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REVISITING “TIN IN SOUTH-EASTERN EUROPE?”

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Abstract. – The important role of the Balkans in the origin and development of metallurgy is well established with respect to copper. In addition, Aleksandar Durman, in his 1997 paper “*Tin in South-eastern Europe?*”, essentially initiated studies into the role of the Balkans in Europe’s Bronze Age tin economy. He identified six geologically favourable sites for tin mineralisation and associated fluvial placer deposits in the former Yugoslavian republics, and suggested that these may have added to the tin supply of the region. The viability of two of these sites has been confirmed (Mt Cer and Bukulja, Serbia) but the exploitation potential for the other locations has remained untested. River gravels from these four sites (Motajica and Prosara in Bosnia and Herzegovina; Bujanovac in Serbia; Ogražden in North Macedonia) were obtained by stream sluicing and panning. The sites of Prosara and Bujanovac were found to be barren with respect to cassiterite (SnO_2). Streams flowing from Motajica and Ogražden were both found to contain cassiterite, but in amounts several orders of magnitude less than at Mt Cer and Bukulja. Although it is possible that minor tin recovery occurred at Motajica and Ogražden, it is unlikely that they could have contributed meaningfully to regional tin trade. This is supported by the fact that the isotopic signature ($\delta^{124}Sn$) of cassiterite from Motajica is highly enriched in light isotopes of tin compared to that associated with Late Bronze Age artefacts of the region.

Key words. – Cassiterite, Placer, Tin, Bronze Age, Balkans, Sn Isotopes

The quest for the origin of the earliest metallurgy in southeast Europe dates to the first half of the 20th century when O. Davies published descriptions of the remains of two mining shafts in Jarmovac in south-western Serbia.[1] The period after the Second World War was marked by papers that discussed the prehistoric mining in the context of geology and possible exploitation.[2] The discovery and excavations of the prehistoric copper mines of Rudna Glava in eastern Serbia and Ai Bunar in Bulgaria in the late ’60s, and Mali Šturac in the ’80s,[3] resulted in several publications that addressed various aspects of prehistoric copper mining, such as the material culture, chronological positioning, and technological processes[4], primarily based on stylistic and typological analyses of archaeological material (e.g. potsherds, crucibles, copper beads and malachite remains) from Neolithic and Eneolithic sites.[5] Papers focused on physical-chemical analyses followed in the ’90s.[6] While the

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[1] Davies 1937.

[2] Simić 1951; Simić 1969.

[3] The excavations at the site of Mali Šturac are still ongoing. For previous results refer to: Antonović, Vukadinović 2012, with cited literature; Antonović 2018, with cited literature.

[4] The most complete overview of the history of research and current data and progress is provided in Богосављевић-Петровић 2005 and Antonović 2018.

[5] *cf.* Jovanović 1971; Черних, Радунчева 1972; Јовановић 1974; Jovanović, Ottaway 1976; Јовановић 1978; Černych 1978; Черньх 1978; Jovanović 1985.

[6] Pernicka et al. 1993; Begeman et al. 1995.

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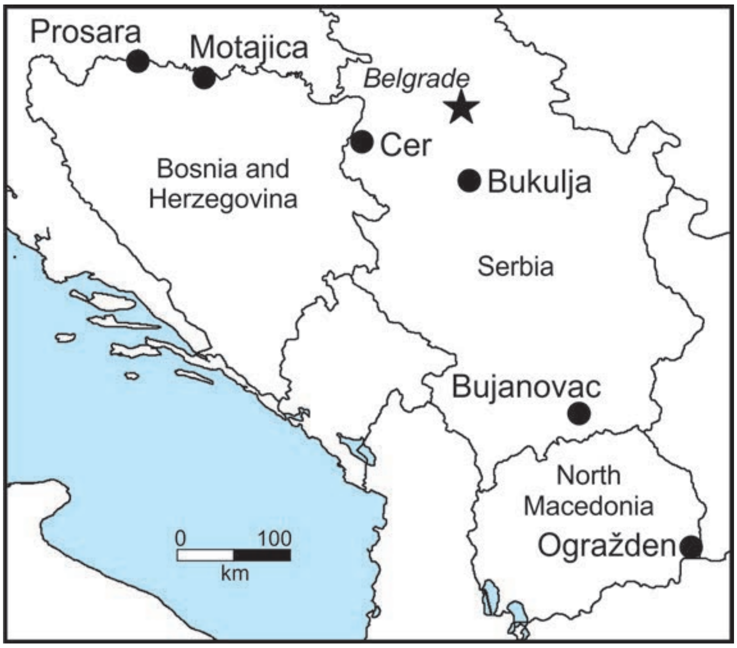

Fig. 1. Potential tin sources identified by A. Durman (1997)

Сл. 1. Потенцијални извори калаја према А. Дурману (1997)

search for the source of tin, necessary for the production of tin-bronze, was ongoing in western Europe throughout the 20th century, it was essentially neglected by local authors in south-eastern Europe.

