

# Hidden riches in the Early Medieval Rhine Delta: Iron working at Merovingian Oegstgeest

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22 The Early Middle Ages were a time of major societal, political and economic upheaval in the Rhine Delta. Though on the outskirts of the Roman world, the collapse of the Western Roman Empire led to 23 significant changes in the region's ethnic and cultural make-up (Wood 1994; van Es and Verwers 24 25 2010). These changes, combined with the lack of written sources, have led to this period being one of 26 the most poorly understood in Dutch history. Archaeological study has shed light on the daily realities 27 of this time, but many questions persist. In particular, the role of "marginal communities" (Loveluck and 28 Tys 2006) remains unclear. Many studies have stressed the role played by secular and religious elites 29 in overseeing the economy of the Early Middle Ages (Pirenne 1937; Besteman et al. 1990) but the study of rural settlements has revealed considerable prosperity outside of such power structures 30 (Henning 2007). Merovingian Oegstgeest is an example of such a settlement. At first glance, the site 31 32 appears to be an isolated backwater but the finds paint a more nuanced picture.

An in depth overview of the site has already been undertaken by Bruin et al. (2021). Rather than re-iterate the results of this work, this paper approaches the settlement through the remains of a specific material: iron. Clear evidence of ironworking was found at Oegstgeest, including iron slag, metallic forge scrap and finished objects. The study of this material can provide considerable insight into the socio-economic role of the settlement and shine light on the realities of forging in the Early Medieval Rhine Delta.

39

#### Iron in the Early Medieval Netherlands

The material from Oegstgeest has already undergone initial macroscopic study (Disser 2019; Theuws et al. 2021b) which, in combination from the study of the wider site (Bruin et al. 2021) found no evidence for primary iron production at Oegstgeest. This suggests that the iron worked on site came from elsewhere, making an understanding of iron production and iron working in the Early Medieval Netherlands crucial to fully understand the settlement within its wider context.

1 The Netherlands is not known for its mineral resources but there is clear evidence of Early 2 Medieval iron production in the region (Joosten 2004). Many Dutch smelting sites relied on bog iron 3 ore, accumulations of iron rich minerals precipitated in sandy, silty or clayey sediments (Thelemann et 4 al. 2016) but there is also evidence for the use of other kinds of mineral deposits. This is especially 5 true in the Central and Eastern Netherlands with the most prominent Early Medieval example being the Veluwe (Gelderland). Wide spread iron smelting in this area (with more than 70 slag heaps 6 identified) became a phenomenon as early as the 7<sup>th</sup> century and this continued until the mid-9<sup>th</sup> 7 8 century (Joosten 2004). in the region, as well of open cast mining pits and deposits of rattle stone ore 9 in certain strata. Alongside these local sources, it is equally plausible that smiths in the Early Medieval 10 Netherlands were using iron from further afield. Bruin et al. (2021) found extensive evidence for 11 medium to long distance trade at Oegstgeest both along the Rhine and Meuse Rivers, and across the 12 North Sea, complementing similar evidence in other Dutch sites from this time (Loveluck and Tys 13 2006; Deckers and Tys 2012).

14 There has been very little study of smithing activities in the Early Medieval Netherlands. What 15 studies exist typically focus on the typology and microstructure of individual finds rather than wider 16 questions of production. Furthermore, centres of trade and power such as Dorestad and Nijmegen 17 typically take centre stage with rural contexts being ignored (c.f. Ellis 2002; Willemsen et al. 2021). 18 Worked iron has been identified and characterised at some smelting sites; such as Heeten in Overijssel (Godfrey and van Nie 2004) but these again focus on individual finds. Joosten (2004) also 19 documented some possible smithing slags from the Veluwe but these were not characterised further. It 20 is for this reason that the study of a rural site like Oegstgeest is of such interest, as it offers a window 21 22 into two understudied contexts.

#### 23

#### Merovingian Oegstgeest

The Merovingian settlement at Oegstgeest is in South Holland near the mouth of the Oude Rijn, 2 km west of Leiden and 3 km south of the modern town of Oegstgeest. The site underwent a series of excavations, led mostly by Leiden University, between 2004 and 2018. These campaigns uncovered much of the site, making it one of the best excavated Early Medieval settlements in the Western Netherlands (Kars et al. 2018).

29 Buildings at Oegstgeest were split into a series of yards of one to two houses each, distributed 30 across the settlement in five clusters (see Figure 1, clusters marked A-E). The waterlogged conditions 31 at Oegstgeest were ideal for wood preservation, allowing for widespread dendrochronological study 32 (Theuws et al. 2021a). The earliest attributed date is 555 CE, which matches the pottery recovered 33 from the settlement though some sherds suggest that the first occupation may have been slightly earlier. A lack of dates from the early 7<sup>th</sup> century was interpreted by Theuws et al. (2021a) as evidence 34 that Oegstgeest was briefly abandoned due to widespread flooding in the Rhine Delta at this time. The 35 settlement was re-occupied by 650 CE at the latest and the second half of the 7<sup>th</sup> century seems to 36 have been the heyday of the site, with the majority of datable finds and buildings being from this 37 38 period. The last reliable dendrochronological date is 700 CE (de Bruin 2021; Theuws et al. 2021a) 39 though the presence of sceattas dating to later periods suggest that the settlement may not have been 40 have been fully abandoned until c. 725-730 CE. The precise reason for the abandonment of the 41 settlement is unclear though Theuws et al. (2021a) theorise that it could be related to a migration of 42 the Rhine River around this time.

43 The landscape around Oegstgeest was regularly in flux and the settlement itself was built on a 44 series of gullies connecting to the main river. Flooding would have been a perennial risk, which 45 combined with the lack of trees in the area, encouraged heavy reuse of wood. This contributed to the 46 difficulties experienced by Theuws et al. (2021a) in establishing a precise end date for the settlement. 47 Between this reuse and the relatively short occupation (175 years at most), establishing clear phases of occupation was not possible. However, Theuws et al. (2021a) suggest that while there may have 48 been buildings in each cluster at any given time, the main centre of the settlement appears to have 49 50 shifted during the occupation. They believe that cluster B is the oldest section of the settlement, due to 51 its association with the earliest dendrochronological dates, with cluster D possibly being occupied last. 52 Due to this uncertainty, giving a population estimate for the settlement is difficult. Nevertheless,

