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A detailed post-IR IRSL dating study of the Niuyangzigou loess site in northeastern China

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In this study, we report standard quartz SAR OSL and post–IR infrared (IR) stimulated luminescence (post-IR IRSL; $pIRIR_{290}$) measurements made on sand-sized quartz and K-feldspar extracts from the loess-palaeosol sequence at Niuyangzigou in northeastern China. The quartz OSL characteristics are satisfactory. Extensive $pIRIR_{50,290}$ dose recovery tests were performed by adding doses on top of the natural dose. We found that dose recovery ratios improve significantly when the test dose ranges between ~15 and ~80% of the total dose, and good dose recovery (within ±5% of unity) can be obtained up to ~800 Gy. Otherwise, the dose recovery ratio deviates from unity. The D_e values also depend on the test dose size so we conclude that the effect of test dose size should be routinely considered in pIRIR dating. First IR stimulation plateau $pIRIR_{290}$ results are compared with multiple elevated temperature -pIRIR (MET-pIRIR) data. It appears that the low temperature MET-pIRIR data are strongly affected by poor dose recovery, but this is not the case for the $pIRIR_{290}$

23 results. Natural signal measurements at the highest (first IR) stimulation temperature
24 on a sample expected to be in field saturation, suggest ~10% signal loss is present in
25 pIRIR signals. Long term laboratory bleaching experiments (>80 days) show that a
26 constant (or very difficult to bleach) residual $pIRIR_{290}$ signal is reached after ~300 h,
27 corresponding to a dose of 6.2±0.7 Gy. Quartz OSL and feldspar $pIRIR_{50,290}$ ages are
28 in good agreement at least back to ~70 ka. Beyond this the quartz ages begin to
29 underestimate but the feldspar ages are in agreement with the expected Last
30 Interglacial age palaeosol.
31
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41 Northeastern China is located in the East Asian monsoon region and lies near the
42 present-day limit of the summer monsoon (Fig 1); as a result it is sensitive to the
43 global climate systems of both the high and low latitudes. Since the 1980s this area

has been characterised by a pronounced regional temperature increase and a decrease in precipitation compared to the rest of China (Sun *et al*. 2007; Gao *et al*. 2008). Because of these factors it is a very suitable place to study climate change and environmental evolution during the late Quaternary. The considerable area of loess deposits in north China forms one of the largest and most important aeolian records on Earth. Loess/palaeosol sequences contain detailed archives of terrestrial palaeoenvironmental changes and are highly sensitive to climatic changes, specifically to shifts in the Asian summer and winter monsoon and/or Northern Hemisphere westerly circulation (Liu & Ding, 1998). However, research into loess deposition and past climate change in northeastern China is limited due to the lack of independent age control (i.e. radiometric dating).

Luminescence dating has proved to be particularly successful for dating aeolian sequences (e.g. Stevens *et al*. 2006; Buylaert *et al*. 2008; Lai 2010;Lai & Fan 2014). These studies are all based on equivalent dose estimation using the single aliquot regenerative (SAR) dose protocol developed for fast-component dominated quartz OSL (Wintle & Murray 2006). However, its use is typically limited to samples with equivalent doses up to 150-200 Gy. This restricts the OSL dating of quartz to loess deposits (typical dose rate of between 3 and 4 Gy ka^{-1}) from within the last 50-70 ka (Buylaert *et al*. 2007; Roberts 2008; Chapot *et al*. 2012; Timar-Gabor & Wintle 2013). The infrared stimulated luminescence (IRSL) signal from feldspar (Hütt *et al*.1988)

has the potential to extend the datable age range because it saturates at much higher doses compared to quartz OSL (Huntley & Lamothe 2001). However, it is now widely accepted that IRSL measured at ambient temperature suffers from anomalous fading (e.g. Spooner 1994; Huntley & Lian 2006; Buylaert *et al*. 2011, 2012). Recent advances in the understanding of feldspar as a luminescence dosimeter (Thomsen *et al*. 2008, 2011; Murray *et al*. 2009; Jain & Ankjærgaard 2011) have led to new single-aliquot regenerative dose (SAR) dating protocols (so called post-IR IRSL protocols; e.g. Buylaert *et al*. 2009), in which a high temperature IRSL signal is measured at an increased temperature after a first IR stimulation at some lower temperature, usually close to ambient temperature. These post-IR IRSL (pIRIR) signals appear to be much less prone to fading than the conventional IRSL signal. Various single-aliquot based pIRIR dating protocols for feldspar have been developed; these include a two-step (e.g. Thomsen *et al*. 2008; Buylaert *et al*. 2009; Thiel *et al*. 2011a) and a multiple elevated temperature (MET) (Li & Li 2011; 2012a) pIRIR stimulation procedure. These procedures have been shown to give accurate ages both for young (<10 ka, Fu & Li 2013; Reimann *et al*. 2011, 2012) and old (>100 ka, Buylaert *et al*. 2012; Li & Li 2011, 2012a; Kars *et al*. 2012; Zander & Hilgers 2013) samples. Even though pIRIR measurement protocols have become the preferred method to measure feldspar equivalent doses, there are still remaining issues concerning dose recovery results (Roberts 2012), the determination of a potentially unbleachable component (e.g. Buylaert *et al*. 2011, Stevens *et al*. 2011; Murray *et al*.

2012; Kars *et al*. 2014a) and signal (in)stability (Li & Li 2012b; Thomsen *et al*. 2011).