In 1997, Aleksandar Durman published the article “*Tin in South-eastern Europe?*”. This breakthrough paper proposed that the long-sought source of tin during the Bronze Age of south-eastern Europe need not have been the large Variscan deposits of the Erzgebirge or Cornwall, nor an exotic source in Central Asia. Rather, he suggested small placer cassiterite (SnO_2) deposits in the Balkans may have provided tin for regional bronze production. Based on communications with Serbian geologist Dr. Antonije Antonović, he identified two Serbian sites as the most likely sources for prehistoric exploitation, Mt Cer[7] (20 km east-northeast of Loznica) and Bukulja[8] (8 km west-southwest of Aranđelovac) (Fig. 1).

Mining companies and national geological surveys have conducted feasibility studies of these two placer deposits. As reported by Durman (1997), Mihajlović (1978) estimated the gravels associated with Bukulja’s Cigankulja stream contain sufficient cassiterite to produce approximately 225 tons of tin metal. Tin placer deposits of Mt Cer were found to be significantly larger than those of Bukulja, and this was confirmed by Tomić (1991), who projected that the alluvial deposits of the Lešnica and Cernica rivers contain sufficient cassiterite to produce 2,700 tonnes of tin. Thus, only 0.1% of Cer’s current ore reserves could have produced 30 tonnes of bronze, sufficient to supply the entire central Balkan bronze production of the Late Bronze Age.

Subsequently, highly disturbed archaeological sites yielding predominantly fragments of Late Eneolithic and Bronze Age pottery were documented adjacent to the richest tin gravels at Mt Cer, and these include rare fragments of technical pottery with tin-bearing vitreous surfaces.[9] The Sn isotopic composition of Late Bronze Age Serbian artefacts defines a regional cluster south of the Danube River and west of the Morava River (coincident with the location of Mt Cer) that is consistent with those of the ores of Cer’s Milinska, Cernica, and Lešnic rivers.[10] Accordingly, Mason et al. (2020) concluded that mining activities at Mt Cer likely contributed significantly to tin metal production of the region at that time.

In addition to Mt Cer and Bukulja, Durman (1997) identified four other locations of interest.[11] He reported an unpublished account of detrital cassiterite in rivers at Bujanovac in Serbia, and a report by Tućan

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[7] Durman, 1997, 10.
[8] Durman, 1997, 8.
[9] Huska et al., 2014, 487–488.
[10] Mason et al., 2020.
[11] Durman, 1997, 9–10.