1 Theuws et al. posit that there may have been 60 people living at Oegstgeest at any moment of its 2 existence.

3 Despite the settlement's proximity to the coast, Oegstgeest was primarily a riverine site. At the 4 time, the Oude Rijn was one of the largest distributaries of the Rhine Delta and this had a major effect 5 on the character of the settlement. Oegstgeest appears to have been a logical stopping point between 6 the North Sea and the lands upriver (Theuws et al. 2021a), a connection reinforced by the large 7 number of imports identified during excavation. These include simple finds, such as wooden bowls and 8 everyday utensils but also more complex objects. For example, 122 glass shards were recovered from 9 the settlement, the majority of which came from vessels or other containers (Van Wersch 2021). These were typologically consistent with vessels dating to the 6<sup>th</sup> and 7<sup>th</sup> centuries, suggesting that 10 glass was imported during the entire occupation of the site. Other finds included glass beads; some 11 12 produced locally, some from as far as the Mediterranean, worked amber from the Baltic, as well as 13 several exceptional finds, including a silver bowl from the Mediterranean. Theuws et al. (2021a) also 14 found evidence of imported foodstuffs. Several wooden barrels were found to have contained wine, 15 likely from the middle Rhine and further study confirmed the presence of grain, rye, grapes, figs and possibly walnuts, none of which are likely to have grown in the region. Evidence of cross North Sea 16 17 trade was also present, visible through 24 silver sceattas, mostly of the D and E series, a type found in 18 both England and the Netherlands. In addition, an English style horse bit was uncovered, though it is 19 unclear whether this was imported from England or made locally in English style.

There was also clear evidence of local production. Three soled leather shoes, a rarity at this period, were found at Oegstgeest alongside the materials and tools required to make them (Theuws et al. 2021a). Additional evidence for local production included metalworking; 3 gold ornaments and over 200 finds related to copper working were uncovered. Most of these were brooches though several crucible fragments were also found, one of which was attached to a copper alloy fragment. These metal finds were fragmented and seem to have been in the process of being recycled (Theuws et al. 2021b). Furthermore, there was extensive evidence for iron working at Oegstgeest.

As of yet, no forge or metal workshop has been identified at Oegstgeest but the evidence for iron working is indisputable. Over 43 kg of iron slag was recovered alongside 2.4 kg of metallic finds (see figure []). The findings of the preliminary study of this material is synthesised here (Disser 2019; Theuws et al. 2021b).

## 31 Slag finds

32 Disser (2019) established that that 17 kg of the slag from Oegstgeest, about 40% of the corpus, 33 was smithing slag, with the remaining slag being mostly amorphous (see figure []). Three guarters of the smithing slag recovered from Oegstgeest weighed less than 300 g though 39 larger hearth 34 35 bottoms were uncovered. In addition, a very small quantity of slag (303 g) was identified by Disser as 36 possible tap slag but this was determined to be too small to ascertain that smelting was taking place 37 on site. Of the hearth bottoms, 15 were plano-convex, 4 were biconvex, 4 were faceted, 3 were planar, 38 2 were concave and 1 was conical. The remaining hearth bottoms were irregular shapes. Average 39 slag length and width were 93 and 70 mm respectively and average thickness was 45mm.

40 Macroscopic petrographic study established that SGD (Scorie Grise Dense, dense grey slag) was 41 the most represented facies in the corpus, with 51% of the slags containing at least some SGD (see 42 Serneels 1993; Anderson et al. 2003; Serneels and Perret 2003; Eschenlohr et al. 2007 for details on 43 slag facies). However, 33% of the slags also contained SFR (Scorie Ferreuse Rouilée, rusty ferric 44 slag) with this facies being dominant in 21%. SAS (Scorie Argilo-Sableuse, clay and sand slag) was 45 present in 36% of the corpus but was only the dominant facies in 10 hearth bottoms (25% of the corpus) (see figure []). In addition, Disser identified a potential fourth facies, which he dubbed SCD 46 47 (Scorie Crystalline Dense, dense crystalline slag) in 13% of the corpus. This facies is superficially similar to SGD though the hearth bottoms had flat, well-preserved sides, which Disser associated with 48 the possible presence of macro-crystals. 49

50 Finally, Disser (2019) used this information to quantify smithing activities at Oegstgeest. As no 51 dedicated forge was identified, Disser was unable to establish the quantity of iron waste in the relevant contexts and instead based his reasoning on the slag and other scoriaceous waste found on site.
From this, he found a loss between 9.5 and 12 kg of iron during forging. With careful comparison with
similar sites in the literature (Mangin et al. 2000; Perret 2002) he gives a cautious estimate of 90 to
432 kg of iron objects produced over the lifetime of the site.

## Iron finds

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6 In total, 221 iron finds were found at Oegstgeest with nails making up 82% of the overall corpus 7 (see figure []). During excavation, these nails were split into two broad categories, "nails" and "boat 8 nails" as boat production appears to have taken place on site. This categorisation was established 9 based on the size of the nails, which ranged from 24 to 122 mm in length and from 2 to 9 mm in cross 10 section. Establishing typological categories for the iron finds was difficult as almost all of the metal was highly corroded. For this reason, Disser (2019) simply retained the original distinction between "nails" 11 12 and "boat nails". The remaining 15% of iron finds included scrap and other fragments related to 13 forging, as well as finished objects such as knife fragments, a fire striker and an axe head.

From these observations, Disser (2019) concluded that post smelting activities, specifically object creation and upkeep were the primary forms of iron working on site, though the small quantity of tap slag could potentially indicate some smelting in the wider area.

With this context in mind, this paper has two aims: to contribute to the existing data on Oegstgeest by providing the first in depth look into the iron working at this settlement, and to use the lens of iron working at Oegstgeest to contribute to the discussion surrounding the role of such sites within their wider socio-economic context.

To reach these goals, this paper sets the following objectives. Firstly, to establish the forge techniques used at Oegstgeest. Secondly, to establish the provenance of the iron used at Oegstgeest and finally, to consider the implications of these results on the nature of iron working at Oegstgeest. This will consider these can reveal about the role of the settlement as a place of iron production in the Early Medieval Netherlands and also what they imply for the role of the settlement within its wider context.

26 Of the remains of metalworking at Oegstgeest, 36 finds were selected for this study (see Table [] 27 and figure []). Among the slag, 15 hearth bottoms were chosen, including at least one instance of each 28 of the four facies identified by Disser (2019). Additional hearth bottoms were chosen to reflect the distribution of facies at Oegstgeest with complete hearth bottoms being privileged. This was 29 complemented by three pieces of amorphous slag, one of which was found in the same context as one 30 31 of the hearth bottoms. The remaining two were selected in the hopes of characterising an unidentified surface deposit. Due to the generally poor condition of the metal finds from Oegstgeest, iron samples 32 were selected on two criteria: that they had not already been selected for museum display and their 33 likelihood of containing metallic iron. No X-ray imagery was performed and this was instead 34 35 established by magnet response and apparent density. Thirteen iron finds were chosen for the final 36 corpus: 8 nails, including 2 boat nails, 2 pieces of forge scrap to characterise the smithing activities, 2 complex objects in the hopes that these might reveal some more advanced forge processes that may 37 38 have taken place on site and 1 unidentified sample.

