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research article

Mechanical Properties of Medieval Bloomery Iron Materials - Comparative Tensile and Charpy-tests on Bloomery Iron Samples and S235JRG2

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Abstract

Ductility, toughness and strength of medieval bloomery iron materials were highly important mechanical properties, strongly affected by their microstructure and chemical composition. An attempt was made to characterize the most important mechanical properties of representative samples of main bloomery iron materials extracted in smelting experiments and compare them to the well know reference modern steel of S235JRG2. It was confirmed that notching and the stress concentration effect of slag inclusions strongly decrease all the characteristic values of ductility and toughness of bloomery iron materials. Typical medieval bloomery P-iron is a brittle material with almost zero or very low characteristic values of ductility and toughness but revealed similarly high strength as hardened and tempered bloomery steel.

Keywords

Bloomery iron, phosphoric iron, mechanical properties, mechanical testing

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1 Introduction

Based on numerous investigations carried out on both archaeological and experimentally yielded bloomery iron artefacts, it can be stated that the microstructure and the chemical composition of bloomery irons and steels differ from those of modern steels. Since microstructure and chemical composition as well as purity strongly affect mechanical properties, these must differ from those of modern steels as well. Therefore, even for well-trained material engineers it might have been difficult to reliably assess mechanical properties (mainly toughness and strength) of such historic objects as swords, which in addition often reveal high complexity in terms of materials and their various combinations used within the manufacture of a single object.

Naturally, a good understanding of materials, their structures and corresponding effect on mechanical properties would enable us to understand the single objects themselves far better. For that reason the issue of mechanical properties of bloomery irons and steels is certainly worthy of investigation.

1.1 Medieval bloomery iron alloys

It is well known that archaeological iron was produced in a one-step bloomery (direct) process, in which molten slag could not be fully separated from the solid, spongy iron bloom. Bloomery iron therefore always contains much more and bigger slag inclusions than modern steels. The bulk chemical composition of the bloomery iron could only be controlled during the smelting process in the bloomery furnace. It was deduced by smelting experiments that carbon content could be well controlled by air supply and by the ratio of charcoal/iron ore [[1,](#page-4-0)[2](#page-4-1), etc.]. The hardening of steels was also a well-known technique.

Besides carbon, phosphorus was the second most common alloying element. Phosphoric iron (P-iron) with a wide range of average phosphorus content 0.4-1.4wt% [\[6\]](#page-4-2) was deliberately used in medieval metal-working for aesthetic purposes in the manufacture of ostentatious blades, e.g. pattern-welded ones [\[3](#page-4-3)-[5,](#page-4-4) etc.]. On the other hand, P- iron can appear in medieval tools and weapons (with an average content of P up to 0.3- 0.85wt%) unintentionally either as a result of the lack of non

Table 1 Samples for mechanical testing

Sample	Nr of samples	Description	Micro-structure	Chemical composition	
				C wt%	$P wt\%$
$Fe-0.05C(n)$	5	bloomery wrought iron in normalised state	ferritic with little pearlite	0.05 ± 0.02	
$Fe-0.21C-1.05P(n)$	3	bloomery phosphoric iron in normalised state	inhomogeneous, ferritic with ferrite- pearlitic layers	0.21 ± 0.06	1.05 ± 0.02
Fe-0.64C (n)	$\overline{4}$	bloomery steel in normalised state	inhomogeneous, pearlite with proeutectoid ferrite	0.64 ± 0.03	
$Fe-0.64C$ (h-t)	4	bloomery steel in hardened and tempered state	inhomogeneous, tempered martensite	0.64 ± 0.02	
S235JRG2(n)	5	reference modern steel in normalised state	ferritic with little pearlite	0.17 ± 0.00	

P-iron or because of the use of unsorted heterogeneous or scrap iron [[7](#page-4-5)[,8](#page-4-6)]. P content could be controlled by charging limestone into the furnace [\[9\]](#page-4-7).

Elevated concentration of arsenic could also be detected in a few iron artefacts. Although arsenic also decreases ductility and toughness, it appears only in particular bog iron ores [\[10\].](#page-4-8) Other elements such as nickel, cobalt or copper are often present in bloomery iron but only in traces or increased concentration in forge welding lines so they have no significant effect on mechanical properties [\[11\]](#page-4-9). Three main sorts of bloomery iron/ steel were used in the manufacture of medieval iron objects, usually intentionally for their specific properties. Using archaeometallurgical terms, these are wrought iron (not hardenable carbon steel), steel (hardenable carbon steel) and phosphoric iron (low carbon steel containing more than $0.1wt\%$ of P [\[12\]](#page-4-10)).

1.2 Mechanical properties of bloomery iron

Since modern steels do not contain a high level of slag inclusions or P, there is relatively little work carried out on mechanical properties of steels with similar microstructural chemical properties to medieval bloomery iron. From the perspective of archaeometallurgy, the most important research on the mechanical properties of phosphoric iron was carried out by Stewart et al. [\[13\],](#page-4-11) who concluded that increasing the amount of phosphorus decrease the haracteristic values of ductility and toughness, but increases the strength of the iron. Already Stewart et al [\[13\]](#page-4-11) pointed out but Sahoo and Balasubramaniam [\[14\]](#page-4-12) explained in detail that under special heat treatment of P-iron with 0.25-0.5wt% of P it is possible to maintain ductility and toughness by avoiding P segregation to the grain boundaries, which is responsible for brittleness. The samples were not prepared with the bloomery process but P-alloys were melted in the both cases [[13](#page-4-11),[14\]](#page-4-12).

