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IRON SMELTING SLAG FORMATION AT TELL HAMMEH (AZ-ZARQA), JORDAN

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1. ARCHAEOLOGICAL BACKGROUND

At Tell Hammeh (az-Zarqa), Jordan, a wide range of metallurgical debris from an early iron smelting operation (ca. 930 CalBC) was excavated (van der Steen 1997; van der Steen 1997; van der Steen 2001; van der Steen 2001; van der Steen 2001; van der Steen 2003; van der Steen 2003; Veldhuijzen 1998; Veldhuijzen 1998; Veldhuijzen 1998; Veldhuijzen 2000; Veldhuijzen and van der Steen 1999; Veldhuijzen and van der Steen 1999; Veldhuijzen and van der Steen 2000). Tell Hammeh is a relatively small site in northern Jordan, located where the Zarqa river valley opens into the Jordan Valley. It is close to several of the larger tells in this part of the Jordan Valley as well as the main iron ore deposit of the wider region at Mugharet al-Warda (Abu-Ajamieh *et al.* 1988; Bender 1968).

Several periods have been attested at the site, starting with Chalcolithic (ca. 4500-3000 BC) and Early Bronze Age (ca. 3000-2000 BC) material, followed by more substantial layers of Late Bronze Age (ca. 1600-1150 BC) material, to Iron Age I (ca. 1150-1000/900 BC) and Iron Age II (ca. 1000/900-586 BC) (van der Steen 2003).

It is at the transition of Iron Age I and Iron Age II that metallurgical activity took place at the site. As iron production finds predating the Roman period are very rare in the Near East, Hammeh allows a unique insight into the earliest developments of iron smelting technology in this region.

2. THE IRON PRODUCTION PHASE

The large deposit of iron production debris at Hammeh forms a stratigraphically well defined phase. It has a complex internal layering, possibly indicating seasonal activity over an extended period of time.

Based on elevation levels of the remaining tell, half or more of the original production area may have been lost due to bulldozer activity. Less



than 7% of the projected production area has been excavated, yielding roughly 500 kg of slag. The excavated area reveals no indication of domestic or non-metallurgical use of the site, nor of any distinct activity areas, except possible furnace structures.

Radiocarbon analysis of two charcoal samples (identified by Eleni Asouti as olive wood (*Olea europaea*), pers. comm.), provides a date at 930/910 Cal BC (±40 years; 1 sigma ranges of 1000-900 and 940-850 Cal BC; AMS Analysis with C13-C12 correction). With due caution (see: "van Strydonck *et al.* 1999), these dates place Hammeh among the earliest known finds of iron smelting in the Near East (Pleiner 2000; Pleiner 2000; Pleiner 2000; Snodgrass 1980 p.338; Waldbaum 1999). Taking these dates as a *terminus post quem*, production at Hammeh may cover a period from around 910 BC to ca. 800 BC.

3. CLASSIFICATION

Initial classification of the finds took place during excavation, based on macroscopic features that can be observed in the field such as colour, shape, presence or absence of rust, flow patterns, and surface inclusions (Bachmann 1982 p. 2-6; pers. comm.; Sperl 1980; Sperl 1980). This was examined further using various laboratory techniques (Veldhuijzen and Rehren 2005).

Several types of material were identified, the bulk consisting of slag and tuyère fragments. The slag types range from black/grey, free flowing slag (tap slag; ca. 50%), through less dense glassy slag (possible ceramic origin) and plano/concavo-convex slag-cakes (furnace bottom and possible primary smithing) to rusty lumps (bloomery furnace mess; ca. 45%). The non-slag material ranges from tuyère fragments (approx. 350) to charcoal and possible furnace structures. The tuyère fragments are characterised by a vitrified nozzle and a 5 x 5 cm square section with a 1 cm bore (Veldhuijzen in press).

4. SLAG FORMATION

AAnalyses of the main type of slag demonstrate that clay is a major contributor to the slag formation in the Hammeh iron smelting process (Veldhuijzen 2003; See also: Serneels and Crew 1997; Crew 2000).

