



Material description of a unique relief fibula from Poland

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Abstract

A unique relief fibula dated to the Migration Period (first half of the sixth century) was found in Radziejów, Poland. This stray find changes previous opinions on the lack of settlement in central Poland at that time. As the find is the only one of such type in Poland, a special attention was paid to possible analogies, mainly finds from Scandinavia and Western Europe. The fibula underwent technological analyses in order to reveal its technology of manufacture. For this purpose, the chemical and elemental composition of the alloy was studied. Several physico-chemical complementary techniques such as optical microscopy (OM), scanning electron microscopy with energy dispersive X-ray (SEM–EDX) spectroscopy, energy dispersive X-ray fluorescence (ED–XRF) analysis, X-ray diffraction (XRD) analysis and micro-hardness testing (HV0.2) were used to study the technology of the find. The investigations revealed that this artefact was made from brass (4–17 wt% Zn) with an admixture of Sn (2–12 wt%). Two technologies were used: casting for the bow and forming for the part with the axle of the spring. The artefact's surface was tinned in the hot-dipping process. The physical structural analyses demonstrated that the artefact was cast and ornamented by surface stamping under a relatively low temperature (about 500 °C).

Keywords Relief fibula · Migration Period · Scandinavian metal handicraft · Ancient metallurgy · SEM–EDX · ED–XRF · XRD

Introduction

Apart from traditional typochronological or stylistic examinations of archaeological artefacts, the role of technological analyses of finds has been more and more appreciated by archaeologists. Such analyses can often provide crucially important data on the technology of manufacture or provenance of raw materials. They can also supply otherwise unavailable pieces of information on the chronology of artefacts. A Migration Period relief fibula which was discovered in Radziejów, Poland, is in

every respect a unique find. Due to this, apart from a ‘traditional’ archaeological discussion on the artefact, we also decided to carry out a series of technological examinations of the find with the use of different analytical techniques. Another reason is that the number of technological analyses of analogous artefacts is still far from satisfactory. Therefore, each analysed find may provide valuable data for future comparative examinations.

Circumstances of discovery

The relief fibula was found in 1990 in the town of Radziejów, located in the region of Cuiavia. The discovery was made during a historical battle re-enactment. The artefact was simply kicked from the ground by one of the participants in the event. The fibula, being a stray find with no archaeological context, was located in the topsoil layer. The discovery was made at the border of a wood in the area enclosed between Sportowa and Armii Krajowej Streets (Fig. 1).

Archaeological background

Settlement in the vicinity of Radziejów has recently been discussed by Kontny (2016). The entire Cuiavian settlement

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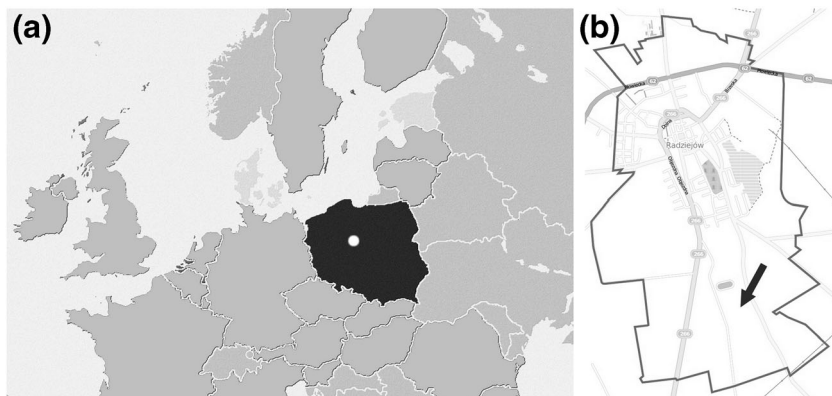
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Fig. 1 **a** The town of Radziejów in the map of Poland. **b** Contour map of the town with the place of discovery



cluster which survived unexpectedly in the Migration Period at least until the mid seventh century is a matter of intensive studies by a team of scholars—participants in a multidisciplinary research project ‘Migration Period between the Odra and Vistula’ (<http://www.mpov.uw.edu.pl/pl/>). Its unique character is related to the fact that according to previous opinions, a major part of the territory of today’s Poland (except for its north-east part) was almost totally abandoned in the beginning of the Migration Period. New discoveries, including the relief fibula from Radziejów, change this image drastically.

The relief fibula weighs 51 g with metal alloy density over 7.5 g/cm^3 . The artefact survived in a good condition. Due to this, we were able to study the shapes of the relief ornamentation (see Fig. 3). The dimensions of the surviving fragments are as follows: length—7 cm, width of the head-plate—5.5 cm, length of the head-plate—2.8 cm, width of the bow—1.8 cm, height—2.5 cm, height of the catch-plate—1 cm and length of the catch-plate—1.7 cm.

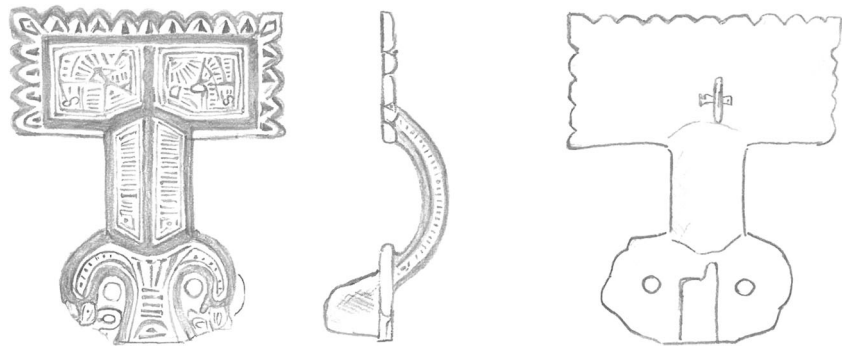
The relief brooch survived only in parts (Fig. 2): two fragments have been glued together, but still a large part of the foot-plate is missing. The head-plate is rectangular with a distinct ornamental area of two parts bordered from the top and sides with a line of arches. Their arms are directed inwards and there are triangles inside them. Both decorative zones are separated with double grooves. Inside the zones, there is a

decoration in Animal Style I after Salin (1904). Highly stylised flat images of parts of animal and human bodies are filled with parallel convex strokes. It is possible to recognise a bird’s head, an animal paw and multiple elements of bodies, possibly also heads (double ovals symbolising eyes with upright hair) and head fragments with eyes. Unfortunately, a lot of motifs seem to be unidentifiable. Generally, the composition is asymmetrical but some motifs are repeated symmetrically, i.e. in similar places of both images (humanoid heads, eyes, arrangement of body fragments; see Fig. 3). The bow of the fibula is distinguished at the edges and along the central axis with elongated laths; the surfaces confined with the laths are filled with short horizontal convex lines. In the lower part of the bow, there is a representation of a pair of eyes. The above motif, i.e. a centrally incised beak, is continued in the upper part of the foot-plate. Only the upper part of the foot-plate has survived. It bears an image of a pair of animal heads with the necks outlined as a contour, filled with parallel strokes. Their eyes are oval and are underlined with grooves. A simplified image of ears is also visible. There are tongues which slide out of the muzzles and are outcurved backwards. The heads of the beasts are preserved partially. Between the heads, there are short convex horizontal lines and a centrally situated fragment of an unrecognisable motif (a zigzag bordering the decorated surface, the image of an eye?). The

