By the hand of the smelter: tracing the impact of decision-making in bloomery iron smelting

Ivan S. Stepanov1,2 · Lee Sauder3 · Jake Keen4 · Vanessa Workman5 · Adi Eliyahu-Behar1,6

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Abstract
Slag analyses from archaeological iron smelting sites are common. Rigorous analyses of iron and slag from successful experimental smelting, however, are still rare. Furthermore, thorough analyses from a series of smelts, and of the slag produced in different phases of the smelt, are exceedingly rare. The present study investigates the effect of an iron smelter’s decision-making and skills on the products of the smelting process: iron and slag. Four smelting experiments were carried out in a shaft furnace with slag tapping using iron ores from the Southern Levant. Using various analytical techniques, including portable X-ray fluorescence, optical and electron microscopy, metallography, and hardness tests enabled us to correlate the properties of the final products with adjustment of various parameters during the smelting process. The latter include airflow and charging rate, temperature, residence time in the reducing zone, ore-charcoal ratio, and control of the slag characteristics. Results obtained allowed us to empirically demonstrate the direct impact of decisions made by the smelter during the complex technological practice of bloomery smelting. Analysis also highlights the benefits of moderately reducing conditions controlled by the smelter to produce good-quality, low-carbon iron, which is particularly relevant within the geological setting of the Southern Levant.

Keywords Bloomery iron smelting · Shaft furnace · Slag analysis · Experimental archaeology · Southern Levant

Introduction
A series of iron bloomery experiments was conducted over two seasons during February 2019 and February 2020, in Mesheq Hanan (Kidron, Israel). Experiments were conducted as part of an ongoing research, which investigates the beginning and development of iron production during the Iron Age of the Southern Levant. Three main aims were put forward for the experiments: (1) to test the viability of iron ore deposits, from the northern Negev region (Southern Israel) for bloomery smelting; (2) to produce consolidated bloom, slag, and metal bar samples in order to develop and assess the methodology of iron and ferrous metal provenance using osmium isotope composition (Os IC); and (3) to replicate the archaeological evidence and the type of iron production debris produced during the early Iron Age of the Southern Levant.

The smelting experiments were supervised and operated by two professional smelters, Mr. Lee Sauder and Mr. Jake Keen, and were designed to fulfill the aims specified above, using a slag tapping shaft furnace (described below), and various designs of bowl furnaces. The latter were built using local clay mixtures and were operated without slag tapping. Insights gained from the bowl furnace experiments were recently summarized (Workman et al. 2021). An emphasis was given to the deformations and vitrification these installations underwent through the smelting process, and the
implication this has for understanding the lack of preservation of such installations in the archaeological record.

Results of the osmium isotope analysis of ore, bloom, and metal produced in the first three shaft furnace experiments were recently published and confirmed the potential of osmium isotopes as a useful tool for the provenance of archaeological ferrous metals (Brauns et al. 2020).

The focus of the present paper is to refine and explore some intriguing questions related to the role of human choices and decisions taken in bloomery smelting, for which these smelting experiments using a shaft furnace provided an excellent opportunity.

Iron smelting is a complicated technology, involving many steps and decisions. The multitude of choices offered by the chaîne opératoire of iron production has been well-documented, and these studies serve as a suitable framework to diagram the various process steps in antiquity as well as those of the present experiments (for examples, see diagrams in Serneels 1993; p. 35, Fig. 35; Bauvais and Fluzin 2009: Fig. 5).

Each objective in our experimental trials directed the decisions and choices made for each of those stages, which can generally be divided into three categories: the choice of raw materials, the technical design of the furnace, and the smelting protocol. The choice of ores was dictated by the local geological environment and ores accessibility. Three geologically similar deposits located in the northern Negev region in southern modern Israel (Fig. 1) were selected based on surveys and analyses already initiated within the framework of ore prospecting by the Israeli Government during the 1950s (Ilani et al. 1985, 1988). These deposits (and others) were previously investigated for their viability in crucible smelting under laboratory conditions and were found promising for the bloomery smelting experiments (Stepanov et al. 2020a).

Although any evidence for past exploitation of the Negev ores has not yet been identified, these ores are geologically and mineralogically similar to the Mugharet el-Wardeh ore deposit in the Ajloun region in modern Jordan—the only source in the region currently known to be exploited in the early first millennium BCE (Veldhuijzen and Rehren 2007; Al-Amri 2008; Al-Amri and Hauptmann 2008; Dill et al. 2010).

Both the design of the smelting furnace and the smelting protocol were largely dictated by our major goal—to produce a well-consolidated, forgeable bloom of soft iron that would allow the completion of the entire chaîne opératoire—from smelting the ore to the forging of a final object—a bar. This assured the necessary samples for developing the osmium IC provenance method, including production debris.

Parameters related to the design of the smelting furnace and its operation protocol were determined solely by the head smelter—Mr. Lee Sauder—relying on his vast experience, and determined by many years of experimental trials, in which he investigated practical techniques for bloomery iron production (Sauder and Williams 2002; Sauder 2013). Moreover, the methodology developed by Sauder and utilized in our smelting experiments has become a common practice in contemporary bloomery smelting experiments all over the world (e.g., Benvenuti et al. 2016, and several smelts conducted during Woodford Furnace Festival in 2018–2020).

One of the main benefits of this protocol (described in detail in “The smelting protocol”) is the production of a dense, forgeable bloom of soft iron. Archaeological evidence of similar shaft furnaces operated by slag tapping has been identified in many different parts of the world (e.g., Africa, Europe, Eurasia: Chirikure and Bandama 2014, Pleiner 2000: pp. 172–175; Vodyasov 2018). However, similar evidence has so far not been identified in the Southern Levant;
indeed, archaeological evidence for any furnace remains is generally lacking in this region (Veldhuijzen and Rehren 2007; Eliyahu-Behar et al. 2012, 2013; Yahalom-Mack and Eliyahu-Behar 2015; Workman et al. 2020, 2021).

In this study, we observe and investigate the technical choices the smelter must make while operating the furnace by employing empirical observations alone. Then, using metallography, microstructure, and chemical composition analysis of each of the smelting products (the bloom, the bar, and the slag), combined with observations made during the experiments, we make direct connections between particular decisions made and the results obtained. As will be shown, technical choices made by the smelter during operation are especially manifested in the management of slag, air blowing rates, ore-to-charcoal ratio, and charging rates.

This in turn shows how the skills and decisions of the ancient smelters, which tend to be invisible parameters in the archaeological record and therefore rarely appear in the center of archaeometallurgical studies, may complement the more common emphasis given to the composition of raw materials, slag, and furnace remains in our search for reconstructing ancient metallurgical processes (for e.g. Eliyahu-Behar et al. 2012, 2013; Rehren et al. 2007; Serneels and Crew 1997; Joosten et al. 1998; Charlton et al. 2010; Charlton and Humphris 2019; Crew 2013; Doonan and Dungworth 2013; Stepanov et al. 2020b, 2021).

### Description of the smelting experiments

In this section, we describe the various parameters and choices made in relation to raw materials, furnace design, the smelting protocol, and its rationale. These are summarized in Table 1 and discussed in detail below. Decisions regarding the smelting protocol are given particular attention in the study.

#### Shaft furnace design and construction

The shaft furnace used in our experiments was built under the direction and according to a design developed by Sauder (2013). It is a simple, relatively small cylindrical shaft

### Table 1  Decisions and their rationale affecting smelting experiments

| Decision                                      | Rationale                                                                 |
|-----------------------------------------------|---------------------------------------------------------------------------|
| **Raw materials**                             |                                                                           |
| Ores: epigenetic goethite-hematite ores of the Dead Sea Transform | Local source; availability; morphological and chemical indication of high iron content; successful outcome in laboratory trials |
| Kaolin clay for furnace construction          | High refractoriness to maintain good control of the smelting process, avoid contamination of Os signatures due to melting of furnace walls. Durability allowing multiple reuses of the furnace |
| Commercial charcoal                            | Availability                                                             |
| **Furnace parameters**                        |                                                                           |
| Shaft furnace with slag tapping               | Effective reduction, proven results, Smelter’s experience                |
| Electric air blower                           | Adequacy and control of air flow without long training of bellows operators |
| Copper tuyère                                 | Durability and stability—no contribution of clay melting for Os signatures |
| Tuyère angle (17°)                            | Optimal bloom shape in this furnace design                               |
| Total input of ore per single smelt (30–45 kg) | Furnace volume                                                           |
| **Smelting protocol**                         |                                                                           |
| Ore preparation and roasting, ore particle size ca. 1–2 cm, charcoal lump size 0.5–4.0 cm | Ore quality and type; smelter’s experience                                |
| Furnace pre-heating time (30 min–1 h and 30 min) | Time required for other preparatory chores                               |
| Ore-charcoal charging rate (9–14 min/2 kg); residence time of the ore in the reducing zone | Color of the hot zone of the furnace; balancing reduction and slag chemistry |
| Airflow rate and furnace temperature         |                                                                           |
| Ore-charcoal ratio (c. 1:1) and redox conditions |                                                                           |
| Monitoring of the slag composition            | Visual observation of viscosity and color of the slag after tapping       |
| Timing of slag tapping                        | Observing slag composition and formation of stable slag bowl             |
| Duration of the smelt and time of bloom extraction (3 h and 10 min–4 h and 40 min) | Judging density of bloom by watching slag flow; desired size of bloom |
| Forging and sectioning of bloom immediately after the smelt | Initial test of the metal quality: smaller pieces for multiple forging experiments |
furnace, ca. 1 m high, with 26 cm inner diameter, operated with forced air. Figure 2 shows a schematic representation of the furnace design. Although the cylindrical shaft furnace is probably the most common type in both ancient and modern metallurgy, its size and proportions may vary significantly. The choice of a relatively small-diameter shaft used in this case is reasoned by the intent of the smelter to achieve a moderately sized bloom that could be easily refined using hand hammers.