Mass balance calculations, based on a haematite ore sample from Warda and smelting tap slag from Hammeh (see table 1), indicate that 100 kg of ore contain theoretically enough wüstite (FeO) to enable the production of ca. 48 kg metallic iron, while retaining 38 kg of slag of an iron oxide level comparable to the Hammeh slag. Other oxide components in this theoretical slag, however, do not match the actual slag composition, particularly in their lime content, which is too high, and alumina levels, which are too low. The addition of a further ca. 20 kg of local and tuyère clay to the system,



simulating the absorption of furnace wall and tuyère material, balances the theoretical and archaeological slag composition much more closely.

	SiO ₂	Al ₂ O ₃	FeO	TiO ₂	MnO	CaO	MgO	K ₂ O	P_2O_5	S
Mugharet al-Warda Ore	8.50	0.38	82.32	0.07	0.05	8.29	0.15	0.03	0.21	0.00
Local Clay/Tuyère average (3 samples)	58.76	12.13	5.05	1.05	0.05	17.04	2.54	2.82	0.32	0.24
Hammeh Tap Slag average (6 samples)	24.05	5.47	52.80	0.31	1.32	11.26	2.30	0.74	1.42	0.33
Hypothetical slag ceramic-free	22.98	1.03	52.20	0.18	0.15	22.39	0.41	0.09	0.56	0.00
Hypothetical slag with ca 20 kg ceramic	26.31	3.65	52.37	0.36	0.08	15.19	0.86	0.77	0.35	0.06

Table 1. Main components of Hammeh slag formation related materials (Warda ore, local clay and tuyère ceramic), actual Hammeh slag, hypothetical ceramic-free slag and hypothetical slag with ca. 20 kg of ceramic contribution. Results are expressed in weight % and normalised to 100%. Samples were measured by (P)ED-XRF on Spectro XLab Pro 2000 with "Slag_fun" calibration method (Veldhuijzen 2003). See text for details on hypothetical slag composition.

This addition though reduces the iron metal yield to ca. 33 kg, i.e. less than 2/3 of the amount calculated without the addition of ceramic. While there is little doubt that a major ceramic contribution did in fact occur during the smelting operation, one wonders why the design in particular of the tuyères was done in such a way as to actually promote the absorption of ceramic material, which reduces the iron yield.

5. DISCUSSION

One possible explanation for this seemingly counter-productive feature may be found in the relevant phase diagram CaO-FeO-SiO₂ (see figure 1), and in particular in the morphology of the liquidus surface of this system.

The hypothetical ceramic-free slag has been calculated to about 20 wt% SiO_2 , 50 wt% FeO and 20 wt% CaO; alumina being a major contributor to the remaining 10 wt%, but not exceeding 2 wt% on its own. Reducing such a slag to its three main components (or rather six, adding Al_2O_3 to SiO_2 , MnO to FeO and MgO to CaO) and plotting it into the phase diagram indicates a melting temperature in excess of 1250 °C, probably beyond the thermal ability of an early furnace and the thermal refractority of the local clay.

The addition of clay/ceramic to the system, in quantities of ca. 1/5 of the ore weight, lowers the estimated liquidus temperature to around 1120 °C if not less, much more in line with the assumed performance characteristics of





Figure 1. CaO-FeO-SiO2 Phase diagram. Plotted are six Hammeh tap slag (in green) and their average composition (yellow), the local ore (red) and average local clay/ceramic composition (light blue), the hypothetical, ceramic free slag (dark blue) and the hypothetical slag when ca. 20 kg of local ceramic is added to the system (purple) (for numerical values see table 1); values are plotted with MgO added to CaO, MnO added to FeO and Al₂O₃ added to SiO₃.

an early furnace. This difference of ca. 100 °C makes the difference between thermal survival of the technical ceramic and fuel consumption and sufficient fluidity to allow the metal/slag separation to occur.

