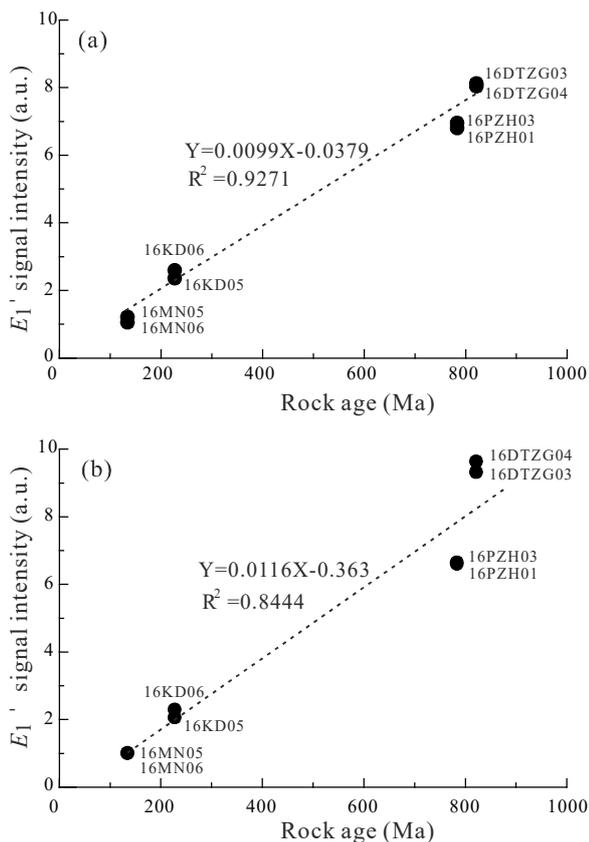


Fig. 5. Correlation between rock age and its heat-treated E_1' signal intensity. (a) heating temperature is 300°C; (b) heating temperature is 330°C.

sedimentary and metamorphic source rocks (Wei *et al.*, 2017), combined with the heat-treated E_1' centre would be useful in distinguishing quartz of different origins extracted from different lithological rocks with various formation ages. Because quartz is stable both physically and chemically, it forms the major component of the fluvial sediment, especially in large river sediment. Both of the two parameters discussed above are not affected by the weathering process and very easy to measure.

As the ESR signal parameters of heat-treated and natural E_1' centre we measured are from the same sample, the results are less influenced by the pretreated physical and chemical processes. Moreover, we reduced the uncertainties in heating temperature by using the same heating condition and controlling the accuracy temperature. Thus, compared with other provenance tracers, the heat-treated E_1' centre can effectively represent the quartz formation age, the natural E_1' centre can effectively distinguish the different lithology source rocks. Therefore, a combination of the two ESR signals offers a powerful approach for tracing the sediment quartz originated from different source regions. In conclusion, we propose the heat-treated E_1' centre, combined with other lithology discrimination indicators, can also serve as an effective provenance tracer in river drainage.

4. CONCLUSIONS

Responses of the E_1' centre of quartz extracted from fluvial sediment and its source rock to the artificial heating at different temperatures were investigated. It was revealed that the quartz heat-treated E_1' centre of fluvial sediment also could reach the maximum value on heating at about 300°C for 15 min, which is consistent with the previous works. The significant correlation between quartz heat-treated E_1' signal intensity and its formation age showed that the heat-treated E_1' centre could directly reflect the quartz formation age. The statistical analysis indicates that the ESR signal intensity of quartz heat-treated E_1' centre heated at 300°C has better linear correlation with various original formation age of source rocks. In conclusion, this study demonstrates that the heat-treated E_1' signal intensity (oxygen vacancies) can be a stable indicator for provenance research on fluvial sediments.

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