39 Sample preparation was based on similar studies (Pagès 2011; Disser et al. 2016; Berranger et al. 40 2017; Disser et al. 2020). The slag samples were cut using a Struers Unitom-2 circular saw. The iron 41 samples were cut using a Struers Accutom-50 saw along their length to provide an overview of the 42 piece. Two larger samples (OEG 164 and 221, boat nails) had approximatively 1.5 cm cut from the 43 end, which was then cut in half. OEG 968 sub 1 (fire striker) had two triangular sections removed at 44 different heights to examine the full width of the piece while preserving its structural integrity. Once cut, 45 the larger samples were embedded in Struers EpoFix epoxy resin and smaller samples (>2cm in length) were embedded in transparent acrylic under pressure for five minutes using a Metkon 46 Ecopress 52. The resulting samples were polished using abrasive disks and diamond solution (grade 47 48 P80 paper to 1µm diamond solution) to obtain a mirror finish.

49

Optical microscopy

1 Metal samples were etched first using Nital (4 vol% HNO<sub>3</sub> in ethanol) then polished and eteched 2 again using Oberhoffer's reagent. In both cases, the surface was studied by reflected light microscopy 3 using a Zeiss Axio Imager A2 Vario. Low magnification micrographs were taken across the surface of 4 the sample using a Zeiss Axiocam camera. These were assembled to create a mosaic image of the 5 surface microstructure using Zen Core v. 3.3. Areas typical of the microstructure of the sample, as well 6 as any areas of metallurgical interest were photographed. The proportion of pearlite was used to 7 establish the approximate carbon content of each sample (c.f. Pagès 2011; Gosselin 2021). Slag samples were analysed and photographed using the same setup to establish mineral compounds 8 9 present within the slags.

#### 10 SEM-EDX

11 A 20 nm carbon coat was applied to the samples using an Agar Auto Carbon Coater, which were 12 then subjected to elemental analysis by energy dispersive X-ray spectrometry coupled with scanning 13 electron microscopy. This was accomplished using a JEOL SEM-FEG 7001 equipped with a silicon-14 drift detector. Accelerating voltage was set at 15 kV, working distance at 10 mm and probe current 15 around 8 nA. This allows for reliable quantification of elements over 0.3-0.5 wt%, depending on the 16 element in question. An automated filter was applied to each spectrum to remove the background 17 contribution. Quantification of chemical elements was processed using XPP matrix correction routine 18 (with a Phi-Rho-Z function). O, Na, Mg, Al, Si, P, S, Cl, K, Ca, Ti, V, Cr, Mn, Fe and Cu were 19 systematically quantified, and results were normalised to 100%. The iron samples were studied using 20 the same protocol as Disser et al. (2020): slag inclusions in the iron matrix were systematically 21 analysed by backscattered-electron imagery using x300 magnification. This process generated a 22 spreadsheet detailing spatial (location, size, and shape) and chemical data for each identified particle. 23 This was used to compliment the metallurgical study by as it allows the differentiation of forge and slag 24 inclusions, as well as identifying areas made from separate blooms (c.f. Disser et al. 2020).

25 Slag samples containing a microstructure seen in all or most of the respective slag facies (SGD, SAS, SFR and SCD) were also analysed by SEM-EDX (see Table []). Additional SEM-EDX analysis 26 27 was performed on slag samples containing phases that were unable to be identified by optical 28 microscopy. A 2 cm<sup>2</sup> sample of the area of interest was identified using optical microscopy and 29 sampled using a Brot geological saw. These were then re-embedded in Struers EpoFix resin, carbon 30 coated and analysed using the pre-established parameters combined with the cartography tool in Aztec. The results of these findings are summarised in table [] and table [] for iron and slag 31 32 respectively. Full results are available in the supplementary material.

## 33 LA-ICP-MS

34 Establishing possible origins for the iron used at Oegstgeest was based on trace element content in slag inclusions. This was quantified by Laser Ablation Inductively Coupled Plasma Mass 35 36 Spectrometry at the IRAMAT Centre Ernest Babelon in Orléans. Set up and methodology was 37 modelled on that used by Bérard et al. (2020), itself based on the protocol detailed in L'Héritier et al. 38 (2016). Measurements were performed using a sector field Thermo Scientific ICP-MS Element XR 39 coupled with an ArF\* excimer laser ( $\lambda$ =193 nm, RESOlution S155-E, Resonetics). Beam diameter was 40 set at 80 µm with a pulse duration of 4-5 ns and a repetition rate of 7 Hz. Energy rate was 34 µJ per pulse. The ablated particles were carried to the plasma torch using an Ar/He carrier gas (1.01 L·min<sup>-1</sup>) 41 which was used to form the plasma. Cooling gas flow rate was 16 L min<sup>-1</sup> with an auxiliary gas flow 42 rate of 1 L·min<sup>-1</sup>. Measured isotopes were: <sup>9</sup>Be, <sup>28</sup>Si, <sup>47</sup>Ti, <sup>51</sup>V, <sup>60</sup>Ni, <sup>63</sup>Cu, <sup>69</sup>Ga, <sup>72</sup>Ge, <sup>75</sup>As, <sup>85</sup>Rb, <sup>88</sup>Sr, <sup>89</sup>Y, <sup>90</sup>Zr, <sup>93</sup>Nb, <sup>118</sup>Sn, <sup>133</sup>Cs, <sup>178</sup>Ba, <sup>139</sup>La, <sup>140</sup>Ce, <sup>141</sup>Pr, <sup>146</sup>Nd, <sup>147</sup>Sm, <sup>153</sup>Eu, <sup>157</sup>Gd, <sup>159</sup>Tb, <sup>161</sup>Dy, <sup>165</sup>Ho, <sup>167</sup>Er, <sup>169</sup>Tm, <sup>172</sup>Yb, <sup>175</sup>Lu, <sup>178</sup>Hf, <sup>181</sup>Ta, <sup>182</sup>W, <sup>208</sup>Pb, <sup>232</sup>Th and <sup>238</sup>U. Time per sample was 10 ms with 43 44 45 46 100 samples per peak. Due to the varying size of slag inclusions, the quantity of ablated material was 47 a function of inclusion size so the net-recorded signal for each trace element was normalised using <sup>28</sup>Si as an internal standard. This was measured using Faraday detection mode with a segment 48 duration of 100 ms. All other isotopes were measured using triple detection mode with a segment 49 50 duration of 40 ms.

51 The samples were placed in ablation cells alongside a NIST610 standard reference glass for 52 calibration. Bérard et al. (2020) found this to be the closest approximation to slag inclusions. At the

1 start of each session, a blank signal was obtained for 20 s to measure the background noise. The 2 NIST610 was then measured 3 times followed by 18 measurements of the archaeological samples, 3 after which this process was repeated. Once all samples were analysed, a final blank and 3 4 measurements were performed on the NIST610. In total, 12 slag inclusions per sample were selected 5 for analysis. The goal was to choose inclusions representative of the sample as a whole so the choice 6 of slag inclusions was informed by the results of SEM-EDX analysis: for samples made from multiple 7 blooms, an equal number of inclusions were chosen on both sides of the weld line. Large slag 8 inclusions were preferred to limit accidental ablations of the metal matrix.

