REVEALING THE SURFACE PATTERN OF MEDIEVAL PATTERN WELDED IRON OBJECTS – ETCHING TESTS CONDUCTED ON RECONSTRUCTED COMPOSITES

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Pattern welding is a forging technique used for making and employing laminate composites that reveal a surface pattern after polishing and etching. The technique was widely used within the period of the late 2nd to the 14th century AD in the manufacture of ostentatious swords, scramasaxes, knives and spear-heads. Although pattern welding derived from piling wrought iron and steel together, deliberately created surface patterns were, since the 2nd century at the latest, revealed by composites employing phosphoric iron as the basic constituent. As the readability of the patterned surfaces is significantly enhanced by etching, one can assume that historical pattern welded objects were somehow etched. Hence, the basic question arises: which material combination and what etching parameters lead to the most contrasting and visible pattern? Another important question is: how can archaeometallurgists and conservator-restorers reveal surviving pattern welded elements of archaeologically excavated iron objects without a risk of misinterpretation and destabilization of the objects studied?
In an attempt to answer these questions, samples detached from patterned welded rods combining phosphoric iron, wrought iron and steel were ground and etched using six different acids (which could be available in the 2nd–14th centuries) under various conditions concerning acid concentration, temperature and etching time. The etching test revealed that the most visible pattern appears in the case of composites combining phosphoric iron and tempered steel, when hydrochloric acid is applied as etchant. When concerned parts of archaeological iron objects are subjected to etching, applying a weak solution of nitric acid in gradually increased concentration or temperature seems to be the most convenient method for the particular purpose.

Keywords: *etching – pattern welding – archaeometallurgy – phosphoric iron*

VYVOLÁNÍ POVRCHOVÉ KRESBY STŘEDOVĚKÝCH SVÁŘKOVĚ DAMASKOVANÝCH PŘEDMĚTŮ – TESTOVÁNÍ LEPTADEL NA REPLIKÁCH DAMASKOVÝCH KOMPOZITŮ

Svářkové damaskování je technika zahrnující výrobu a aplikaci laminovaných kompozitů, na jejichž leštěném (broušeném) a leptaném povrchu se objevuje kresba pravidelného vzorování. Jde o techniku užívanou prokazatelně v době od 2. do 14. století n. l. při výrobě honosných mečů, saxů, skramasaxů, nožů a hrotů kopí. Přestože svářkové damaskování vychází z techniky vzájemného paketování železa a oceli, cíleného povrchové vzorování bylo nejpozději od 2. století n. l. dosahováno pomocí kompozitů, jejichž základním prvkem bylo železo se zvýšenými obsahy fosforu (fosforové železo).
Čitelnost vzoru damaskovaného povrchu bývá významně umocňována leptáním, lze proto předpokládat, že historické damaskované předměty byly nějakým způsobem leptány. Otázkou zůstává, kterou kombinací materiálů a jakými parametry leptání bylo možné dosáhnout nejvíce kontrastní a viditelné kresby (vzorování). Další důležitou otázkou je, jak dnes může archeometalurg a/nebo konzervátor-restaurátor zviditelnit dochované damaskové elementy v archeologických předmětech bez rizika chybné interpretace a destabilizace sledovaného předmětu.
Při pokusu nalézt na dané otázky odpověď byly experimentálně připravené damaskové kompozity kombinující fosforové železo, prosté svářkové železo a ocel broušeny a za různých podmínek leptány šesti různými kyselinami, dostupnými v době od 2. do 14. století n.l. Experiment prokázal, že nejlépe viditelné vzorování se objevuje u kompozitů kombinujících fosforové železo a popuštěnou ocel, leptaných 20% kyselinou chlorovodíkovou. Je-li toho třeba, při průzkumu archeologických předmětů se zdá být nejvýhodnější užití slabého (2%) roztoku kyseliny dusičné mající pokojovou (18 až 24°C) nebo vysokou teplotu (kolem 70°C).