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REFERENCES

- Abu-Ajamieh, M.M., Bender, F.K. and Eicher, R.N. 1988. Natural Resources in Jordan, Inventory-Evaluation-Development Program. Amman: Natural Resources Authority.
- Bachmann, H.-G. 1982. The Identification of Slags from Archaeological Sites. Occasional Publication 6 London: Institute of Archaeology Occasional Publication 6.
- Crew, P. 2000. The influence of clay and charcoal ash on bloomery slags. in Tizzoni, C.C. and Tizzoni, M., (Eds). 38-48.
- Pigott, V.C. (1999) The archaeometallurgy of the Asian Old World. Ephrata, Pennsylvania: University Museum, University of Pennsylvania.
- Pleiner, R. 2000. Iron In Archaeology. The European Bloomery Smelters. Praque: Archeologick_ Ústav Av Cr.
- Serneels, V. and Crew, P. 1997. Ore-slag relationships from experimentally smelted bog-iron ore. in Crew, P. and Crew, S., (Eds). 78-82. Maentwrog: Plas Tan y Bwlch Occasional Paper 3.
- Tizzoni, C.C. and Tizzoni, M. (2000) Il ferro nelle alpi. Giacimenti, miniere e metallurgia dall' antichità al XVI secolo. Atti del convegno/Iron in the alps. Deposits, mines and metallurgy from antiquity to the XVI century. Proceedings of the conference. Bienno: Commune di Bienno.
- van der Steen, E.J. 2001. Excavations at Tell el-Hammeh. Studies in the History and Archaeology of Jordan VII, 229-232.
- van der Steen, E.J. 2003. Tribes and Territories in Transition: the central East Jordan Valley and surrounding regions in the Late Bronze and Early Iron Ages: a study of the sources. Groningen: Rijksuniversiteit Groningen.
- van Strydonck, M., Nelson, D.E., Crombé, P., Bronk Ramsey, C., Scott, E.M., van der Plicht, J. and Hedges, R.E.M. 1999. What's in a 14C Date. Rapport du Groupe de travail : Les limites de méthode du carbone 14 appliquée à l'archéologie, 433-440. Rennes: Mémoires de la Société Préhistorique Française, XXVI, 1999 et Supplément 1999 de la Revue d'Archéométrie.
- Veldhuijzen, H.A. 1998. Early Iron Smelting. Analysis and Interpretation of Late Iron Age Iron Smelting Remains from Tell Hammeh az-Zarqa, Jordan. Leiden: Leiden University, Faculty of Archaeology, unpublished M.A. Thesis.
- Veldhuijzen, H.A. 2003. 'Slag_Fun' A New Tool for Archaeometallurgy: Development of an Analytical (P)ED-XRF Method for Iron-Rich Materials. PIA (Papers from the Institute of Archaeology) 14, 102-118.
- Veldhuijzen, H.A. 2004 (in press). Technical Ceramics in Early Iron Smelting. The Role of Ceramics in the Early First Millennium BC Iron Production at Tell Hammeh



(az-Zarqa), Jordan. In Proceedings of EMAC '03, (Ed.). Série Monográfica, Instituto Português de Arqueologia (IPA).

- Veldhuijzen, H.A. and Rehren, Th. 2005 (submitted). Early Evidence for Iron Smelting in Jordan; how classification in the field relates to analyses in the laboratory. IAMS Journal 25. London: Institute for Archaeo-Metallurgical Studies.
- Veldhuijzen, H.A. and van der Steen, E.J. 1999. Iron Production Center Found in the Jordan Valley. Near Eastern Archaeology 62 (3), 195-199.
- Waldbaum, J.C. 1999. The Coming of Iron in the Eastern Mediterranean. Thirty years of Archaeological and Technological Research. in Pigott, V.C., (Ed.). 16, 27-57. Philadelphia: MASCA, University Museum, University of Pennsylvania.