## 9 Raman spectrometry

10 The study of OEG 180A revealed inclusions that were unable to be fully characterised by optical microscopy or SEM-EDX analysis. These were further studied using Raman spectrometry to establish 11 12 the precise mineral compound. This was accomplished using a Renishaw Invia Reflex spectrometer 13 equipped with a doubled Nd-YAG laser  $\lambda$ =532 nm) and a Leica DMLM microscope using a x50 lens to focus the beam on the sample surface. A silicon reference wafer was used for calibration with the 14 reference peak at 520 cm<sup>-1</sup>. Spectral resolution was on the order 2 cm<sup>-1</sup> and power was limited to 15 under 100 mW to avoid heating the surface and affecting sample composition. Beam area was set to 16 17 around 1 µm<sup>2</sup>. Spectra were obtained from five 30 s acquisitions over the inclusion and five 30 s 18 acquisitions over the metallic matrix. The signal from the metallic matrix was then subtracted from the 19 inclusion signal. Spectrum acquisition was performed in Wire 3.4 and the baseline was established 20 using the included Cosmic Ray Remover software. Data processing was undertaken in Origin Pro 21 2016. The obtained spectra were compared with published reference spectra (Weber et al. 2015).

Due to the large quantity of data produced by SEM-EDX and LA-ICP-MS, data processing was performed using programs written by Alexandre Disser (Disser et al. 2016; 2020). Because of the effect that differences of magnitude can have on descriptive multivariate analyses when applied in Euclidian space, the data underwent an initial transformation to limit this impact. The log ratio has been found to be the most effective method of data transformation for slag inclusions in archaeological iron; specifically a transformation to  $X_{ij}$  values (Charlton et al. 2012; Leroy et al. 2012). The transformation is calculated using the following formula:

$$X_{ij} = \log_{10}([E]) - \frac{1}{N} \sum_{k=1}^{N} \log_{10}([E_k])$$

Where [*E*] is the measured quantity of each element in a given inclusion and  $[E_k]$  is the quantity of each of the selected elements.

31 For major element data, values with a standard deviation over 10% the measured value were 32 removed though the small number of inclusions in OEG 968 sub 2, 2540, 2546 and 5355 required this 33 limit to be raised to 50% for these samples. The remaining entries were converted to oxide values 34 (assuming iron was present under valance II) and converted to  $X_{ij}$  values. The resulting data was run through a Hierarchical Clustering Analysis (HCA) to sort entries into classes based on chemical 35 36 similarity (see Husson et al. 2010). A threshold of chemical similarity was decided by the user upon 37 study of the resulting dendrogram, this was unique to each sample and chosen to best reflect the data. 38 Samples with a common branch below this threshold were placed in the same group while samples 39 with a common branch above the threshold were placed in a separate group. The results were plotted 40 over the mosaic of the sample surface, which allowed easy identification of zones within the sample 41 with differing inclusion chemistry. The resulting data was run through a Principal Component Analysis 42 (PCA) to convert the results into Cartesian coordinates. The full results are available in the 43 supplementary material.

#### 44

#### Provenance attribution

Provenance was established by direct comparison of trace element content of slag inclusions with smelting slag and iron ore of known origin. This method requires consistent concentrations of elements between ore, slag and slag inclusions. As such, not all elements detected by LA-ICP-MS are usable. Some elements (known as *siderophiles*) preferentially bond with the iron during the smelt and are therefore overrepresented in the metallic iron. LA-ICP-MS analysis often accidentally ablates a small portion of the iron matrix meaning that any provenance attribution relying on *siderophile* elements will be skewed by this overrepresentation. In addition, some elements can show excessive variance even after conversion to  $X_{ij}$  values. The choice of elements for this study was based on the findings of Leroy (2010) and Disser (2014): Y, La, Ce, Yb, Nd, Pr, Gd, Sm, Dy, Eu, Th and U.

5 This study follows the example of Disser et al. (2016) and uses an unsupervised method of data 6 analysis. A supervised method would require manual categorisation of the data prior to analysis. This 7 approach works best when all finds are known to be of a single origin, but it can bias interpretation. 8 For example, a preliminary categorisation of ore based on geographic origin could obscure differences 9 between chemically distinct but geographically close ores and vice versa. Unsupervised methods of 10 data analysis allow a categorisation based directly on chemical data: samples are ranked by chemical 11 similarity and the obtained data is then reviewed manually to establish groups that best reflect the 12 samples. The method of analysis chosen for this study was Hierarchical Clustering of Principal 13 Components (HCPC, c.f. Husson et al., 2010). Once converted to X<sub>ij</sub> values, a preliminary PCA was 14 performed to "de-noise" the dataset. The results were run through an HCA, which was the primary 15 method of establishing provenance. Provenance attribution was the same as group attribution for the major elements: if two analyses were attributed to a common branch of the resulting dendrogram 16 17 below the user-defined threshold, they were assumed to have a common origin. Finally, the results 18 underwent a second PCA to represent the data in Cartesian coordinates.

19 Provenance was established in two stages. No method of analysis can account for the entirety of 20 chemical variation of slag produced from a single source of ore. As such, a direct comparison of 21 inclusions from Oegstgeest with the ore and slag could result in confusion due to several inclusions from a single sample being attributed to distinct sources. For this reason, an initial "intra-site" 22 23 comparison was undertaken between the Oegstgeest samples without including the ore and slag to 24 establish whether any samples had a common origin. As the "height" values provided by the HCA are 25 indicative, ensuring a meaningful contrast required including a sample known to be of a different origin 26 (in this case a shaped offcut from Messein in Lorraine c.f. Disser 2014). The second "inter-site" stage 27 involved individual comparisons between each sample and the full corpus of analyses of ore and slag 28 of known origin. However, initial runs rarely matched samples with single sources so any potential 29 match underwent the same process a second time including only the entries grouped in the first run. 30 The results of this comparison were synthesised Table [] and are presented in full in the 31 supplementary material.

32

## Selected ore and slag for comparison

Analyses of ore and slag were chosen on the likelihood of a connection with Oegstgeest (see 33 34 Figure []). As detailed previously, the Netherlands, in particularly the Veluwe, are a likely candidate for 35 the iron used at Oegstgeest. However, while ore and slag from the Veluwe has been extensively 36 studied (Joosten 2004), the available data focuses on siderophile elements. Due to the 37 aforementioned risk of over-representation of these elements due to accidental ablation of the iron 38 matrix, this makes the available data for the Veluwe incompatible with the methods of this study. As 39 such, the Veluwe was not included but was borne in mind as a possible origin for otherwise 40 unattributed samples.

