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A ROMAN-PERIOD DENTAL FILLING MADE OF A HARD TISSUE COMPOUND?

BIOARCHAEOLOGICAL AND MEDICAL-HISTORICAL INVESTIGATIONS CARRIED OUT ON A ROMAN-PERIOD BURIAL FROM OBERLEUKEN-PERL (LKR. MERZIG-WADERN / D)

During excavation work carried out in 2001 near the open-air site and museum »Villa Borg« in Oberleuken-Perl (Lkr. Merzig-Wadern / D) the skeletal remains of a single individual were found in a prone position. The archaeological context attested to its dating to the Roman Imperial period. The skeletal remains were recovered and, between 2005 and 2013, underwent anthropological and archaeometric examinations at the Institut für Anthropologie at Johannes Gutenberg-Universität Mainz. Because of its unusual burial position, particular care was taken to identify potential traces of injuries resulting from violence. Another goal of the examination was to study an unusual dental feature which pointed to a possible »dental filling« in one of the teeth. Whilst indirect evidence in the form of dental cavities and possible fillings consisting of bitumen or beeswax is known from as far back as the Upper Palaeolithic and Neolithic (Coppa et al. 2006; Bernardini et al. 2012; Oxilia et al. 2017), confirmed evidence of dental fillings only exists from the 17th century onwards (Hoffmann-Axthelm 1979; Riethe / Czarnetzki 1983; Alt 1993; 2011). This study aims to present a comprehensive bioarchaeological, radiological and material analysis of the exceptional skeletal remains from Oberleuken-Perl using a broad range of methods.

THE ARCHAEOLOGICAL CONTEXT

The Archaeological Park Roman Villa Borg, which the Oberleuken »Auf Schiffels« excavation site was part of, is located between the communities of Borg and Oberleuken in the municipality of Perl in the tri-border region of Luxembourg, France, and Germany. The »Auf Schiffels« site is situated some 300 m north of the actual villa's outbuildings (**fig. 1**) and has so far yielded just a small number of structures, of which only those west of the Roman road have been excavated (Birkenhagen 2010; Birkenhagen / Galla-Feld 2011). In addition to the walls of a relatively large building (building 1), possibly a *mansio*, the remains of three other houses were uncovered (**fig. 2**). Two wells and a feature which might have served as a horse pond also came to light. As shown by the magnetometric examination east of the Roman road, we can expect to find further ground plans (of probably three to four buildings) there.

The excavations at the »Auf Schiffels« site were prompted by the construction of a parking lot. The archaeological fieldwork was carried out in three consecutive campaigns from summer 1999 to summer / autumn 2001. The excavation overall extended over an area of c. 100 m × 60 m from north to south.

The structure and ground plan of building 1 were consistent with that of an inn with a large courtyard for horses and carriages and an accommodation with bathing facilities for travellers (in other words, a *mansio*). The surrounding buildings might have served as living quarters for tradesmen and staff. Post-excavation

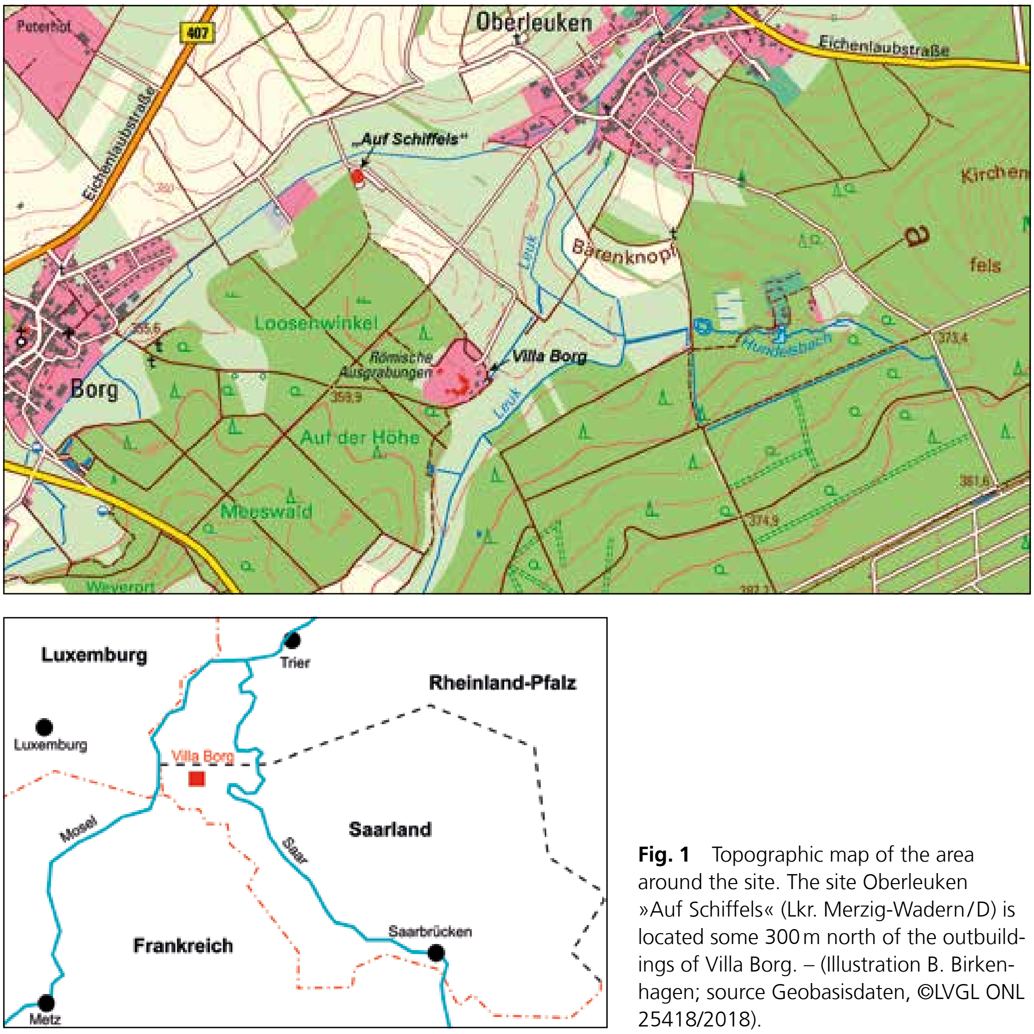

Fig. 1 Topographic map of the area around the site. The site Oberleuken »Auf Schiffels« (Lkr. Merzig-Wadern/D) is located some 300 m north of the outbuildings of Villa Borg. – (Illustration B. Birkenhagen; source Geobasisdaten, ©LVGL ONL 25418/2018).

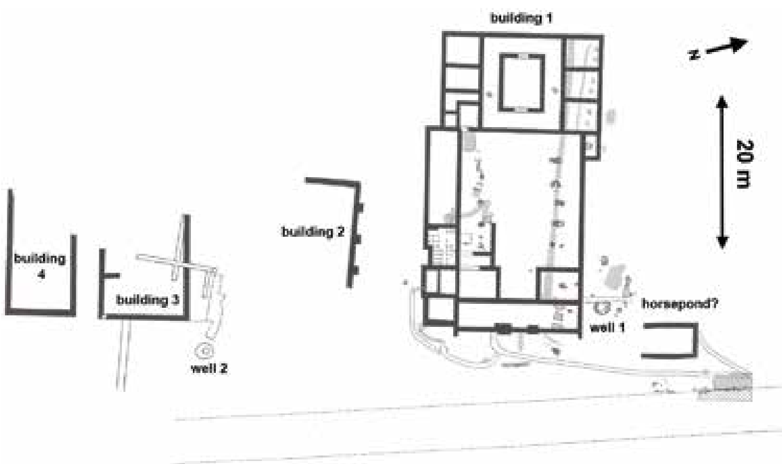

Fig. 2 Oberleuken »Auf Schiffels« (Lkr. Merzig-Wadern/D). Plan of the reconstructed Roman buildings. – (Illustration B. Birkenhagen; source Landesdenkmalamt Saarland).

work is still ongoing, but the analysis of the archaeological material is expected to confirm this interpretation of the site (Birkenhagen 2010; Birkenhagen/Galla-Feld 2011).