Fig. 2 Relief fibula from the wood in Radziejów: top view (obverse) and bottom view (reverse) (photo, M. Jakubowski)



Fig. 3 Fibula in different projections with reliefs decorating the obverse. Interpretation is offered below (drawing, T. Jakubowski)



original shape of the foot-plate is impossible to reconstruct. An axle was fixed in the arched plate protruding perpendicularly from the lower side of the head-plate. Only vestigial fragments of the axle and a spring remained stuck in the fixing plate. The catch-plate is solid; it was cast together with the foot-plate and the entire brooch and was then formed (small traces of plastic forming are noticeable on the bottom). This was confirmed by technological examinations (see below).

Originally, the discussed artefact must have been almost twice as long as the remaining part, which situates it in a group of medium-sized fibulae, i.e. 10–13 cm, according to the idea of Høilund Nielsen (2009: p. 61). The fibula is not worn out, so probably it was not used for long.

The fibula represents Type A-2 after Sjøvold (1993), characterised by a rectangular head-plate and an elongated flat foot-plate (non-profiled longitudinally). Theoretically, it could be attributed to variant f (ended with an animal mask) or d (without a mask). However, due to its incomplete state of preservation, its precise attribution is not possible (see Sjøvold 1993: p. 38, pl. 17). As regards the motif of the eyes on the bow and the beak on the upper part of the foot-plate, one may recall the brooch from Vik (II), Fjære, Agder as an analogy, although not very adequate (Sjøvold 1993: p. 62, pl. 17:N19). It is considered a simplification of patterns which were identified on the fibula from Gyland, Bakke, Agder (Sjøvold 1993: p. 62, pl. 17:N20, with further literature); however, the latter lacks the mentioned motif. Moreover, a similarly shaped image of eyes (but doubled, i.e. with a pair of eyes similarly placed in the upper part of the bow) can be found on the brooches with oversimplified ornament (although still in Style I, Phase B) which can be seen on Thuringian finds from Mühlhausen, Görmarke Landstraße, Ldkr. Unstrut-Hainich-Kreis, grave 2 (Schmidt 1976: pl. 114:c) and Beuchte, Ldkr. Wolfenbüttel, grave 1 (Schmidt 1961: Fig. 10:f; Høilund Nielsen 2003: p. 213, Fig. 8:26e). It should be noted here that finding an ideal analogy is generally impossible. This is due to the fact that relief brooches, being an element of female elite clothing, had individualised decorations and morphology. These traits, however, were in agreement with general rules of a given style (see Høilund Nielsen 2009: p.57).

A good analogy is offered by a fibula from Darmstadt-Windmühle, Ldkr. Darmstadt-Dieburg in Hessen. The artefact is provided with the same so-called *Zangenmuster* bordering and an almost identical composition of the head-plate decoration. Even the eye pattern on the bow is placed in the same position. After the devastation that had occurred during World War II, the artefact was reconstructed on the basis of the gypsum mould that survived in the Römisch-Germanisches Zentralmuseum in Mainz (Salin 1904: p.295, Fig. 636; Haselhoff 1981: p.474–477, 479, Figs. 306, 307). Originally, it was 11.6 cm long, so being very much like the find from Radziejów. It is believed to be a continental copy of south Scandinavian origin, made by an artisan in the Middle Rhine area, partly without understanding the concept of the image, which is especially noticeable in the head-plate animal ornamentation (Haselhoff 1981: p.474, 476–477, 479; Høilund Nielsen 2009: p.70–71). There are minor differences: instead of heads with bristled hair, there are animal paws, the motif in the upper right corner of the right ornamental zone seems to be simpler and some fragments of bodies are replaced by limbs. Additionally, there is no representation of a bird's head. However, despite these small discrepancies, it seems obvious that both fibulae were made in the same workshop (despite the lack of metallographic analyses of the missing fibula).

In addition to metallurgical examinations, a quite important field to study is the stylistic analysis of the ornamentation of the Radziejów fibula. Undoubtedly, the decoration of the discussed find was arranged according to principles of Animal Style I after Salin (1904), more accurately to its Phase B after Haseloff (1981: p.180–196). Phase B finds are characterised by low relief, clear accentuation of contours and covering the major surfaces with animal decoration. The bodies depicted in outlines are filled with raised stripes (Haselhoff 1981: p.180). This is clearly visible also on the fibula from Radziejów. Geographically, artefacts representing Phase B with its striped animal bodies are mostly found in the Baltic Sea zone (Haselhoff 1981: p. 707). However, they also appeared as imports in the Gepidic areas or the Lombard territory in Pannonia, coming there from the north across the Odra and Vistula watersheds (Haselhoff 1981: p. 673–674). If we attempt at identifying the fibula from Radziejów more precisely,

it should be assigned to Continental fibulae, Group Kirchheim (besides the general composition, the bristled hair is typical of the group in question). Such fibulae appeared mainly in medium (like the one from Radziejów) but also in small sizes (see Høilund Nielsen 2009: p.70–71, Fig. 13). The same identification refers to a fibula from Darmstadt-Windmühle (Høilund Nielsen 2009: p.70, table 7). As to the bordering, we do not know any exact analogies to the Radziejów fibula, apart from the find from Darmstadt-Windmühle. It looks like a simplified version of the claw-frieze (Germ. *Zangenfries* or *Zangenmuster*), Type B2 (Haselhoff 1981: p. 314–318), which suggests a certain, but not very strong, simplification or stylisation. Another example of such stylisation of the above pattern, although appearing in a different motif, was studied by Hillberg (2009: p.182–186) who discussed it against the central-, north- and west-European background.