The samples that were included came from regions in the broader Rhine and Meuse river systems. In particular, this included ore from the Condroz in southern Belgium, characterised by Pagès et al. (2022). Pagès et al. analysed 39 smelting slag from this region (detailed in Table []), differentiating between slag from the Eastern Condroz and Entre-Sambre-et-Meuse in the Western Condroz. This is because the latter area extends into areas that contained no iron ore but did contain some smelting sites.

Additional samples came from Lorraine in Eastern France, based on ore and slag characterised by Disser et al (2016). Iron production in Lorraine relied on a wide range of metallogenic contexts, different kinds of sedimentary ore, as well as siderolithic and vein ores. Geological and archaeological context of the included provenance spaces are described in Table []. Two provenance spaces described by Disser et al. (2016) were not included. The Bajocian-Bathonian ore corresponded to material from only two sites and the ore and slag from St-Dizier was characterised by only one site. 1 These were determined to be too small to be sufficiently representative and were not included to avoid 2 skewing the results of the provenance study.

3 The final samples included in this study come from across Southern Germany characterised by 4 Dillmann et al. (2017) (detailed in Table []). This region was included in part due to its proximity to the 5 Rhine River, but also because of the connection between the Rhine and Danube rivers in the Early 6 Middle Ages. At this period, these rivers formed a major axis of transportation between Eastern and 7 Western Europe (van Es and Verwers 2010). Given the aforementioned contact established between 8 Oegstgeest and the Mediterranean by Theuws et al. (2021a), it is likely that the objects traded 9 between these regions took this route. It is plausible that iron was traded along the same route, 10 making southern Germany, the land between the Rhine and Danube rivers, an interesting candidate 11 provenance space to explore.

12 The mineral facies identified by Optical Microscopy in the slags from Oegstgeest match similar 13 examples described in the literature. SGD slag was comprised of fayalite with widespread presence of wüstite dendrites. SFR was made of fayalite and globular wüstite as well as large quantities of other 14 iron corrosion products that were unable to be identified. SAS showed a mostly vitrified microstructure 15 16 with occasional iron prills caught in the glassy matrix. The SCD microstructure identified by Disser 17 (2019) was indistinguishable from SGD as it was also comprised of fayalite with wüstite dendrites, 18 though these hearth bottoms did appear to contain more spinels. Subsequent SEM-EDX analysis 19 found that these were all iron oxides at concentrations consistent with magnetite. There was one 20 exception to this trend as OEG 283B presented a different microstructure. Macroscopically, the slag 21 was a complete circular hearth bottom with a thin cross section (~2 cm). Rather than the ubiquitous 22 fayalite and wüstite observed in other slags, its microstructure was dominated by spinels and needle 23 like crystals. SEM-EDX analysis found many of these to be comprised of Oxygen, Iron and Aluminium, 24 suggesting that these are hercynite. However, other minerals identified in the same slag either did not 25 contain iron, or contained higher concentrations of Magnesium and Calcium, suggesting the presence 26 of a wide range of minerals in this slag.

27 Several slag samples contained clear strata between different slag facies and this was especially noticeable in OEG 5047 and 5052. These slags were not hearth bottoms and were included for 28 29 analysis in the hopes of identifying an unusual blue surface deposit. Their microstructure was almost 30 identical with both comprised of a lower SGD stratum and an upper SAS stratum, which contained the blue colouration. Furthermore, optical microscopy identified a beige surface deposit adhering to the 31 SGD strata in both slags. SEM-EDX analysis found this deposit to be an iron sulphide, seemingly the 32 33 same in both samples, possibly pyrite (FeS<sub>2</sub>, see table []). The lower SGD strata contained fayalite 34 and wüstite dendrites though the latter presented unusually angular edges, not noted in any of the 35 other slags. This could indicate that the wüstite is euhedral. Despite the blue colouration, the SAS 36 strata optical microscopy was unable to distinguish the SAS stratum with other such examples seen in 37 the slags from Oegstgeest, though SEM-EDX did find higher concentrations of calcium.

38 The last slag of note was OEG 603, a hearth bottom initially identified as SAS due to the sediment 39 adhering to its surface. Observation of the cross section and optical microscopy found it to be 40 consistent with SGD ("rusty" appearance, presence of fayalite and globular wüstite alongside iron 41 corrosion product). SEM-EDX analysis also found the glassy matrix surrounding these minerals to 42 contain unusually high concentrations of Mn (up to 11.28 wt%, see table []). In addition, the hearth 43 bottom contained several green prills (~1 mm diameter, see figure []). SEM-EDX analysis found these to be copper oxides. One of these prills was found encircling another and both presented an 44 45 enrichment on their surfaces with elements common in iron slag (Fe, Si and Al). Further analysis by 46 SEM-EDX found smaller metallic copper prills (~1 µm diameter) in the sediment adhering to the 47 surface of the slag.

48 3.2.

49 Nails

50 No clear distinction was found between the nails and boat nails, either by optical microscopy or 51 major element HCP. Both boat nails had relatively low carbon content with OEG 164 being ferrite with

1 some patches of hypo-eutectoid steel and OEG 221 being entirely ferrite. No welds were identified in 2 either sample. These same features were observed in almost all the other nails and are consistent 3 with bloomery steel samples described in the literature. The only exception was OEG 2611, which was 4 a mixture of ferritic iron and hypo-eutectoid steel but contained a weld identified by major element 5 HCP. Inclusions on either side of the weld line were attributed to separate groups, suggesting the use of multiple blooms. Etching with Oberhoffer's reagent revealed ghost structures in all but one of the 6 7 nails (OEG 2540) indicating the use of phosphoritic iron. The extent of these structures varied 8 considerably from sample to sample, from a minimal presence in OEG 180B to extensive structures 9 across the entire sample in OEG 2546.

Two nails (OEG 180B and 2540) presented an unusually high copper content in several of the slag inclusions. SEM-EDX analysis did find high copper in both the slag and iron samples but in most cases, these could be discounted as either instrument error (as indicated by the high variance of the respective analyses) or accidental measurement of the copper tape used to complete the circuit within the electron microscope. However, the presence of copper in these samples was confirmed by LA-ICP-MS.