The skeleton was found during the final excavation campaign in the area of building 3 (area 52/45, site 180; **fig. 3**). No burial pit or soil stain was visible in the top layers. The skeleton was only found when the earth was removed, and the back of the skull came to light. When the remains were fully exposed, it became obvious that the deceased had been buried on his stomach with his head pointing towards the north (**fig. 4**).

According to the initial observations made by the excavator, Dr. Salomé Galla-Feld, the skull was located some 7 cm beneath the surface on the same level as a »stone paving«. On closer inspection, the latter appears to be the bottommost layer or the remnants of a wall, which apparently belonged to an early construction phase of building 3. The wall is located immediately adjacent to another wall foundation (site 190), which was still intact. The upper body was covered by local limestone and appears to have lain on top of the same type of material. From the waist down, the cover stones decreased in number and became smaller. The context indicated that a wall had been stripped down to its foundations. The remains of the foundation trench and a small section of the bottommost foundation layer had been the only elements that remained extant. This »pit« had been used to deposit the body, which had then been covered with any available stones and rubble. Unfortunately, the excavation did not continue after the recovery of the skeleton and the ground beneath it was not examined any further. The records, therefore, do not indicate whether the skeleton had lain on another archaeological layer or the natural soil. The section drawings do not provide any further information either.

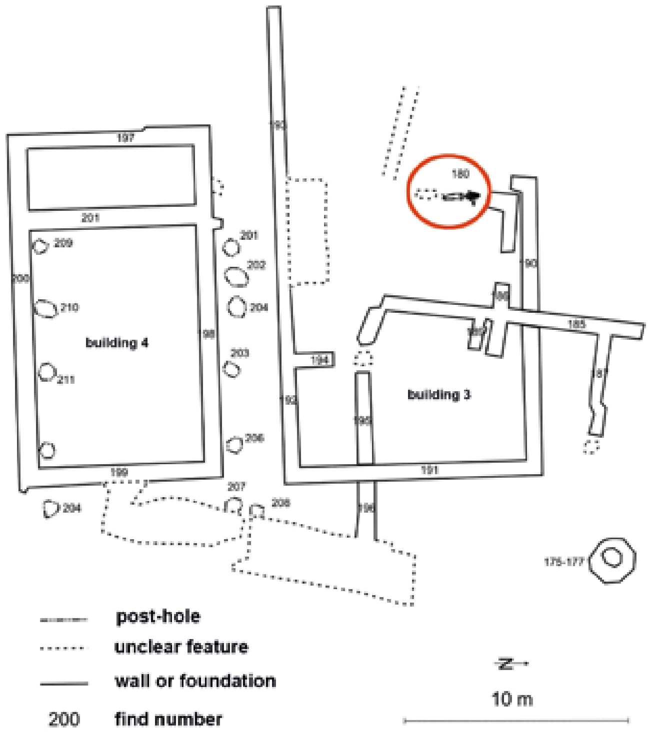

Fig. 3 Oberleuken »Auf Schiffels« (Lkr. Merzig-Wadern/D). Plan of buildings 3 and 4, showing the location of the skeleton (site 180) in building 3. – (Illustration B. Birkenhagen; source Landesdenkmalamt Saarland).

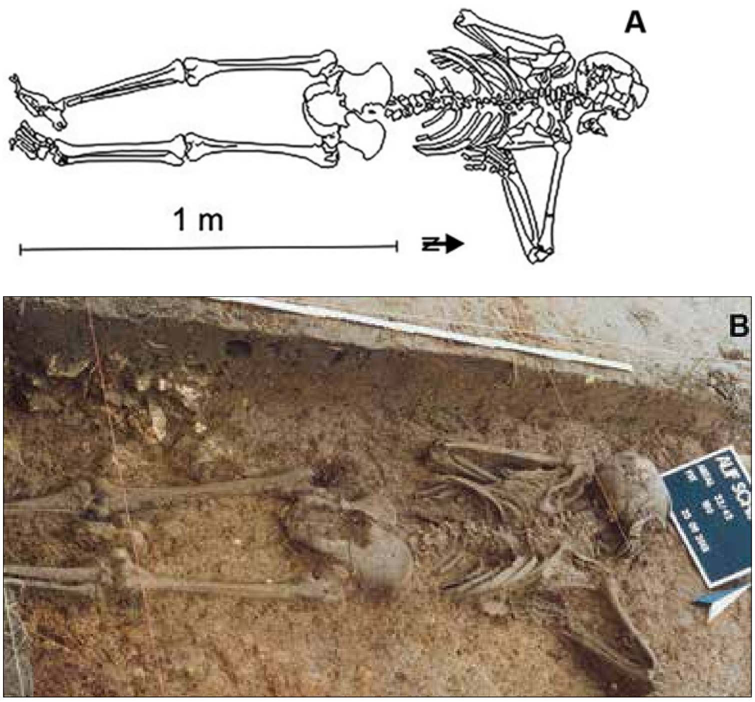

Fig. 4 Oberleuken »Auf Schiffels« (Lkr. Merzig-Wadern/D). Original position of the skeleton (site 180) lying face-down with its head pointing north. Ink drawing (**A**) and photographic documentation (**B**). – (Illustration and photo B. Birkenhagen; source Landesdenkmalamt Saarland).

MATERIALS AND METHODS

Osteological examination

To begin with, the skeletal remains were washed and dried, fragmented bones were reconstructed and the state of preservation was recorded. Age and sex are some of the basic data obtained from an osteological examination (Ferembach / Schwidetzky / Stloukal 1980; Buikstra / Ubelaker 1994; White / Folkens 2005). Sex determination was carried out using the classic features of the skull and pelvis (Phenice 1969; Ferembach / Schwidetzky / Stloukal 1980). Age determination was obtained by assessing the extent of closure of the cranial sutures (Meindl / Lovejoy 1985), the degree of dental wear (Miles 1963; Lovejoy 1985), the age-related changes to the sternal rib ends (Iscan / Loth / Wright 1984) and the auricular surface of the pelvis (Lovejoy et al. 1985). Measurements of the long bones were used to determine the individual's body height (Pearson 1899). Because of the unusual location and context, special attention was paid to potential pathological changes and injuries to the bones, particularly to determine the circumstances of death (Steinbock 1976; Aufderheide / Rodriguez-Martin 1998; Ortner 2003). Beyond the conventional anthropological study, methods of biogeochemistry and material analysis were also employed. These were used to identify the composition of the possible dental filling.

Radiocarbon dating

In 2014, a bone sample was taken from one of the femurs and sent for radiocarbon dating to the Klaus-Tschira-Labor für Physikalische Altersbestimmung at the Curt-Engelhorn-Zentrum Archäometrie gGmbH (Mannheim), to obtain an absolute date for the feature (MAMS 20490). Collagen was extracted using the modified method developed by R. Longin (1971), the desired fraction (> 30 kD) was separated by ultrafiltration, freeze-dried and then combusted. The CO_2 was converted to graphite by catalytic reduction and the ^{14}C content measured using the MICADAS-AMS system (Kromer et al. 2013).

Isotope analyses

Strontium isotope ratios ($^{87}Sr/^{86}Sr$) in the dental enamel are indicators of human mobility. During the formation of a person's dental crowns, the dental enamel stores information regarding the geological environment of the location where they spent their childhood and early adolescence from the food and water they consumed there (Knipper 2004; Bentley 2006). The analysis was carried out at the Curt-Engelhorn-Zentrum Archäometrie using the methodology described by C. Knipper et al. (2012). In this case, tooth enamel samples were extracted from three molars (teeth 16, 17, 18) and a bone sample was taken from the left femur. A bone sample from a pig was used as a local comparative sample. After grinding, purifying and incinerating the samples, the strontium was extracted under cleanroom conditions using Sr-Spec ion-exchange resin; the Sr concentration in the resulting solution was then measured using a single collector ICP (inductively coupled plasma) mass spectrometer. A multi-collector ICP mass spectrometer (VG Axiom) was used to identify the isotope ratios. The data obtained were normalised to $^{88}Sr/^{86}Sr = 8.375209$ based on the exponential mass fractionation law.