Relief fibulae served as an element of a female dress. It is believed that they were status symbols of female sorceresses or priestesses. Such fibulae were worn across the upper part of the bosom which can be seen, e.g. at the image on a pendant from Aska in Sweden (Olsen 2006: Fig. 7). It could also be concluded from the position of similar fibulae in graves, e.g. Sejflod, Aalborg Komm., Region Nordjylland, grave DI (Jørgensen, Nørgard Jørgensen 1997: Fig. 46:g). Such fibulae were not only artefacts of practical function but were also ornamental pieces of jewellery of symbolic value. As a probable attribute of goddess Freya, they may have been worn during sacrifices carried out by noble ladies from the top of the society, in the course of erotic rituals or while interpreting the omens (Olsen 2006: p.496–497, 503).

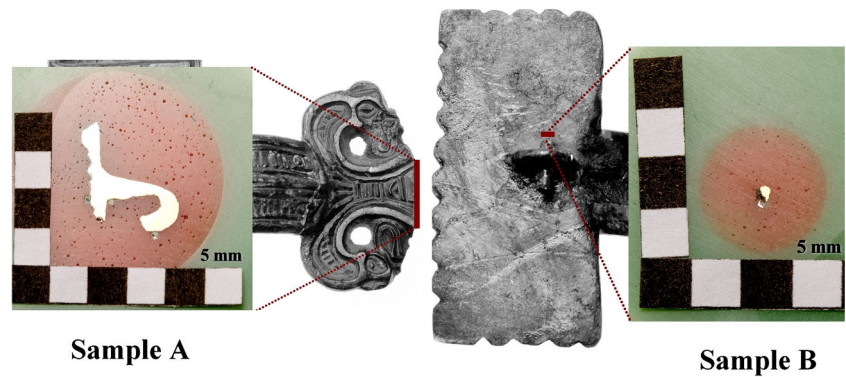
As regards their chronology, relief fibulae have been included in Type E1 after Høilund Nielsen, (1997, 76), characteristic of Phase 1A for Bornholm materials. In most cases, they appear in pairs in rich female graves (Høilund Nielsen 1987: p. 70–72, Fig. 10). Their absolute chronology generally covers the first half of the sixth century, though a precise distinction between Phases 1A and 1B (lasting until ca. AD 600) seems to be problematic (Høilund Nielsen, 1997: p.69, Fig. 18). Bitner-Wróblewska places them in Phases 3 and 4 according to her idea, i.e. a late stage of Phase D and Phase E. This generally means the second half of the fifth century and the first half of the sixth century. This stands in agreement with earlier Scandinavian perspectives (Bitner-Wróblewska 2001: p.120, pl. II, LIX). For instance, they are attributed to Gotlandic Phase VI:2 after Nerman (Nerman 1935: p.64, pl. p.32–34), i.e. ca. AD 475/500–550 (Nerman 1935: p.119–121; 1969; 1975; see Bitner-Wróblewska 2001: p.16). Relief cast square-headed fibulae from England were also produced between AD 500 and 575, with a decline in numbers after AD 550 (Olsen 2006: p.479; Hines 1997: p.301; Høilund Nielsen 1997: p.79–80, Fig. 28). However, we have to remember that the latest ones were embellished in Animal Style II. The finds from central Germany could be later than the Scandinavian

relief fibulae. Schmidt attributed them to his Group IIIb, which falls to AD 560–600 (Schmidt 1961: Fig. 49); however, such dating seems dubious in particular cases and should be shifted for earlier stages (Høilund Nielsen 2009: table 7–8). The ‘Lombard Phase’ finds from Bohemia are attributed to the period of AD 530–560/70 (Droberjar 2009: Fig 4). The Italian brooch from Nocera Umbra is dated to the earliest Stage 1 (AD 572–590) of the Lombard stay in Italy. Together with single costume elements made in Style I, they are considered products of Pannonian workshops from the period before the end of the Lombards’ migration (see Bierbrauer 2005: Fig. 3; 2008: 113, Fig. 2). Taking into consideration the chronology of Group Kirchheim, it should be remembered that there are no exact dates for its production (the Kirchheim grave itself must be a very late deposition from AD 580–600, see Høilund Nielsen 2009: 86, table 7). However, on the basis of stylistic traits, one is tempted to link it with Stage A after Høilund Nielsen, i.e. 460/80–510/25 AD. Copies (made in the Middle Rhine area) come from Stage B, i.e. AD 510/25–565, and further imitations stem from a not datable context (Høilund Nielsen 2009: p.82–84). Therefore, the dating of the fibula from Radziejów should be specified as the first half of the sixth century, but having in mind the fact that it is not worn out, an earlier stage of this period is probable.

We should underline that the artefact in question most probably belongs to imported fibulae, perhaps from Scandinavia or maybe the Middle Rhine area. This is due to the fact that it is not as sophisticated as many (but not all) finds from the north. It has some stylistic analogies in Thuringia, e.g. the eyes pattern on the bow and the general shape of biting beasts on the foot-plate. On the other hand, the former trait is also proved for Scandinavia, while the latter seems to be connected with the process of overall simplification of the original motif. Copy-level fibulae are also known from Scandinavia. The bordering looks like a simplified version of a claw-frieze. There is one very close analogy for this in the Middle Rhine area and it is considered a copy of Scandinavian origin. However, it is not entirely clear whether we are really dealing with a Merovingian copy or a copy-level fibula made in south Scandinavia. The latter identification is supported by the fact that fibulae of that and larger size (over 14 cm) are mostly typical of southern Scandinavia (Høilund Nielsen 2009: p.61, Fig. 5). It seems slightly more probable that the discussed artefact came here from Scandinavia rather than from a more distant place, i.e. the Middle Rhine region. However, the problem cannot be solved at the present stage of research.

So far, it is the first relief fibula found in the territory of Poland. We know only one fragment of gilded foot of a relief fibula from Brzostowo, Piła district. This artefact is either a Scandinavian original or an import of simplified form which was manufactured in the Merovingian cultural circle (Rudnicki 2014, p.287–288, Fig. 6:1). It seems interesting that no finds of relief brooches are known among numerous

Fig. 4 Samples A and B with their sampling areas in the fibula



imported fibulae from the West Balt Circle, including the Elblag Group and the Olsztyn Group (Hillberg 2009: p.188). This is in spite of the fact that Scandinavian fibulae and belt fittings are proved here, namely, in the Elblag Group but also in the Sambian-Natangian area (Kontny 2012: p.61–65). Furthermore, Animal Style I is also represented in the Olsztyn Group and in the Sambian-Natangian area (Hillberg 2009: p.190–195).