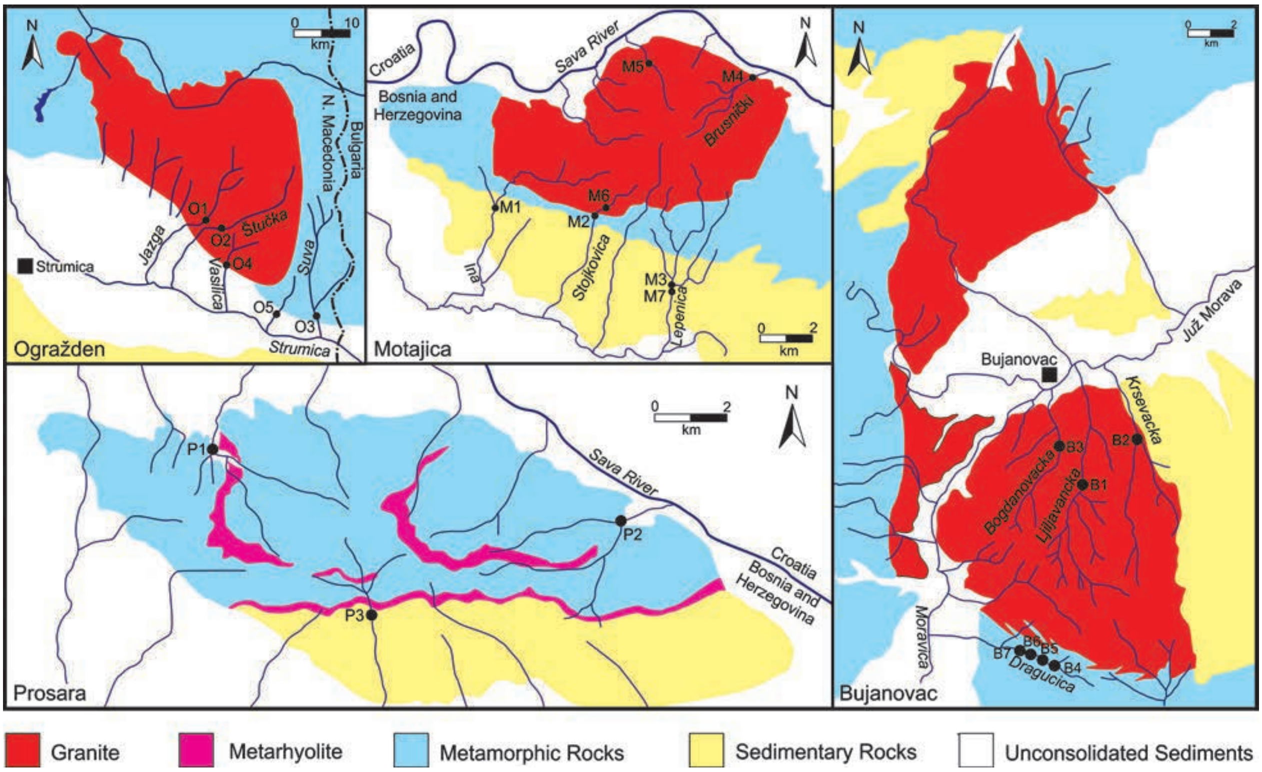

Fig. 2. Sample Location Maps
(Geology of Ogražden is based on Boev et al. 2002; Geology of Motajica and Prosara is based on Ustaszewski et al. 2010; Geology of Bujanovac is based on geological map of Vranje K34–56)

Сл. 2. Локације на којима је вршено узорковање
(геолошке подлоге засноване су: Ограждена – на Boev et al. 2002; Мотајице и просаре – на: Ustaszewski et al. 2010; Бујановца – на геолошкој карти Врања, исечак К34–56)

(1957) of cassiterite in streams that flow from the Ogražden granite near Strumica in North Macedonia (Fig. 1). In addition, he noted that the granites that lie beneath Motajica and Prosara in northern Bosnia are similar in composition to those of Mt Cer and Bukulja, and so could potentially host similar mineralisation. However, none of these four sites had been investigated in detail to confirm whether detrital cassiterite is present, and if so, whether sufficient quantities would have allowed for production of a significant mass of tin. The purpose of this study was to examine each of these sites and evaluate their viability as potential prehistoric placer tin mining sites.

Sampling and Methods

The bedrock geology and river drainage pattern of each site was examined to identify the most likely sites for placer tin accumulations (i.e., confluences of rivers with larger watersheds that cross-cut granite bodies). For each stream that had sufficient water flow to allow a sluice to operate, river gravels were sieved (<2 mm) to produce approximately 30 litres of sand, which was then fed through a portable sluice box. The sluice output was panned on site to a “black-sand” concentrate. An Olympus Delta portable x-ray fluorescence device (pXRF) was used to determine the tin content of the “black-sand” concentrate on site.

Initial sampling was undertaken at Motajica (M1 through M3) and Prosara (P1 through P3) in June 2017, and reported on by Cruse et al. (2017). The remaining samples, including a resampling of Motajica, were collected in May 2018 when streamflow was more conducive to sluicing. A total of 22 samples were taken from 17 streams at the four study locations (Fig. 2): four streams at Bujanovac (Bogdanovačka, Dragučica, Krševačka and Ljiljanovačka rivers), five at Ogražden

(Jazga, Štučka, Suva, Vasilica, unnamed stream), five at Motajica (Brusnički, Ina, Lepenica, Stojkovica, unnamed stream), and three at Prosara (Busovača, Gasnica, Jablanica). In addition, one sample was taken from both Mt Cer (Milinska) and Bukulja (Dugačko) for comparison. At each sample site one bulk sample was taken.

