Thesis Abstracts

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Thesis Title: Luminescence dating of heated silex – Potential to improve accuracy and precision and application to Paleolithic sites
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Thermoluminescence (TL) dating of heated silex artefacts represents an important chronometric tool for Middle to Upper Paleolithic archeological contexts. Since the firing of these lithics can legitimately be attributed to human agency, this method provides direct age estimates of the occupation of a site. However, in relation to other dating methods, the precision of TL dates is comparatively low, and the often observed "overdispersion" in ages of obviously syndepositional finds indicates additional sources of scatter mainly disregarded so far. This thesis examines potential sources of both imprecision and inaccuracy of age estimates as well as the capability of alternative approaches to overcome or reduce these shortcomings of the TL method applied to heated silex.

Besides assessing the influence of spurious luminescence signal contributions from sample carriers on determined dose, focus is set on investigating strength and uniformity of the internal dose rate of silex samples and resulting effects on the age. Being constant over burial time, self-dosing may either decrease the influence of elusive and variable external radiation – in case of homogeneous radioelement distribution within the sample – or introduce systematic errors and enhanced data scatter, if radiation is concentrated in "hot spots". With α- and β-autoradiography and laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS), both qualitative and quantitative approaches served to draw a detailed picture on presence and spatial distribution of hot spots and associated impurities in thick sections of over 20 silex specimens. While the low β-activity of most samples is rarely visible on respective autoradiographs, recorded α-tracks revealed zones and spots with strongly increased Th and U concentrations. Comparison of autoradiographs with sample surfaces shows a clear relationship between track density and impurities or filled cracks. These findings are confirmed by LA-ICP-MS: In contrast to the low radioactivity of pure silex, most kinds of impurities are related to strongly increased radiation. If this is not accounted for, micro-dosimetric effects may lead to random and systematic errors in age determination.

Additionally, the performance of TL single-aliquot regenerative-dose (SAR) protocols for dating small-sized samples and as a diagnostic tool for non-uniform dose distribution in the samples was tested. For both the blue (~475 nm) and red (~630 nm) TL emission it was found that the degree of adequate sensitivity-correction by test dose monitoring strongly depends on the properties of individual samples. However, laboratory doses could be well reproduced for most specimens. By comparing De distributions from natural dose measurements and dose recovery tests and regarding the value range of sensitivity corrected natural signals (L_n/T_n), assessment of sample homogeneity and SAR dose response behavior is enabled, respectively.

Unlike for commonly used TL, few is known about optically stimulated luminescence (OSL) of silex. However, access to specific, optically sensitive trap populations (e.g. slow-components) and more gentle heat treatment in the course of SAR sequences may yield the benefits of increased saturation dose levels (and hence upper dating limits) and reduced systematic errors, respectively. Therefore, fundamental properties such as signal composition and thermal long-term stability of silex OSL were studied and its applicability to heated samples evaluated. First-order fitting of linearly modulated (LM-) OSL curves showed best results for five components in most cases. Pulse annealing experiments and the varying heating rate method, however, attested only the best bleachable component sufficient thermal stability. Successful dose recovery tests and OSL ages in agreement to TL ages of the same samples validate the general applicability of this OSL component for dating. However, since not all silex varieties show an optical signal at all, this approach may be regarded as complementary to TL and reassuring for important samples.

In the applied part of this thesis, several sets of heated artefacts from Middle and Upper Paleolithic

sites in Portugal, Spain, Romania and Egypt were TL and OSL dated. Gravettian samples from Vale Boi (Portugal) were submitted for dating after termination of the excavation, so that environmental radiation could not be determined accurately. This is very likely the reason for the significant deviation between radiocarbon dates of the same layer and TL ages, because SAR and multiple-aliquot additive-dose (MAAD) ages of the same sample agree and other influencing variables such as the α-efficiency or the moisture content have too little impact on the calculated age as to explain the observed discrepancies. The SAR approach allowed dating the relatively small silex samples from the cave site Las Palomas (Spain). Here, successful dose recovery tests indicated the suitability of the measurement procedure, and in-situ measurements of γ-radiation provided more accurate dose rate information. As a result, TL ages between ~51 and ~84 ka allow a first chronometric assessment of the formerly undated archeological remains. Artefacts from the Aurignacian site Româneşti-Dumbrăviţa I (Romania) proved to be more problematic, as part of them had to be discarded due to poor dose reproducibility. SAR measurements, dose recovery tests and their detailed analyses suggested internal non-uniformities of most samples, so that only rigorous data filtering allowed extraction of reliable age estimates. Due to the luminescence behavior of dated samples, the occupation of the site could, however, not be narrowed down to a range smaller than ~39-45 ka, giving a weighted average age of 40.6 ± 1.5 ka for the Aurignacian find layer. Furthermore, two TL emissions (blue and red) and two protocols (SAR and MAAD) were applied to heated silex from Sodmein Cave (Egypt). Dose recovery tests showed uncorrectable sensitivity changes of the TL signal, rendering the TL SAR sequence inaccurate for these samples. Consequently, age estimates are based on TL MAAD and OSL SAR data; for the smallest sample only a TL SAR maximum age can be given. Coming from different depths of a huge hearth, TL ages of dated samples indicate a large time span to be condensed in the archeological deposits, probably reaching from MIS 5e to MIS 5a.

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