Concluding, the fibula is a unique discovery in Poland and it comes from a period which is still poorly studied for this country. Due to the discussed characteristics of the artefact, it was decided to carry out a series of technological examinations. In the future, their results will allow for a more accurate provenance study of fibulae of such type.

Methods

Instrumentation

Preliminary structural observations were carried out with a Nikon SMZ 800 optical microscope and with a Nikon Epiaphot 200 inverted metallographic microscope at $\times 50$ – 500 magnifications. Two samples (see below) were taken and their surfaces were etched with 1 part of perchloric acid HClO_4 and 2 parts of acetic acid anhydride $\text{C}_4\text{H}_6\text{O}_3$ solution for 10 s.

Scanning electron microscopy with energy dispersive X-ray (SEM–EDX) analyses (Goldstein et al., 2007, Barbacki 2007) of sample cuts were performed using a Carl Zeiss EVO MA10 scanning electron microscope equipped with a EDAX X-Flash Detector 5010 with 123 eV spectral resolution (Zeiss, Poland; www.zeiss.com). Image analyses were made using a secondary electron (SE) detector with resolution up to 2.0 nm. Accelerating voltage of 20 keV was applied; other characteristics of current, field magnifying and type of sampling were adopted to the type of studied surface area. Spectra were registered by an energy dispersive spectrometer that collects the entire spectrum of X-ray from 0 to 20 eV, with resolution of 123 eV. Then, average concentrations of elements were obtained. The measurement time was set to 120 s for multi-point analysis and to 5 min for mapping. Measurements were carried out for eight areas of a few micron size. Concentrations of copper (Cu), tin (Sn), zinc (Zn), iron (Fe), lead (Pb) and other metallic and light elements were studied using SEM–EDX examinations. Prior to taking samples, the SEM–EDX analyses of the entire artefact were also performed. However, due to the surface contamination by soil ingredients like silicon (Si), aluminium (Al), oxygen (O), calcium (Ca) and other light elements, as well as due to a low depth range (up to 2 μm) of electrons of excitation source, the analyses did not allow to obtain reliable information about the raw alloy composition.

An X-ray generator designed and built in the NCNR in Świerk, Poland, was used for energy dispersive X-ray

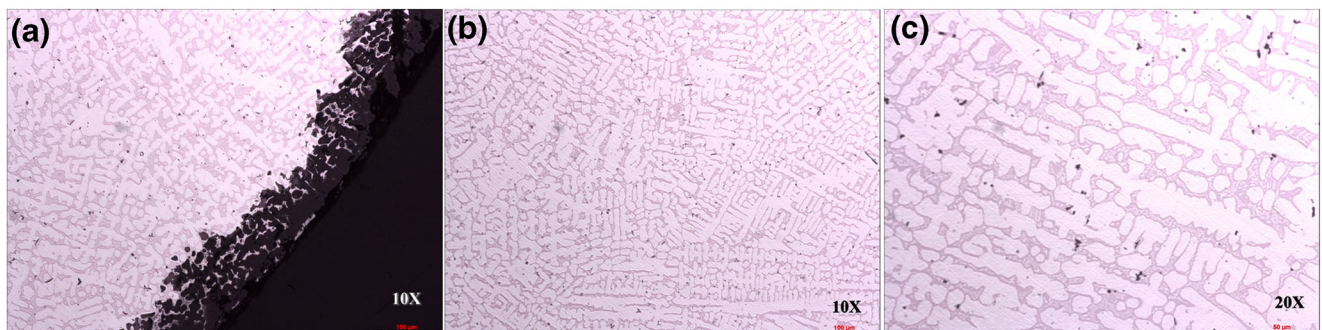


Fig. 5 Sample A. OM images of metal alloy structure obtained after etching at **a**, **b** mag $\times 10$ and **c** mag $\times 20$. There is an internal core dendritic microstructure (two-phase multiple components system) with a porous corrosion layer up to 100 μm thick in the surface (**a**)

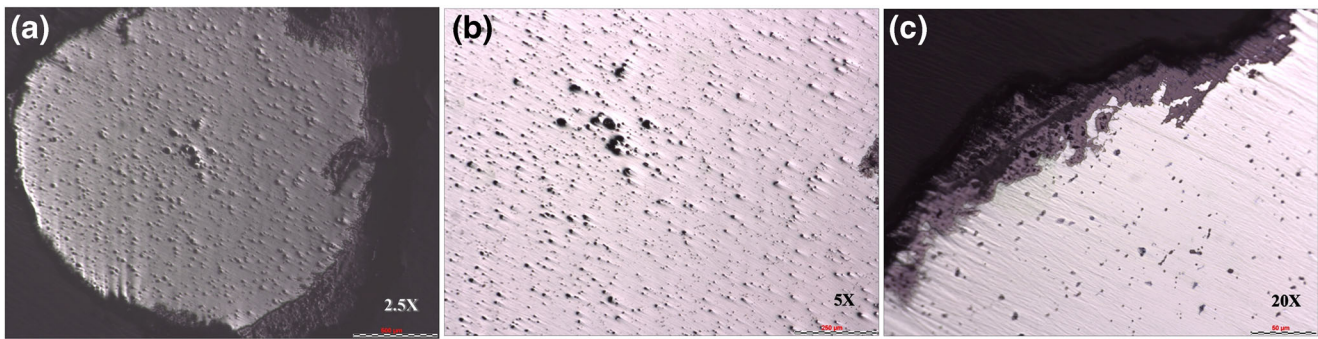


Fig. 6 Sample B. OM images of metal alloy structure obtained after etching at **a** mag $\times 2.5$, **b** mag $\times 5$ and **c** mag $\times 20$. One-phase system and an external porous corrosion structure (up to 50 μm thick) can be seen

fluorescence (ED-XRF) measurements. This compact X-ray generator (weight 0.835 kg, length 430 mm) was constructed with the use of transmission X-ray tube technology. A focused electron beam bombards a silver (Ag) anode target generating X-ray radiation. The silver anode has been sputtered on beryllium. The maximum accelerating voltage is 50 kV. The beam current and accelerating voltage can be smoothly adjusted.