The present work focuses on the Niuyangzigou (NYZG) loess-palaeosol sequence in northeastern China (Fig. 1). The luminescence characteristics of quartz SAR OSL and K-feldspar pIRIR signals ($pIRIR_{290}$) are documented in a SAR-based methodology. The pIRIR signal measured at 290°C is then investigated in detail to (i) test the dependence of dose recovery and D_e on test dose size, (ii) determine the size of an (un)bleachable residual component and (iii) check the stability of the signal (including MET-pIRIR signals) using a sample expected to be of non-finite age. Finally, quartz OSL and $pIRIR_{290}$ ages are compared and a luminescence chronology for the upper part of the NYZG section is presented.

Geological setting, stratigraphy and sampling

Northeastern China extends from 40° to 59° N and 110 to 135° E, it includes the provinces of Heilongjiang, Jilin and Liaoning, and the Inner Mongolia Autonomous Region east of 110° E (Fig. 1). The major topographical features in this area are the extensive Northeast Plain surrounded by a series of mountains and hills. The Daxingan Mountains lie along the western side while the Xiaoxingan Range with a northwest-southeast orientation lies to the north.

The NYZG section (41°55′ N, 118°43′ E, 774 m.a.s.l) is situated in KaLaQin County, Chifeng city, in the northeastern part of the Inner Mongolia Autonomous Region (Fig. 1). The whole section is a 36.6 m thick series of loess intercalated by palaeosols; it is accessible through a natural exposure of the upper 26 m and a 10.6 m deep exploratory well. The magnetic characteristics of the Matuyama–Brunhes palaeomagnetic boundary (0.78 Ma; Cande & Kent 1995) have been identified towards the bottom of the sequence and some evidence for the Jaramillo sub-chron is exposed in a basal complex of palaeosols (Zeng *et al*. 2011).

Fifteen luminescence samples were collected using light-tight steel cylinders (diameter 5 cm, length 20 cm) from the upper 3.2 m of a freshly excavated profile. Based on field observations, this section comprises the upper Holocene soil (S_0), the Last Glacial loess (L_1) and the Last Interglacial palaeosol (S_1) (Fig.1). One additional sample (sample code 1535) was collected near the B/M boundary (Zeng *et al*. 2011) to provide a feldspar sample of non-finite luminescence age (expected burial dose >2000 Gy).

Sample preparation and analytical facilities

Samples were opened under subdued red light conditions and material from the outer ends of each tube was used for dose rate and water content measurement. The

non-light exposed material from the middle part of the tube was treated with HCl (30%) and H_2O_2 (30%) to remove carbonates and organic matter, respectively. Grains in the range 63 to 90 µm were obtained by wet-sieving. Pure quartz grains (no significant IRSL signals) were obtained after a 40 min HF (40%) etch and 40 min 10% HCl rinse. For K-rich feldspar extraction, a portion of the initial 63-90 µm sieved fraction was cleaned with 10% HF for 20 min to remove coatings and the outer alpha irradiated layer, and then rinsed in 10% HCl acid for 20 min to remove any precipitated fluorides. K-rich feldspars were floated off using an aqueous heavy liquid (sodium heteropolytungstate 'LST Fastfloat'; density 2.58 $g.cm^{-3}$).

Luminescence measurements employed Risø TL/OSL readers model DA-20 (Bøtter-Jensen *et al*. 2003) equipped with blue LEDs (470 nm, ~80 $mWcm^{-2}$) infrared (IR) LEDs (870 nm, ~135 $mWcm^{-2}$); each reader was equipped with a calibrated ^{90}Sr /^{90}Y beta source. Quartz OSL signals were collected through 7.5 mm of Schott U-340 (UV) glass filter and feldspar (post-IR) IRSL through a combination of Corning 7-59 and Schott BG-39 glass filters (blue-violet part of the spectrum). Quartz grains were mounted as large (8 mm) aliquots on stainless steel discs and K-rich feldspars as small (2 mm) aliquots on stainless steel cups; Silicone oil (Silkospray) was used as an adhesive. For quartz OSL, the signal was derived from the first 0.16 s of stimulation and an early background (0.16-0.32 s) to minimize the influence of slow and medium components (Ballarini *et al*. 2007; Cunningham & Wallinga 2010). Feldspar (post-IR)

IRSL signals were derived from the integral of the first 2 s of (post-IR) IRSL stimulation, less a background based on the last 50 s; for measurements made using an MET-pIRIR protocol these intervals are 1 and 25 s, respectively.

Dosimetry

The environmental dose rate was calculated from the uranium, thorium and potassium concentrations, measured by neutron activation analysis (NAA). The *in situ* water content (mass of moisture/dry mass) was determined by weighing the sample before and after drying, and was assigned an absolute uncertainty of ±5% (e.g. for a water content of 10% we have used (10±5)%). Using the revised dose rate conversion factors of Guérin *et al*. (2011) and water content attenuation factors (Aitken 1985), the elemental concentrations were converted into effective dose rate. Calculation of the cosmic dose rate is based on Prescott & Hutton (1994). For K-feldspar dose rates a K concentration of 12.5±0.5% and Rb concentration of 400±100 ppm was assumed (Huntley & Baril 1997) consistent with earlier work on sand-sized K-feldspar from Chinese loess (Zhao & Li 2005; Li *et al*. 2008). A small internal dose rate contribution from U and Th of 0.03±0.015 Gy ka^{-1} and 0.06±0.03 Gy ka^{-1} was included for quartz and K-feldspar respectively (Mejdahl 1987; Zhao & Li 2005; Vandenberghe *et al*. 2008). Table 1 summarises the uranium, thorium and