A circular layer of modern building bricks was set as a foundation to build the furnace on, giving it stability and raising it above the ground surface to facilitate slag tapping and bloom extraction (Fig. 2a). The bottom part of the furnace was filled with sand and wood ash pounded into a semi-spherical bowl inclining towards the opening through which the slag was to be tapped, and bloom removed.

The clay mixture was made by mixing kaolinite commercial clay (as powder), commercial sand, and shredded straw (in app. 13:33:1 ratio by weight). Refractory kaolin clay was chosen for several main reasons: better control of the smelting process by preventing significant melting of furnace walls, thus enabling multiple reuses of the furnace, and avoiding contamination of the osmium isotopic signal of the ore and products.

The clay walls of the furnace were constructed around a cylindrical form of bamboo bundled together, both to produce an accurate and regular interior diameter, and to prevent the clay from slumping (Fig. 3a). Lumps of wet clay (ca. 10 × 6 × 6 cm) were densely packed around the wooden core and additionally stabilized by binding with twine. The bamboo form was then burned which helped to dry and fire the furnace walls prior to smelting.

Bloom forging was conducted in a small hearth, specifically built for this purpose using the above-described clay mixture. Forging comprised re-heating and hammering quarters of blooms into bars (Fig. 3g and Fig. 5).

**Air supply**

During smelting, air was supplied via an electric blower and a copper tuyère. The choice for this system instead of a more authentic hand-operated bellows was underlain by the difficulty of maintaining a consistent supply of air via bellows over the duration of several hours: a skill which many modern experimentalists do not have (Doonan and Dungworth 2013; Crew 2013; Humphris et al. 2018). An inconsistent airflow rate may significantly disrupt the quality of the final product, while a stable and constant airflow promotes a better reduction process.

The tuyère, with an inner diameter tapering from 5 cm at the blower end to 2 cm at the furnace end, was inserted at an angle of c.17°, 25 cm above the brick furnace base (see schematic in Fig. 2). The tip of the tuyère protruded 6 cm inside the furnace, and its rear end was connected via a plastic hosepipe to an electric air blower (Makita UB1101, 50–60 Hz, 600 W, air volume: 0–2.8 m³/min) equipped with an airflow regulator (rpm: 0–16,000). During the smelt, the airflow was not directly measured, though the airflow regulator was set to c. 15–30% of the blower’s maximum capacity corresponding to c. 0.4–0.8 m³/min.

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**Fig. 2** a Schematic design of the shaft furnace built for the experiments. b An image of the furnace during operation (FEXP-1)
Another, although indirect, estimation of air input is based on the rate of charcoal consumption, which was approximately set to a rate of 2 kg of charcoal consumed every 9–12 min for the first three experiments (FEXP-1, FEXP-5, and FEXP-6), and 12–15 min for the fourth experiment (FEXP-100). The c. 10-min charcoal consumption rate was chosen based on the experience of Sauder, as best suited for production of a well-consolidated bloom of soft iron.

**Raw materials: iron ores and charcoal**

Three sources of iron ores from the Negev region in southern modern Israel were used for the experiments. The geological formation of these ores is related to epigenetic mineralization associated with major trending faults of the Dead Sea Transform (Ilan et al. 1985, 1988). The ore sources used are Zavar: IP-49 (FEXP-1 and FEXP-100), Nekarot-Evos: IP-43 (FEXP-5), and Nekarot: IP-61 (FEXP-6) (for ore locations, see map in Fig. 1). The chemical, mineralogical, and isotopic compositions of these ores were studied and results were previously published (Brauns et al. 2020; Stepanov et al. 2020a).

All three ore sources have common mineralogical and geochemical properties; they are relatively rich in iron oxide (Fe₂O₃, 70–80 wt%) predominantly composed of goethite with small amount of hematite, and are associated with quartz (as chert), calcite, gypsum, and barite, which is reflected in the levels of SiO₂ (14–29 wt%), CaO (1.1–4.6 wt%) SO₃ (1–4), and BaO (0.1–4.0 wt%). Ores IP-43 and IP-61 share significant compositional similarities appearing in elevated concentrations of CaO, BaO, SO₃, and V, with respect to ore IP-49, which is located several dozen of kilometers away and is characterized by a higher silica content. The chemical composition of the ores after roasting, measured by portable X-ray fluorescence (pXRF), is presented in Table 2.

Prior to roasting, the ores were broken into ca. 4–7-cm fragments (Fig. 3b). These were visually inspected, and parts appearing to have more nonferrous (white-yellow-colored) impurities were discarded. Although experiments 1 and 100 used the same ore deposit (IP-49), some
variability is observed, perhaps also due to differing ore grading skills. The crushed ores were roasted in several consecutive batches (c. 50 kg each) in a pile of wooden logs for 4–8 h and were subsequently further crushed into smaller pea-size fragments ca. 0.1–2.0 cm, without removal of the fine fraction. The choice to use this particle size was based upon past experience of Keen and Sauder and was also proven effective during smelts of limonite and siderite ores by other experimentalists (Crew 1991; Tylecote et al. 1971).

Over the course of many experiments with this furnace type, Sauder has found that the total amount of ore to be charged, resulting in a high-quality iron, easily forgeable by a hand hammer, and a practical and economic yield, usually lies in the range of 32 to 44 kg, depending on the richness of the ore. This deliberate limitation of the bloom’s size also helps to preserve the furnace for multiple uses. These considerations determined the amount of ore prepared for each experiment.

Commercially purchased charcoal, imported from South America, was used for both smelting and smithing. This decision was merely based on convenience and availability. However, it was later found out to bear some unexpected consequences, regarding sulfur contamination (discussed later). In accordance with Keen and Sauder’s experience, the initially large charcoal lumps were chopped down to ca. 3–5 cm and the fines and dust sifted out with a 1-cm net. This decision is in accord with the suggestion that the optimal charcoal size should be of 8–10% of the inner furnace diameter (Rehder 2000: pp. 17, 66).

### Furnace temperature

The furnace temperature was directly controlled by the air flow (charcoal combustion rate) and indirectly by the fuel to ore ratio. The smelter can judge the temperature of the hot zone by peering through the tuyère and observing its color. While to the casual observer the color might look like a blazing white at these temperatures, the practiced smelter can discern gradations of red, yellow, green, and even blue within the white that allow for fairly subtle temperature distinctions. This in fact was proven a rather reliable estimation.