The XRF spectrum was induced by X-ray irradiation on a spot with a diameter of 2.1 cm. Such a large analysing area yielded well-averaged values of the elemental composition. The X-ray generator was operated with an anode voltage and current of 40 kV and 30 μA , respectively. Fluorescence spectra of studied objects were recorded with an Amptek Si-PIN detector. The energy resolution of the detector was assumed to be 149 eV at 5.9 keV photon energy. The measured energy resolution was found to be 152.9 ± 0.3 eV. The distance between the X-ray source and measured objects (SO) and between objects and the detector (OD) was set to 9 and 11.5 cm, respectively. Energy spectra were analysed with an Amptek ADMCA multi-channel analyser. Data on contents of copper (Cu), tin (Sn), iron (Fe), lead (Pb), zinc (Zn), silver (Ag) and gold (Au) was obtained. Moreover, μ -XRF analyses of elemental composition were carried out on sample cuts (sample A and sample B, see below).

XRD method was used for phase identification. Powder X-ray diffraction measurements were performed with Cu $K\alpha$ radiation ($\lambda = 1,5418 \text{ \AA}$) using a Siemens D500 diffractometer equipped with a Si:Li semiconductor detector cooled with liquid nitrogen and a ICDD PDF4 2014 database. The powder diffraction pattern was measured in $\theta/2\theta$ scanning mode with a step of 0.02 and integration time of 10 s/step.

Vickers micro-hardness (HV0.2) was measured on polished surfaces of the cuts, using a 200-g load applied for 12 s. At least 10 measurements per data point were made. A Zwick Roell ZHU0.2/Z2.5 machine was used. The tests were carried out according to the ASTM E384 standard with a constant distance of 2.5 diameter of a stamp between every stamp.

Description of the samples

Complementary instrumental physico-chemical techniques were used to examine the material structure (for a similar methodology, see, e.g. Miazga 2014, Mišta et al. 2015 & Mišta et al., 2016, Meeks 1993, Ashkenazi et al. 2015, Giunilia-Mair 2005) of this unique fibula. The analyses were performed on the unchanged artefact and on the cuts of two samples. Figure 4 shows the cuts with areas from where the samples were taken. The samples were mounted in epoxy resin. They were ground using Premium SiC abrasive paper (for 5 s with P220, P500, P800 and P120 type: <http://www.metallographic.com/Brochures/SiCpaper.pdf>; accessed on 8 November 2017) and polished using Al_2O_3 powder for 2 min. The surfaces of the prepared cuts were etched in 1 $\text{HClO}_4/2\text{C}_4\text{H}_6\text{O}_3$ solution for 10 s. Elemental and phase components of the prepared samples which correspond to the artefact's raw alloy without corrosion, degraded surface and subsurface structure were determined. Furthermore, the cuts correspond to the parts of the fibula which were manufactured separately, i.e. the cross-section of the bow (sample A) and part of the axle of the spring (sample B).

Optical microscopy (OM) images of the cuts' surfaces show the crystallographic metal alloy system with corrosion structures on the surface layer. In Fig. 5, there are results of microscopic examinations.

Scanning electron microscopy (SEM) revealed information about the surface condition (see Fig. 7). Moreover, using EDX spectrometry allows to determine elemental composition in the cuts. XRD and HV0.2 analyses were also performed for sample A and sample B. XRF analysis was carried out in micro scale.

X-ray fluorescence spectroscopy (XRF) does not require sample preparation (see Gójska & Mišta 2016, Mišta & Gójska 2016). The ED-XRF measurements were done on the entire artefact in order to determine the elemental composition of the external alloy layer (up to ca. 20 μm : Henke et al. 1993) by recording signals from components.

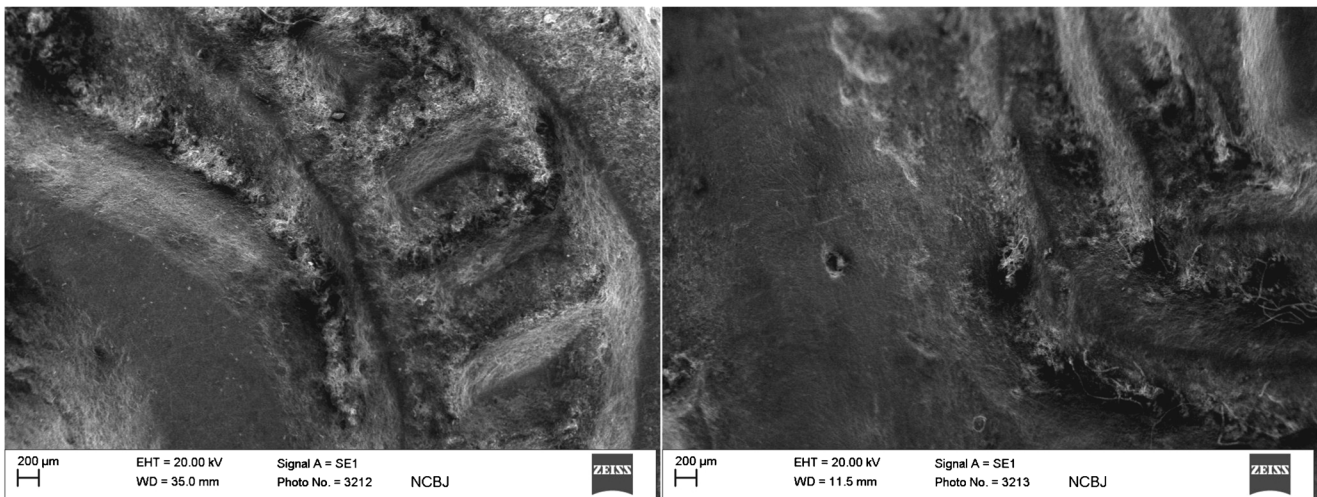


Fig. 7 SEM–SE images of a fragment of the higher disc fragment (obverse side = obv.). Surface relief ornament, soil contamination and corrosive degradation can be seen

Results and discussion

A preliminary SEM–EDX study of the fibula shows its surface degradation which was invisible to the naked eye. The results (Fig. 7) demonstrated that it was necessary to work with pure metal alloy whose structure corresponds to the raw material. Below are examples of SEM images obtained for the fibula surface with relief ornaments (obverse side). Irregular cloudy shapes correspond to the corrosion and soil contamination of the structure which is mainly built of light element oxides. The external surface is also rich in iron and copper oxides (up to ca. 50 wt% in total).