It has become a standard process in archaeometry to analyse stable carbon ($\delta^{13}C$) and nitrogen isotopes ($\delta^{15}N$) from the archaeological source material. The main foundations were laid by M. J. DeNiro and

M. J. Schoeninger, who established a connection between the carbon and nitrogen isotope ratios in food and the ratios in consumer tissue (collagen in the bone) (DeNiro/Epstein 1978; 1981; Schoeninger/DeNiro 1984). In the case presented here, a long bone (humerus) was sampled and some 300 mg of bone were analysed. Sample preparation was carried out using the specification set out by R. Longin (1971) as modified by P. Semal and R. Orban (1995) and by T. Brown et al. (1988). The long-chain collagen extracted in this manner was analysed using a mass spectrometer (MAT Finnigan® Delta XL plus) for light stable isotopes coupled with an element analyser (University of Bradford, UK). Horse, sheep/goat, red deer and hare bones were used as reference samples for comparison with the human sample. All animal bones and teeth were found during excavations around the Roman Villa Borg and fit into the same time frame.

X-ray diffractometry and energy-dispersive micro X-ray fluorescence

Examinations using X-ray diffraction (XRD) and energy-dispersive micro X-ray fluorescence (μ-XRF) were carried out at the geoscience laboratories of the Goethe-Universität Frankfurt (Institut für Geowissenschaften). The blackish-brown deposit in the tooth was analysed on one hand by means of energy-dispersive micro X-ray fluorescence (Eagle II, X-ray analytical measurement technology; analysis: S. Klein) and on the other by means of X-ray diffractometry (X-ray powder diffractometer, Panalytical X'Pert Pro; analysis: R. Petschick). Both procedures allowed us to obtain information about the composition of phases and elements.

Micro X-ray fluorescence was carried out using a rhodium X-ray tube with a voltage of 40 kV and an amperage of 95 μA (with a detector dead time of 30-40 %). The measuring time was 150 seconds per measurement. Wherever possible, the measuring spot was set to 300 μm in diameter. Because the object can be placed without preceding preparation or mounting in the sample chamber and the X-ray exposure does not consume the sample, this method is non-destructive. The atoms of phases in the sample surface are excited by the radiation and react in a manner that is characteristic of each element. This allows us to identify the major and minor components in the percentage range with a detection limit of c. 0.2 %. In this case, the analysis provided the inorganic chemical composition of the sample. The results were semi-quantitative because the standardless fundamental parameter method was applied. The elements were calculated to oxides.

For X-ray diffraction examination, a sample was taken from the potential dental filling using a spatula and the gained portion was then carefully ground using an agate mortar and pestle. From the sample preparation, it was obvious that the hardness of the material was heterogeneous. Whilst part of the material was very soft and almost earthy, other was harder. The resulting powder was placed in a special PVC sample holder suitable for minute sample sizes (< 50 mg). The holder was locked into position and the X-ray analysis carried out. Data assessment consisted of comparing the highest peaks with published reference lists (Powder Diffraction Files) to determine the phases (compounds) present by means of an iterative process.

Infrared spectroscopy and micro-CT analysis

FTIR (Fourier transform infrared spectroscopy) and micro-CT analyses were carried out at the Institut für Geowissenschaften, Johannes Gutenberg-Universität Mainz.

For the FTIR analysis, a sample of approx. 1 mg was taken from the possible »dental filling« using a scalpel and mixed with 150 mg potassium bromide. The homogenised mixture was formed into a pellet using an

8-ton press and then analysed using an FTIR spectrometer by Thermo Scientific – Nicolet (Model 6700) in the mid-infrared region of 4000 to 400 cm^{-1}.
For the micro-CT analysis, the tooth with the possible filling was examined using the micro-computed tomography system CT Alpha by the company Procon X-Ray at a voltage of 90 kV and an amperage of 110 µA, coupled with a 0.5 mm aluminium filter. The micro CT produced images of the tooth at a 360-degree rotation on 1200 projections at 2-second exposures each, allowing for a spatial resolution of 10.2 µm. The CT detector (Hamamatsu CMOS Flat Panel Sensor C7942SK-05) translates the intensity of the radiation, which is weakened when entering the sample, into so-called greyscale values. These result from the radiation energy, the extent of weakening of the radiation in the different sample materials and the sensitivity of the scintillation crystals in the detector. High values represent high density, low values represent low density.

Scanning electron microscope and EDX analyses

Initial examinations had already been carried out in 2006 at the Curt-Engelhorn-Zentrum Archäometrie in Mannheim using a conventional scanning electron microscope (SEM). Repeat analyses were undertaken in 2013 using a ZEISS device (EVO 60 MA 25) with a variable pressure range (10-400 Pa) for non-conductive samples; energy-dispersive X-ray spectroscopy (EDX) was carried out using a Silicon Drift Detector (SDD). A standardless quantification method was employed in this case using the peak-to-background method (BRUKER QUANTAX Esprit 1.9.4).

RESULTS AND INTERPRETATION

Osteological examination

The skeleton was well represented with almost completely preserved long bones (**fig. 4**). The pectoral girdle and the pelvis area were also well preserved. However, some of the skeletal elements were in a rather fragmented state. These included the torso with the spine and ribs as well as the skull, which could not be fully reconstructed. The remains belonged to the same individual whose sex was identified as male. The age of the individual was determined to have been 30-40 years. Based on the right femur, the individual's height was reconstructed as approx. 168.2 cm (± 3.3 cm).
The remains examined bore no injuries or pathological changes that could have pointed to the cause of death. Traces of wear and tear on the joints or vertebrae were slight, which was consistent with the age determined. Any activities that involve a particularly high degree of physical stress and lead to premature arthritis were largely excluded for this individual.
The jaw bones and teeth were also examined. In contrast to the mandible, the maxilla was highly fragmented. The right maxillary wisdom tooth (tooth 18) exhibited a radicular cyst (*Radicula appendiciformes*) which, however, would have had no pathological significance for the individual concerned (Alt 1997).
The mandibular second molar on the left (**fig. 5**) appeared to be of particular relevance. The molar (tooth 37 FDI notation) exhibited a dark discolouration on the mesial approximal surface, which was reminiscent of a dental filling and stood out from the surrounding dental enamel (**fig. 5C**). The surface of the black-

Fig. 5 Oberleuken »Auf Schiffels« (Lkr. Merzig-Wadern/D). Mandible of the skeleton with remaining teeth and tooth 37 (second left molar) with a possible dental filling; occlusal view (**A**) and left lateral view (**B**). Detail of tooth 37 with the possible dental filling in the mesial approximal area of the dental neck including the efflorescence in the area of the preserved dental enamel (**C**). – (Photos P. Held).

ish-brown to dark-grey defect was rough to the touch. The adjacent enamel exhibited efflorescence of identical colour. The first molar (tooth 36 FDI notation) had been lost or extracted during the individual's lifetime. The extent of bone formation in the tooth socket indicated that the tooth had been lost a long time before the individual had died (fig. 5A-B). Other teeth with carious lesions were found in the right maxilla (teeth 14, 15, 17, 18) and mandible (tooth 45). None of these, however, bore traces of manipulation or filling.

Results of the radiocarbon dating

A radiocarbon date of 1967 ± 19 BP was obtained with the calibrated range (2σ) falling between 36 cal BC and AD 76 (cal 1σ: AD 19-64). The burial therefore dated from the Roman Imperial period, and more precisely, from the 1st century AD.

sample	individual	skeletal region	material	ppm Sr	$^{87}Sr/^{86}Sr$	2σ
1.1	human	first upper molar (M1)	enamel	93	0.70975	0.00003
1.2	human	second upper molar (M2)	enamel	81	0.70952	0.00005
1.3	human	third upper molar (M3)	enamel	63	0.70976	0.00006
1.4	human	right femur	bone	137	0.71270	0.00002
2.1	pig	third lower molar (M3)	enamel	79	0.71626	0.00002
2.2	pig	mandibula	bone	151	0.71501	0.00003

Tab. 1 Results of the strontium isotope analyses of three teeth and a bone from the male individual and of a tooth and mandible bone from a pig.

Isotope analyses

Strontium isotope analysis to ascertain the individual's origins

The $^{87}Sr/^{86}Sr$ ratios of the individual's first, second and third molars yielded very similar values of 0.70975, 0.70952 and 0.70976 (**tab. 1; fig. 6**). A change of locality during his childhood and early adolescence is therefore highly unlikely unless the new locality had very similar geological conditions.

