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Climate of the Past
Discussions

Interactive comment on "Geochronological reconsiderations for the Eastern European key loess section at Stayky in Ukraine" *by* A. Kadereit and G. A. Wagner

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First of all, we would like to thank the referees for their both critical and very constructive comments. Several points were addressed by the referees, which are fundamental to the controversially discussed topic. These include differences in the basic philosophies underlying correlations of terrestrial loess-palaeosol sections with marine and ice core records as well as different assessment of, on the one hand, established and, on the other hand, novel luminescence dating techniques.

Both referees suggest adding a graph that outlines the main characteristics of the pedosedimentary sections at Stayky, Nussloch and Schwalbenberg which would help the

reader to better follow the argumentation. This is a welcome suggestion.

Referee-#1 disapproves of regarding the Lohne Soil resp. Vytachiv Soil as marker horizons 'not unless they have been dated as the same age using radiometric means'. Contrary to that opinion, in European loess research the Lohne Soil serves as a pedostratigraphic marker horizon, which at its upper boundary marks the classical boundary of the terrestrial Middle Pleniglacial to the Upper Pleniglacial of western Europe sensu Schönhals et al. (1964, Eiszeitalter & Gegenwart 15, 199 - 206). In the literature, the Lohne Soil (and its equivalents) is regularly addressed as a 'marker horizon', e.g. in:

- Semmel (1995) in Eiszeitalter & Gegenwart 45, page 59: Die von Schönhals et al. (1964) und Semmel (1969) publizierte Würmlößgliederung für Hessen hat sich in den letzten Jahrzehnten als im westlichen Mitteleuropa in vielen Fällen anwendbar erwiesen. [...] Besonders markante Leithorizonte sind der "Eltviller Tuff" (Semmel 1967) und der "Lohner Boden" (Schönhals et. al. 1964).

- Terhorst et al. (2001) in Quaternary International 76/77, page 237: "[...] correlation with marker horizons, like the Lohner soil or the older Gräselberg soil [...]"

- Wagner, B. (2011) in Eiszeitalter & Gegenwart 60/1, page: 28: "[...] or maps, displaying the distribution of marker horizons like the Lohne soil [...]

- Zöller & Semmel (2001) in Earth-Science Reviews 45, page 23: "The Lohne soil, which terminates the Middle Würmian" in sensu Schönhals et al. (1964), corresponds, pedologically and stratigraphically, to the Brauner Verwitterungshorizont (brown weathering horizon) of Brunnacker (1954, p. 85.), a marker horizon interpreted at that time as the W I/II soil formation.

Also, in scientific practice and fieldwork, it is treated like a marker horizon when, e.g., Antoine et al. (2009, QSR 28, pp 2859 - 2973) and Rousseau et al. (2011, Clim. Past 7, 221 - 234) start counting of Dansgard-Oeschger events resp. Greenland Interstadials (GIS) and Greenland Stadials (GS) from above the Lohne Soil. It is a practical way to gain orientation in geological time within a pedosedimentary section despite the fact that up to now the likely time window for the development of the Lohne Soil ranges between GIS8 and GIS5 and a more precise timing is still under discussion. Traditionally, a 'marker horizon' denotes a more or less easily recognizable stratigraphic unit, for which more or less isochronic development in different parts of the world may be assumed, even if it has not been radiometrically dated (cf. respective entries/definitions, e.g., in Neuendorf, Mehl & Jackson (eds., Glossary of Geology, published by AGU 2005) and Whitten, D.G. A. & Brooks, J.R.V., The Penguin Dictionary of Geology, 1982, p. 283). We added 'pedostratigraphic' to include readers, who use the term 'marker horizon' exclusively for radiometrically dated strata/horizons and not in a stratigraphic sense. Irrespective of whether the Lohne Soil will finally be attributed to GIS8 or GIS7-GIS5, its upper boundary will still delineate the terrestrial Middle Pleniglacial/Upper Pleniglacial (MPG/UPG-) transition, and therefore a (conceptual) time boundary. If, in practice, the Lohne Soil was not treated as a marker horizon, results for the Lohne Soil from the key loess sections like, e.g., Nussloch would not be transferred to other profiles. Also, other pedosedimentary strata/horizons are treated as markers before they are radiometrically dated (cf., e.g., Rousseau et al. 2013, Clim. Past 9, 2213 – 2230).