Samples were subsequently dried and the heavy mineral concentrate was further purified by extracting light minerals (<2.9 g/cm^3) using flotation separation with a solution of sodium polytungstate ($3Na_2WO_4$• $9WO_3$•H_2O). The tin content of this concentrate was determined by pXRF. The heavy mineral assemblages were then fed through a Frantz Isodynamic Magnetic Separator after magnetite was removed using a hand magnet. Subsamples were taken at 0.5A, 0.7A, 1.0A, 1.75A, and the remaining non-magnetic fraction, the non-magnetic fraction being that into which cassiterite accumulates. The Sn content of the non-magnetic fraction was determined by pXRF and then mounted on adhesive carbon stubs for SEM examination and mineral identification.

A Hitachi TM3030Plus scanning electron microscope operating at 15kV and an Oxford Instruments AZtec energy dispersive spectrometer with the AZtec One software platform were used to identify all heavy mineral grains mounted on each stub. These quantitative analyses were used to define the major element components of each mineral grain so that mineral formulae/identities could be deduced stoichiometrically, in conjunction with the physical features observable under the SEM (form and cleavage) and binocular microscope (colour, lustre). Emphasis was placed on the identification of cassiterite and other Sn-bearing minerals.

Isotopic analyses of cassiterite from Cer and Bukulja were presented recently in Mason et al. (2020), and, so, analyses were not repeated in this study. Of the samples from the four remaining sites, only sample M6 from the Lepenica at Motajica contained a sufficient mass of cassiterite to allow for the separation of a cassiterite concentrate that could be used for isotopic analysis. Grains of cassiterite (0.3–0.5 mm) were identified by SEM-EDS analysis and then handpicked to form a cassiterite separate of >100 grains. The cassiterite sample was digested following the procedure of Mathur et al. (2017, p17): 0.1 g of -100 mesh cassiterite powder was mixed with 0.5 g of KCN and heated at 850°C for one hour in graphite crucibles contained within capped alumina crucibles. The resulting reduced Sn metal beads were dissolved in heated ultrapure 11N HCl overnight. A small aliquot of this solution was removed and dried and redigested in ultrapure 1M HCl. This solution was purified using the ion exchange chromatography described in Balliana et al. (2013, 2981–2982) and employed by Mason et al. (2016) and Mathur et al. (2017).

Analysis was conducted on the Neptune MC-ICPMS at Rutgers University. Solutions were measured at 50ppb Sn with 50ppb Sb ICP-MS standard, as described in Mathur et al. (2017). Mass bias was corrected for using Sb doped solutions and an exponential mass bias correction defined in Mathur et al. (2017). The corrected values were then bracketed with the NIST 3161A Sn standard (Lot# 07033). One block of 30 ratios was collected. Data is presented relative to the NIST 3161A Sn standard (Lot# 07033) in per mil notation defined as:

$$\delta^{1xx}\text{Sn‰} = \left(\frac{\left(\frac{1\,xx_{Sn}}{116_{Sn}}\right) sample}{\left(\frac{1\,xx_{Sn}}{116_{Sn}}\right) NIST\ 3161} - 1 \right) * 1000$$

Instrumentation 2σ error for δ^{124}Sn is 0.02%. Whole procedural 1σ errors for analysis (ample variability, reduction, dissolution, purification, and analysis) are δ^{120}Sn= 0.08‰ and δ^{124}Sn= 0.16‰. Note that the δ^{124}Sn and δ^{120}Sn are reported relative to ^{116}Sn, with a difference of 8 amu and 4 amu, respectively.

Results

The results are summarised in Table 1. In the panned black sand concentrates, tin was detectable in only the Milinska at Mt Cer (1.4 wt%) and the Dugačko at Bukulja (0.9 wt%). Tin was detected in post-flotation heavy mineral separates at Cer (2.6 wt%), Bukulja (1.2 wt%), two streams at Motajica (Brusnički and an unnamed stream, each with 0.33 wt%), and two streams at Ogražden (Jazga 0.31 wt% and Vasilica 0.24 wt%). In the non-magnetic mineral fractions tin was detected in samples from two additional sites, the Lepenica at Motajica and an unnamed stream at Ogražden. No tin was detected in samples from either Prosara or Bujanovac