Klíčová slova: *leptání – svářkový damask – archeometalurgie – fosforové železo*

1. INTRODUCTION

In general, pattern welded objects are those revealing decorative surface patterns through specifically treated composite materials produced by the forge welding of alternating sheets of steels that differ in chemical composition. In the past, composites employed for decorative purposes combined phosphoric iron with either non-phosphoric iron (wrought iron) or steel. One should not confuse these pattern welded composites with *wootz* (sometimes called *Damascus steel*), which is a sort of hypereutectoid crucible steel, whose controlled cooling and forging leads to the formation of large cementite particles scattered in a pearlitic matrix, and consequently to the development of a surface pattern resembling watered silk *(Williams 2012, 36–37; Verhoeven et al. 1998).*

The visibility and contrast of pattern welding is significantly higher in an etched state, therefore it is generally accepted that pattern welded parts of iron objects were etched in the past, albeit it is yet unknown exactly how. As a better knowledge of possibilities to enhance the visibility of patterned parts would meaningfully help in understanding how pattern welded objects were manufactured and maintained, series of etching tests were conducted in order to find out which combination of materials and what etching parameters result in the most visible and contrasted pattern. The presentation of this research and of the results obtained is the main goal of this paper. Besides, the results allow the discussion of the most convenient method for revealing details of pattern welded parts of archaeologically excavated objects subjected to archaeometallurgical and/or conservation-restoration surveys.

1.1. Pattern welding techniques

The idea to produce pattern welded composites, which alternate high-phosphoric iron (from 0.4 to 1.4wt% P; a material not used in modern industry (*Thiele – Hošek in press)*) with non-phosphoric iron or steel and which reveal as rule a regularly alternating pattern when ground and etched, was undoubtedly born from the knowledge of piling numerous small pieces of iron or steel in order to achieve one large billet of reasonable homogeneity and purity. Because the first objects displaying evidently deliberate pattern welding (with a twisted pattern and with pattern welded strips extending in nearly the entire length of blades) are Roman swords dated to the second half of the 2^{nd} century AD (*Hošek et al. 2011; Gilmour 2007*), it seems that the technique was developed by smiths working in the territory of the Roman Empire. In the following centuries, Roman blade-smiths managed to produce a number of very complex patterns, suggesting that they perfectly mastered this technique *(Biborski – Ilkjær 2006, 288–289)*. Pattern welded swords then reached the peak of popularity around the 7^{th} century *(Kucypera in press)*, but since around the $8^{th}/9^{th}$ century, pattern welding gradually turned into the form of an iron inlay forming various inscriptions and signs on blades made entirely or almost entirely of steel, see Fig. 1 *(Moilanen 2009; Hošek et al. 2012; Williams 2012, 62)*. Pattern welding disappeared from sword-making around the $10^{th}/11^{th}$ centuries *(Kucypera in press)*. Nonetheless, from the 10^{th} until the 14^{th} century it appears on knife blades, thus the technique was continuously used for at least twelve centuries. Besides swords and knives, a number of pattern welded scramasaxes and spear-heads is known *(Pleiner 2006, 214–222; Anteins 1973; Hošek – Šilhová 2006)*.