We agree that there is controversy on the rhythmicity of Dansgard-Oeschger events and on the reaction and relaxation of the terrestrial geomorphic systems, which may be complex or even chaotic, possess internal thresholds as significant steering parameters etc. However, for the loess-palaeosol sections in question – and especially those parts that are dominated by accumulation not showing any evidence of intermittent erosion – which were situated in a periglacial environment with temperature being a major limiting factor for pedogenesis, it seems legitimate to correlate loess strata with the colder periods resp. GS and soils with the warmer periods resp. GIS.

Both reviews state that it would be beneficial to have more chronometric data for the discussed eastern European key loess section, and we fully agree to that opinion. In our manuscript we suggested intensifying dating in future studies. This way

the presently likely interpretation could either be supported or challenged. Following referee-#1, one to one correlations of palaeosols with Greenland interstadials (GIS) have to be tentative, and we fully agree to this, as stated in our manuscript. To our mind, however, a tendency whether a palaeosol corresponds rather to GIS8 or GIS5, and thus whether the MPG/UPG transition belongs rather into an earlier or a later phase of MIS3 may be deduced from the available data.

Although we strongly support the idea to produce more good data for Stayky and other important loess sections we think that in the foreseeable future it will not be possible to establish chronometric frameworks of similar resolution and precision for the terrestrial pedosedimentary archives as they already exist for the marine and ice-core records. This argument is independent of the likely reliability of certain dating protocols, which is another point of controversial discussion addressed below. Even with highly precise (and accurate) dose-equivalent (DE) determination, luminescence ages of natural sediments will always have quite large errors, due to, e.g., the influence of water-content estimations on age calculation. Therefore, we do not conform to referee-#1 who critically notes that we use 'tentative stratigraphic supporting evidence' in addition to the chronometric data. We think that for this type of terrestrial archives, numerical chronometry alone will never be sufficient to provide good correlations. Rousseau et al. (2011) analyzed the profile in a multiply stratigraphic manner, and this multiplicity is exactly what allows (tentative) correlation with the marine or ice-core stratigraphy and deduce (tentative) models on how the terrestrial palaeoenvironment reacts to climate change. Numerical dating can provide only chronometric tie points, which help place (parts of) a studied section into a likely correct time window. Depending on the error margins, the width of a respective window is narrower or broader. Once this general placement has been done, other means have to be used for the chronological fine tuning. There are cases, in which the stratigraphic data may challenge the results of the numerical data and/or vice versa (cf. e.g. V-S1 at Crvenka, in Stevens et al. 2011, QSR 30, 662 - 681). But in general, the numerical chronometry is a most important starting point for a detailed and reliable interpretation of the stratigraphic data. A recent example for the same kind of approach is the study of Rousseau et al. 2013: the numeric time windows provided by luminescence dating are too wide to unambiguously correlate the markers with any unique events; this can only be done if in addition stratigraphic information/correlation is accepted, which is usual practice. On this issue, we have a different philosophy as referee-#1.

Both reviewers point to the fact that in the original publication of Rousseau et al. (2011) the dating method was not sufficiently described which leaves room for speculations on the reliability of the dating. However, dating of the Stayky samples was performed in the established luminescence dating laboratory of Bayreuth/Germany. Multiple aliquot (MA) measurements were done detecting the blue feldspar emission around 410 nm using the glass filter combination of BG39, 2 x BG3 and GG400 (Schott) as recommended by Krbetschek et al. (1996). In between sample irradiation and IRSL-readout, samples were stored at room temperature for at least 4 weeks in the dark following Lang et al. (1996, Ancient TL 14, 7-11) and Mauz et al. (2002, Ancient TL 20/2, 53 - 61) or a minimum of one week at 70 °C following Berger (1987, Canadian Journal of Earth Sciences 24, 1975-1984). Prior to IRSL-readout samples were preheated for 60 s at 270 °C (information on measurement parameters by courtesy Ludwig Zöller/Bayreuth). Thus, precaution was taken to avoid anomalous fading. Several publications, both for TL and IRSL, had shown that observed signal losses reach a (measurable) standstill after either (1) sample storage at room temperature for several weeks or (2) shorter storage at elevated temperature (e.g. Berger 1987; Lang 1996, HGA 103, 137 pp.). Equally important, however, is the choice of a detection window suitable for luminescence dating that focusses on a stable emission, as, e.g., the blue emission around ca. 410 nm, and does not include any instable signal, as, e.g., the 280 nm emission (cf. e.g. Krbetschek et al. 1996; Lang 1996). Considering these rules, with multiple aliquot additive (MAA) protocols, IRSL ages from a few ka to ca. 120 ka may be gained, which are in agreement with independently derived ages (compilations, e.g., by Lang 1997, 4th International Conference on Geomorphology, Bologna, cf. Suppl. Di Geografica Fisica e Dinamica Quaternaria, Suppl. III-1997, p. 241; Rieser & Wang 2011,