For documentation purposes, the furnace temperature was measured throughout each smelting experiment by four K-type thermocouples (within permissible temperature range up to c. 1260 ± 2.2 °C), positioned at relative heights from the bottom of the furnace; T1 = 33 cm (just above tuyère level), T2 = 55 cm, T3 = 75 cm, and T4 = 91 cm (ca. 10 cm below the top of the shaft). The penetration depth of each thermocouple was c. 4.5 cm from the inner surface of the furnace wall. Each measurement lasted for approximately 3 min until the reading stabilized. Within 30 min of the first ore charge, the temperature at the tuyère level (T1) usually reached more than 1350 °C. As this temperature exceeds the thermocouple permissible range, some were melted and lost their function (at c. 1370 °C); hence, we report here only the temperatures recorded by T2 and T3. T4 showed higher fluctuations and is therefore not reported (Fig. 4; for the thermocouple position, see Fig. 2a).

| Table 2 | pXRF analyses of major elements of roasted ores and charcoal ash used in the present experiments (given in wt% except where noted). The analysis of reference materials is given for comparison. (*Matching ore in Stepanov et al. 2020a, Zavar = Negev 3–4, Nekarot-Evos = Negev 1–2, Nekarot = Negev 5–7) |
|---------|---------------------------------------------------------------|
| Sample  | Fe₂O₃ | MgO | Al₂O₃ | SiO₂ | P₂O₅ | SO₃ | K₂O | CaO | TiO₂ | MnO | BaO | Tot | V (ppm) |
| Roasted ore | | | | | | | | | | | | | |
| IP-49 (FEXP-1) | | | | | | | | | | | | | |
| Zavar* | 78 | bdl | 1.1 | 27.7 | 0.1 | 1.8 | 0.2 | 1.1 | 0.1 | 0.1 | 0.1 | 110 | 281 |
| IP-49 (FEXP-100) | | | | | | | | | | | | | |
| Zavar* | 75.2 | 2.6 | 1.2 | 29.3 | 0.1 | 3 | 0.2 | 1.6 | 0.1 | 0.2 | 0.1 | 111 | 258 |
| IP-43 (FEXP-5) | | | | | | | | | | | | | |
| Nekarot-Evos* | 79.4 | 2.4 | 1.3 | 13.8 | 0.2 | 2.6 | 0.3 | 4.6 | 0.1 | 0.1 | 1.3 | 101 | 1999 |
| IP-61 (FEXP-6) | | | | | | | | | | | | | |
| Nekarot* | 69.4 | 2.5 | 1.3 | 23.4 | 0.2 | 4.8 | 0.2 | 3.3 | bdl | 0.1 | 2.7 | 103 | 1960 |
| Charcoal ash | | | | | | | | | | | | | |
| - | 10.8 | - | 0.8 | 2.3 | 10 | 5.1 | 65.9 | 0.1 | 0.3 | - | 95 |
| 0.1 | 14 | - | 0.7 | 1.5 | 5.8 | 3 | 77.5 | 0.2 | 0.4 | - | 103 |
| Standards | | | | | | | | | | | | | |
| CRM180-649 | Cert. value | 4.8 | 2.4 | 13.9 | 64.8 | 0.2 | - | 2.5 | 2.7 | 0.6 | 0.1 | 0.1 | 110 |
| Meas. value | 4.9 | 2.5 | 13.2 | 63 | 0.3 | - | 2.1 | 2.8 | 0.6 | 0.1 | 0.1 | 90 | 179 |
| CRM180-706 | Cert. value | 2.6 | 0.5 | 12.5 | 73.4 | 0.1 | 0.2 | 5 | 0.8 | 0.3 | 0.1 | 0.1 | 25 |
| Meas. value | 2.5 | 0.7 | 11.9 | 80.5 | 0.3 | 0.6 | 4.5 | 0.8 | 0.3 | 0.1 | 0.1 | 102 | 74 |
The smelting protocol

As mentioned earlier, the head smelter followed a specific, previously developed strategy (Sauder and Williams 2002; Sauder 2013). At the base of this technique lie two specific goals: producing an easy to forge, well-consolidated soft iron bloom, and assuring the formation of sufficiently fluid smelting slag, with relatively low viscosity to optimize tapping and minimize carburization. Some advantages of slag tapping are rather obvious: assisting bloom consolidation, providing more room for the bloom to grow, and avoiding clogging of the tuyère (Rostoker and Bronson 1990: pp. 81, 94; Rehder 2000: p. 127). Less obviously, formation of low viscosity, iron-rich slag is needed for efficient transport of reduced metal particles into the bloom, protecting the bloom from oxidation, preventing carburization, and avoiding the reduction of phosphorus (Sauder and Williams 2002; Sauder 2013; Sauder forthcoming). Some of these observations were also supported by results of crucible smelting experiments (Stepanov et al. 2020a).

According to Sauder, the optimal slag should be relatively heavy and have a grey/black color with dull metallic luster. This indicates wüstite in excess of fayalitic composition. A more black, glassy slag indicates a near fayalitic composition. In contrast, a lighter, greenish slag with a more vitreous luster is undesirable, indicating a low iron content. Similar classification of slag has been suggested by others (Serneels 1993; Bachmann 1982). By judging the visual appearance of the slag as smelting proceeds, the smelter decides upon his actions of increasing or decreasing air blast or altering the charcoal to ore ratio and charging speed.

Description of smelting experiments

Four field experiments (FEXP) were carried out over two sessions. Three experiments (FEXP-1, FEXP-5, and FEXP-6, February 2019) were conducted under very similar conditions, using roughly the same airflow rate, temperature, and charging intervals, each with a different ore. In contrast, the fourth experiment (FEXP-100), conducted a year later, in February 2020, was operated using a slower charging rate (see below). In all experiments, the same furnace design was used.

The amount of charcoal and ore used in each experiment, the duration of the smelt, the time of the first slag tapping, and the weight of bloom and slag, as well as other related parameters, including calculated yield, are summarized in Table 3. Figure 4 shows the furnace temperature profile of each experiment in correlation with ore charging intervals.

The slag was first tapped after about 2/3 of the total charge was consumed, by opening a 15 × 7-cm channel at the base of the tapping arch. From that moment onwards and until the end of the smelt, the slag ran freely out of the tapping door. Figure 4 shows a constant temperature rise until this moment, followed by a gradual decrease until the last ore charge. This was probably caused by the accumulating pool of liquid slag keeping the temperature high until tapping. It could also indicate that the hot zone was moving downward, rather than an actual temperature decrease.

Between 32 and 41 kg of ore was used in each experiment. The ore-to-charcoal ratio charge was generally kept at ca. 1:1, with consumption rate of 2 kg of ore and 2 kg of charcoal per an average time of 9–15 min (as discussed in more detail in “Air supply”). Charging was generally done...
### Table 3  Initial parameters and produced results of smelting experiments

| Field experiment no | Used roasted ore (kg) | Used charcoal (kg) | Total duration | Charcoal burning rate | Tapping time | Total metal (kg) | Yield (%) | Total slag (kg) | Slag viscosity (visual assessment) | Forging |
|---------------------|-----------------------|-------------------|----------------|-----------------------|--------------|-----------------|-----------|----------------|----------------------------------|---------|
| FEXP-1              | 32                    | 16                | 03:10          | 11*–10**              | 01:40        | 4.8             | 0.6       | 16.9           | 16                               | 1.4     | Average: viscosity improved after the increase of ore to fuel ratio | Relatively easy to forge to 1 cm thickness |
| Ore, IP-49, Zavar   |                       |                   |                |                       |              |                 |           |                |                                   |         |
| FEXP-100            | 35                    | 10                | 04:44          | 15*-12**              | 02:55        | 7.6             | 1.3       | 25.5           | 17                               | 0.1     | Good | Metal became brittle after it was thinned to 2 cm |
| Ore, IP-49, Zavar   |                       |                   |                |                       |              |                 |           |                |                                   |         |
| FEXP-5              | 40.3                  | 14                | 04:26          | 13*-12**              | 02:06        | 5.9             | 2.4       | 20.6           | 16                               | 2.1     | Good | Relatively easy |
| Ore, IP-43, Nekarot-Evos |               |                   |                |                       |              |                 |           |                |                                   |         |
| FEXP-6              | 41                    | 14                | 03:53          | 12*-9**               | 02:08        | 7.1             | 1.1       | 20.4           | 20.4                            | 0.2     | Average; tapping improved after the increase of ore to fuel ratio | Metal became brittle after it was thinned to 3 cm |
| Ore, IP-61, Nekarot |                       |                   |                |                       |              |                 |           |                |                                   |         |

*Total duration is the time since 1st ore charge until bloom removal

*Charcoal burning rate is the time to consume 2-kg charcoal and 2-kg ore: before (*) and after (**) tapping

*Time of first tapping, since 1st ore charge

*Fragments broken off during hot cutting of the bloom: metal 60–80 vol%, slag 40–20 vol%

*Yield calculated by dividing the total metal (bloom and parts detached during hot cutting) by the total of ore charged
in the following manner: rather than administering the full charge at once, the ore and charcoal were continuously poured over the top of the shaft every time the furnace burden lowered/was consumed, leaving a 10-cm gap at the top of the shaft. The 2:2 measurements were used as a marker of consumption rate. The furnace was kept full throughout the smelt until the last charge which was left to burn down through the shaft (last chance for reduction). This procedure was followed for all four experiments. However, in the first three experiments (FEXP-1, FEXP-5, and FEXP-6), the smelter increased slightly the ore-to-charcoal ratio (from 1:1 to 1.25:1) for a number of charges shortly after first slag tapping. For smelting experiment 100, we chose to work at a slightly lower consumption rate (2 kg of ore per 13–15 min) to test the effect of this change on the final product. Sauder expected this change to increase yield and raise carbon content, and perhaps decrease the ease of forging.