Table 1 shows the results of the quantitative elemental analysis carried out with the use of different methods. All

the methods are based on measurements of X-ray spectra. The characteristic energy of X-ray corresponds to a given element in the sample. The difference between these methods consists in another type of X-ray excitation: for EDX, it is electron beam and for XRF, it is X-ray from an X-ray tube. The range of penetration depth for electrons and X-ray is different so we can study surface and subsurface layers (Mišta et al. 2016). This penetration depth is highly dependent on the material density. Furthermore, the areas of sampling are also different. For ED-XRF, it is a field with a diameter of 2.1 cm, while for EDX and μ-XRF, it is a field within a micrometre area. The obtained numerical data were recalculated and are offered in Table 1 as normalised weight percent taking into account the alloying elements.

Table 1 Results of the SEM–EDX, ED-XRF and μ-XRF quantitative elemental composition analyses (wt%) (low detection limit LLD = 0.1 wt%)

| Technique | Sampling | No. | Cu | Sn | Zn | Pb | Ag | Fe |
|----------------------------------|----------|--------------|--------------|--------------|--------------|-------------|-------------|-------------|
| For the entire artefact: | | | | | | | | |
| ED-XRF | Obv. | 1 | 59.59 ± 5.96 | 22.46 ± 2.24 | 10.28 ± 1.03 | 4.34 ± 0.43 | 1.58 ± 0.16 | 1.39 ± 0.14 |
| | Rev. | 2 | 52.67 ± 5.23 | 27.09 ± 2.71 | 9.36 ± 0.94 | 5.38 ± 0.54 | 1.84 ± 0.18 | 1.63 ± 0.16 |
| For the cuts of samples A and B: | | | | | | | | |
| μ-XRF | A | 1 | 71.23 ± 7.12 | 10.19 ± 1.01 | 18.58 ± 1.86 | < LLD | < LLD | < LLD |
| | B | 2 | 90.09 ± 9.00 | 2.85 ± 0.28 | 4.18 ± 0.42 | 1.54 ± 0.15 | 0.60 ± 0.06 | 0.79 ± 0.08 |
| SEM–EDX | A | 1 | 69.95 ± 1.94 | 12.34 ± 0.41 | 16.98 ± 0.53 | 0.60 ± 0.10 | 0.13 ± 0.04 | < LLD |
| | | 2 | 66.94 ± 1.85 | 12.57 ± 0.41 | 15.96 ± 0.50 | 4.40 ± 0.27 | < LLD | 0.12 ± 0.04 |
| | | 3 | 71.89 ± 1.99 | 9.83 ± 0.33 | 18.18 ± 0.56 | 0.11 ± 0.06 | < LLD | < LLD |
| | | 4 | 67.25 ± 1.86 | 17.80 ± 0.56 | 14.62 ± 0.46 | 0.33 ± 0.08 | < LLD | < LLD |
| | | 5 | 71.75 ± 1.96 | 9.43 ± 0.31 | 18.58 ± 0.56 | 0.24 ± 0.06 | < LLD | < LLD |
| | Av. | 69.95 ± 1.06 | 12.34 ± 1.49 | 16.98 ± 0.73 | 0.33 ± 0.82 | < LLD | < LLD | |
| | B | 6 | 93.72 ± 2.56 | 2.13 ± 0.10 | 4.15 ± 0.17 | < LLD | < LLD | < LLD |
| | | 7 | 93.45 ± 2.55 | 2.09 ± 0.10 | 4.46 ± 0.18 | < LLD | < LLD | < LLD |
| | | 8 | 92.61 ± 2.50 | 2.03 ± 0.09 | 4.59 ± 0.16 | 0.63 ± 0.07 | < LLD | 0.15 ± 0.03 |
| Av. | | 93.45 ± 0.34 | 2.09 ± 0.03 | 4.46 ± 0.13 | < LLD | < LLD | < LLD | |

Due to a considerable degree of the artefact surface contamination and degradation, it was necessary to perform EDX and (for the sake of comparison) μ -XRF analyses on cross-section cuts (samples A and B). Figure 8 shows a juxtaposition of the SEM–EDX mapping analyses performed for sample A. The maps demonstrate the homogeneity of distribution of the main determined elements: copper (Cu), zinc (Zn) and tin (Sn) in the alloy core of sample A.

The results of the quantitative elemental composition analyses of samples A and B are given in Table 1 while Fig. 9 offers an example of registered EDX spectra.

The elemental composition of sample A corresponds to the alloy composition of the raw material of the fibula bow, as shown in Table 1. The main elements determined by SEM–EDX are (in descending order) copper (av. 69.95 ± 1.06 wt%), zinc (av. 16.98 ± 0.73 wt%) and tin (av. 12.34 ± 1.49 wt%), while lead is present as trace element (av. 0.33 ± 0.82 wt%). The results are confirmed by the μ -XRF analysis of sample A (Table 1). This elemental composition corresponds to the bi-phase of casting brass with a significant addition of tin. Structures of this type can be seen in the OM images in Fig. 5 and in the diffractogram in Fig. 10 (see below) (Czermański 1978: p.375, Dobrzański 2002: p. 719). Addition of tin (at the level of 1–2%) prevents zinc escape from the alloy due to its easy evaporation above 907°C (Chodkowski 1976: p. 340), while the addition of lead (1.5–4%) improves the machinability of the brass alloy (Dobrzański 2002: p. 719). However, in this case, the addition of tin is significant (at about 12 wt%). This leads to the inference that it was introduced to the alloy in order to provide it with a gold colour. This is also the case with the so-called Mannheim gold used in jewellery production. In its basic form, the alloy consists of 80% Cu and 20% Zn but also occurs with addition of tin at the level of 9% (<http://www.gutenberg.czyz.org/word.44746>, accessed on 8 November 2017).

In sample B, which is part of the axle of the spring, the main elements are copper (av. 93.45 ± 0.34 wt%), zinc (av. 4.46 ± 0.13) and tin (av. 2.09 ± 0.03 wt%) with trace amounts of other elements being below 0.1 wt% (in average value of the EDX measurement done for $n = 3$ micro-sampling points). However, results of μ -XRF analyses (see Table 1) indicate the presence of trace amounts of lead (1.54 ± 0.15 wt%), silver