Poster 13th LED Torun, cf. Book of Abstracts, p. 169). Practically, storage can be performed only with MA protocols, but not with single aliquot regeneration (SAR) protocols (Murray & Wintle 2000, Rad. Meas. 32, 57 - 73). To compensate for the lacking storage time, prior to IRSL stimulation/readout the latter require adjusted/stronger thermal pretreatment within the luminescence reader (so called 'preheat'). Another important difference between MAA and SAR protocols is that, apart from the additional laboratory irradiation, treatment of the sample for measurement of the natural luminescence and treatment of the sample for measurement of the growth curve dose points is identical, and no possible sensitivity changes have to be corrected for. From this it follows that measurement results gained with SAR protocols are not a priori comparable with those gained with MAA protocols. This includes results from fading measurements. Therefore we do not follow the philosophy of the referees, who, based on several SAR studies which report significant fading for polymineral fine grains, question the reliability of MA measurements resp. any uncorrected IRSL ages. In our opinion, comparison is reasonable only to MAA measurements with similar measurement parameters (i.e. comparable storage time, preheat procedure and detection window; e.g. Lang et al. 2003, QSR 22, 953 - 959) and to 'plain' SAR measurements with comparable measurement parameters (i.e. comparable detection window; adequate preheat procedure; IRSL detection at a moderate temperature; no additional optical and/or thermal treatment of the sample not necessary for IRSL DE determination; e.g. Lomax et al. 2012, QI (in press), 1-10). This is why we favor the study of Lomax et al. (2012) for comparison, and not the other studies quoted by the referees.

Fading measurements in addition to the fading prevention measures would be desirable for the Stayky samples. However, basic skepticism of uncorrected IRSL ages appears to be unnecessary. In the following, we explain our dissenting opinion with reference to the study of Vasiliniuc et al. (2013, QI 293, 15 - 21), which both referees cite as an example. The study is adequate as it is technically well conducted and each step is clearly documented.

The study is on polymineral fine grains (4 – 11 μm) extracted from twelve loess samples collected at a loess-palaeosol section at Mircea Voda in SE-Romania. Following Banerjee et al. (2001, Rad. Meas. 33, 73 - 94), the authors used a double-SAR protocol resp. a combined IRSL- and post-IR BLSL-protocol. The authors found significant fading rates, both for the IRSL signal and the post-IR BLSL-signal. The g-value (in % per decade, assumed to denote the athermal resp. anomalous signal fading of a sample/aliquot per decade; cf. Aitken 1985) of the IRSL signal being ca. twice as high as the g-value of the post-IR BLSL-signal (ca. 4.0 $\pm$ 0.1 % versus 1.8 $\pm$ 0.2 %). Therefore the authors corrected the equivalent doses of both the IRSL-dating as well as the post-IRSL BLSL-dating using the method provided by Auclair et al. (2003, Rad. Meas. 37, 487 – 492). As a result, both the IRSL- and the post-IR BLSL-ages were in agreement, with the exception of the upper three samples, for which the IRSL-ages overestimated the post-IR BLSL ages, likely due to insufficient bleaching of the IRSL-signal. Further, the results of the fading corrected IRSL- and post-IR BLSL ages were in agreement with formerly produced BLSL-ages on pure quartz fine-grains (4 – 11 μm), extracted from the same samples and published by (Timar-Gabor et al. 2011, QI 240, 62 - 70). However, the results were not in agreement with BLSL-ages from sand-sized quartz from the same samples, which significantly overestimated the fine-grain ages. Discrepancies between DEs of coarse and fine grains are not understood (Timar-Gabor et al. 2011). Agreement among the fine-grain ages was taken as an indication of the reliability of the younger ages as compared to the older ages from the sand-sized fraction, which were interpreted by Vasiliniuc et al. (2013) to overestimate the true ages of the loess deposition.