169 potassium concentrations and the resulting total dose rates to quartz and K-feldspar
170 grains
171
172 **Quartz OSL characteristics and resulting ages**
173
174 The quartz equivalent doses (D_e) were measured using a standard SAR protocol
175 (Murray & Wintle 2000, 2003; Table 2). Typical dose response curves and OSL decay
176 curves (inset) are shown for the upper (138101) and lower (138115) samples in Fig.
177 2A and B, respectively. The blue-light stimulated OSL signals decrease very quickly
178 during the first second of stimulation, indicating that the signal is dominated by the
179 fast component (Jain *et al*. 2003; Singarayer & Bailey 2003). In order to select
180 appropriate preheat conditions, the D_e dependence on preheat temperature was
181 checked using a preheat plateau test. A plateau was observed for temperatures from
182 180 °C to at least 260 °C (Fig. 2C). The suitability of our adopted SAR protocol was
183 further checked with a dose recovery test (Murray & Wintle 2003). The ratios of the
184 given doses to the measured doses were within 10% of unity over the entire
185 temperature interval (Fig. 2D). Based on these preheat tests, a 260 °C preheat for 10
186 s and 220 °C cut-heat was chosen for final D_e determination. For all samples,
187 recuperation is low (average = 0.06±0.01% of natural, n = 245) and the average
188 recycling ratio is 1.01±0.01 (n = 233) indicating that the adopted SAR protocol
189 successfully corrects for laboratory sensitivity changes.

In order to ensure that only those aliquots that are capable of measuring the dose of interest have been included in the calculation of the mean D_e, aliquots with $2 \times D_0$ values that are smaller than the average dose (derived from all aliquots) are not used in the derivation of D_e, irrespective of the D_e value of the individual aliquot (based on Thomsen *et al*. 2016; note that for simplicity our data were approximated by a single saturating exponential function). For the upper four samples (<150 Gy) no aliquots were rejected based on this criterion. Below this level (1.20 m) the percentage of aliquots that are rejected ranges between 6 and 31%. Over all the 15 samples the average D_e was decreased by ~3% because of this selection criterion; the biggest decrease was 16% and the largest increase was only 2%. Table 1 summarises the resulting D_e values and quartz OSL ages. The calculated D_e values range from 92±3 Gy for the upper sample to 270±13 Gy for the lowest sample; the corresponding ages are 30±2 and 99±7 ka. We note that the bottom four samples all have D_e values ≥200 Gy. Several papers suggest that the upper limit of SAR based quartz OSL dating lies around ~200 Gy in loess and ages derived from these high doses should be interpreted with caution because they are likely to be underestimates (e.g. Buylaert *et al*. 2007, 2008; Zhou & Shackleton 2001; Lai 2010; Chapot *et al*. 2012, Timar-Gabor & Wintle 2013; Lai & Fan 2014).

Feldspar luminescence characteristics

Dose response curve and L_x/T_x plots

The SAR $pIRIR_{290}$ protocol proposed by Thiel *et al*. (2011a) and tested by Buylaert *et al*. (2012) was used to measure the K-feldspar dose in these samples (Table 2). Aliquots were preheated at 320 °C for 60 s followed by IR diode stimulation (90% power) at 50 °C for 200 s (the choice of this temperature is discussed in the next section) to recombine nearby electron-hole pairs (Jain & Ankjærgaard 2011). Subsequently, a more stable IRSL signal is measured at 290 °C for 200 s (referred to as $pIRIR_{290}$); this is the dating signal of interest. The response to the test dose is measured in the same manner and is followed by an IR illumination at 325 °C for 200 s at the end of each SAR cycle, to reduce recuperation. Representative K-feldspar $pIRIR_{290}$ and quartz OSL dose response curves (normalised to the fitted saturation values) are presented for the lowermost sample (138115) in Fig. 3A. It can be seen that the feldspar $pIRIR_{290}$ dose response curve has a much more extended dose range compared to quartz OSL, 86% of saturation is reached at 1250 and 310 Gy, respectively. This extended feldspar range indicates its usefulness for dating samples beyond the quartz OSL limit.

One of the main assumptions when using the SAR protocol to measure a dose is that the test dose luminescence sensitivity is directly proportional to the preceding regenerative dose; i.e. it can correct for sensitivity changes (Murray & Wintle 2000). We have constructed L_x-T_x plots for the $pIRIR_{290}$ signal for five samples down our

232 section by repeating SAR cycles using a fixed regenerative dose and test dose (Fig.
233 3B); there is clear proportionality between regenerative and test dose signals
234 indicating that the test dose successfully corrects for sensitivity changes. This is also
235 supported by the mean recycling ratio of 1.031±0.002 (n = 109, RSD = 2%; 15
236 samples). If no test dose is used for sensitivity correction, the resulting recycling ratio
237 is 1.099 ± 0.011 with a much higher standard deviation of 10%. For all samples,
238 recuperation is small (<3% of the natural signal) showing that our high temperature
239 clean-out is sufficiently stringent. However, the most stringent test for any SAR
240 protocol is the dose recovery test (Murray 1996; Wallinga *et al*. 2000; Murray & Wintle
241 2003) and this is addressed in the next section.
242
243 ***Dose recovery test***
244 Although several studies have presented good/acceptable dose recovery results for
245 $pIRIR_{290}$ protocols on a variety of sediments (e.g. Buylaert *et al*. 2011, 2012, 2013;
246 Nian *et al*. 2012; Thiel *et al*. 2012; Tsukamoto *et al*. 2014), there is also considerable
247 evidence for poor dose recovery results (e.g. Stevens *et al*. 2011; Lowick *et al*. 2012;
248 Roberts 2012; Thiel et al. 2011b, 2014 ; Murray *et al*. 2014). Usually, these poor dose
249 recovery ratios were significantly greater than unity. Some authors report difficulties in
250 bleaching natural $pIRIR_{290}$ signals (using SOL2 simulator or natural daylight); these
251 can result in poor dose recovery because of incorrect residual dose estimation (e.g.
252 Stevens *et al*. 2011; Alexanderson & Murray 2012). To avoid potential complications