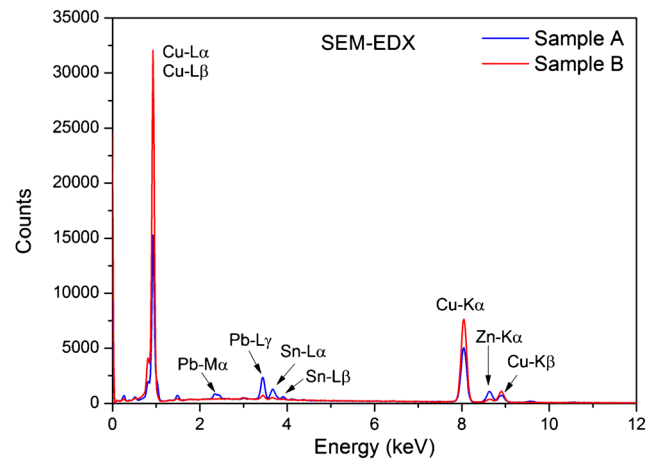


Fig. 9 EDX spectra obtained for the cross-section cuts of the fibula: sample A (blue, no. 2 EDX in Table 1) and sample B (red, no. 6 EDX in Table 1)

(0.60 ± 0.06 wt%) and iron (0.79 ± 0.08 wt%) in the alloy. Due to the fact that these elements were determined by ED-XRF measurements at a significant weight percent level on the artefact surface (see Table 1: ED-XRF results and Fig. 12), their trace amounts in the inner alloy can be linked with partial melting from the surface tinning coat (this issue is discussed below). Therefore, with regard to the contents of the main elements, the composition of sample B corresponds to the composition of brass dedicated for a technique of plastic metalworking called die forming (Czermański 1978: p.375, Dobrzański 2002: p. 719). This type of brass is generally made of two components with small admixtures with a mono-phase crystallographic system (see Fig. 10).

X-ray diffraction analysis (XRD) of metal alloy determined the crystallographic system of samples A and B (Fig. 10). It corresponds to the elemental composition of the matrix alloy of both parts of the artefact (see Table 1) which represents the technological elements (see Fig. 4). Generally, in both diffractograms, there are visible reflections at approximately 42° and 50° . These can be identified as fcc (face-centred cubic cell) solid solution, with a (Cu, Zn) and/or (Cu, Sn) structure, respectively. It follows from the elemental analysis (see Table 1) and from the increased value of the lattice parameter with respect to pure copper. For sample A, the obtained value was $a = 3.655 \text{ \AA}$ and for sample B, it was $a = 3.642 \text{ \AA}$, while

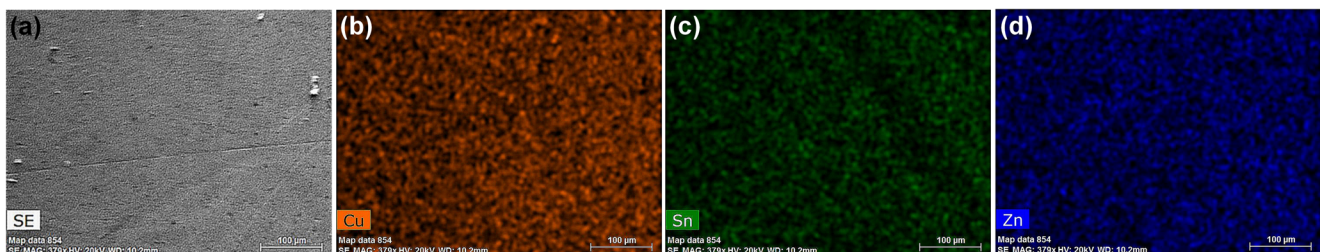


Fig. 8 **a** SEM-SE image (mag $\times 379$) of the surface of the cross-section cut (sample A) and **b–d** (mag $\times 379$) EDX maps with a regular distribution of the main constituting elements: **b** copper—Cu, **c** tin—Sn and **d** zinc—Zn in the alloy

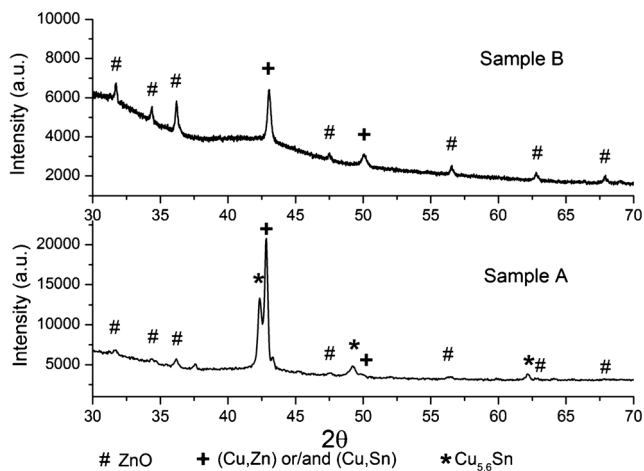


Fig. 10 XRD diffractograms of samples A and B

standard lattice constant for metallic copper is $a = 3.615 \text{ \AA}$. This is due to larger radii of tin (Sn) and zinc (Zn) than copper (Cu), so their participation in the structure expands the crystal lattice of pure copper. Additionally, in sample A, there are several crystallographic phases. This is due to the fact that the fcc structure contains an intermetallic $\text{Cu}_{5.6}\text{Sn}$ phase (or a similar one, having a ‘deformed’ fcc crystal lattice), which is observed in the dendrite structure in Fig. 5. In sample B, its structure is quite uniform and more fine crystalline in comparison with sample A, as it can be seen in optical images in Fig. 6. The ZnO reflections visible in smaller sample B as bigger intensity originate from zinc corrosion.

The low HV0.2 values indicate that the melting process was carried at a not very high temperature.

No layer dislocation can be seen in OM photos (Figs. 5 and 6), which suggests the use of a method of mould casting without treatment at a higher temperature.

Generally, the HV0.2 values decrease in the subsurface area. This corresponds to weakening of the alloy structure due to the annealing process. This process is necessary to form

the relief ornament on the surface and forming the crumple zone like the curvature in the fibula catch-plate (samplings 4 and 5 in sample A) (Ashkenazi & Iddan 2012, Moriera and Tschiptschin, 2016 in Fig. 5).

Summing up, brass with addition of tin and trace amount of lead (sample A) is used as casting alloy, while brass for forming (sample B) is more robust and has better malleable properties (Wesołowski, 1972: p.396–430, Doomke 1982). The selection of alloys demonstrates an intentional use of technological and functional criteria. The bow was made of soft alloy to make it easier to shape the artefact’s form and the relief ornament. On the other hand, moving parts like the spring and pin, which had to be durable, were made of harder metal. Such features are confirmed by the average values of micro-hardness which are shown precisely for each measurement point in Fig. 11. The micro-hardness is $160 \pm 2 \text{ HV0.2}$ for sample A and $166 \pm 2 \text{ HV0.2}$ for sample B. These differences in micro-hardness are the results of variations of proportions of copper, zinc, tin and lead (see. Table 1).