Along with the change of the ore-to-charcoal ratio, the smelter also slightly accelerated the charcoal consumption rate immediately after the first slag tapping, from 10 to 11 min to 9 min per 2 kg charge. This reflects the need to compensate for a sudden removal of mass from the furnace once the slag was tapped; otherwise the furnace would have eventually become empty and lost its heat.

At the end of each smelt, ca. 25–45 min after the final charge, the bloom was extracted by opening the furnace “front door” (Fig. 3e). While still hot, the bloom was cut in half and one of those halves was cut again to produce two quarters (Figs. 3f and 5d). Immediately after that, all produced materials: bloom, furnace, and tap slag, were collected, weighted, and documented (for some representation of the outcome, see Fig. 5).

A quarter of each bloom was subject to cycles of heating and forging, to produce a bar in a small smithing hearth using an iron anvil and hammer (Fig. 3g). Due to varying quality and mechanical properties of the metal and especially the presence of sulfur (discussed in “Analysis of metal: blooms and bars”), the forged bars are nonuniform in size and shape, and therefore do not fully allow the comparison of metal losses during forging (bars are presented in Fig. 5). The full record of each smelt and subsequent forging is provided in Supplementary File S1.

**Methods of analyses**

As described above, four experiments were conducted, each producing a bloom and a bar as well as a sequence of tap slag (from first to last tapping) and furnace slag which remained inside the furnace. Forging slags were also produced; however, these were not included in this study.

Two orthogonal sections from a quarter of each bloom were initially cut using a band saw. Due to their large size, and to facilitate optical (OM) and scanning electron microscopy (SEM) analyses, each of these sections was additionally sawn into two parts (making four sections in total), embedded in epoxy resin, ground, polished, and prepared for metallographic analysis. Debris detached from the bloom during hot cutting at the end of each smelt was also investigated. A cross section of each bar was prepared in a similar manner. From each experiment, at least three tap slag samples representing the tapping sequence: early, middle, and late tap slag, plus one sample of furnace slag were prepared for metallographic analysis.

**Optical and scanning electron microscopy**

Optical and electron microscopy were the primary methods for investigation of the experimental products, both metal and slag. We studied the microstructure and phase
composition of the smelting slag, the metallographic structure of the etched metal, and the inclusions of sulfide within the metal. Optical (OM) analyses were performed on a polarized microscope Zeiss Axioscope 5 using a Clemex vision lite software.

SEM–EDS analyses were performed on a Tescan MAIA3 Triglav electron microscope. The EDS analyses were carried out and processed using AZtec 3.2 Oxford Instruments software. The accuracy of analyses was verified by internal EDS standards. The purpose of SEM–EDS investigation was to determine phase and chemical composition of slag and metal. Tap slag was analyzed as full-areas for bulk composition \( (N = 3–6 \text{ areas of } 0.1 \text{ mm}^2 \text{ per polished sample of } 3 \times 3 \text{ cm}^2) \) and for spot composition of comprising mineral phases. The composition of the furnace slag was not as systematically analyzed, due to its heterogeneity. To determine the composition of the blooms and bars, bulk areas \( (N = 7–15 \text{ areas of } 0.8 \text{ mm}^2 \text{ per polished sample}) \) were analyzed.

**Portable X-ray fluorescence analysis**

X-ray fluorescence analysis was performed on iron ores and slag to determine their bulk chemical composition using a Thermo Gould-III instrument (Thermo Scientific) with a silver (Ag) anode and a set of secondary targets. Quantification was made using a “mining” calibration setting in the system. Certified reference standards CRM 180–649, 180–706, were used to assure the quality of data and estimate the accuracy of the measurements.

For the analysis of iron ores, a starting sample of about 1 kg of roasted ore was crushed, split, and quartered multiple times to obtain ca. 50 g of representative sample that was further ground to a fine powder using an agate mortar and pestle. Powders were placed in an XRF cup with a 4-µm proline film. Under these conditions, accuracy of analyses is generally better than 10% for elements present in the analyte at more than 5 wt%. However, the data for low-Z elements such as Mg, Al, Si, S, and P, especially when present in low concentrations, were treated with caution due to the relatively low sensitivity of the pXRF.

Tap slags were measured for their chemical composition by preparing polished sections (without embedding into epoxy resin). For each experiment, we measured multiple slag samples representative of the complete smelting sequence, denoted here as early, middle, and late slag.

**Indentation Vickers hardness**

Metal samples were subjected to indentation hardness testing using a Future-Tech FV-810 digital Vickers hardness tester. Each measurement was carried with a 3-kg load to an indenter producing an average indentation with a diagonal of c. 130 µm. The quality of analyses was assured by repeated measurements of a certified reference sample (with a hardness of 800 HV). The measurements were performed on the un-etched metallographic samples of blooms and bars formerly analyzed by SEM. Between 5 and 20 points were measured for each bloom/bar sample. Average values and standard deviations are given in Table 5 (Full data is provided in supplementary Table S5).

**Results**

All experiments resulted in a similar outcome: a well-consolidated bloom and tap slag, thus indicating consistency of the smelting protocol used (Table 3 and Fig. 5). Despite these similarities, some variations were observed between experiments. For example, the total metal yield varied within a close range of 17–26%. FEXP-1 undertaken under the highest temperatures produced the lowest yield (17%), while FEXP-100, in which the air blowing and charcoal combustion rate was the lowest, produced the highest yield (26%). The forgeability of the metal also differed: blooms FEXP-6 and FEXP-100 appeared to be particularly difficult to work due to their high brittleness, while blooms FEXP-5 and FEXP-1 showed better workability. In a similar manner, based on visual observations, the slag viscosity varied within and between the experiments.

These results can be associated to variations in ore composition and to slight changes in the process parameters implemented at the smelter’s discretion. Below, we report the results of the scientific investigation of products and by-products of the smelting experiments, and with these results attempt to decipher the impact of the smelter’s decisions and the degree with which the smelter can exercise control over the process.

**Analysis of slag**

For this study, we distinguish two slag types formed during the smelting process: tap slag and furnace slag (Fig. 5a, b). Tap slag constitutes the majority (> 90%). Solidified outside the furnace, it is characterized by distinct flowing textures. However, some slag, especially in FEXP-1 and FEXP-6, started to solidify halfway through the tapping channel and is therefore devoid of the characteristic flow texture. Such slag had to be mechanically raked out to clear the way for the outgoing stream of slag.

Furnace slag, which remains in the furnace (an example is shown in Fig. 5b) or is partly removed along with the bloom, comprises the minor portion of the total slag in each experiment, with its amount reaching up to 10% of the total slag (by weight) in FEXP-1 and FEXP-5. In contrast to tap slag,
the furnace slag shows a rough surface texture caused by the presence of incompletely reacted ore fragments. Below is a more detailed description and analysis of each slag type.

**Tap slag**

To determine the chemical and mineralogical composition of the slag, and identify variations in its composition throughout the smelting sequence, we divided the slag from each experiment into three groups: early, middle, and late tap slag, and analyzed each group, by both pXRF and SEM–EDS.

Table 4 summarizes the results of the SEM–EDS analysis for early and late slag, while typical microstructures of each slag are shown in Figs. 6 and 7. Chemical composition measured by pXRF was used to investigate the variability within the slag throughout the tapping sequence. The complete pXRF analyses are given in Supplementary Table S2, while results are visually summarized in Fig. 8 (For individual analyses of mineral phases within the slag, see Supplementary Table S3).

In general, the average mineralogical and chemical composition of the tap slag reflects the chemical composition of the ore used in each experiment. For example, slag from FEXP-5 and EXP-6, produced from Nekarot deposits (IP-43 and IP-61), contain higher amount of CaO (8.5 wt%), BaO (1.5–3.9 wt%), and V (ca. 500 ppm) than slag produced from Zavar ore (IP-49, FEXP-1 and FEXP-100).

The FEXP-5 slag generally has a higher FeO content (52–68 wt%), and its mineralogy is dominated by wüstite, kirschsteinite/fayalite, and Ba-rich glass (Fig. 7a–c). In contrast, slag from FEXP-6 has lower FeO content (36–49 wt%) and higher sulfur content (1.9–2.3 wt%), resulting in rather unusual mineralogy, consisting of ferrosilite, kirschsteinite, hedenbergite, Ba-rich glass, and pyrrhotite (Fig. 7d–f).