## 16 Forge scrap

17 The two pieces of forge scrap presented very different microstructures; OEG 583 (offcut) was 18 entirely ferrite with equiaxed grain (~200 µm diameter). The piece showed a clear uptick on one end, characteristic of an offcut (Anderson et al. 2003). In contrast, OEG 5355 (bar) presented a mixed 19 20 microstructure: two bands of ferritic iron surrounding a central band of steel. The low proportion of 21 inclusions in this sample (only 2.3% of the sample surface, most of which were corrosion product) made meaningful attribution of inclusions to chemical groups by HCP difficult. The forge inclusions 22 23 along the weld were attributed to a separate group but there were too few inclusions in the rest of the 24 sample to effectively interpret the data. The presence of a weld in this sample is evident from optical 25 microscopy though it is uncertain whether the iron was folded around a steel core or if two separate 26 pieces of iron were used. Oberhoffer etch found ghost structures in the upper ferrite band of OEG 27 5355, making it the only non-nail to present such structures. This same section showed a mixture of fine grain ferrite with bands of hypo-eutectoid steel. The lower ferrite section was entirely large grain 28 29 with no carbon structures visible from nital etching. The central band was eutectoid steel with a slight 30 decrease in carbon content along the centre (see figure []).

## 31 Composite objects

32 OEG 968 sub 2 (possible knife fragment) contained a very small number of slag inclusions, 33 covering only 0.5% of the sample surface. However, the most prominent of these inclusions formed a 34 line along the centre of the sample. This same zone corresponded to a faded line in the Oberhoffer 35 etch, suggesting a weld. This was confirmed by the major element HCP as the inclusions in this zone 36 were attributed to a distinct class (see figure []). The entire sample was eutectoid steel, verging on 37 hyper-eutectoid and presented a highly homogeneous microstructure.

38 OEG 968 sub 1 (fire striker) had the highest carbon content of any sample as the entire piece was 39 either eutectoid or hyper-eutectoid steel. The spine and the working edge were separated by a long series of inclusions that appear to run the length of the sample. These were attributed to a distinct 40 class by the major element HCP due to differences in Mn and Na content (see figure []). However, 41 42 these inclusions do not appear to be a weld. Their large size, lack of wüstite and elongated rather than globular shape all indicate that these are smelting inclusions. The entire working edge of the sample 43 44 presented martensitic structures, indicating that the piece had been quenched. However, a section of 45 the working edge presented cementite grain boundaries. (see figure [])

## 46 Unidentified sample

47 OEG 180A was neither a finished object nor forge scrap; it was entirely ferrite though without a 48 clear grain structure. Instead, the ferrite had a globular appearance and disaggregated into the 49 extensive inclusions (29.8% of surface) as well as into patches of the slag gangue that adhered to the 50 surface. This sample contained very few silicon rich slag inclusions, as seen in the other samples, and 51 instead contained spherical beige inclusions that were unable to be identified by OM. SEM-EDX analysis found that these were iron sulphides (67 wt% Fe and 33 wt% S). Additional characterisation was undertaken by Raman spectrometry. The resulting spectrum was unclear and lacked the characteristic peaks of iron sulphides between 250 and 300 cm<sup>-1</sup> though these results are consistent with Pyrrhotite (Fe<sub>7</sub>S<sub>8</sub>) (see figure []). However, the lack of resolution in this spectrum, combined with the measured Fe and S content being different than the predicted values for pyrrhotite (60.4 wt% Fe and 39.6 wt% S) indicate that multiple iron sulphides are likely present.

7

## Intra-site comparison

8 Initial runs of the trace element HCPC found that the chemical variation of inclusions from 9 OEG 180A was very large and it was decided to remove this sample from the intra-site comparison to 10 avoid skewing the overall results. Intra-site comparison established 7 groups though the high chemical similarity of inclusions within individual samples could indicate that each sample should be attributed 11 12 to individual groups (see figure []). The only sample to be attributed to two separate groups was OEG 13 968 sub 2, which is unsurprising in light of the weld identified by major element HCP in this sample. A quarter of the inclusions from this sample matched with OEG 2611 and 5355, meaning that the 14 resulting group contained most entries from samples known to contain forge inclusions. Subsequent 15 16 visual confirmation by optical microscopy found that the respective ablations were overwhelmingly performed on forge inclusions, meaning that these are unfortunately unexploitable for the provenance 17 18 study.

## 19 Inter-site comparison

20 Individual comparison of finds against the full range of candidate provenance spaces found very 21 few clear groupings of slag inclusions from Oegstgeest with these sources. Only three samples were 22 placed in a common group with entries other than those from the same sample. Two samples, OEG 23 221 and 435, which were grouped together by the intra-site comparison, matched with samples from 24 Bayerische Schwaben. However, the second round of HCPC attributed both of these sets of inclusions to groups containing only entries from the respective samples. The only firm attribution, in which the 25 inclusions from the sample were indistinguishable with the entries of ore and slag, was OEG 968 sub 1 26 27 (fire striker). Initial HCPC comparison against the full range of candidate provenance spaces grouped 28 the sample with entries from Swabia, Barrois, Minette and Fer Fort. A second HCPC was undertaken 29 with these entries which grouped OEG 968 sub 1 with entries from Fer Fort. Subsequent HCPC was 30 unable to distinguish between these entries, suggesting a common origin.

Given the predominance of SAS and SFR forge slag, combined with the presence of forge scraps and finished objects, the results of this study complement Disser's (2019) conclusion that object creation and upkeep of objects was the primary form of iron working at Oegstgeest.

34 The metallurgical study revealed relatively advanced techniques such as welding and guenching 35 without any of indications of poor execution (peripheral oxidation, trapped corrosion product or splitting along the weld line). Furthermore, OEG 5355 is a direct weld between iron and steel; these metals 36 37 weld at different temperatures and maintaining both pieces at the correct heat while undertaking the 38 weld would have required considerable experience (Fluzin 2002). This could indicate that the smith (or 39 smiths) at Oegstgeest were sufficiently experienced to undertake these operations though it is difficult 40 to confirm whether this would have taken place at the settlement. It possible that finds such as OEG 41 968 sub 1 (fire striker), could have been imported to the site as finished objects. This possibility seems 42 all the more plausible given the extensive pre-existing evidence for trade and importation of finished 43 goods at Oegstgeest. In contrast, it seems less likely that pieces of forge scrap, like OEG 5355 were 44 imported as such finds are typically associated with iron working in their immediate vicinity. 45 Furthermore, the presence of SAS slags, such as OEG 2001 sub 1 do indicate that such high temperature activities did take place on site. 46

The metallurgical study revealed another interesting aspect of the iron at Oegstgeest, namely the use of homogeneous steel, often with high carbon content. The steel used in OEG 968 sub 1 (fire striker), 968 sub 2 (possible knife fragment) and 5355 (iron bar) was eutectoid to hyper-eutectoid. Examples of such high carbon steel in the ancient world do exist (Godfrey and van Nie 2004) but these tend to be relatively rare, or attributed to specific kinds of objects. For instance, the presence of homogeneous high carbon steel in OEG 968 sub 1 (fire striker) was predictable as similar microstructures have been previously observed (Terekhova and Zavyalov 2019). The use of such homogeneous steel in OEG 968 sub 2 and 5355, as well as in lower carbon pieces such as OEG 968 sub 3 and 2540 was not expected and its use could indicate that metal of this quality was sufficiently available at Oegstgeest to be used in objects as common as nails. In any case, these results suggest that these materials, or the finished objects made from such materials, were available to the inhabitants of Oegstgeest.