A quite homogenous crystallographic structure and uniform values of HV0.2 parameter for sample A and sample B demonstrate that each part, that is the bow and moving parts used for attaching the fibula to the garment was made separately. The bow was made by casting in a mould after melting, and the axle of the spring was made by die forming in a temperature range of 200–800 °C. In the case of Cu–Sn, this corresponds to dendritic structure (see phase diagram of Cu–Sn by Johnson in Dobrzański 2002: p. 729, Fig. 7.15). The Cu–Zn system is homogeneous in the range up to 40% Zn in a temperature range up to the melting point of pure copper (phase diagram of Cu–Zn by Howkins in Dobrzański 2002: p. 719, Fig. 7.13, Wesołowski 1974: p. 404).

Below, Fig. 12 offers ED-XRF qualitative analysis results obtained for the artefact’s surface, for the obverse and reverse side of the fibula, respectively. Table 1 offers the results of quantitative analysis.

Fig. 11 Micro-hardness test results for sample A and sample B, measurement error $\pm 2\text{HV0.2}$. Stamp places in the samples can be seen on the left

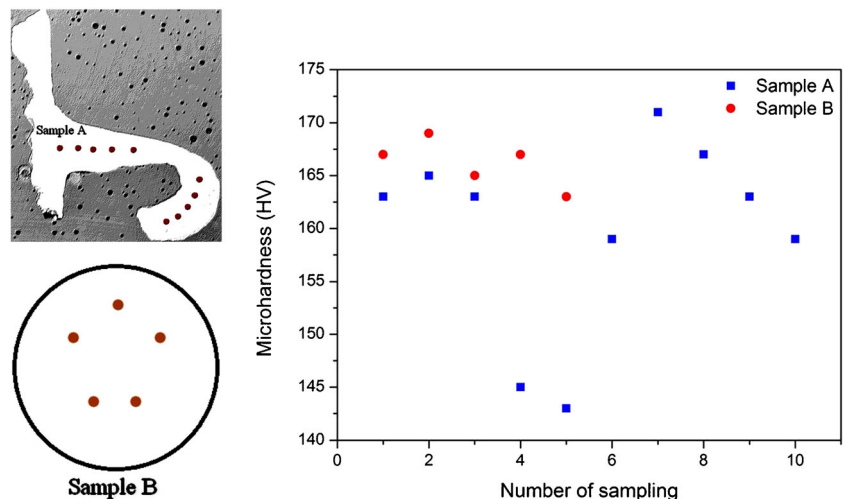
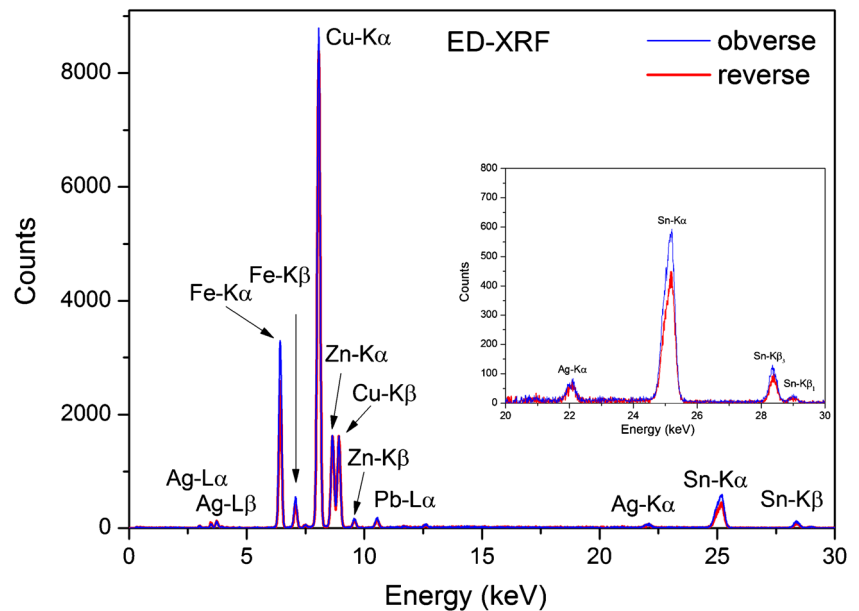


Fig. 12 ED-XRF spectra obtained for the fibula, for the obverse side with the relief ornament (blue) and for the reverse side (red), respectively



In comparison with the base alloys identified in samples A and B, at both sides of the surface (to the depth 20 μm : Henke et al., 1993), there is a noticeable tin (Sn) enrichment. Tin concentration in the surface is above 20 wt%, while in the base alloy in samples A and B, it is at the level of 12 and 2 wt%, respectively (see Table 1). Additionally, in the surface, there are higher contents of silver (Ag) and lead (Pb). Furthermore, traces of iron (Fe) are detected. A higher content of tin can indicate the use of hot-dipping tinning in order to decorate the surface of the fibula (e.g. Miazga 2014, Šmit et al. 2008, Meeks 1993, Giunlia-Mair 2005). The addition of silver and lead can originate from silver ores (galena PbS) which could be added to the tin solution used for coating the artefact by dipping it into the molten tin solution. On the other hand, the presence of iron should rather be related to soil contamination of the surface.

Conclusions

This archaeological examination and the technological analyses of the unique fibula found in Radziejów in Poland can be a background for similar studies of other artefacts mentioned in the introduction. In the future, such studies can shed more light on the presence and transformation of fibulae of such types in the Baltic Sea region. Furthermore, the study of the elemental composition of the artefact, combined with physical micro-hardness tests and crystallographic examinations, allowed for an identification of the technological process which was used in the manufacture of artefacts of this type.

The most important conclusion is that the fibula from Radziejów was tinned, as evidenced by the XRF analysis of the obverse and reverse of the find. Usually, fibulae of this type were gilded in the discussed period. The raw alloy was

determined as a brass with different contents of tin, zinc and trace amounts of lead. The elements were mixed depending on the functional requirements of each part of the artefact. Casting brass was used in the manufacture of the bow. On the other hand, forming brass as a base alloy was applied for the axle of the spring, which was a moving part of the fibula. The HV0.2 value is higher for mounting parts, that is for the pin and other parts of the attachment mechanism (sample B). The relatively low HV0.2 value and the presence of fcc network for Cu/Zn and Cu/Sn crystallographic system (which corresponds to the identification of the elemental composition) indicates that the casting process was performed in a not very high temperature. The presence of the zones with lower HV0.2 values proves the use of the annealing process for surface ornament forming.

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