However, the values do differ significantly from the more radiogenic (higher) isotope ratios in the individual's femur (0.71270) and in the third molar and a bone sample taken from the pig (0.71626 and 0.71501) (**fig. 6**). These comparative data indicate that the locally available strontium is characterised by geologically very old Hunsrück shale. The isotope ratios in the dental enamel of the individual were very different and showed that he would not have spent his childhood and adolescence in Oberleuken or its immediate environs. The fact that the three molars yielded very similar values, which differed from the isotope ratios typically found in the region of Oberleuken, indicated that the change of locality took place after the crown on his wisdom tooth had fully formed (in other words after the age of 14-18). However, it was not possible based on the data available to ascertain whether the man had arrived in Oberleuken shortly before his death or whether he had been in the area for a longer period. Potential distances of migration and thus his region of origin were hard to ascertain because the strontium isotope ratios of the teeth that were examined were all between 0.709 and 0.710, which is a range that occurs very often. This range is characteristic of Loess regions, which are very widespread, and can also be found in areas with other types of sedimentary rock.

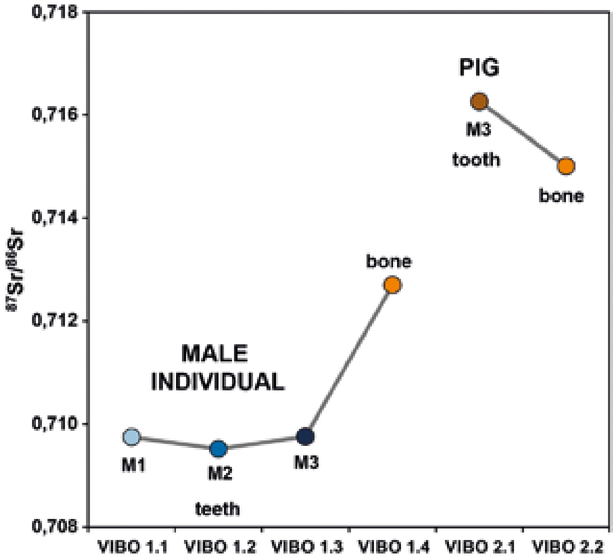

Fig. 6 Oberleuken »Auf Schiffels« (Lkr. Merzig-Wadern/D). Direct comparison between the strontium isotope data from the burial and from a pig. The results of the dental analyses (M1, M2, M3) indicate that the man experienced a change in locality as an adult. – (Illustration C. Knipper).

Carbon and nitrogen isotope analysis to reconstruct dietary habits

The analysis of the human sample yielded a carbon isotope ratio ($\delta^{13}C$) of –20.1‰ and a nitrogen isotope ratio ($\delta^{15}N$) of 9.7‰ (**tab. 2; fig. 7**). Compared to the animal samples analysed, the human $\delta^{13}C$ values were around 1.5‰ elevated and the $\delta^{15}N$ values around 4.5‰ higher.

Tab. 2 Overall average of the $\delta^{13}C$ and $\delta^{15}N$ values of the male individual and of domestic and wild animals for comparison.

sample	species	skeletal region	$\delta^{13}C$ (‰)	$\delta^{15}N$ (‰)
H-1	human	femur	–20.1	9.7
A-1	horse	left metacarpal	–22.1	5.4
A-2	sheep/goat	left tibia	–21.5	8.5
A-3	red deer	right metacarpal	–22.2	4.9
A-4	hare	right tibia	–21.4	4.6

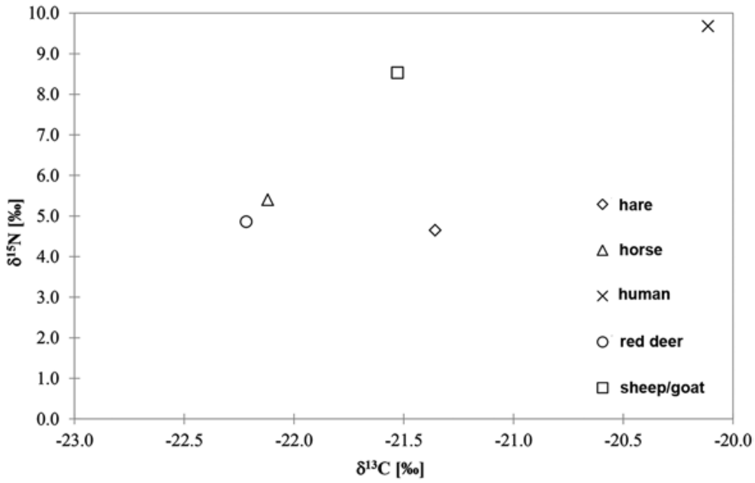

Fig. 7 Oberleuken »Auf Schiffels« (Lkr. Merzig-Wadern/D). Graphic representation of the $\delta^{13}C$ and $\delta^{15}N$ values of the species examined. Compared to the animals, the man's $\delta^{13}C$ values are approx. 1.5‰ higher and his $\delta^{15}N$ values are elevated by approx. 4.5‰, which points to a mixed diet made up of locally available animal protein and C_3 plants. – (Illustration O. Nehlich).

The carbon isotope values measured in the animal samples lay between –22.2 and –21.5‰, which is a discrete range and points to a forested habitat (Schoeninger/Iwaniec/Glander 1997). The relatively high $\delta^{15}N$ values in the animal samples, ranging between 4.6 and 8.5‰, point to a particular type of habitat. Such enriched values are usually found in closed forest habitats where the nitrogen in the plants is continually recycled within the ecosystem. The goat/sheep sample, which could not be identified any further, was characterised by a particularly high $\delta^{15}N$ value (see **fig. 7**). It may have been a kid or lamb, which would be at a higher trophic level than adult animals because of its mother's milk. However, due to the fragmented state of the bone, it was not possible to determine the animal's age. The isotope values of the male individual ($\delta^{13}C$: –20.1‰; $\delta^{15}N$: 9.7‰) were consistent with a contribution of meat or dairy products from those of the animals to his diet. Moreover, the results fit in well with the data published for other sites from the same period. The man's diet would have been a typical mixed diet made up of the available animal protein and C_3 plants.

Attempts at identifying the material of the possible »dental filling«

Radiology and micro CT

A conventional X-ray image of the left mandible from Oberleuken-Perl showed a distinct hard-tissue defect in the mesial approximal area of tooth 37 (**fig. 8**). The resulting cavity appeared to have been filled with a »substance«. The X-ray density of this substance lay somewhere between that of the dental enamel and dentine.

The micro-CT analysis yielded more detailed information (**fig. 9**). From a structural point of view, it was very difficult to distinguish between the dentine and the possible filling, except for the fact that the surface of

Fig. 8 Oberleuken »Auf Schiffels« (Lkr. Merzig-Wadern / D). X-ray image of the left mandible showing changes in the mesial area of the crown of tooth 37 (arrow). – (X-ray image M. Wolf / E. Enzmann).