Slag from FEXP-1 and FEXP-100 are compositionally similar, and some minor differences noticed are likely related to variations of ore preparation, resulting in slightly

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**Table 4** Bulk SEM–EDS analyses of early and late tap slag (given in wt% of oxide). Data is normalized

| Sample  | No. of areas | Na<sub>2</sub>O | MgO | Al<sub>2</sub>O<sub>3</sub> | SiO<sub>2</sub> | P<sub>2</sub>O<sub>5</sub> | SO<sub>3</sub> | K<sub>2</sub>O | CaO | TiO<sub>2</sub> | MnO | FeO | BaO | Total |
|---------|--------------|----------------|-----|-----------------|-------------|----------------|---------|--------|-----|-------------|-----|-----|-----|-------|
| FEXP-1  | Early tap    | n=4            | 0.1 | 1.1 | 1.8 | 39.1 | 0.3 | 0.8 | 1.5 | 6.6 | - | 0.2 | 48.5 | 100   |
|         | Late tap     | n=6            | 0.2 | 0.6 | 1.7 | 31.2 | 0.2 | 1.2 | 1.3 | 4.5 | 0.2 | - | 58.9 | 100   |
| FEXP-100| Early tap    | n=5            | 0.2 | 1.1 | 2.2 | 35.4 | 0.3 | 1.1 | 1.6 | 8.2 | - | 0.4 | 49.7 | 100   |
|         | Late tap     | n=4            | 0.1 | 0.8 | 1.2 | 27.5 | 0.1 | 1.9 | 1.2 | 4.9 | - | 0.3 | 62   | 100   |
| FEXP-5  | Early tap    | n=4            | 0.2 | 1   | 3   | 28.2 | 0.5 | 1.5 | 1.1 | 10.5 | - | - | 52.4 | 1.3  |
|         | Late tap     | n=3            | 0.1 | 0.8 | 1   | 18.9 | 0.3 | 1.7 | 0.7 | 6.6 | - | - | 67.7 | 1.7  |
| FEXP-6  | Early tap    | n=3            | 0.3 | 1.5 | 2.3 | 43.2 | 0.4 | 1.9 | 1.5 | 9   | - | - | 35.6 | 4.6  |
|         | Late tap     | n=4            | 0.2 | 0.9 | 1.7 | 35.8 | 0.3 | 2.3 | 1   | 8   | 0.1 | - | 46.1 | 3.3  |

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**Fig. 6** Typical microstructures by SEM BEI of tap slag from the early (ETS) and late (LTS) tapping sequences. Smelts a–c FEXP-1 and d–f FEXP-100. a ETS. Structure composed of fayalite (Fa) lath and interstitial glass (Gl). b Magnified area of a rectangle from a. c LTS. Structure composed of fayalite laths, wüstite (Wu) dendrites, and interstitial glass. d ETS. Structure composed of fayalite laths, magnetite/wüstite ex-solution (Mt/Wu), hedenbergite (Hed) micro-crystallites, interstitial glass, and pyrrhotite (Po). e Magnified area of a rectangle from d. f LTS. Structure composed of fayalite, wüstite, kirschsteinite (Kir), glass, and pyrrhotite.
higher concentrations of Fe, Ca, Al, and S in FEXP-100 slag. This is in turn reflected in lower amounts of fayalite and higher amounts of wüstite, glass, and/or micro-crystalline hedenbergite and pyrrhotite (Fig. 6a–f).

Variations in slag composition and mineralogy along with the smelting sequence (from early to late tapping) are best observed when comparing the relative concentrations of the major elements, FeO, SiO\textsubscript{2}, CaO, and Al\textsubscript{2}O\textsubscript{3} and the corresponding mineralogical phases in each experiment (Figs. 8 and 9). An increase in the amount of FeO and a decrease in the silica content and other lithophile elements (most notably, CaO and Al\textsubscript{2}O\textsubscript{3}) are observed from early to late tap slag, in all experiments. This is also reflected in the mineralogy, with an increase in the amount of wüstite in the slag towards the end of the smelt. These patterns are most pronounced in FEXP-5 and FEXP-100, while in FEXP-1 and FEXP-6, variations are slightly more random. Moreover, the mineralogy of late tap slag from FEXP-5 and FEXP-100 is dominated by wüstite in respect to the wüstite/magnetite mixture of the earlier slag reflecting changes in the smelting redox atmosphere.

It was a stated goal of the smelter to produce an iron-rich, low-viscosity slag, especially in the last phase of the smelt, to obtain a more easily forgeable product, even if at the expense of a better yield. To assess the success of this goal, the liquidus temperature and viscosity of produced slag were theoretically estimated by plotting the bulk chemical composition (based on the pXRF analysis) of early, middle, and late tap slag of each experiment on a ternary diagram (Fig. 10a). Since lime, silica, and iron oxide comprise more than 90 wt% of the slag, the ternary system FeO-SiO\textsubscript{2}-CaO(+)BaO is used.

Commonly, most analyses plot within the liquidus region of 1100–1250 °C, although, given the presence of other oxides, such as, MgO, Al\textsubscript{2}O\textsubscript{3}, K\textsubscript{2}O, and SO\textsubscript{3}, the real temperature might be slightly different. As previously observed, slag from FEXP-1 and FEXP-100 show very similar composition, reflecting the use of the same ore (IP-49). Slag from FEXP-5 plots in a slightly higher temperature region, while slag from FEXP-6 plots in a lower temperature region. Here, again, most noticeable are changes along the tapping sequence of each experiment—with the late tap slag generally plotting closer to the FeO triangular vertex, corresponding to a slightly higher liquidus temperature.

However, plotting the chemical composition on the isoviscosity diagrams indicates different patterns (Fig. 10b). Significantly, it appears that slag from FEXP-6 and early tap slag of FEXP-1 and FEXP-100 have higher viscosity than late tap slag from the same experiments. This is supported by empirical observations made during smelting. For example, in FEXP-1, a slower slag flow was observed at the
Fig. 8 Variation of Wt% of FeO, SiO₂, Al₂O₃, and CaO throughout the slag tapping sequence (early, middle, and late tap slag) in four undertaken experiments.

Fig. 9 Change in the phase composition of the slag throughout the tapping sequence, from early to late tap slag. *For the early tap slags FEXP-100 and FEXP-5, orange designates wüstite and magnetite ex-solution. The pyrrhotite is not included in the diagram due to the small amount of this phase causing difficulties for accurate quantification.
beginning of slag tapping, causing the smelter to increase the ore-to-charcoal ratio and promote a slightly faster combustion rate, thus resulting in a decrease in slag viscosity (see “The smelting protocol” and “Description of smelting experiments,” Table 3, and Supplementary File S1).

**Furnace slag**

Furnace slag comprises less than 10% of the total slag mass in each experiment. Based on its appearance and microstructure, it is clearly composed of partially or fully reduced ore fragments that have failed to coalesce into the bloom. These fragments preserve the original euhedral shape of the ore grains, often exhibiting a reduced metallic outline and a wüstite core. Thus, reflecting the vector of reduction from the periphery inwards (Fig. 11a, c). Similar structures were also observed in laboratory smelting experiments using the same ores (Stepanov et al. 2020a: Fig. 3a, b). In some wüstite-rich areas, large spherical pores are present, which are likely the result of intensive degassing during reduction (Fig. 11b). Nital etching performed on metallic grains reveals ferritic composition, consistent with etched structures observed in the bloom (see “Analysis of metal: blooms and bars”).

**Analysis of metal: blooms and bars**

Four well-consolidated blooms were produced. All blooms share similar morphologies, generally convex at the bottom and slightly concave at their top, apart from bloom FEXP-5, which has a flatter shape. A similar pattern of pore and slag inclusion distribution was observed; the bottom contains more pores and slag, while the central and top parts are generally cleaner and denser. The cross sections studied from each bloom are presented in Fig. 12.
Comparable patterns were also noted by others (e.g., Tylecote et al. 1971; Rehder 2000: p. 125).

**Microstructure**

Microstructures of the studied blooms are presented in Fig. 13. They are predominantly composed of low-carbon soft ferritic iron. FEXP-1 bloom is somewhat different, containing some carburized areas of c. 0.2–0.3 wt% C and more rarely of higher carbon content in the range of 0.4–0.8 wt% C (Fig. 13a–c). The carburization of bloom FEXP-1 mostly happened near the surface and at its core, but not at the bottom where the structure is ferritic (Fig. 13a). Similar carburized distributions were produced by Tylecote, which he explained by an increase of reducing conditions towards the end of the smelt (Tylecote et al. 1971).

Pieces purposely broken off the bloom by the smelter before hot cutting reveal a composition of 60–80% metal (surface area), dominated by ferritic iron, occasionally carburized areas near the surface, and 20–40% slag (not shown), similar to the bottom (or peripheral) part of the bloom.