253 related to bleaching natural samples, a dose recovery test can also be performed on
254 modern/young samples by adding a beta dose on top of a relatively small natural
255 dose (e.g. Buylaert *et al*. 2011); this is the approach taken in this study.
256
257 We investigated the dependence of dose recovery ratios on test dose size by
258 adding beta doses (ranging from 99 to 1593 Gy) to aliquots of the uppermost sample
259 (138101) which has a $pIRIR_{290}$ D_e value of ~100 Gy. The dose recovery ratio was
260 calculated as the measured dose divided by the sum of the natural and the given
261 dose. Qin & Zhou (2012) suggested that the dose recovery ratio is dependent on the
262 test dose size (their Fig. 3B) but their data are limited in test dose range and
263 inconclusive (the data for different test dose sizes does not differ significantly). Fig 4A
264 shows the dose recovery ratio as a function of test dose size over a wide test dose
265 range (5-260% of the total (natural+added) dose). For small test doses (<15%), the
266 dose recovery ratio is significantly greater than unity. In contrast, large test doses
267 (>80%) yield ratios lower than unity. The best dose recovery ratios (within ±5% of
268 unity) are found when the test dose ranges between ~15 and ~80% of the total dose.
269 The data of Fig. 4A are also shown in Fig. 4B but now as a measured versus added
270 dose plot; these are so-called Single Aliquot Regenerative Added dose (SARA;
271 Mejdahl & Bøtter-Jensen 1994; Wallinga *et al*. 2001) dose response curves. As
272 expected from the dose recovery ratios, the slope (1.01 ± 0.02) of the fitted line is
273 indistinguishable from unity when test doses between 15 and 80% are used. It is

274 interesting to note that at least for added doses larger than about 600 Gy, the data
275 measured with small (<15%) or large (>80%) test doses do not follow a simple
276 straight line relationship between measured and added dose, rather the measured
277 dose increasingly deviates from the known added dose; this occurs because of a
278 systematic change in D_0 with test dose.
279
280 We conclude from the data of Fig. 4 that, at least for these samples, an
281 acceptable dose recovery ratio is best ensured by using a test dose in the range
282 15-80% of the total (natural + added) dose.
283
284 ***Equivalent dose***
285 ***Effect of test dose size.*** Fig. 5 shows the dependence of D_e on test dose size for the
286 uppermost (138101), middle (138108) and lowermost (138115) samples from the
287 section. Sample 138101 shows a clear D_e plateau for test doses ranging between 5
288 and 80% of the D_e value; the D_e value of the older samples appears insensitive to test
289 dose size over a shorter range, up to only ~60%. The shape of the dose recovery
290 ratio versus test dose curve (Fig. 4A) broadly resembles the shape of the D_e versus
291 test dose curve but it appears that especially for low test doses (<20%) the D_e value is
292 less sensitive to test dose size than the dose recovery value. Nevertheless, for these
293 samples, it appears to be inappropriate to use very small (<10%) or very large (>60%)
294 test doses because of poor dose recovery ratios. For the remainder of this study we

adopt a test dose size of ~30% for D_e measurements; in this range the dose recovery ratio is within ±5% of unity and the D_e values are consistent with the plateau regions in D_e versus test dose graphs.

Comparison with MET-protocol. Based on the model prediction by Jain & Ankjærgaard (2011) that the stability of the post-IR IRSL signal could be dependent on the first IR stimulation temperature and/or wavelength, Buylaert *et al*. (2012) have suggested the use of a first IR stimulation temperature plateau to investigate whether a stable signal was reached (interval over which D_e is insensitive to prior IR stimulation temperature). Fig. 6A,B shows D_e as a function of prior IR stimulation temperature for the top (138101) and bottom (138115) samples of the section, respectively; from these data it seems that for D_e values up to ~400 Gy the D_e is insensitive to first IR stimulation temperature and a stable signal is observed. This is in agreement with the data of Li & Li (2012b) who compared $pIRIR_{50,290}$ D_e values with $pIRIR_{200,290}$ D_e values and MET-$pIRIR_{250}$ results for Luochuan samples and showed that the three methods are indistinguishable back to ~400 Gy; they also showed that beyond this a low temperature IR cleaning at 50 °C is apparently not sufficient to recombine all nearby electron-hole pairs. Beyond ~400 Gy, $pIRIR_{200,290}$ is consistent with the MET-250 data. It is claimed by Li & Li (2011, 2012a, b) and Fu & Li (2013) that the MET-pIRIR protocols have an advantage over the two-step protocols (Buylaert *et al*. 2009, 2012; Thiel *et al*. 2011a) because of the possibility to construct