In addition to Cer and Bukulja, cassiterite was identified in the non-magnetic fraction of one sample from Motajica (M6 Lepenica) which contained 0.47 wt% Sn, and two samples from Ograzden (O3 Suva, O5 unnamed stream), with 0.05 and 0.12 wt% Sn, re-

Sample	Stream	Panned	Heavy Liquid	Non Magnetic	Minerals of Interest
B1	Ljiljavancka	nd	nd	nd	
B2	Krsevacka	nd	nd	nd	
B3	Bogdanovacka	nd	nd	nd	
B4	Dragucica	nd	nd	nd	
B5	Dragucica	nd	nd	nd	
B6	Dragucica	nd	nd	nd	
B7	Dragucica	nd	nd	nd	
M1	Ina	nd	nd	nd	Sn-bearing rutile, euxenite, bastnaisite
M2	Stojkovica	nd	nd	nd	Thorite, scheelite, euxenite, bismuth, brookite
M3	Lepenica	nd	nd	nd	Bastnaisite
M4	Brusnički	nd	0.03		
M5	Unnamed	nd	0.03		
M6	Lepenica	nd	nd	0.47	Cassiterite, ixiolite, euxenite
M7	Stojkovica	nd	nd	nd	
O1	Jazga	nd	0.03	0.04	
O2	Štučka	nd	nd	nd	
O3	Suva	nd	nd	0.05	Cassiterite, euxenite, scheelite
O4	Vasilica	nd	0.02	nd	
O5	Unnamed	nd	nd	0.12	Cassiterite, thorite, scheelite
P1	Busovaca	nd	nd	nd	Scheelite, wolframite
P2	Gasnica	nd	nd	nd	Scheelite, wolframite
P3	Jablanica	nd	nd	nd	Scheelite, wolframite
Cer	Milinska	1.4	2.6	18.7	Cassiterite, euxenite, microlite
Bukulja	Dugačko	0.9	1.2	11.6	Cassiterite, wolframite, thorite

Table 1. Tin concentrations in various sample fractions in percent, as determined by pXRF, along with key minerals identified through SEM-EDS analysis

Табела 1. Концентрације калаја у узорцима различитих фракција, изражени у процентима, концентрације су утврђене путем pXRF анализе, а главни минерали путем SEM-EDS анализе

spectively. Although these are the most Sn-rich samples found in this study, their tin concentration is two to three orders of magnitude less that that found at Mt Cer and Bukulja.

Although several other samples from Motajica and Ogražden (M4, M5 and O4) had detectable Sn based on pXRF analysis, no cassiterite was found. This suggests that the Sn occurs as a component in other minerals. This has been documented at Mt Cer, where Sn is present within a microlite-series mineral ($[Ca,Sn,U]_2[Ta,Nb]_2O_6(OH,F]$)[12] and within rutile $[(Ti,Sn)O_2]$ in sample M1 from the Ina at Motajica. All samples from Bujanovac and Prosara were found to be barren with respect to tin in any form.

The cassiterite concentrate from sample M6 from the Lepenica at Motajica yielded mass-dependent isotopic compositions of δ^{120}Sn=-0.44‰ and δ^{124}Sn= -0.83‰, relative to NIST 3161A. This contrasts with the more heavy isotope weak to moderate heavy isotope

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[12] Powell et al., in press.