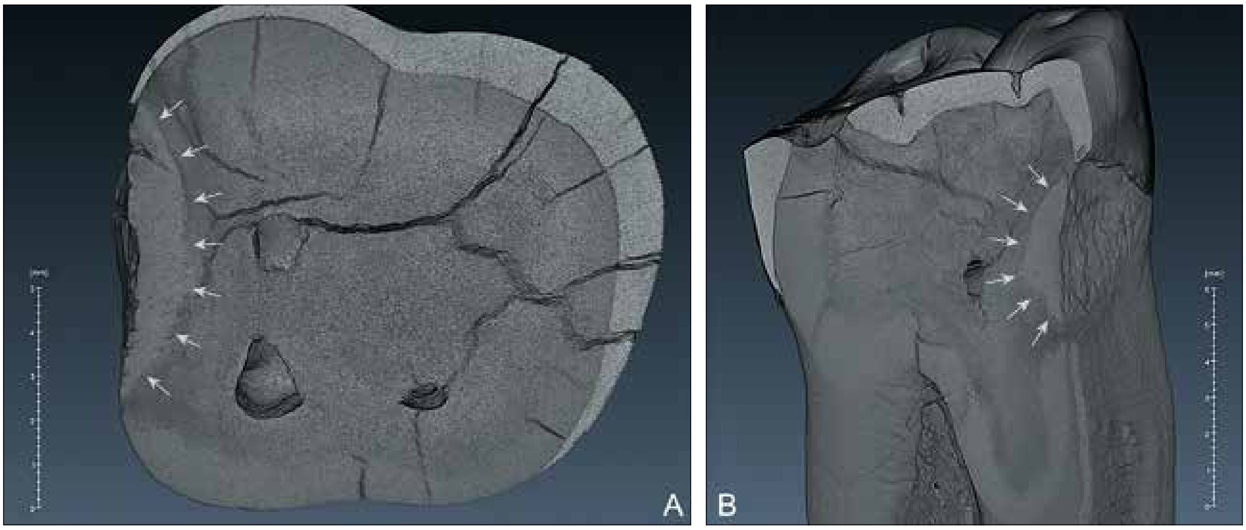

Fig. 9 Oberleuken »Auf Schiffels« (Lkr. Merzig-Wadern / D). Micro-CT images of tooth 37. – **A** section through the tooth parallel to the chewing surface. There is a clear boundary between the dental enamel and the dentine on the (upper) buccal and right (distal) side. The possible dental filling can be distinguished from the dentine as a slightly lighter, crescent-shaped area (arrows). – **B** longitudinal section through the tooth. Because of its slightly higher density, the dental enamel is slightly lighter in colour than the dentine in the areas of its occlusal surface and on its mesial and distal sides. The possible filling (right exterior) is distinguishable from the dentine by its slightly lighter greyscale (arrows); the dental enamel in the mesial area is intersected by the upper edge of the cavity. – (Photos M. Wolf / E. Enzmann).

the latter was significantly coarser than the surface of the dentine or dental enamel. Some of the desiccation cracks in the dentine continued into the possible filling but did not lead to spalling, which also points to very similar material properties (fig. 9A).

Figure 9B shows a cross-section of the three-dimensional CT image of the tooth. It is a vertical section at the level of the possible filling (right exterior), which in this image reached a maximum thickness of 1.8 mm; the pulp is just visible to the left of it. The varying greyscales in the image result from the different densities of the materials in the tooth, or more precisely, from their linear attenuation coefficient. The dental enamel (top) with its slightly higher density is of a lighter colour, the dentine with its slightly lower density is somewhat darker. The filling is just a little lighter in colour than the dentine, which indicates very similar densities. The difference in the materials was almost impossible to distinguish from the images, which is why a seg-

Tab. 3 µ-ED-XRF analysis of the intact tooth. The values were calculated for the oxide forms using the fundamental parameter method. – Wt % = weight percent; At % = atomic percent; I-Error % = alpha error; BG = detection limit; Wt-Error = weight error.

intact tooth					
oxide	Wt %	At %	I-Error %	BG	Wt-Error
Al_2O_3	0.6	0.43	8.71	1.92	0.05
SiO_2	1.22	1.51	3.1	2.08	0.04
P_2O_5	39.13	20.42	0.37	2.08	0.64
K_2O	0.44	0.35	2.6	2.4	0.01
CaO	58.39	77.12	0.2	2.56	0.94
TiO_2	0	0	0	2.83	0
MnO	0.04	0.04	11.22	3.87	0
Fe_2O_3	0.07	0.03	7.55	4.83	0.01
ZnO	0.1	0.09	3.96	7.19	0

mentation was not possible. This, in turn, prevented us from identifying the volume of the »filling« or creating an isolated three-dimensional image of it. The transition between the possible filling and the dentine was almost seamless, both within the tooth and on the surface. The only exceptions were fine hairline cracks between both materials at the top and bottom edges of the cavity. In some areas, the CT showed a gradual transition between the materials. A step was visible between the possible filling and the dental enamel.

Because the greyscales of the different materials result from a range of factors (e. g. atomic number, radiation energy, detector properties) it is not possible to derive any absolute data concerning material densities. In medical diagnostics, the Hounsfield scale is used to differentiate between organic tissues and to describe the location-dependent attenuation of the radiation when it is absorbed by the tissue. The measurements obtained were in the hard-tissue range but could not be differentiated any further. The slight differences in the greyscales, however, do indicate that the dentine and the »fill material« had different densities.

Material analysis

Results of the µ-ED-XRF and XRD analyses

According to the energy-dispersive µ-X-ray fluorescence analysis, the intact tooth consisted of 58 wt % CaO (calcium), 39 wt % P_2O_5 (phosphorus) and 1 wt % SiO_2 (silicon) (**tab. 3**). From a phase-analytical point of view, the main component of the tooth, as identified by X-ray diffraction, was hydroxyapatite.

In some of the intact areas in the vicinity of the blackish-brown deposit, which was presumed to have been a dental filling, the tooth exhibited a brown efflorescence. Besides being overprinted by the dental substance, these areas contained elevated amounts of aluminium oxide and silicium oxide (5 wt % Al_2O_3 and 11 wt % SiO_2), titanium oxide (0.5 wt % TiO_2), but also a significant proportion of manganese oxide (15 wt % MnO) and iron oxide (2 wt % Fe_2O_3) (**tab. 4**). Various locations of the blackish-brown deposit were also analysed *in situ* (**tab. 5**). The analyses, much like in the efflorescence, yielded an elevated aluminium and substantially higher silicium content (up to 8 wt % Al_2O_3 and 20 wt % SiO_2), but also titanium oxide with up to 0.4 wt %. The manganese values lay at 3-4 wt % MnO, the iron values at 2-7 wt % Fe_2O_3.

From the blackish-brown deposit, a small sample amount (< 10 mg) was analysed for the phase composition by the X-ray diffractometry method. Besides hydroxyapatite, the major tooth components, also rutile (TiO_2), quartz (SiO_2) and goethite (α-FeOOH) were identified. In its crystallised form, rutile is yellowish-brown to reddish, in a pulverised state it is white, as is quartz. Rutile is used as a white pigment, but it does not occur

brown efflorescence on the intact tooth					
oxide	Wt %	At %	I-Error %	BG	Wt-Error
Al_2O_3	5.42	3.6	2.95	1.92	0.18
SiO_2	10.76	12.13	1.34	2.17	0.22
P_2O_5	14.8	7.06	0.92	2.17	0.27
K_2O	1.57	1.13	1.71	2.7	0.04
CaO	49.43	59.73	0.28	2.99	0.8
TiO_2	0.49	0.41	4.29	3.29	0.02
MnO	15.87	15.16	0.37	3.93	0.26
Fe_2O_3	1.51	0.64	1.24	4.27	0.03
ZnO	0.15	0.13	4.98	5.06	0.01

Tab. 4 µ-ED-XRF analysis of the brown efflorescence on the intact tooth. The values were calculated for the oxide forms using the fundamental parameter method. – Wt % = weight percent; At % = atomic percent; I-Error % = alpha error; BG = detection limit; Wt-Error = weight error.

»dental filling« centre brown 1					
oxide	Wt %	At %	I-Error %	BG	Wt-Error
Al_2O_3	1.44	1.06	8.27	0.56	0.12
SiO_2	2.83	3.55	3.45	0.61	0.11
P_2O_5	31.62	16.8	0.77	0.61	0.56
K_2O	0.52	0.42	4.18	0.8	0.02
CaO	52.21	70.23	0.36	0.85	0.86
TiO_2	0.17	0.16	11.07	1.02	0.02
MnO	4.13	4.39	0.96	1.35	0.08
Fe_2O_3	7	3.31	0.71	1.57	0.12
ZnO	0.08	0.07	10.43	2.51	0.01
»dental filling« centre brown 2					
oxide	Wt %	At %	I-Error %	BG	Wt-Error
Al_2O_3	8.55	5.86	1.54	0.83	0.19
SiO_2	20.65	24.04	0.71	1.09	0.36
P_2O_5	18.92	9.32	0.65	1.31	0.33
K_2O	1.75	1.3	1.27	2.73	0.04
CaO	43.57	54.34	0.24	3.25	0.71
TiO_2	0.43	0.37	3.41	4.32	0.02
MnO	3.73	3.68	0.57	5.91	0.06
Fe_2O_3	2.35	1.03	0.71	6.27	0.04
ZnO	0.07	0.06	6.27	7.97	0

Tab. 5 µ-ED-XRF analysis of the possible dental filling. Two different areas were examined. The values were calculated for the oxide forms using the fundamental parameter method. – Wt % = weight percent; At % = atomic percent; I-Error % = alpha error; BG = detection limit; Wt-Error = weight error.

in nature as an individual material, and thus has only been man-made and used as a whitening pigment since the modern period.