However, the need for correction of the IRSL-ages is not a general characteristic of the feldspar dosimeter but it is largely owed to the chosen measurement setup:

(1) Banerjee et al. (2001) had not developed the protocol for IRSL dating of the feldspar component of polymineral fine grains, but for BLSL-dating of the quartz component, after the IR-stimuable feldspar component had been bleached. Therefore, the mea-

surement setup was optimized for quartz detection.

(2) The detection filter (U340, Schott) excludes the stable feldspar emissions around 560 nm (yellow) and 410 nm (blue) but centers in the UV, thus including the possibly existing instable emission around 280 nm. From this it follows that high g-values and underestimating dose equivalents (DEs) are not surprising but to be expected. Therefore, it is not correct to draw a general conclusion that IRSL-ages need correction and cannot be trusted if no correction was applied. Unfortunately, such general skepticism of (uncorrected) feldspar ages seems to become common opinion. Yet, the need and the degree for correction depend largely on the measurement setup.

(3) Additional to the inadequate detection filter (i.e. inadequate only for IRSL/blue detection of feldspar, not for the subsequent BLSL/UV quartz detection), the chosen preheat procedure of 10 s at 240 °C appears too mild to eliminate instable components sufficiently. More rigorous preheating as applied, e.g., by Lomax et al. (2012) is regarded more suitable for IRSL SAR dating of feldspar.

(4) The underestimating DEs (1) may be used to calculate minimum ages or (2) they may be corrected to calculate ages which likely present the dating event. The authors chose the second possibility. In table 2 no errors were given for the OSL-ages. They will probably be in the range of ca. 10 %. Errors of the g-values, however, amount up to 50 %. Therefore, the corrected ages should also have errors (1 sigma) of ca. ? 50 %. However, it does not seem desirable to produce luminescence ages with such large errors. If possible, we would prefer to use a measurement setup which requires less or no correction.

(5) In contrast to this, MAA measurements in the study of Rousseau et al. (2011) were done on the blue feldspar emission around 410 nm (cf. Krbetschek et al. 1996) and IRSL-readout occurred after sufficient sample storage. Therefore, potential g-values may be assumed to be much smaller or negligible (e.g. Lang 1996; Fuchs et al. 2008, Boreas 37, 66 – 73; Necea et al. 2013, Tectonophysics 602, 332 - 354). Measurement

parameters of the Stayky ages are more comparable to the ones in the aforementioned studies as well as the study by Lomax et al. (2012), for which only slight deviations were found for the IRSL-ages as compared to the quartz and 14C-ages. Also, according to the authors it is not clear whether the slight differences are owed to anomalous fading or to other circumstances, like e.g. a-value determination or others. Fading measurements, after all, did not reveal any clear trend.