The microstructures of the forged bars are unsurprisingly similar to their parent bloom (Fig. 14). Bars are generally composed of ferritic iron, while only FEXP-1 bar reveals a small patch (c.3 mm) of mild steel (0.1–0.3%C) near one of its surfaces. Such limited carburization is likely the result of the originally low-carbon content of the bloom, and possibly additional decarburization during hot forging.

**Chemical composition**

The chemical composition of the blooms and associated bars was studied using SEM–EDS. Multiple areas of ca. 0.8-mm² average size were measured for each sample. Apart from metallic iron, only two elements were detectable: sulfur and phosphorous (Table 5).

Sulfur is present in the range of 0.1–1.2 wt% and often occurs as pure iron sulfide, close in composition to pyrrhotite, or as the eutectic of wüstite and iron sulfide, due to the mutual solubility of these phases at high temperatures (Fig. 14e, f). Metallographic investigation shows that sulfide phases precipitate at the boundaries of former austenite grains (Figs. 13h and 14b). The distribution of sulfides in the metal is heterogeneous; however, a slight enrichment is observed at the bottom part of each bloom probably due to sinking of sulfide in the metal in a liquation effect (Tylecote et al. 1971).

Greater iron sulfide content was identified for FEXP-6 and FEXP-100 blooms and bars in comparison to FEXP-1 and FEXP-5. This evidence supports empirical observations made during forging of FEXP-6 and FEXP-100 metal bars, where both showed a tendency to break and split, rendering their forging difficult, resulting in an increased metal loss. This observation is in accordance with the notion that sulfur is particularly harmful impurity for iron alloys, causing brittleness of the metal at forging temperatures in the range of 800–1000 °C. Nevertheless, it is interesting to note that a slight decrease in sulfur content happened from bloom to bar of FEXP-6 and FEXP-100, perhaps due to partial elimination of sulfur in the oxidizing atmosphere of the forging hearth, or perhaps due the highest sulfur portions of the bar breaking off during forging (Table 5).

Two main sources can account for the sulfur and phosphorus content—ore and charcoal. The ores used in the experiments are geologically associated with the sulfate minerals gypsum (CaSO₄) and barite (BaSO₄). SO₄ content measured by pXRF in the roasted ores is between 1.8 and 4.8 wt%. The sulfur content of an ashed charcoal sample was found to have an average of 8 wt% SO₄ (ca. 40 g of charcoal was burned in a graphite crucible at 850 °C for 3 h, producing ca. 1 g of ash, see Table 2). However, based on the analysis of standard materials, this is likely an overestimation. In any case, it is hard to estimate how much of the original sulfur content in the ore and/or charcoal contributed to the metal or was released as volatile matter during smelting. The volatilization of the sulfur was observed empirically as a bright yellow deposit on the top bed of charcoal and on
the upper inner walls of the furnace. Sauder had observed similar effect when smelting with both sulfur-rich ores or sulfur-rich charcoal. In this case, we may be looking at the accumulative effect originating from both fuel and ore.

**Indentation Vickers hardness**

Results of indentation hardness measurements generally accord with the chemical and microstructure composition of the blooms and bars and confirm the softness of the produced metal (Table 5). Blooms from experiments FEXP-5 and FEXP-100 have particularly low hardness (84–103 HV), and despite some limited carburization observed in FEXP-1 bloom, its average hardness is similar to the others. In contrast, FEXP-6 bloom has slightly higher hardness (139 HV), likely, due to higher phosphorus content (0.2 wt%).

Interestingly, an increase in hardness by about 25–40 HV was observed in bars from experiments 1, 100, and 5 relative to their blooms, which is likely the result of cold forging. However, the opposite was observed for FEXP-6. This may be linked to a lower phosphorous content in the bar compared to the bloom, as the metal in both cases contains no carbon. Similarly, to the behavior of sulfur, the decrease in phosphorous content could have been caused by forging under strong hot oxidation conditions, such as those created, when metal is heated up to “glowing white” color (around 1100–1300 °C). Both the cold hammering (below 700 °C, dull red color) and the extended annealing of metal at or above the temperatures of “white heat” were necessary to overcome the severe brittleness of metal which occurs in the regular annealing range (800–1100 °C, red–orange color) caused by the sulfur impurities.

**Discussion**

The smelting experiments described here were undertaken as part of an ongoing research focusing on the advent of iron technology during the Iron Age of the Southern Levant.

In the past decade or so, archaeological evidence for the bloomery process has been identified at major Iron Age sites...
in the Southern Levant, for example, at Hazor, Megiddo, Rehov, Ashkelon, Beth-Shemesh, and Tell es-Safi-Gath in Israel, and Tell Hammeh, in Jordan (Veldhuijzen and Rehren 2007; Eliyahu-Behar et al. 2012, 2013; Yahalom-Mack et al. 2014, 2017; Erb-Satullo and Walton 2017; Erb-Satullo 2019; Workman et al. 2020). In all these sites, iron production was dated not earlier than the Iron Age IIA, the late tenth–early ninth centuries BCE (Fig. 1), and was often associated with administrative buildings, suggesting that it had been controlled by a central authority (Yahalom-Mack and Eliyahu-Behar 2015). Production remains attest to a range of activities and hint to variable forms of technological practice. Several types of production waste were uncovered, such as iron slag (tapped, furnace bottoms, and slag cakes), hammerscales, and a range of “technical ceramics”, mainly tuyeres and vitrified clay lining. The appearance of production debris raised an intriguing question about the provenance of ore resources. Which iron ore deposits were exploited? Was there one or more available resources? And what networks of acquisition and trade were in place in the early periods of ironworking?

Thus, although the newly discovered production sites introduced new and pertinent data to the discussions surrounding the transition from bronze to iron use, the question of provenance remained unresolved. Provenancing iron is exceptionally important especially when the metal

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**Fig. 13** Nital-etched OM microstructures of produced blooms. a–d FEXP-1. Overview photo with mapped carburized areas. b Carbon-rich area (0.4–0.7% C) near the top surface of the bloom. c Mildly carburized (0.2%) area. d Ferritic structure. Ferritic area of the poorly consolidated bottom part of the bloom at the contact with slag composed of fayalite and glass. e FEXP-100. Ferritic structure with pyrrhotite (Po) inclusions and slag composed of fayalite (Fa) and wüstite (Wu). f FEXP-5. Bottom part of the bloom at the contact with slag composed of wüstite and fayalite. g, h FEXP-6. g Ferritic structure with pyrrhotite inclusions and slag composed of fayalite, glass, and pyrrhotite. h Ferritic structure with inclusions of iron sulfide in place at grain—boundaries of former austenite.
Fig. 14 Metal structures from four bars. a–d Nital-etched. All are composed of ferrite. a FEXP-1. Structure with slag inclusions (SI) characterized by different FeO content. One SI is composed of fayalite (Fa) and glass (Gl), and the other SI is composed of fayalite and wüstite (Wu). b FEXP-100. Structure with intergranular areas occupied by inclusions composed of wüstite and pyrrhotite (Po). c FEXP-5. Structure with some slag inclusions composed of wüstite, glass, and pyrrhotite. d FEXP-6. Structure near the major crack of the bar where hot oxidation has developed. e–f SEM, BEI. Microstructure of pyrrhotite from the metal of bar FEXP-6. e Pyrrhotite eutectics. f FEXP-6. Inclusion of pyrrhotite exsolved with wüstite.

Table 5 Chemical composition (SEM–EDS) and hardness measurements of metal: blooms and bars. For SEM–EDS, multiple areas of average size 0.8 mm² were analyzed. Individual analyses are provided in Tables S5 and S6.

| FEXP no | Metal | No. of analyses | P (wt%) | S (wt%) | Vickers hardness, HV |
|---------|-------|----------------|---------|---------|----------------------|
| FEXP-1  | Bloom| AVG (n = 18) | <0.1    | 0.1     | AVG (n = 17) 103 |
|         |       | STD           | -       | 0.2     | STD 29               |
|         | Bar   | AVG (n = 8)   | 0.2     | 0.1     | AVG (n = 9) 142      |
|         |       | STD           | 0.1     | 0.1     | STD 5                |
| FEXP-100| Bloom| AVG (n = 6)   | <0.1    | 0.4     | AVG (n = 5) 89       |
|         |       | STD           | -       | 0.2     | STD 3                |
|         | Bar   | AVG (n = 11)  | 0.1     | 0.3     | AVG (n = 7) 114      |
|         |       | STD           | 0.0     | 0.2     | STD 10               |
| FEXP-5  | Bloom| AVG (n = 6)   | 0.1     | 0.2     | AVG (n = 5) 84       |
|         |       | STD           | 0.1     | 0.3     | STD 10               |
|         | Bar   | AVG (n = 2)   | <0.1    | 0.2     | AVG (n = 5) 102      |
|         |       | STD           | -       | -       | STD 5                |
| FEXP-6  | Bloom| AVG (n = 19)  | 0.2     | 1.2     | AVG (n = 17) 139     |
|         |       | STD           | 0.0     | 0.6     | STD 13               |
|         | Bar   | AVG (n = 9)   | 0.1     | 0.6     | AVG (n = 5) 113      |
|         |       | STD           | 0.0     | 0.3     | STD 4                |
becomes prevalent in daily use. In the Southern Levant, this occurred concurrently with the rise of Iron Age territorial kingdoms, such as Judah and Israel (Bunimovitz and Lederman 2012; Yahalom-Mack and Eliyahu-Behar 2015). The control over the iron ore resources was likely a strategic military and economic advantage at that time.