According to the µ-ED-XRF analysis, iron and manganese oxides, both red and brownish-black in colour, were barely identified in the intact tooth (< 0.1 wt %), but were found in significant quantities in the possible dental filling (4 wt % MnO) and in the efflorescence-affected areas of the intact tooth (15 wt % MnO). Unexpectedly, it was not possible, using XRD, to identify an inorganic crystallised manganese-rich mineral phase; manganese may have been present in other than a crystallized inorganic phase, maybe as part of a complex or organic compound.

The following conclusions can be drawn from the results of the µ-ED-XRF and XRD analyses: the fact that the area in question is distinct from the intact tooth can be used to make a case for the existence of a dental filling. The material was, although heterogeneous in hardness, in total much softer in this area, thus

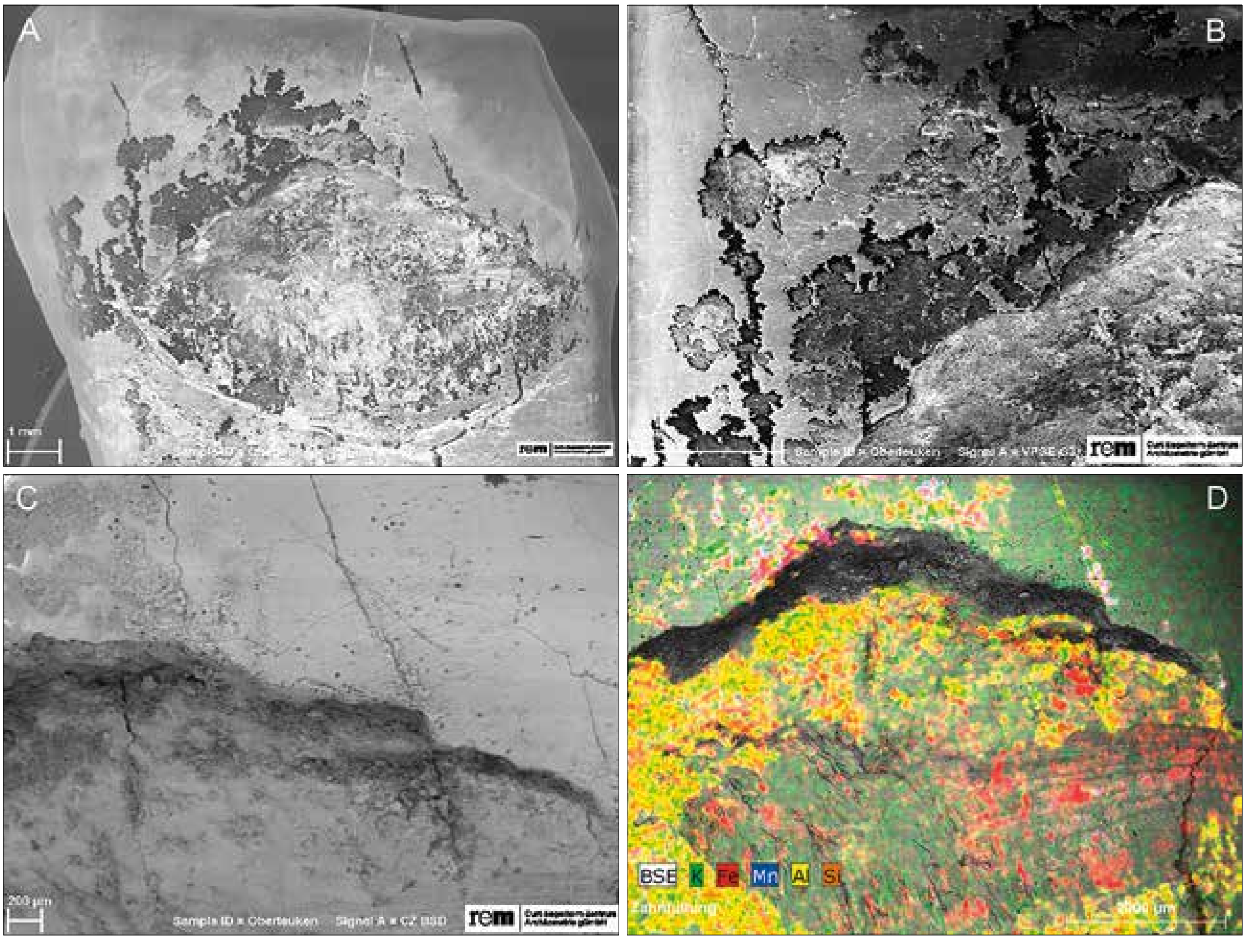

Fig. 10 Oberleuken »Auf Schiffels« (Lkr. Merzig-Wadern/D): **A** secondary electron image of the filling. – **B** transition between the tooth and the possible filling. – **C** the edges between the dental enamel and the »filling material« are made visible on the backscattered electron image by shading, whilst the tooth and the possible filling have almost identical backscatter coefficients. – **D** a combination between backscattered electron imaging and qualitative element mapping without the main components calcium and phosphorus. – (Photos R. Schwab).

pointing to the presence of some sort of fine-grained material. The entire area in question became discoloured over time: manganese-rich substances staining the intact tooth as dark-coloured efflorescence by developing from the weakened cavity surface into the direction of the intact tooth. It is obvious that the areas of the efflorescence were all located along the edges of the cavity.

If this was an intentional dental filling, it may have consisted of the white components rutile and quartz, both of which were identified using XRD. These could have been mixed and bound by an organic substance such as egg, oil or casein to form a paste, which would have been similar in colour to the tooth. As for manganese and iron, it should not be ignored that proportions might have been also subsequently absorbed by the possible dental filling from the surrounding soil since fine-grained material is more absorbent than intact dental enamel. The only question, in this case, would be how the hardening of the paste could have been achieved. Another theory would be that the white components were mixed with the Roman »universal remedy« wood tar, a hydrophobic material that preserves well when it is deposited in the ground over a long period of time (pers. comm. Dr. U. Baumer, Doerner Institut, Munich, 2012). Once it has dried off, the consistency of wood tar is similar to modelling clay; it could easily have been mixed with quartz and rutile powder and would have been well suited for the use as a filling, though it would not have been of a similar

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_2	K_2O	CaO	TiO_2	MnO	FeO
»filling«	0.6	0.6	1.2	2.7	35	0.3	0.6	54	0.4	2.7	2.6
s	0.2	0.2	0.7	1.3	1.3	0.1	0.1	2.4	0.1	1	0.2

Tab. 6 Mean values (rounded up) obtained by EDX analyses (in percent by mass) recalculated for the usual oxides with the respective standard deviations (s).

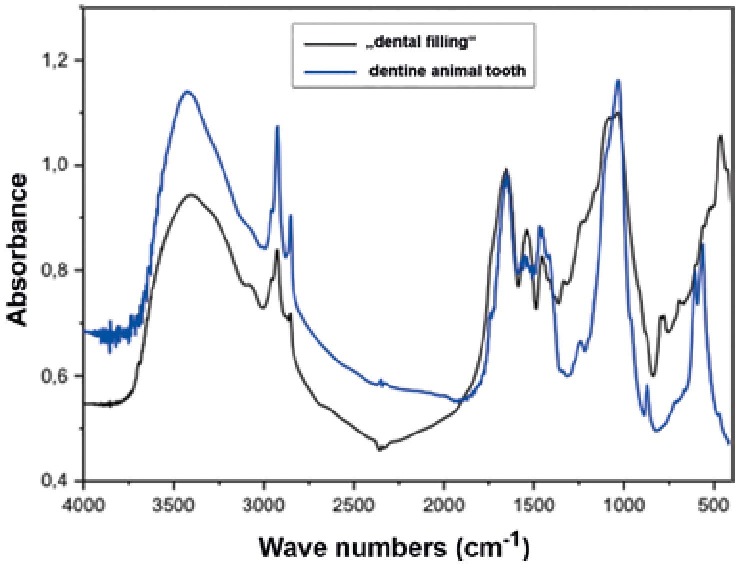

Fig. 11 Oberleuken »Auf Schiffels« (Lkr. Merzig-Wadern/D). Infrared spectroscopy. Spectrum of the »dental filling« as compared to the animal dentine (Weiner database). – (Illustration T. Häger).

colour to the surrounding tooth. The fact that quartz and rutile are harder than dental enamel (dental enamel: 5, rutile: 6-6.5 and quartz: 7 on the Mohs hardness scale) would make the result more durable and resistant to mechanical stress. Organic components were not identified, though modern softening agents in plastic wrappers, synthetics in labels or coatings, remnants of adhesive tape etc. (which were used in this case to keep the samples separate), could lead to distorted results from such analyses.

Scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDX)

Backscattered electron imaging shows local differences in the composition based on the different backscatter coefficients from the individual elements of a material: compounds of elements with higher atomic numbers appear in a lighter colour than elements with lower atomic numbers. The backscattered electron images in this case clearly showed that the filling must have had a near-identical composition to that of the tooth, whilst a substance of a different composition had accumulated in scratches and in between the filling and the tooth (**fig. 10**). According to the electron microanalysis, the possible filling consisted mainly of calcium phosphate which, based on the presence of manganese, iron, silicon, and aluminium, was probably contaminated by a sediment that had accumulated in depressions (**tab. 6**). The sediment remains were not just found in the filling but on the tooth as well and were therefore subsequently introduced.

Infrared spectroscopy (FTIR = Fourier transform infrared spectrometer)

The spectra recorded by means of 64 scans showed hydroxyapatite and collagen to be the main components of the possible filling, as is typical of the tooth and/or bone material. **Figure 11** shows the spectrum of the »dental filling« compared to animal dentine (Weiner database). The outstanding feature is that there are clear differences between the two spectra. The double peak at 603 and 563 cm^{-1} that is typical of hydroxyapatite and is visible on the right-hand side of the figure, is not visible in the spectrum of the »filling«. The »splitting factor« would normally be calculated to indicate the state of preservation of bone and teeth. The absence of a visible double peak, in this case, could mean either that it was not hydroxyapatite or that the spectra of several components were superimposed. It was not possible by means of FTIR to identify a »binder« or any other foreign substance.

DISCUSSION

The remains were found in a prone, and therefore irregular, burial position. None of the individual's personal effects were preserved and no remnants of clothing or metal artefacts came to light. We may, therefore, assume that the man was stripped of his belongings, such as jewellery or a belt, and that he was placed in the burial pit more or less unclothed. The context indicates that the body had to be »disposed of« as quickly as possible. Neither the body position within the pit nor the findspot within the settlement or the deposition without grave goods is consistent with funerary rites known from the Roman period. In archaeological research, any burial rites that differ from the regular, culturally relevant funerary practices are classed as deviant or atypical burials; examples are known from all cultures and periods (Wahl 1994; Orschiedt 1998). It is known from a range of comparable archaeological sites that individuals who died of unnatural causes, be it as a result of an accident or a violent encounter, were often buried in a different manner than was customary in the communities they belonged to (e.g. Berszin/Wahl 2002; Meyer et al. 2013; Pichler et al. 2013).

Irregular burials were not uncommon at this time, as evidenced by numerous skeletal finds from wells or ditches (e.g. Lange/Schultz 1982; Wahl 1991; 1997; Czysz 2003). Two regional examples should be mentioned here. An excavation carried out at Rubenheim (Saar-Pfalz-Kreis/D; Reinhard 2002) uncovered a similar feature to the Oberleuken find. There too, a skeleton was found in a prone position directly beside the foundations of a Roman building. In that case, however, it was evident that the individual had been the victim of a violent attack. There was also a complete absence of any belongings, and it was not until the bone material was radiocarbon dated that it was confirmed to date from the Roman period. The archaeologists presumed that the deceased had been the victim of a nasty attack and was then hastily buried in the ditch of the foundation wall (Reinhard 2002). Another example can be cited from the excavations carried out at the Gallo-Roman theatre of Dalheim (Ct. Remich) in Luxembourg (Henrich 2015). The incomplete skeleton of an older man found there showed various injuries to the skull (Held/Alt 2015). The man's violent death has been linked to the Germanic invasion in the 3rd century (Henrich 2015, 225).

It is quite likely that the Oberleuken individual suffered a similar fate, though the skeletal remains exhibited no evidence of violence. The absence of injury to the bones does not exclude the possibility that the man's premature death was caused by violence. Injuries to the internal organs or any of the larger vessels can also quickly lead to a person's death (Walker 2001; Cappella et al. 2014). How the individual was deposited allows us to draw conclusions concerning the relationship between the deceased and the »burial community« (Komar 2008). A quick and careless »disposal« of the body does not point to a high degree of respect for the deceased. A particularly interesting aspect in this context is the fact that the strontium isotope ratios measured in the teeth were not consistent with the biologically available strontium in the area, and thus is a sign of the man's non-local origin. He probably arrived in the region as an adult and may not have had a sufficient degree of social rootedness in the local community. It was not possible to make any statements regarding his actual region of origin.

The possible dental filling is particularly interesting for the medical history. The macroscopic analysis and X-ray results both indicated that a mesial defect in the approximal area of the second molar of the left mandible (tooth 37), which had probably been caused by caries, was filled with a dark substance, which may have been discoloured as a result of taphonomic processes. The occurrence of caries in the area of contact between two teeth is not unusual because remnants of food often become trapped in these locations and the absence of the appropriate dental care – tooth brushes were not widely used until the early 19th century – sooner or later lead to dental caries. It was therefore almost the norm before the personal hygiene became common practice that one's teeth were lost to caries throughout one's lifetime (e. g. Alt 2001; Lehmann / Hellwig 2005). The missing first lower molar was likely lost due to the reasons outlined above.
Because of the dating of the feature to the Roman Imperial period, however, it seems highly unlikely, though not beyond the realms of possibility, that a cavity caused by caries would have been filled as part of dental treatment. The earliest concrete evidence for a dental filling in Europe concerned Anna Ursula von Braunschweig und Lüneburg who died in 1601 at the age of 28. Two fillings were identified in that case, one made of gold, the other of amalgam, both in the same right upper molar (Riethe / Czarnetzki 1983). A small number of metal fillings made of tin, lead, gold, copper and cadmium date from no earlier than the 16th century. Amalgam is believed to have been known in China since the 7th century, and in Europe, it is mentioned for the first time in a prescription by the Ulm physician Johannes Stocker (Riethe 1980). Dental fillings became more widespread from the 18th century onwards, though at that time they were still limited exclusively to members of the upper classes who could afford such luxury procedures (Alt 1993; 1994; Cox et al. 2000). This social aspect is even more pronounced concerning the use of dental prostheses (Alt 2011).
More recent studies, however, have emphasised the fact that even prehistoric people did not simply accept painful dental defects, but that they used various means to either treat themselves or get treatment from healers. These were not routine medical procedures but rather measures to relieve pain and other ailments. In a case study on a Neolithic find from Slovenia, F. Bernardini et al. (2012) have interpreted the evidence as pointing to a therapeutic palliative treatment which involved closing a fracture in a tooth using beeswax. Much earlier finds from an Early Neolithic context in Pakistan (Coppa et al. 2006) and a Late Palaeolithic context in Italy (Oxilia et al. 2015) have shown that carious defects were treated by scraping out the affected tooth or even drilling into it. Material analyses carried out on a Palaeolithic find from Italy have provided evidence of bitumen being used as a component in a dental filling (Oxilia et al. 2017); bitumen is known to have been used in medical contexts throughout Antiquity.
The material analyses carried out on the possible dental filling from Oberleuken have identified hydroxyapatite as the main component, which is the primary natural mineral component in teeth and bone. In addition, traces of rutile, quartz, goethite, manganese, and iron were found. None of the historical or modern dentistry references consulted for the purpose of this study indicated that the material components that were observed in this case would have been used in dental fillings, though the use of rather exotic materials such as cadmium, gallium, and antimony in fillings was not, in principle, unheard-of (Bremer 1969; Krämer 1964). In terms of the ancient Roman medicine, however, it is in fact doubtful that fillings were ever used to treat carious teeth. A text reference to this effect by Martial (c. AD 40-103) »*Eximit aut rificit dentem Cascellius aegrum*« (Mart. X, 56, Epigrammata X, 1, 10.53; Schnur 2003: Cascellius extracts or repairs a painful tooth; literally: »remakes it«) was interpreted as a reference to a »prosthetic« tooth by H. L. Strömgren (1935), who postulated that *reficere* in this case could be translated as »replace«.
Much like the traces of manganese and iron, rutile, quartz, and goethite could, in principle, also have derived from the human remains coming into contact with the soil in which they were deposited. However, although this possibility cannot be excluded, it is not very likely because efflorescences are rarely found in