Both referees also quote a study of Stevens et al. (2011) who use another type of novel SAR protocol which is presently under development for the dating of feldspar resp. the feldspar component of polymineral fine grains (e.g. Buylaert et al. 2009, Rad. Meas. 44, 560 – 565; Thiel et al. 2013, QI 234, 23 – 31). DE-determination is based on IRSL-readout at elevated temperature (Buylaert et al. 2009: 225 °C; Stevens et al. 2011/Thiel et al. 2013: 290 °C) following rigorous preheating (Buylaert et al. 2009: 60 s at 250 °C; Stevens et al. 2011/Thiel et al. 2013: 60 s at 320 °C). Prior to IRSL-readout at 290 °C for 200 s, the IRSL-signal is read out for 200 s at 50 °C, in order to allow any unstable traps recombine with holes in nearby recombination/luminescence centers. This means that, prior to post-IR50 IRSL290 measurement, the signal traditionally used for IRSL dating has been bleached away. As the post IR50 IRSL290 signal is difficult to bleach, this hard to bleach background has to be corrected for. Additionally, for thorough clearance of the IRSL-traps in between individual SAR-cycles an IRSL-hotbleach for 200 s at 325 °C is performed. Thus, the IRSL-DE which is read out at a moderate temperature is determined as a by-product of the post-IR50 IRSL290 DE-determination, i.e. in between several measurement impacts on the aliquot which are not necessary for IR50-DE-determination but may influence the measurement results. g-values are significantly higher for IR50 than for postIR50 IRSL290. However, g-values of ca. 1 % and more per decade are also observed for postIR50 IRSL290 measurements. As this is inconsistent, e.g., with the observation that geologically old samples, e.g., from around the Brunhes/Matuyama boundary are found to be in saturation, g-values are (partially) regarded as laboratory artifacts (e.g. Thiel et al. 2013). Using these for age corrections, would lead to age overestimation. Another aspect of

the study of Stevens et al. (2011) is that the expected age of the V-S1 soil (expected to represent MIS5) is not met. This is in contrast, e.g., to the IRSL MAA dating for Nussloch which reproduced the expected MIS5-age, and the IRSL SAR dating for Krems-Wachtberg which met the 14C-age of ca. 31 ka within error margins. Therefore, we think that the post-IR IRSL protocols are most valuable developments, but at present they are not sufficiently understood to deduce from them general findings which could justify general criticism of established MAA protocols in the age range of the last ca. 120 ka. Anyhow, in their final discussion, Stevens et al. (2011) discarded their own IRSL- and post-IR IRSL SAR ages up to ca. 50 ka preferring the quartz OSL ages. But they included the IRSL ages from Surduk, which were produced with a traditional MAA protocol by Fuchs et al. (2008).

The two studies by Vasiliniuk et al. (2013) and Stevens et al. (2011) show that it is important to improve and further develop luminescence dating techniques. But still, dating results may be not unequivocal, possibly in conflict with stratigraphic information and therefore open to evaluation and interpretation.

Summarizing there are different philosophies: (1) With respect to the use of ‘marker horizon’ being (a) reserved for radiometrically precisely dated units only or (b) including also stratigraphic units of likely isochronic age serving as practical guiding lines e.g. in fieldwork. (2) Whether supportive stratigraphic information from pollen etc. is welcome to establish chronologies for (the mostly fragmentary) terrestrial pedosedimentary archives and their correlation with marine and ice core records, or whether numerical dating alone with a higher sampling resolution will suffice. (3) Concerning an assumed obsolescence of IRSL MAA protocols and a priori superiority of pIRIR and other derivatives of the SAR protocol for the dating of polymineral fine grains. (4) Whether uncorrected IRSL feldspar ages in the range up to ca. 120 ka are per se suspicious to underestimate the true ages severely, or whether the need and degree of correction may be owed to the applied dating protocol.

Lastly, we would like to point to two findings, which are notable with respect to the likely temporal placement of the MPG/UPG-boundary. Considering the quartz OSL data preferred by Stevens et al. (2011) to establish a chronology for the upper part of the Crvenka loess section, loess accumulation on top of VL1-S1 starts around 33 $\pm$ 3 ka, thus providing an estimate for the onset of the Upper Pleniglacial. Although, in our manuscript we stuck to sections further north, as not to mix areas influenced mainly by the Atlantic regime with areas under Mediterranean influence, the Crvenka section may play a special role in connecting the two. Recently, Spoetl et al. (2013, JQS 28/6, 552 - 558) published a new 14C-based chronometry for the type site of the onset for the Upper Wurmian at Baumkirchen/Austria. They constrain the change from MPG- to UPG palaeoenvironmental conditions to 32 – 33 cal ka BP. These results are in agreement with the results of Lüthgens (2010, Quat. Geochronology 5, 237 - 243) who suggest an early LGM ice advance into northern Germany after ca. 34 ka, the post-Lohne Soil onset of loess accumulation at Nussloch ca. 31 – 32 ka, and the change of palaeoenvironmental conditions as they might - at least tentatively - be deduced for Stayky from the data reported by Rousseau et al. (2011).

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