To the current date, the only known archaeological evidence of iron ore exploitation within the Southern Levant derives from the Mugharet el-Wardeh ore deposit mentioned earlier. Nevertheless, a range of smaller deposits, geologically and mineralogically similar to Wardeh ores, exists in the region, notably in the Negev desert.

Thus, a major aim of the current experiments, apart from producing the necessary materials for developing the osmium (Os IC) provenance methodology, was to explore the compatibility of the Negev ores with the bloomery smelting process.

Hence, four bloomery smelting experiments were undertaken by an experienced smelter/blacksmith using a technique that he developed and perfected over more than 20 years. The blacksmith exercised a high degree of control over the smelts, constantly making decisions influencing the reduction process, resulting in well-consolidated blooms and bars. The extensive experience of Sauder with both smelting and smithing bloomery iron provided the rationale behind the technical decisions he implemented during the smelt. Systematic recording and sampling of the smelting and subsequent smithing products allowed us to trace the smelter’s technical steps and their impact on the resulting slag and bloom. These have broader ramifications for understanding past human choices and strategies implemented in iron-making technologies.

Many parameters of the smelting process, including the choice of raw materials, their preparation procedures, and the furnace design, were pre-set by the aims of the project and the head smelter and were not tested for variability during the present experiments.

One important variable introduced in these experiments is the ore source. Three localities within one geophysical and geological setting were utilized allowing us to test the effect of compositional variability within these local ores. These slight variations were overcome by the smelter’s high ability to control the reduction process by adjusting its parameters which is demonstrated by the very consistent results obtained. The main parameters consciously and directly manipulated by the smelter were charging and airflow rate, ore-to-charcoal ratio, and the slag tapping regime. These, in turn, influenced the furnace temperature and slag composition. Notably, the slag, judging by its appearance alone, served as an indirect marker to control the process. The impact of these deliberate choices and manipulations is reflected in the composition of the final products and are discussed below.

### Charging and airflow rate, temperature, and yield

Charging rate is one of the key parameters controlling the smelting process. Essentially, this is the rate at which the charcoal burns. Here it was empirically controlled by adjusting the time needed to consume 2 kg of ore and 2 kg of charcoal. This in turn was manipulated by adjusting the amount of air introduced into the furnace by the blowing apparatus, a stronger airflow obviously producing a faster charging rate. Moreover, both charging and airflow rates are directly linked to the furnace temperature and manipulation of one of these parameters will affect the other. The significance of the air blowing rate in iron smelting has been previously noted (Sauder and Williams 2002; Crew and Charlton 2007).

It must be stressed that no special measurement of air flow was carried out during the process and the impact of this parameter can be only indirectly assessed through the change in the charging rate.

According to Sauder’s experience, a 10–12-min rate using the shaft furnace described here is most likely to produce a soft, consolidated bloom with sufficiently high yield, which is a reliable starting point for work with an unfamiliar ore. Controlling the airflow rate and temperature to assure the desired charging rate is an intuitive skill, which depends on many variables: the blowing apparatus, type of charcoal and ore used, and even the weather and elevation. It can be mastered through repetitive trial and error, relying on senses and empirical observations, such as sound, flame color, and the color glimpsed through the tuyère.

Thermocouple measurements taken during our experiments show a consistent profile throughout the smelt, with a gradual temperature increase from initial ore charging, peaking just before first slag tapping, and decreasing immediately thereafter until the end of the smelt. The ore-charcoal charging time log was also very consistent, which, along with the production of a well-consolidated bloom, indicates that the smelter effectively mastered control of all three parameters - charging, airflow rate, and temperature - in this type of furnace. It should be emphasized that thermocouples were only used to monitor the smelter’s decisions, while the smelter himself did not require thermocouples to masterfully perform the experiments, relying only on his senses and experience. While three of our experiments were conducted using a charging rate of c. 10–12 min, only during experiment no. 4 (FEXP-100), this parameter was increased up to 12–15 min. The purpose of this change was to create conditions favoring the production of a steely bloom. Given that the same ore (Zavar, IP-49) was smelted in FEXP-1 and FEXP-100, we were able to observe the effects of adjusting the airflow rate, and consequently the charging rate, on the final metal product.

Based on his previous empirical observations, Sauder’s original assumption was that a slower charging rate in a
given type of furnace promotes higher yield and higher carbon content in the bloom. However, the metallographic and hardness investigation of FEXP-100 bloom revealed a pure ferritic composition, in contrast to the heterogeneously carburized bloom formed in FEXP-1. Thus, we could not confirm the proposed correlation between charging rate and carbon content. In contrast, rather the opposite can be deduced from our experiments: the faster charging rate linearly correlates with the stronger airflow promoting higher temperatures and production of a more carburized metal, as also supported by the results of previous experiments (Sauder and Williams 2002; Crew and Charlton 2007; Tylecote et al. 1971; Thiele and Török 2019).

Nevertheless, under some conditions too high airflow rate can apparently have a negative impact on the outcome of the smelt, such as significant melting of the furnace wall and/or too high reducing conditions and too viscous slag, consisting of high amount of glass and fayalite and/or pyroxene/melilite. This results in poor consolidation of the metal and its loss in the slag (Leroy et al. 2020; Leroy et al. 2015: p. 340, Table 2; Thiele and Török 2019). Furthermore, the high airflow promotes greater reducibility of undesirable impurities such as phosphorous or sulfur, in case these are present in the system, which was also relevant for our raw materials.

Although a slower charging rate did not produce the desired steely bloom, it resulted in a higher yield: 27% in FEXP-100 vs. 17% in FEXP-1. Obtaining a higher yield can possibly be explained by the longer residence time the ore spent descending the furnace shaft before reaching the tuyère level, as it extends the reaction of the ore with the furnace gases promoting a more efficient process (Rehder 2000: 93). It is likely that the shorter residence time in FEXP-1 did not allow for complete reduction, as was indicated by the larger amount of partly reacted furnace slag and the presence of reduced metallic filaments in the tap slag of FEXP-1. According to Sauder several factors can contribute to increased metal yield including: the length of the smelt, the use of high charcoal to ore ratios, and the smaller ore particle size. Furthermore, practical experience shows that higher yield can in fact be less important than a good forgeability of the produced metal, given the significant time and efforts invested in bloom forging to transform the nonconsolidated metal into a working tool. Sauder points out the need to consider not only the forgeability of the metal itself, but also the viscosity of the slag included within it, as it affects the forgeability of the metal. Slag depleted of iron oxide causes many difficulties in forging.

Thus, the most frequently used measure for efficiency and yield, based on quantification of the amount of iron oxide remaining in the slag, may not always be relevant from the blacksmith’s practical perspective. This idea was also suggested by Rehren et al. (2007). In fact, the smaller bloom produced in FEXP-1 appears to be much more valuable than the larger bloom from FEXP-100 due to the better forgeability of the former.

The importance of malleable, easily forgeable iron can also be tracked in the archaeological record. For instance, it has been suggested by Crew (2013) that the common practice of narrowing the ends of iron semi-products (iron bars/billets) demonstrated the good mechanical properties of the metal. Evidence for this practice has been shown as early as the Iron Age in the Near East and Europe, for example, the bipyramidal ingots (e.g., from Assyria or Celtic Europe) and the spoon-shaped bars with a flat extremity from Norway (Curtis et al. 1979; Pleiner 2006: p. 27, Fig. 6; Bauvais et al. 2018; Buchwald 2005: p. 151, Fig. 154). Indeed, as confirmed by the forging of FEXP-6 bloom, the thinning of a metal ingot containing harmful impurities such as S (and probably P or As), excessive or uneven carbon content, or iron-poor slag inevitably reveals noticeable cracks downgrading the value of such metal.