the dental enamel of archaeological skeletal finds. The value ranges identified by means of the FTIR analysis eliminated the presence of foreign substances almost completely, and this included the use of possible binders, though it must be borne in mind that the latter may have evaporated over time. Overall, the evidence strictly speaking only allows us to state that there was either no »dental filling« or that the »filling« itself consisted of human or animal tooth or bone material.

Although there was no such thing as a dental filling in the modern sense of a dental restoration treatment before the early modern period, ancient literature (Pliny [AD 23-79], *Naturalis Historia*) does include mentions of »filling materials« in a Graeco-Roman context. In a medical-historical treatise on »Pliny and dentistry. Textual references in the *Naturalis Historia*«, Ch. Freitag (1994, 114) describes »a series of remedies [...] as ›*fillers in hollow teeth*‹«. The ancient Greeks and Romans did practice some form of dental hygiene involving mouth washes and toothpastes, at least amongst the upper echelons of the society, and used a pumice stone and the shells of oysters or eggs as well as a bone meal to clean their teeth (Burrell 1999). In recent times, various forms of the bone meal have once again been more widely used in bone tissue repairs or bone regeneration and as additives in the toothpaste (Vajrabhaya et al. 2016; Venkatesan et al. 2018). It is therefore perfectly feasible that it could also have been used in the case of our possible »dental filling«, but this could probably only be proved by way of molecular genetics, i. e. by means of aDNA analyses. Both animal and human tooth or bone meal would have to be considered since the teeth of dead animals and humans have been used since Antiquity as tooth replacements (Alt 2011). Oyster and egg shells are carbon-based and could therefore be clearly distinguished from dental apatite. However, it was not possible, for the reasons outlined above, to eliminate the use of tooth or bone meal. It is worth noting that the FTIR spectra did not yield any evidence of a binder. It is not very likely that a potential binder dissolved completely because this would have led to the tooth or bone meal to lose its cohesion. Even a minimal amount of binder is necessary to ensure material cohesion. It is possible, however, that the context is of such a high complexity from a chemical and mechanical point of view that even the use of extensive technological procedures did not allow us to completely solve this particular conundrum.

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Zusammenfassung / Summary / Résumé

Eine römerzeitliche Zahnfüllung aus Hartgewebe-Komposit?
Bioarchäologische und medizinhistorische Untersuchungen an einer römerzeitlichen Bestattung aus Oberleuken-Perl (Lkr. Merzig-Wadern / D)

In der Nähe des Archäologieparks Römische Villa Borg bei Oberleuken-Perl im Saarland wurden 2001 die beigabenlosen Skelettreste eines Menschen aufgefunden. Nach Radiokarbondaten gehört der Befund in das 1. Jahrhundert n. Chr. Die osteologische Untersuchung ergab, dass es sich um das Skelett eines adulten Mannes handelt. An den Knochen fanden sich weder Spuren von Verletzungen noch Anzeichen schwerer Erkrankungen und somit auch keine Hinweise auf die mögliche Todesursache. Während die Kohlenstoff- und Stickstoff-Isotopenverhältnisse ein orts- und zeittypisches Nahrungsspektrum anzeigen, sprechen die Ergebnisse der Strontium-Isotopenanalyse zumindest für eine Ortsfremdheit in Kindheit und Jugend. Die Zahnkrone des zweiten Molaren im linken Unterkiefer zeigt im mesialen Kontaktbereich einen Hartgewebsdefekt auf, der Ähnlichkeiten mit einer Zahnfüllung aufweist. Um festzustellen, ob es sich tatsächlich um eine Zahnfüllung handelt, und um Aufschluss über die Art und Zusammensetzung des Füllmaterials zu erhalten, wurden bildgebende und materialanalytische Untersuchungen durchgeführt. Als Hauptbestandteile des »Füllungsmaterials« wurden Hydroxylapatit, Rutil, Quarz und Goethit identifiziert, es fehlen jedoch Hinweise auf ein Bindemittel. Da zermahlener Knochen und Zähne seit der Antike vielfältig eingesetzt wurden, wäre eine Benutzung als »Füllungsmaterial« im vorliegenden Fall denkbar.

A Roman-period Dental Filling Made of a Hard Tissue Compound?
Bioarchaeological and Medical-historical Investigations Carried out on a Roman-period Burial from Oberleuken-Perl (Lkr. Merzig-Wadern / D)

The skeletal remains of an individual were found in 2001 in the vicinity of the Roman open-air museum at »Villa Borg« near Oberleuken-Perl, Saarland. Radiocarbon analysis dated the feature to the 1st century AD. No belongings or grave goods were found. The osteological examination showed that the skeletal remains were those of an adult male. The bones bore no evidence of injury or disease, which meant that it was not possible to determine the cause of death. Whilst the carbon and nitrogen isotope ratios were consistent with the local dietary range in the Roman period, the results of the strontium isotope analysis suggested that the individual had spent his childhood and adolescence elsewhere. The second molar of the left mandible exhibited a defect or cavity which contained a substance resembling filling material. Imaging techniques and material analyses were used to determine whether this was indeed a dental filling and to obtain information about the nature and composition of the material used. Hydroxyapatite, rutile, quartz and goethite were identified as the main components, though there was no evidence of a binder. Since crushed bone and teeth have been widely used for various purposes since Antiquity, it is conceivable that the substance analysed was a »filling«.

Une obturation dentaire romaine en composite de tissu dur?
Recherches bioarchéologiques et médico-historiques effectuées sur une sépulture d'époque romaine à Oberleuken-Perl (Lkr. Merzig-Wadern / D)

Près du musée en plein air de la »Villa Borg« romaine près d'Oberleuken-Perl en Sarre, les restes osseux d'une personne sans sépulture ont été retrouvés en 2001. Selon les données radio carbone, le squelette appartient au 1er siècle apr. J.-C. L'examen ostéologique a révélé qu'il s'agissait d'un homme adulte. Il n'y avait aucune trace de blessures ou de signes de maladies graves sur les os et donc aucune indication de la cause possible de la mort. Bien que les rapports isotopiques du carbone et de l'azote indiquent un spectre alimentaire local typique pour la période, les résultats de l'analyse des isotopes du strontium indiquent a minima un déplacement durant l'enfance et l'adolescence. La couronne de la deuxième molaire de la mâchoire inférieure gauche présente un défaut tissulaire dur dans la zone de contact mésiale qui est similaire à une obturation dentaire. Des examens d'imagerie et d'analyse des matériaux ont été effectués pour déterminer si l'obturation était réellement une obturation dentaire et pour obtenir des informations sur le type et la composition du matériau d'obturation. L'hydroxyapatite, le rutile, le quartz et la goethite ont été identifiés comme étant les principaux composants du »matériau de remplissage«, mais aucun liant n'a été trouvé. Étant donné que les os et les dents broyés ont été utilisés de différentes manières depuis l'Antiquité, leur utilisation comme »matériau d'obturation« serait envisageable dans le cas présent.

Traduction: L. Bernard

Schlüsselwörter / Keywords / Mots clés

Saarland / Römische Kaiserzeit / Bioarchäologie / Zahnbehandlung / Zahnfüllung
Saarland / Roman Imperial period / bioarchaeology / dental treatment / dental filling
Sarre / Romain impérial / bioarchéologie / traitement dentaire / remplissage de dents

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