#### Copper content

9 The copper prills observed in the hearth bottom OEG 603 are broadly similar with examples noted in the literature (Eliyahu-Behar et al. 2012; Vodyasov et al. 2021). However, the described examples 10 were metallic copper rather than copper oxides and were an entire order of magnitude smaller (10-100 11 12 µm compared to ~1 mm diameter prills observed in OEG 603). Whether these prills were already 13 corroded upon entry to the slag or whether they oxidised during sample preparation is difficult to establish. However, given that one of these prills is surrounded by a second, and that they both 14 present the same surface enrichment, it seems likely that these formed sequentially as liquid copper. 15 The smaller metallic copper prills found at the surface of the slag are more consistent with those 16 described in the literature. It is possible that this surface sediment is formed in part by the remains of 17 18 the hearth lining, in which case it would suggest that copper was sufficiently present in the hearth for this sediment to find itself attached to the resulting slag. 19

20 There are two plausible sources for the origin of this copper. The first is brazing, in which deliberate 21 use of copper to attach two pieces of iron could have resulted in a portion dripping into the hearth. However, given the clear evidence for copper recycling at Oegstgeest (Theuws et al. 2021b), the 22 23 possibility of tandem iron and copper working in the same hearth seems more likely. It is possible that 24 a spill would have led to the presence of metallic copper in the heath which would have then been 25 incorporated into the forge slag. One of the main questions surrounding polymetallurgy at Oegstgeest 26 was whether iron and copper were being worked during the same phase of occupation. If the 27 presence of these prills is indeed an indication that iron and copper were worked in tandem, this not only confirms that the metals were worked during the same phase of occupation, but potentially even 28 29 the same day. Furthermore, it implies that Oegstgeest had sufficient resources to either maintain two 30 artisans working in parallel, or that at least one smith had experience with both metals.

It is tempting to try and link the copper observed in OEG 180B and 2540 (nails) with this 31 32 polymetallurgy and attempts at brazing iron using copper as a filler could theoretically result in copper 33 being present in some of the forge inclusions. Confirming or refuting this hypothesis would require 34 further study. In the event that brazing was not taking place, it is highly unlikely that this copper is of 35 the same origin as that noted in OEG 603 as any copper in the smelting inclusions could only have entered the metal during the smelting process and are unlikely to have contributed to the copper in 36 this sample. While a small portion of such slag inclusions could have entered the hearth due to the 37 forging of samples like OEG 180B and 2540, this would be a negligible contribution to slag 38 39 composition when compared with the forge scale, hearth lining and fuel. As such, the copper in these 40 samples is likely of a different origin.

41 42

8

## Sulphur content

43 Despite the differing stoechiometric ratios of iron and sulphur between these samples, the 44 presence of the iron sulphide deposit in OEG 5047 and 5052 could be explained by the forging of 45 samples which contained large numbers of iron sulphide inclusions, such as OEG 180A (Craddock and Lang 2005). However, as no other such samples were found at Oegstgeest, this remains 46 speculative. Nevertheless, OEG 180 A remains interesting as the sample most resembles a "gromp", a 47 48 small fragment of iron bloom. This could be taken, alongside the possible smelting slag found at Oegstgeest, as evidence of some smelting activities in the areas. However, the distinct lack of 49 smelting structures preclude the possibility of smelting within the settlement and both of these finds 50

are [outliers], meaning that this interpretation can again, only be speculative. Nevertheless, its
 presence at Oegstgeest does raise the question of the origin of these sulphide inclusions.

3 High sulphur concentration in iron is typically provided by the ore (Craddock and Lang 2005) but 4 some sulphur rich fuel sources can produce iron sulphides, specifically coal. Within the Netherlands, 5 coal use is documented during the Roman period with evidence of smithing slags containing coal in 6 the settlements of Vechten and Kesteren (Joosten 2001, 2004, Hessing et al. 1977). Coal use 7 decreased in the Early Middle Ages, a trend noted across Western Europe at this time (Jagou 2020) 8 but it is possible that small-scale coal use continued into the Merovingian period. However, coal use is 9 typically limited to forge activities and the shape and distribution of these inclusions indicate that they 10 originated during the smelt. Furthemore, none of the forge slags from Oegstgeest match the typical facies of coal forge slags, i.e. no coal or jarosite inclusions and relatively dense morphology (Jagou 11 12 2019). This does not completely rule out the possibility of sulphur contamination from the fuel as Stepanov et al. (2022) found that sulphur contamination of wood charcoal can lead to the formation of 13 14 pyrrhotite inclusions in the iron bloom.

15 This leaves the ore as a possible source of sulphur, which raises several questions. Stepanov et 16 al. (2022) also found that despite a beneficiation regiment (roasting, hydro panning and sorting), it is possible for sulphur rich ores to retain a portion a significant portion of the sulphur during the smelt. It 17 18 is also possible that the smelters were using especially sulphur rich ores in the first place. Turniati et al. (2005) studied a collection of 9<sup>th</sup> century slags produced using Servette-Chuc ore, a Pyrite (FeS<sub>2</sub>) 19 rich ore with occasional "flames" of chalcopyrite (CuFeS<sub>2</sub>) from the Val d'Aosta in Northern Italy. 20 21 Rather than spreading evenly throughout the bloom, sulphur was found as troilite (FeS) inclusions 22 caught within the iron matrix. This raises some possibilities as to the origin of this ore: most Dutch ores 23 are found in sedimentary contexts (Joosten 2004) which can result in some pyrite deposits though the 24 use of chalcopyrite could also potentially explain some of the copper observed in the slag inclusions of 25 OEG 180B and 2540 (nails). This copper would require a volcanic origin, placing the closest possible source of ore in the Eifel. This would be a considerable distance to transport metal unfinished 26 27 materials, and also raises the question of why the settlement would have bothered importing such 28 poor iron when the even carbon distribution noted in many of the samples suggests that quality metals 29 were available, but this hypothesis remains consistent with the results of this study.

30

The provenance study was largely inconclusive. Despite the firm link established between OEG 968 sub 1 (fire striker) and Lorraine (Fer Fort), none of the other samples were able to be definitively associated with any source of iron. It is likely that the iron used at Oegsteest came from sources not considered by this study. As previously mentioned, the wider Netherlands, specifically the Veluwve, are an extremely likely candidate for any worked at Oegstgeest and should compatible characterisation of this ore be published, it is probable that at least one of the objects from Oegstgeest will match this candidate provenance space.