316 “age-temperature” plots from individual aliquots; the presence of an “age plateau” at
317 higher stimulation temperatures is used to determine whether a stable signal was
318 identified. We have also measured the MET-pIRIR signals (following Li & Li, 2012 a)
319 for the top and bottom sample of our section and the data are also shown in Fig. 6A,B
320 (open symbols). As expected the MET-pIRIR D_e values are consistent with the
321 $pIRIR_{290}$ D_e measurements when stimulation temperatures of >200 °C are reached. It
322 should be noted that the plateau identified by Li & Li (2011, 2012a) and Fu & Li (2013)
323 is usually limited to two (or maximum three) datapoints over a limited temperature
324 interval of 50-100 °C (e.g. Li & Li, 2011, 2012a). The same observation is made here.
325 For sample 138101 we have carried out a dose recovery test as a function of prior IR
326 stimulation temperature (Fig. 6C). It can be seen that dose recovery is satisfactory
327 over a wide prior IR stimulation temperature interval between 50 and 260 °C for the
328 $pIRIR_{290}$ data. This is in contrast with the MET-pIRIR dose recovery data which shows
329 a pronounced increase in of dose recovery between 50 and 150 °C IR stimulation
330 temperature; the shape is very similar to the MET-pIRIR D_e data shown in Fig. 6A for
331 the same sample. We conclude that the MET-pIRIR D_e data are significantly affected
332 by dose recovery problems at low IR stimulation temperatures (a suggestion to this
333 effect was made in Li & Li 2011). This is consistent with the observations of Kars *et al*.
334 (2014b) who showed that high preheats (>300 °C) should not be used with low
335 temperature IR stimulations. Because of this, we suggest using first IR stimulation

temperature plateaus instead of the MET-method to determine whether a more stable signal has been reached.

Stability. Thiel *et al*. (2011a) were the first to show a natural $pIRIR_{290}$ signal in saturation on the laboratory dose response curve for a sample collected below the B/M boundary; this led them to conclude that the $pIRIR_{290}$ signal is a stable signal. Other studies have made the same observation for $pIRIR_{290}$ (Buylaert *et al*. 2011; Thomsen *et al*. 2011) and the high-temperature MET-pIRIR signals (Li & Li 2011, 2012a). In order to investigate this for our material, we collected a sample (code 1535, 27.30 m) below the B/M boundary defined using palaeomagnetic measurements (Zeng *et al*. 2011). The expected burial dose is >2000 Gy and all natural signals are expected to be in field saturation.

Natural signals and dose response curves were measured for both the $pIRIR_{290}$ signals (using different prior-IR stimulation temperatures) and the MET-pIRIR signals. The natural signals are shown in Fig. 7, expressed as a fraction of the saturation level of the laboratory dose response curves. Note that for simplicity a single saturating exponential was fitted through the data; Guralnik et al. (2015) have shown that for their sample a single exponential fit gives a D_0 indistinguishable from that obtained from their theoretically based generalised growth curve. Because most earlier work

356 tended to use relatively small test doses, in this experiment we use both a large (500
357 Gy, approximately equal to D_0) and a small (50-60 Gy) test dose.

358 The results obtained using a 500 Gy test dose are discussed first (Fig. 7A). For
359 the $pIRIR_{290}$ protocol, lower first-IR stimulation temperatures (50-140 °C) yield
360 fraction of saturation values of less than 0.90 (average = 0.87±0.01) and higher
361 temperatures (170-260 °C) yield higher values (average = 0.92±0.01). The
362 MET-pIRIR data measured with a 500 Gy test dose does not seem to show a clear
363 plateau at higher temperature (previously reported at 250-300 °C; see Fig. 3 in Li & Li
364 2012a). At 250 and 300 °C the fraction of saturation is 0.93±0.01 and 0.965±0.004,
365 respectively. Both for $pIRIR_{290}$ (measured with prior IR stimulation ≥170 °C) and the
366 MET-pIRIR protocols, the data are not consistent with unity (~4% below saturation
367 light level for MET and ~8% for $pIRIR_{290}$) which might suggest instability in the
368 $pIRIR_{290}$ signal and the high-temperature MET-pIRIR signals. For the $pIRIR_{290}$ signals
369 measured with low prior-IR stimulation temperatures this underestimation increases
370 to ~13%.

371

372 Fig. 7B shows the results when a small test dose of 50-60 Gy is used. For both
373 $pIRIR_{290}$ (only measured at 50 and 200°C prior-IR stimulation temperature) and the
374 MET-pIRIR signals the fraction of saturation appears to have increased significantly.
375 For the $pIRIR_{200,290}$ this value is 0.99±0.02, consistent with the MET-pIRIR signals
376 measured at 250 and 300 °C (0.96±0.02); this could be interpreted as suggesting

377 negligible instability. However, given the test dose dependence of the dose recovery
378 and D_e results, it is not surprising that the fraction of saturation reached by a natural
379 signal is also a function of test dose size. One must be critical of saturation
380 measurements made with a test dose of only 60 Gy (<<10% of the natural dose). The
381 measurements made with a 500 Gy test dose (~D_0) are more likely to yield accurate
382 D_e measurements and so more accurate measurements of saturation level. We
383 conclude that the apparent stability of signals (e.g. Fig. 1 in Buylaert *et al*. 2012 -
384 pIRIR; Li & Li 2012a – MET-pIRIR) measured with a small test dose is likely to be an
385 artefact of the dose recovery dependence on test dose size, and that the small but
386 finite apparent instability (up to ~8% below saturation) observed at saturation using a
387 large test dose is probably real.
388
389 To further investigate the stability of pIRIR signals we have measured fading
390 rates (g_{2days} values) on sample 138106 using a 50 and 200 °C prior-IR stimulation
391 temperature. The resulting g_{2days} values are 0.75±0.10%/decade (n = 6) and
392 0.37±0.13%/decade (n = 6) for $pIRIR_{50,290}$ and $pIRIR_{200,290}$ respectively. It appears
393 that the $pIRIR_{200,290}$ fading rate is lower than the $pIRIR_{50,290}$ fading rate; this is
394 consistent with the saturation measurements on sample 1535 presented earlier. Li &
395 Li (2012b) have also argued that $pIRIR_{200,290}$ is more stable than $pIRIR_{50,290}$.
396
397 ***Bleaching characteristics***