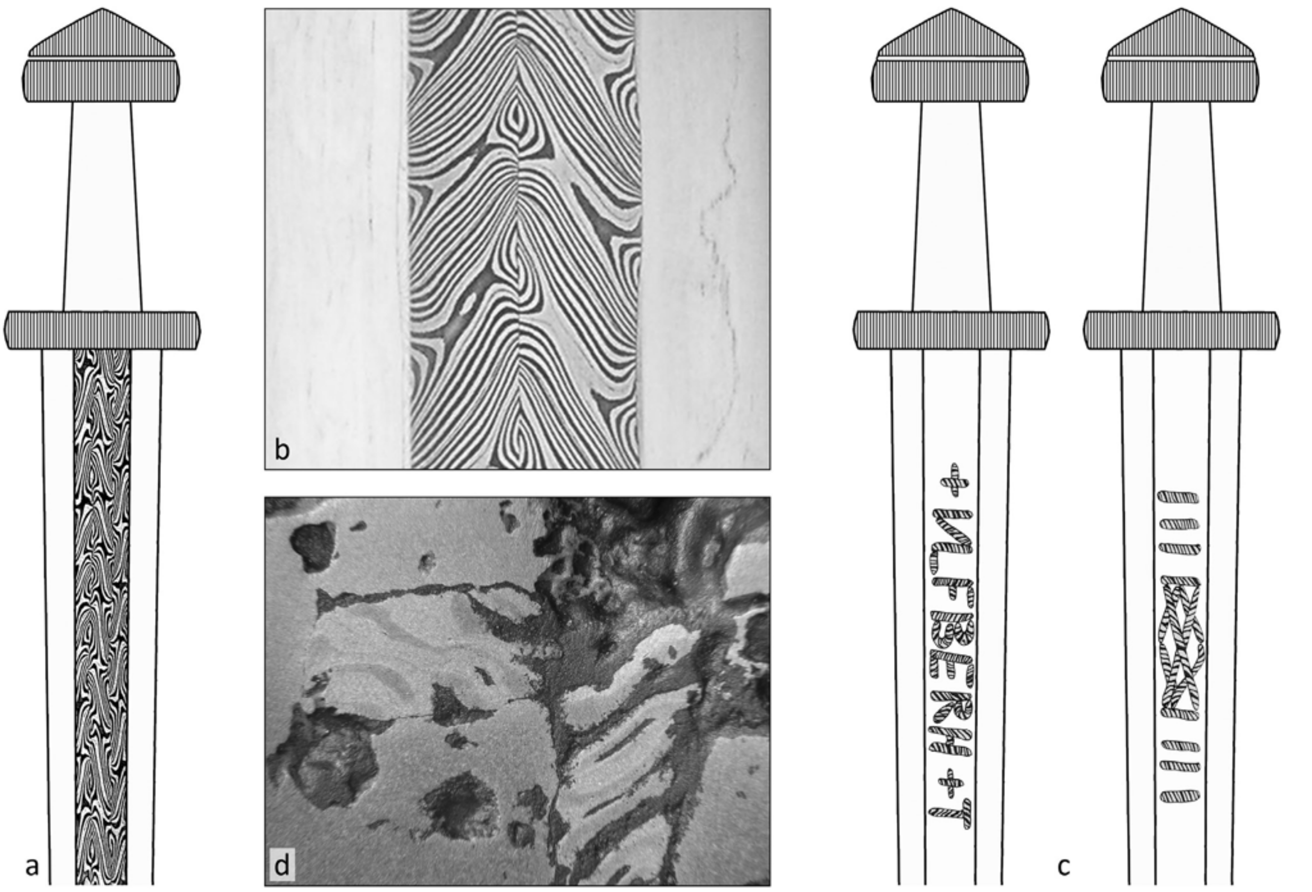

Fig. 1: Pattern welding and possibilities of its application to historical sword blades: a – pattern welding in the central part of the blade; b – an example of the real appearance of a pattern welded blade (modern replica made by P. Bárta); c – pattern welding applied in the form of an inlaid inscription and motives; d – an example of the real appearance of an inlaid pattern welded cross found on an archaeologically excavated blade

The aesthetic role of pattern welding is indisputable, but its positive effect on mechanical properties, especially in case of long sword blades, is often the subject of discussion. Nevertheless, research recently conducted by the authors evidences that genuine pattern welding did not have an important positive effect on the mechanical properties of sword blades *(Thiele et al. in press)*. The main reason for this is that high-phosphoric iron, and hence pattern welded composites themselves (irrespective of the pattern), has low dynamic and static toughness, mechanical properties that are highly valued in the usage of swords. Therefore, it can be stated that the pattern welding technique was actually applied almost distinctly for aesthetic purposes and etching must have played a very important role in the manufacturing process.