It is possible that different Dutch sources, such as the Veluwe and bog iron ore, could account for the full range of chemical variation seen at Oegstgeest but the clear evidence of wider contacts seen at Oegstgeest through the study of other materials (Kars et al. 2018; Theuws et al. 2021a) indicates the possible use of iron of other sources. Two regions stand out in particular:

The presence of English artefacts at Oegstgeest suggest contact with the British Isles. Cross North-sea trade is also well documented at this period (Loveluck and Tys 2006) and it is entirely possible that iron was traded alongside other goods. As of yet, English ores have not been as extensively studied as their continental counterparts and if and when compatible data becomes available, it would be interesting to compare it with the finds from Oegstgeest.

Another conceivable source of iron is the Danube. Data from this region was not included in this study due to its geographic distance from Oegstgeest. However, in the Early Middle Ages the Rhine and the Danube formed the main axis of communication linking Eastern and Western Europe (van Es and Verwers 2010). Contact between Oegstgeest and Eastern Europe seems likely given its location at the mouth of the Rhine River and the presence of Mediterranean artefacts within the settlement. To reach the Rhine (and by extension Oegstgeest), these artefacts would have passed through the land between the Upper Rhine and the Upper Danube, which is Swabia. While OEG 221 and 435 were unable to be formally associated with Swabian ore and slag, it is possible that Danubian iron (either as finished objects or semi-products) could have been traded to Oegstgeest.

5

6 The study of both the slag and metal samples supports the conclusions of Disser (2019): the 7 creation and upkeep of finished objects appears to have been the primary form of iron working at 8 Oegstgeest The presence of homogeneous steel at Oegstgeest, as well as complex objects like fire 9 strikers, suggests that quality goods and materials were available to the inhabitants of the settlement. 10 This is backed by the other imported goods found at Oegstgeest (Theuws et al. 2021a). Furthermore, the use of advanced techniques like direct welding of iron and steel, as well as likely tandem iron and 11 12 copper working, suggests that at least one of the smiths had the training required to produce complex 13 objects using these materials.

The presence of sulphur rich inclusions in OEG 180A remains confusing as it is unclear how they could have formed, though the possibility of coal use can be discarded. These inclusions could be the result of smelting pyrite rich ores, an explanation that could potentially explain the anomalous presence of copper in OEG 180B and 2540 but many questions persist. Further study will be required to understand these samples properly.

Fer Fort from Lorraine was established as a likely source of iron used at Oegstgeest and an additional tentative connection was made with Swabia. However, most of the samples remain unattributed and these appear to be made from iron of several different sources. Should compatible data become available, these would benefit from a comparison with the Netherlands, England and the Danube.

In summary, the findings of this study further support the conclusions of Kars et al. (2018) and Theuws et al. (2021a): far from being a poor and isolated settlement lost in a "marginal landscape", the site was well-connected, both within its local region and within wider networks of exchange spanning the European sub-continent. At least from a metallurgical point of view, Oegstgeest was fully integrated into both local regional networks of exchange.

29

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#### Figure titles and descriptions

2 Figure 1. Map of the Merovingian settlement at Oegstgeest (Theuws et al. 2021a).

3 Figure 2. a. OEG 221, SCD hearth bottom from Oegstgeest retaining the clear shape of the hearth. b.

4 OEG 2352. One of the tap slags from Oegstgeest with a knotted surface and broken edges.

5 Figure 3. List of the different sources of ore and slag compared to the Oegstgeest samples and their 6 geographic location.

Figure 4. One of the copper oxide prills in OEG 603. This specific prill is comprised of one larger prill
surrounding a smaller prill (a.), suggesting that these formed sequentially, likely in the hearth. The
surrounding facies was typical SFR (b. wüstite dendrites, c. glassy matrix). The composition of the prill
is summarised in Table 3.

11 Figure 5. OEG 5047. a. Macroscopic view showing the vitrified blue SAS patches on its surface. b. 12 Boundary between the SAS and the SGD strata. The neat delineation between the two strata indicates 13 that they formed sequentially but given that the fayalite continued to form further into the SAS stratum, 14 this must have been while the SAS was at least partially liquefied. The yellow/white microstructure on the right was identified as an iron sulphide compound by SEM-EDX. Given that the edge of this 15 surface enrichment is perfectly aligned with the split between SGD and SAS, this deposit seems to 16 17 have formed after the SGD section but before the SAS section, suggesting that it likely took place in 18 the hearth.

- Figure 6. The microstructure of OEG 2352. This piece contained relatively little wüstite compared to the forge slag but it did have lines of wüstite running through the sample. These were likely surfaces exposed to air, which oxidised before being covered with fresh slag.
- Figure 7. Etched microstructure of a. OEG 968 sub 3 and b. OEG 2540. Both samples were made of hypo-eutectoid steel and had unusually even carbon distribution across the piece.
- Figure 8. OEG 2611. SEM-EDX analysis split the inclusions from this sample into three groups that are located in three discrete regions across the sample surface, suggesting that the sample is likely made of two blooms (red and blue inclusions) separating by forge inclusions (yellow).
- 27 Figure 9. OEG 5355. This offcut contained a clear weld between iron and steel.

Figure 10. OEG 968 sub 2a. The three inclusions marked in red were attributed to a separate class by HCPC. The other three inclusions were in the corresponding location on the other side of the piece, which was analysed as a separate sample (OEG 968 sub 2b, see supplementary material).

- Figure 11. Microstructure of OEG 968 sub 1. a. Tempered martensite located along the working edge of the piece. b. Hyper-eutectoid steel located along the rear edge. c. Cementite that formed along the grain boundaries in the lower edge of the quenched working edge.
- Figure 12. Iron sulphide inclusions in OEG 180A. Most inclusions were small and spherical but some were larger and disaggregated into the iron matrix.
- 36 Figure 13. Results of Raman spectroscopy of iron sulphide inclusions in OEG 180A.
- Figure 14. Dendrogram resulting from HCPC of intra-site provenance. The red line indicates the threshold above which objects are considered to belong to a separate group. Objects grouped together are circled in red and attributed a group number.
- Figure 15. Crater resulting from LA-ICP-MS ablation in one of the inclusions in OEG 2611. The linear shape and large amount of wüstite in this inclusion could indicate that it is a forge inclusion. The analysed inclusions in OEG 2611 and 5355, as well as the six inclusions identified as separate in OEG 968 sub 2 showed similar facies. This suggests that these were all accidental ablations of forge inclusions and therefore cannot be used in the provenance study.

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## Table titles and descriptions

- 3 Table 1. List of samples analysed in this study, specifying the methods by which they were studied.
- 4 Table 2. Synthesis of petrographic study.
- 5 Table 3. Composition of the copper prill shown in Fig. 4. a. as established by SEM-EDX.
- 6 Table 4. Synthesis of the metallurgical study.
- 7 Table 5. Synthesis of provenance results.