Comparison of quartz OSL, feldspar IR_{50} and $pIRIR_{290}$ bleaching rates. Fig. 8A summarises the quartz OSL, IR_{50} and $pIRIR_{290}$ ages for all 15 samples. All the IR_{50} data are consistent with a smooth curve passing through the origin and lying below the 1:1 line; this is what would be expected from samples for which both signals are well-bleached at deposition but for which the IR_{50} signal is significantly unstable compared to $pIRIR_{290}$ (Buylaert *et al*. 2013). This result is not surprising given the prolonged and almost ideal light-exposure received by wind-blown dust before final deposition. Note that this relationship does not arise because of a dose-dependent change in initial sensitivity of the IRSL signals (see Fig. S1). Although the scatter in the quartz and pIRIR age relationship is larger, the data are consistent with the fitted curve and there is again no evidence for $pIRIR_{290}$ outliers at larger ages.

To illustrate the likely initial conditions of this material had it not been well-bleached, we exposed quartz and feldspar aliquots of the oldest sample (138115) to a SOL2 spectrum at a lamp-sample distance of ~80 cm for various lengths of time. It took ~130 s for the $pIRIR_{290}$ dose, ~20 s for the IR_{50} dose and <2 s for the quartz OSL dose to be reduced by 50%. The resulting residual dose are shown as open circles (IR_{50}) and open stars (quartz OSL) in Fig. 8B, plotted against the residual $pIRIR_{290}$ doses. The differential bleaching rates of the three signals are obvious and each dataset has been fitted with a single and double exponential; when the residual quartz OSL dose is less than 10% of its initial value the IR_{50} dose is ~55% of its initial

value and the $pIRIR_{290}$ is >80%, comparable to the results described by Murray *et al*. (2012). As expected both curves point at the origin – sufficient light exposure will bleach all three signals so that the apparent residual doses are close to zero. Had our loess samples been incompletely bleached in nature at deposition then the observed IR_{50} and $pIRIR_{290}$ doses would have grown along a line parallel to the solid line but starting from some point on the dashed line passing through the open circles, depending on the degree of incomplete bleaching. Given the inevitable variation in the degree of incomplete bleaching with time this would have resulted in scattered points lying below the observed solid line (as was seen by Buylaert *et al*. 2013). Since our data do not show such scatter we conclude that the $pIRIR_{290}$ signals (and thus the IR_{50} and quartz OSL signals) from these samples were all well-bleached at deposition.

Residual component. Thomsen *et al*. (2008) and Buylaert *et al*. (2009) have shown that some pIRIR signals can be readily bleached (with residual doses <2 Gy); these levels are insignificant when older sediments are dated. However, more recent work has found a much wider ranges in residual doses (2 to >20Gy) after natural and laboratory bleaching of the $pIRIR_{290}$ signal (Thiel *et al*. 2011a, b; Buylaert *et al*. 2011, 2012; Stevens *et al*. 2011; Lowick *et al*. 2012; Murray *et al*. 2012, 2014; Sohbati *et al*. 2012; Kars *et al*. 2014a). The curve fitted through the open circles in Fig. 8B suggests that it is possible to bleach the $pIRIR_{290}$ signals to relatively small doses but it is not

clear whether there may be an unbleachable component. To investigate the degree to which our $pIRIR_{290}$ signal is bleachable, we have repeated the experimental procedures described by Sohbati *et al*. (2012). A set of 225 aliquots was prepared for each of three samples from the top, middle and bottom of the of the loess sequence (138102, 138108, 138115). These were exposed in groups of three to twelve aliquots per sample for various lengths of time (3.7 to 4171 h) in a Hönle SOL2 simulator at a lamp-sample distance of 80 cm. The apparent dose was measured using the $pIRIR_{290}$ protocol. The results are summarized in Fig. 9A ($pIRIR_{290}$) and Fig. 9C (IR_{50}). There is a clear tendency for the residual doses to increase with equivalent dose (D_e). Extrapolation of these residual doses to a D_e of zero Gy can be used to predict the average residual dose that would have been present in these samples before burial had they been exposed to these light levels (Buylaert *et al*. 2012; Sohbati *et al*. 2012; Kars *et al*. 2014a). The inset to Fig. 9A shows this relationship for all 15 samples for a 15 h Sol2 bleach. The intercepts of the three samples bleached for various lengths of time are plotted against exposure time in Fig. 9B,D for $pIRIR_{290}$ and IR_{50} signals, respectively. In both cases this residual appears to be consistent with a constant (or very difficult to bleach) dose after a bleaching time of ~300 h. In our view it is likely that these apparently constant difficult-to-bleach signals were present in these samples at the time of deposition and accordingly a $pIRIR_{290}$ residual dose of 6.2±0.7 Gy has been subtracted from all measured D_e values used to calculate the $pIRIR_{290}$ ages given in Table 1.

A chronology for the NYZG section: comparison of quartz and feldspar ages

Fig. 10A presents the quartz and $pIRIR_{290}$ feldspar ages on a simplified section of the NYZG site. All of the quartz and feldspar ages are consistent with each other down to a depth of 2.60 m corresponding to an age of ~70 ka. The quartz dose in the 71±5 ka sample (138112 at 2.60 m) is 212±10 Gy, consistent with the ~200 Gy limit suggested by Buylaert *et al*. (2007, 2008). This agreement is clearer in Fig. 8A which shows that there is no easily detectable systematic deviation between quartz and feldspar below ~80 ka. Only the oldest three samples show significant deviation.