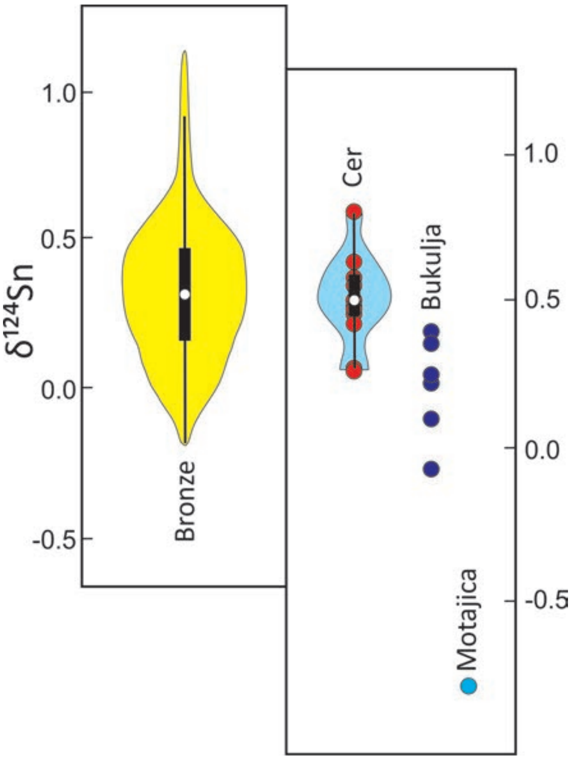

Fig. 3. δ^{124}Sn values for Late Bronze Age bronze artefacts from Serbia and Bosnia and Herzegovina, as reported in Huska et al. (in press) after a -0.2‰ shift to correct for smelt induced fractionation, as compared to cassiterite from Mt Cer, Bukulja, and Motajica. (Cassiterite data for Mt Cer from Huska et al., in press; Bukulja data from Huska et al., in press; Berger et al., 2019)

Сл. 3. Вредности δ^{124}Sn за предмете позног бронзаног доба из Србије и БиХ (Huska et al., in press), кориговане са -0.2‰ како би се одстранили ефекти фракционације проузроковане топљењем, у поређењу са вредностима добијеним за каситерит са Цера, Букуље и Мотајице. (Вредности за каситерит преузете су из: за Цер – Huska et al., in press; за Букуљу – Huska et al., in press; Berger et al. 2019)

enrichment displayed by similar multi-grain placer samples from Mt Cer and Bukulja (δ^{124}Sn of 0.3 to 0.8‰ at Cer; δ^{124}Sn of -0.1 to 0.4‰ at Bukulja).[13]

Conclusions

Of the six potential tin placer deposits identified by Durman, Mt Cer and Bukulja are by far the richest, with both yielding percent-level concentrations of tin in the panned black-sand concentrate. In addition, cassiterite was documented to occur in stream sediments at Ogražden and Motajica, but at concentrations several orders of magnitude lower than Cer and Bukulja. Although containing other rare elements, including tungsten, Prosara appears to be barren with respect to tin. The Vranje geological map (K34–56) notes the presence of Sn in the Dragučica River, along the south contact of the Bujanovac pluton. However, four separate samples taken along the same stretch of this river failed to yield any trace of tin, as was the case for the other three rivers samples at Bujanovac. This may indicate that Bujanovac hosts very minor tin mineralisation, certainly less than would have been necessary for the development of a mineable placer deposit.

Mason et al. (2020) demonstrated through isotopic composition and geographic correlation that ore from Mt Cer likely supplied much of the tin economy of Late Bronze Age Serbia south of the Danube. Although Bukulja is a viable source of tin ore, there remains no archaeological evidence that these deposits were exploited in prehistory. Nor is there a clear correlation between the Sn isotopic composition of Bukulja ores and local artefacts, particularly when the 0.2‰ correction associated with smelt-related enrichment of heavy isotopes in metal products is taken into account, as recommended by Berger et al. (2019), Powell et al. (2019) and Mason et al. (2020) (Fig. 3).[14] Thus, it appears that Bukulja either was not mined in the Late Bronze Age, or was a minor contributor to the tin economy of Serbia relative to Mt Cer and the Erzgebirge at that time.

The Sn istotopic composition of the multi-crystal composite sample of cassiterite from Motajica is far more enriched in light isotopes of Sn than that of Late Bronze Age metal artefacts from the region (Fig. 3). The full range of Sn isotopic composition of cassiterite from Motajica cannot be determined from a single analysis. However, the placer sample was composed of at least a hundred individual sand- to silt-sized detrital

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[13] Mason et al. 2020

[14] Mason et al., 2020.

mineral grains. Thus, the composition of this sample represents a multi-crystal average and, as such, is likely to approach the central tendency of the deposit’s isotopic composition. To support this assertion, the mean and standard deviation of the set of nine placer samples from the Erzgebirge reported by Haustein et al. (2010) is 0.56±0.12‰, as compared to 0.59±0.38‰ for the total of 43 samples. Accordingly, it is reasonable to conclude that the Motajica mineralisation is highly enriched in light isotopes of Sn. Given that none of the 336 artefacts analysed by Mason et al. (2020) yielded δ^{124}Sn values < -0.04 (Fig. 3), Late Bronze Age artefacts with light isotope enrichment appear to be absent from the region. Therefore, if the low-grade placers from Motajica were exploited at all at that time, they did not contribute to the regional tin economy.