Redox conditions and slag properties

The redox atmosphere, defined by CO/CO₂ ratio inside the furnace, is another key parameter of the smelting process and can be influenced by varying the ore-charcoal ratio during each charging episode. Sauder relied on operating the furnace under rather low charcoal to ore ratio of 1:1, resulting in a moderately reducing atmosphere. The efficiency of this practice has been shown here (and previously) by the production of a soft and sufficiently forgeable metal. In contrast, had the charcoal-ore ratio been increased, the dynamics of the smelting process would be altered, with the likely outcome of a more heterogeneously carburized bloom and a fayalite-glassy slag. All these aspects would complicate the subsequent forging process, extending its length and fuel consumption, increasing the amount of metal loss, therefore decreasing yield and resulting in a generally less efficient process.

We previously mentioned that after first tapping, the ore to charcoal ratio was intentionally increased by up to 1.25:1 per charge while the charging rate was simultaneously sped up by 1–3 min. The rationale behind these changes was to deliberately offset the increase of CO/CO₂ ratio towards the end of the smelt to avoid carburization of the bloom. This technique has been previously demonstrated (Tylecote et al. 1971). Whether a similar practice was performed in the past would generally be difficult to identify. However, variability within the blocks of pit-slag produced at Kinanisi site of Buganda Kingdom (eighteenth–nineteenth c. AD) of Uganda may have resulted from a similar practice (Humphris et al. 2009).

Redox environment is also defined by the furnace temperature. The higher the temperature, the more CO reducing gas is generated (Rehder 2000). Our first experiment
FEXP-5 and FEXP-100 behaved as such; higher amounts of silicate—glassy phases (Ji et al. 1997). The slag produced in and fayalite, while more viscous slag is dominated by other low-viscosity slag plots in the iron-rich region of wüstite SiO$_2$-FeO system. When plotting this on ternary diagrams, chemical composition that can be described within the CaO-volcanic glass system, enabling efficient bloom consolidation, with a first and foremost manifested in the properties of the slag needed to verify this assumption.

Overall, iron smelting under moderately reducing conditions, as demonstrated here, bear several advantages:

1) Avoiding overheating and melting of furnace walls, allowing repeated use.
2) Economizing on fuel due to lower charcoal to ore ratios.
3) Decreasing the passage of harmful impurities, such as P, S, As, and Cu, from the ore to the metallic iron (as previously noted in experimental studies; Crew and Charlton 2007; Sauder 2013; Sauder Forthcoming).
4) Maintaining an iron-rich slag chemistry, with all the advantages noted above.

All these factors seem relevant for the geological environment setting of the Southern Levant, which is characterized by the abundance of poor refractory calcareous clays (mostly smectite of the 2:1 clay family), aridity, and iron ore sources generally associated with evaporite (S-bearing) deposits (Dill et al. 2010; Grosz et al. 2006).

The association of the local iron ores with sulfur impurities deserves further attention. Apart from reducing less sulfur, smelting under moderately reducing conditions promotes admixture of oxygen into the iron sulfide eutectics, appearing as ex-solution of wüstite and iron sulfide (FeS). An ex-solution mixture composed of c. 68 wt% FeO and c. 32 wt% FeS liquefies already at 918 °C, which is lower than the melting temperature of 988 °C of the Fe-FeS eutectic (Shishin and Decterov 2012: Fig. 5). This can potentially limit formation of cracks in the metal while forging under the temperatures of white heat (1000–1200 °C). More empirical smelting and forging experiments with S-rich ores are needed to verify this assumption.

All the above-described decisions and operations are first and foremost manifested in the properties of the slag formed. The present experiments yielded a relatively low-viscosity slag, enabling efficient bloom consolidation, with a chemical composition that can be described within the CaO-SiO$_2$-FeO system. When plotting this on ternary diagrams, low-viscosity slag plots in the iron-rich region of wüstite and fayalite, while more viscous slag is dominated by other silicate—glassy phases (Ji et al. 1997). The slag produced in FEXP-5 and FEXP-100 behaved as such; higher amounts of wüstite in the slag resulted in lower viscosity and vice versa, as was empirically observed during smelting.

Changes described in the sections above, are reflected in the chemistry and viscosity of the obtained slag. This was especially observable in the charging rate, the increased ore-to-charcoal ratio after first slag tapping, and the consistency of slag flow in the second half of the smelt. Generally, late tap slag, in contrast to early tap slag of the same smelt, is more iron-rich and less enriched in major lithophile elements (Si, Ca, Al). These results should be born in mind when chemical analyses are employed on archaeological slag. As the chemical composition of a single or even several fragments of slag are not always representative of the full slag sequence of a smelt. Moreover, archaeological slag often appears in piles, or even mounds, resulting in the mixing of chemical signatures from one smelt and between consecutive operations, disguising some of the more interesting nuances associated with choices and decision-making made by the craftspeople—the ancient smelters.

Bloomery iron smelting controlled by iron-rich, low-viscosity slag was evidently a practice of past societies, including those of the ancient Southern Levant (Veldhuijzen and Rehren 2007; Amri 2008) and other geographical areas (Park and Rehren 2011). It may well be that some of these practices relate to availability and choice of iron ores, such as the use of rich iron ores of the Southern Levant, for example the Mugharet el-Wardeh ore deposits (Amri 2008). But it likely also represents deliberate choices made by the ancient smelters in order to optimize the production process.

Summary
One of the main observations of the present experiments is that through adjustments of airflow rate, charging rate, and ore-to-charcoal ratio an experienced smelter can control the furnace temperature, redox conditions, and slag characteristics, in order to obtain a metal product of a desirable quality. This is particularly the case when the work is performed in a habitual setting with familiar ore, furnace, and blowing apparatus. Small variations seem inevitable, due to challenges assuring a consistent air rate via bellows and when dealing with new variables such as a new ore type or the natural heterogeneity of an ore deposit.

As mentioned, the technique used in the present experiments was perfected by the smelter over many years. However, it needs to be emphasized that it is not based on a particular ancient socio-technological context, nor on one related to the Southern Levant. Obviously, if evidence enabling a full reconstruction of the type of smelting furnace/installation would exist, the smelting technique adopted for these experiments would have been revised accordingly (for the evidence at hand, see Workman et al. 2021).
Apart from the parameters of the smelting process discussed in the present study, various other variables and factors could affect the outcome of a smelt. These include technical parameters such as the inclination angle of the tuyère, the size of the slag tapping channel, and the timing of the slag tapping and bloom extraction. However, the physiochemical processes underlying these variables are less apparent and were not tested in the present framework.

It is agreed that the required metal product, whether malleable soft iron or various grades of steel, would influence the process protocol and decisions made by the ancient smelters. While in the present experiments we aimed at producing soft iron bloom, numerous archaeological and ethnographic evidence exist favoring the deliberate production of steel (Furger 2019; David et al. 1989).

The data presented here has some important implications regarding the interpretation of smelting strategies in antiquity. While the analysis of tap slag (usually from slag mounds) may give information on the general smelting protocol, the lack of contextual information (i.e., early, middle, or late tapping) prevents the identification of subtle variations and decisions taken at mid-process. By contrast, if this information is available, which is more often the case for furnaces with large blocks of pit-slag (Pleiner 2000: 149; Humphris et al. 2009; Vodyasov et al. 2021), it provides a major insight into the past smelting strategies and technical decisions made by the ancient smelters.

Conclusions

Four successful smelting experiments using three similar iron ore deposits from the Negev region of modern Israel, were carried out in a bloomery furnace. Each of these smelts yielded a well-consolidated, forgeable bloom and a low-viscosity, iron-rich slag. The blooms were forged into bars, thus completing the full production cycle. In doing so, we fulfilled one of the major aims of these experiments: we were able to show that the three ores chosen for the experiments were suitable for iron production using the bloomery process.

The success of these experiments was highly dependent on the smelter’s experience, thoughtful choices, and decisions taken during the process. Fine-tuning of the smelting protocol for each specific ore type was necessary to optimize the outcome of each experiment. This was especially evident as the smelter corrected and adjusted his smelting protocol based on the results of the first experiment (FEXP-1). These fine adjustments were reflected in the chemical composition and physical properties of the final products—iron and slag.

These experiments also emphasize the benefits of smelting under moderately reducing conditions for limiting the passage of harmful impurities, especially sulfur, into the metal. These observations may be particularly important for the geological setting of the Southern Levant, for limiting the passage of harmful impurities, especially sulfur, into the metal.

Finally, performing these experiments highlighted the benefits of verifying archaeological inferences with a thorough program of experiment and analysis. Such work can improve our ability to envisage and decode the choices and decisions taken by smelters and blacksmiths of past societies, even if it encourages certain humbleness about what we can discern from archaeometallurgical remains.

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Declarations

Conflict of interest The authors declare no competing interests.

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