The data suggest that the clearly defined soil at the bottom of this section below 2.8 m is consistent with deposition during the MIS 5 (130-75 ka). The sedimentation rate during this period increases dramatically from $0.0063^{+0.0034}_{-0.0017}$ m ka^{-1} during the interglacial to $0.16^{+0.12}_{-0.05}$ m ka^{-1} during the subsequent glacial period. The L_1 loess from ~2.7 up to 1.2 m accumulated over a time period that is small compared to our uncertainties. There may be a discontinuity above 1.2 m where the feldspar age drops from 59±3 to 44±3 ka, and another between 0.8 m (43±2 ka) and 0.6 m (32±2 ka). However, the spatial resolution of the data is insufficient to be confident of these breaks and we confine ourselves to calculating the average sedimentation rate for the

482 top 1 m of 0.031±0.005 m ka^{-1}. The OSL ages confirm that the Holocene soil is
483 missing but the weak soil developed during the warming period of the Last Glacial, i.e.
484 MIS 3, so we cannot tell whether recent deposition rates are comparable with the
485 previous interglacial.
486
487 It appears that sedimentation rate was low during MIS 5 and accelerated at the
488 onset of MIS 4. At MIS 4 there was a period of very high dust deposition followed by
489 slow deposition throughout MIS 3.
490

491 Conclusions

492
493 We have examined both quartz and feldspar from the NYZG loess deposit in
494 northeastern China in detail. The quartz is sensitive, fast component dominated and
495 not surprisingly has excellent luminescence characteristics
496
497 Based on studies of dose recovery and equivalent dose as a function of test dose
498 size measured using the $pIRIR_{290}$ signal, we deduce that the test dose employed
499 should be related to the equivalent dose under investigation. Typically it would appear
500 that a test dose of 15 to 80% of the D_e is most likely to give a satisfactory dose
501 estimate. Furthermore, we question the interpretation of MET-pIRIR data in terms of
502 increasing stability with increasing stimulation temperature. Even in a sample of

503 non-finite feldspar age, the signal resulting from a low first-IR stimulation temperature
504 in a $pIRIR_{290}$ protocol only results in a sensitivity corrected luminescence signal ~5%
505 lower than that resulting from a high first-IR stimulation temperature; contrast this with
506 the ~21% underestimate at low MET-temperatures. It appears that this underestimate
507 is at least in part an artefact of variation in dose recovery with stimulation temperature.
508 Nevertheless, both methods suggest the presence of some instability (~10% signal
509 loss) at field saturation even at the highest (first IR) stimulation temperature (Fig. 7).
510
511 Finally, we have investigated the relative bleaching characteristics of quartz OSL
512 and the IR_{50} and $pIRIR_{290}$ feldspar signals and confirmed that quartz OSL bleaches
513 considerably more rapidly than feldspar, reaching <10% of its initial value when IR_{50}
514 has only lost <50% and $pIRIR_{290}$ <20%. These data support the suggestion that the
515 ratios of the three signals can be used as indicators of the degree of bleaching of the
516 more sensitive signals. Our $pIRIR_{290}$ data are also consistent with an unbleachable
517 residual signal (equivalent to 6.2±0.7 Gy) underlying an exponentially bleached
518 signal.
519
520 At this site, quartz OSL and $pIRIR_{290}$ ages are in agreement back to ~70 ka. For
521 older sediments we deduce that quartz increasingly underestimates the deposition
522 age. Based on our preferred ages we conclude that sedimentation rates increased
523 rapidly during the Last Glacial, peaking at MIS 4 (~60 ka) at $0.16^{+0.12}_{-0.05}$ m ka^{-1}. During

MIS 3 this sedimentation rate was considerably lower but remained high at 0.031±0.005 m ka^{-1} Unfortunately, MIS 2 and 1 are missing at this site.

This study has demonstrated the consistency of quartz and feldspar ages (and probably their reliability) over the last glacial period. Beyond that, quartz is increasingly unreliable and it appears that feldspar ages are consistently more accurate.

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Figure captions

Fig.1. Map of loess distribution in northeastern China showing location of the study site (Niuyangzigou, NYZG) and its stratigraphy. L and S represent loess and palaeosol units, respectively. From the top to the bottom, the loess–soil sequences

716 are named as L_1 and S_1 followed the designation of Liu (1985). Based on the OSL
717 data the Holocene soil (S_0) appears to be missing.
718
719 Fig.2. Luminescence characteristics for coarse-grained (63-90 μm) quartz. A. and B.
720 Dose–response curves for aliquots of samples 138101and 138115 respectively (inset
721 shows the natural decay curves measured at 125 °C and at 90% blue LED power). C.
722 Preheat plateau tests of samples 138101 and 138115. Three aliquots were measured
723 at each temperature and error bars represent 1 standard error. The dash-dot line is
724 drawn at the average D_e over the 180-280 °C interval. D. Dose recovery ratio as a
725 function of preheat temperature for samples 138101 and 138107. Aliquots were first
726 bleached with blue light at room temperature (2×100 s separated by a 10 ks pause)
727 and then given a dose of 78.5 Gy and 156 Gy for 138101 and 138107, respectively.
728 The solid line is drawn at unity and the dashed lines at ±10%. Three aliquots were
729 measured per preheat temperature and error bars represent one standard error. The
730 inset shows a histogram of recycling ratios for all samples (measured as part of D_e
731 determination).
732
733 Fig.3. A. Natural signals and dose–response curves for coarse-grained quartz and
734 K-feldspar of sample 138115. Data were fitted with a sum of two saturating
735 exponential functions of the form $y=a\times(1-\exp(-bx))+c\times(1-\exp(-dx))$. The
736 sensitivity-corrected luminescence (L_x/T_x values) was normalized to the fitted