Based on the results of on-site sluice- and pan-based sampling, of the six potential Balkan tin ore sources identified by Durman, only Cer, Bukulja and Motajica appear to have sufficient cassiterite concentration to have allowed for the potential exploitation of placer gravels. Furthermore, based on the isotopic composition of Sn from these sources compared to Late Bronze Age artefacts, Cer is the only site that appears to have likely contributed substantially to the regional tin economy at that time

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ЈОШ ЈЕДНОМ О „КАЛАЈУ У ЈУГОИСТОЧНОЈ ЕВРОПИ?”

Кључне речи. – сводови, цеви за сводове, керамичке цеви, *tubi fittili*, *Timacum Minus*, технике грађења, римска архитектура, касноантичка архитектура, северијански период, југоисточна Европа

Обновом археолошких ископавања античког кастела Тимакум Минус 2019. године створиле су се нове могућности за тумачења његових грађевина које су истраживане пре више деценија. Међу остацима грађевина око античког кастела *Timacum Minus*-а посебну пажњу привлачи делимично истражен „објекат са хипокаустом”, нарочито у погледу његових конструктивних карактеристика. Поред иначе честих античких конструкција хипокауста и зидног грејања, међу остацима ове грађевине уочена је и посебна врста грађевинских елемената – керамичке цеви за сводове. Велика количина откривених цеви указала је на то да је ова грађевина заиста имала сводове израђене од њих.

Иако је појава цеви за сводове приликом истраживања античких локалитета на тлу југоисточне Европе регистрована, она није довољно документована, као што ни сама функција цеви често није препозната. Један од разлога за то јесте недовољна упућеност истраживача у специфичне карактеристике цеви за сводове и њихову функцију, услед чега се оне мешају са водоводним цевима, тубулусима или калемовима везаним за зидно грејање – будући да сваки од тих елемената припада керамичким производима који су намењени грађевинарству.

У раду су разматране карактеристике цеви за сводове на Тимакум Минусу, као и контекст у коме су пронађене унутар „објекта са хипокаустом”. На основу налаза печата кохорте Аурелије II Дараданорум одређено је да „објекат са хипокаустом” и конструкција сводова од керамичких цеви потичу из III века – у коме је и иначе појава тих сводова широм Римског царства била честа.

Приликом систематизације врста керамичких цеви на Тимакум Минусу посебно је издвојена она које је било највише у „објекту са хипокаустом”. У склопу ње је препознат и сасвим специфичан централни елемент који је омогућавао да се два низа цеви на истом правцу, али из супротних смерова, међусобно споје. Тај елемент је дефинисао облик свода којим су биле покривене просторије чију је реконструкцију основе било могуће извршити.

Архитектонске анализе „објекта са хипокаустом”, као и карактеристике уочене на самим цевима указале су на то да су просторије биле покривене полуобличастим сводом, изграђеним од лучних вертикалних низова цеви које су у темену биле „закључане” централним елементом. Реконструкција изгледа цеви и начина њиховог ређања уклапа се у хронологију извођења објекта и свода током III века. Даљим статичким анализама дошло се до још неколико сазнања. Показало се да је преко свода морао бити нанесен одређен слој малтерне масе да би дебљина свода досегла оптималну вредност у опсегу 20–30 cm. На основу пропорција објекта које су одређене у његовој основи испитана је висина објекта, где је група случајева такође дефинисана пропорционално. Према нашим анализама, зидови просторија „објекта са хипокаустом” у којима су цеви регистроване могли су досезати висину до 3,08 m, док је висина просторија у темену свода могла бити 6,16 m.

Овим истраживањем покушали смо да укажемо на велики значај појединачних архитектонско-грађевинских елемената, а међу њима и керамичких цеви за сводове, којима се често не придаје довољна пажња. Налази керамичких цеви за сводове у Тимакум Минусу, уз извршене архитектонске анализе, употпуњују слику откривеног „објекта са хипокаустом” из више аспеката. Посебно је значајно дефинисање његове висине, које је веома тешко за античке грађевине профане архитектуре на нашем тлу будући да су најчешће сачуване у приземној или темељној зони. Значај налаза керамичких цеви за сводове у Тимакум Минусу велики је стога што је он омогућио како конкретно дефинисање контекста њиховог налаза тако и реконструкцију облика одређених делова грађевине помоћу тог елемента, што до сада није истраживано приликом анализа античке архитектуре на тлу југоисточне Европе.