In this paper, one can find the summarized results of the attempt to set the most important characteristic values of mechanical properties of representative samples of main bloomery iron materials and compare them to the well know reference modern steel of S235JRG2.

2 Samples and methods

In order to be historically accurate, we extracted iron blooms by smelting bog iron ores in the copies of the excavated 10th century Fajszi-type embedded furnaces [\[15\]](#page-4-13) (about smelting experiments cf. details in [\[16\]\)](#page-4-14). Bloomery phosphoric iron (Fe-0.21C-1.05P) was smelted using phosphoric bog iron ore from Inner-Somogy (South-West Hungary). Bloomery wrought iron (Fe-0.05C) was smelted using the same ore, charging limestone into the furnace to decrease the P content of the bloom. Bloomery steel (Fe-0.64C) was produced by the re-smelting and carburizing of pieces of wrought iron. The carbon content of the steel bloom was homogenised by multiple folding and forge-welding. S235JRG2 modern steel was used as reference material [\(Table 1\)](#page-2-0).

Charpy-samples were made of bloomery iron materials and reference S235JRG2 steel, surface-milled and provided with a "V" profile notch. Charpy-tests were conducted according to the International Standard ISO 148- 2:2008(E) (with the exception of the specimen being 10mm longer) at room temperature. All the samples were kept in normalised (air cooled) state apart from four steel sample, which were water-quenched from 900°C and tempered in 300°C for 60 minutes.

Both halves from the specimens broken in Charpy-tests were heated up to 1250°C embedded in cast iron swarf (to prevent the oxidation of the surfaces) in a heat-treating furnace equipped with silicon-carbide rods and subsequently rolled by a rolling mill in four steps into flat specimens of 3 mm thickness. Specimens for the tensile tests were milled out of the flat-rolled specimens. Tensile tests were conducted according to the International Standard ISO 6892-1:2009(E) (except for the specimens' dimensions). Crosshead speed was 20mm/min. Heat treatment of the samples was the same as in the Charpy-test.

The microstructure and chemical composition of the tested bloomery iron materials was established after tensile tests on metallographic cross-sections taken close to the failure surface after fracture ([Fig. 1](#page-3-0)). Carbon content was calculated by means of image analysis while P content was measured by SEM-EDS

Fig. 1 Metallographic macro-photographs of cross-sections of representative specimens of each type

method [\(Table 1\)](#page-2-0). With regard to previously conducted analyses [\[6\]](#page-4-2) it can be stated that all the tested bloomery materials rank among the basic types of material used in the past and all of them are representative from this point of view.

3 Results and discussion

Elongation (Δl (mm)) – force (F (kN)) curves plotted from tensile tests are presented in [Fig. 2.](#page-3-1) Tensile curves of all samples excluding Fe-0.21C-1.05P (n) samples represent ductile materials with the ability to yield and necking, however plastic deformation was much lower in bloomery samples than in reference modern steel. P-iron samples have linear tensile curves typical for brittle materials without any plastic deformation and necking and with almost the same yield- and ultimate tensile strength.

Fig. 2 Tensile diagram of representative samples of each type

The characteristic values of ductility and toughness (impact energy (KV (J)), percentage elongation after fracture $(A \binom{0}{0})$, percentage reduction of area $(Z \, (%)$ and absorbed specific fracture energy (W_c (J/cm3))) resulted from Charpy- and calculated from tensile tests are summarized in [Fig. 3](#page-4-15). It can be stated that Fe-0.05C (n) samples with a similar chemical composition to the reference S235JRG2 ones dispose of only half or quarter of the characteristic values of ductility and toughness. This is caused by the microstructural difference, i.e. the presence of slag inclusions, which provide a notching and stress concentration effect. The average area covered by slag inclusions is 6.5% in case of Fe-0.05C (n) samples while 0.3% in case of S235JRG2 ones (calculated using a photo editing software, Photoshop CS3). The slag inclusions in Fe-0.64C (n) (2.3%) and in Fe-0.64C (h-t) (2.1%) samples also strongly decrease the characteristic values of ductility and toughness (fine grained Fe-0.64C (h-t) samples have higher values). Characteristic values of ductility and toughness of Fe-0.21C-1.05P (n) samples are almost zero due to the slag inclusions (6.8%) and the high P content.

Fig. 3 Characteristic values of mechanical properties of tested materials

Here we can point out the difference between the results of the research carried out by Stewart et al. [13] and Balasubramaniam et al. [\[14\]](#page-4-12). In their experiments they tested P-iron prepared with melting and so avoiding slag inclusions, and the P content of ca. 0-0.5wt% in their samples represents the minimum P content in medieval iron artefacts [cf. 6, 7], therefore they measured and calculated much higher characteristic values of ductility and toughness for P-iron, not representative as medieval bloomery P-iron. As [Fig. 3](#page-4-15) shows, slag inclusions have no effect on characteristic values of strength (yield strength (R_{eff}) (MPa)) and ultimate tensile strength $(R_m(MPa))$) but the strong solid solution strengthening effect of P results in highest yield strength values among the samples. Ultimate tensile strength values are higher in Fe-0.64C (n) and in Fe-0.64C (h-t) samples then in Fe-0.21C-1.05P (n) samples, due to the work hardening effect in the plastic deformation zone.

5 Conclusions

The results of comparative mechanical testing preformed on reconstructed bloomery iron materials and on modern steel allow the following conclusions to be drawn:

1. The notching and stress concentration effect of slag inclusions strongly decrease all the characteristic values of ductility and toughness of medieval bloomery iron materials compared to modern steels.

2. Typical medieval bloomery P-iron (with an average P content of 1wt%) was a brittle material with almost zero or very low characteristic values of ductility and toughness but with high characteristic values of strength.

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