737 saturation values ('a+c'). B. L_x/T_x plots for the $pIRIR_{290}$ signal for a set of samples
738 down the section. Three aliquots were measured for each sample but only one
739 representative aliquot is shown. Sample 138101 (L_x = 100 Gy; T_x = 30 Gy); 138102 (L_x
740 = 999 Gy; T_x = 300 Gy); 138108 (L_x = 216 Gy; T_x = 65 Gy); 138115 (L_x = 362 Gy; T_x =
741 108 Gy) and the B/M boundary sample 1535 (L_x = 999 Gy; T_x = 300 Gy).
742
743 Fig.4. Result of the dose recovery test for $pIRIR_{290}$. A. Plot of the dose recovery ratio
744 versus test dose size. The test dose size various from 5 to 260% of the total
745 (natural+added) dose. The dose recovery ratio was calculated as the measured dose
746 divided by the sum of the natural and the given dose. The solid line is drawn at unity
747 and the dashed-dot lines at ±5%. B. dose recovery results show as the measured
748 dose versus added dose.
749
750 Fig.5. Dependence of D_e on test dose size for the uppermost (138101), middle
751 (138108) and lowermost (138115) samples from the upper 3.2 m section. The
752 dash-dot line is the average of the D_e value for test doses ranging between 15 and 80%
753 of the total (natural + added) dose.
754
755 Fig.6. Comparison of $pIRIR_{290}$ and MET-pIRIR. A. and B. Dependence of D_e on prior
756 IR stimulation temperature for the uppermost (138101) and lowermost (138115)
757 samples from the upper 3.2m section. Three aliquots were measured at each

758 temperature and error bars represent one standard error. The dash-dot line is drawn
759 at the average D_e over the 50-260 °C interval for the $pIRIR_{290}$. C. Dependence of
760 dose recovery ratio on prior IR stimulation temperature for the uppermost sample
761 (138101). Three aliquots were measured at each temperature and error bars
762 represent one standard error. The dash-dot line is drawn at unity on the vertical axis.
763
764 Fig.7. Fraction of saturation for both the $pIRIR_{290}$ and MET-pIRIR signals for a sample
765 from below to the B/M boundary (burial dose >2000 Gy). A. Fraction of saturation
766 measured using the large test dose. B. Fraction of saturation measured using the
767 small test dose. All the data were fitted with a single saturating exponential function.
768 Each data point is an average of three aliquots and error bars represent one standard
769 error. The solid line is drawn at unity and the dashed line at 0.9.
770
771 Fig.8. Comparison of age and bleaching rate for the quartz OSL, feldspar IR_{50} and
772 $pIRIR_{290}$ signals. A. Quartz OSL and feldspar IR_{50} ages plotted as a function of
773 $pIRIR_{290}$ age. The IR_{50} and $pIRIR_{290}$ ages have a residual dose of 0.6±0.1 Gy and
774 6.2±0.7 Gy subtracted from the D_e and are not corrected for fading. The vertical
775 dashed line shows the upper quartz OSL dating limit. B. Residual quartz OSL,
776 feldspar IR_{50} and $pIRIR_{290}$ doses for different Hönle SOL2 solar simulator bleaching
777 times for sample 138115. The solid line was derived from fitting the IR_{50} to $pIRIR_{290}$ D_e
778 values for all 15 samples. Each data point is an average of three aliquots and error

779 bars represent one standard error.

780

781 Fig.9. The relationship between the residual dose and D_e obtained with the $pIRIR_{290}$
782 protocol on three samples (138102,-08,-15). A. and C. Average residual dose
783 obtained after different Hönle SOL2 solar simulator bleaching times as a function of
784 the $pIRIR_{290}$ and IR_{50} D_e, respectively. Each point is the average residual dose of
785 three to twelve aliquots per sample obtained after the given exposure times and error
786 bars represent 1 standard error. The relationship between residual dose and
787 equivalent dose is described by a linear fit for each exposure time. Inset in A. is the
788 same relationship for all 15 samples for a single bleaching time of 15 h. B. and D.
789 Intercept of the linear fits with the y-axis from A. and C. as a function of exposure
790 time.

791

792 Fig.10. Stratigraphy, quartz OSL and feldspar $pIRIR_{290}$ ages for NYZG. The ages are
793 plotted against the section depth. Open circles represent the quartz OSL ages and
794 closed circles represent the $pIRIR_{290}$ ages. Sedimentation rates were derived from
795 linear regression.

796

797 **Table captions**

798

799 Table 1. Summary of sample code and depth, radionuclide concentrations, calculated

dose rates, OSL and $pIRIR_{290}$ D_e values and luminescence ages. The absolute uncertainty on the water content is ±5%. The $pIRIR_{290}$ D_e values have a residual dose of 6.2±0.7 Gy subtracted from the measured value while the $pIRIR_{290}$ ages are not corrected for fading. (n) denotes the number of aliquots contributing to the D_e.

Table 2. Outline of dose measurement protocols used in this study. SAR protocol after Murray & Wintle (2000, 2003), post-IR IRSL protocol after Thiel *et al*. (2011a), MET-post-IR IRSL after Li & Li (2012a). For the 'natural' sample, the give dose = 0. T varies from 50 to 260 ^{o}C. The whole sequence is repeated for several regenerative doses including a zero dose and a repeat dose.