1.2. Medieval etchants

As the surface pattern is revealed by etching in the technique of pattern welding, it is important to study the written sources concerning the etchants existing in the period examined. Although there are no exact medieval written sources about etching technology, it is possible to identify several acids which were available at the time.

Sulphuric acid (H_2SO_4) was well known since ancient times, mentioned in Assyrian and Sumer written sources as well as in the works of Dioscorides (Greek doctor from the 1st century AD.) and Pliny the Elder (Roman polymath 23–79 AD.) *(Karpenko & Norris 2002)*. Early Islamic written sources report the use of sulphuric acid for etching metals in the Middle-East. Ya'qūb ibn Iṣḥāq al-Kindī already recorded in his 9th-century work, that vitriol, i.e. sulphuric acid was used for revealing the surface pattern of sword blades *(Fehér 2000)*. Sulphuric acid, made from the roots of "Rubia tinctorum" by fermentation or by alkaline hydrolysis *(Theophilius 1986)*, could also be known in Europe in the Middle Ages, having been used for dyeing since the antiquity. One of the earliest written sources from medieval Europe which reports vitriol as paint (as opposed to the "atramentum" used as back paint) is the so-called Lucca manuscript, a book on painting from the 8th century (*Compositiones ad tingenda musiva*) *(Stillman 1960)*.

Nitric acid (HNO_3) as well as vitriol could also be well-known in the Middle Ages. Both these acids were identified by Abū Mūsā Jābir ibn Ḥayyān (Arabian scientist, polymath, alchemist, ca. 721–815. AD., Geber in Latin). Geber was the first to mention the technology of distillation, roasting, evaporation, etc., and he was the first who described sulphur, nitric and hydrochloride (HCl), citric ($C_6H_8O_7$) and tartaric ($C_4H_6O_6$) acid as well as aqua regia as the mixture of three parts of nitric acid and one part of hydrochloride acid *(Datta 2005)*. In European written sources, the same technologies and acids appear in the works of Pseudo-Geber (eg. *Summa perfectionis magisterii*), who also mentions that aqua regia was made by solving sal-ammoniac (sal-ammoniacum, NH_4Cl) in sulphuric acid *(Karpenko & Norris 2002)*. Sal-ammoniac appears in a European written source from the 14th century, in which it is stated that the aqueous solution of sal-ammoniac could be used as an etchant of iron („Aqua corrosiva: Nota quod aqua corrosiva minuens corporum pondera fit ex sale armoniaci et coperosa in equali portione distillando aquam per alembicum […] Aqua solvens argentum: Aqua solvens argentum et quidem alia metalla fit ex vitriollo romano et sale armoniaco in equali portione et haec aqua dissolvit ferrum…", *Newman 1991*).

Acetic acid (CH_3COOH) was well-known both in the antiquity and also in the Middle Ages. It was used in medicine against toothaches, to stop bleeding, and to disinfect wounds, while in gastronomy it had a role in the seasoning of food, in pickles and as a preservative. As it was easy and cheap to prepare, it was widely available for the lower classes.

We can read about tannic acid ($C_{14}H_{10}O_9 + 2H_2O$) in Teophilus Presbyter's (ca. 1070–1125) work entitled *De diversibus artibus*. Ligna Spinarum, a plant mentioned in a recipe for ink is a thorny tree whose crust contains a high amount of tannic acid *(Teophilus 1986)*. Phosphoric acid (H_3PO_4), also known as "rust converter", which turns the reddish-brown rust (Fe_2O_3) into black ferrous phosphate ($FePO_4$), cannot be found in its natural state, but its salts are common. A strong oxydizing agent, e.g. nitric acid, turns phosphorus into phosphoric acid.

1.3. Revealing pattern welded parts of archaeologically excavated objects

The investigation of archaeological objects such as swords, knives, spearheads or seaxes requires the knowledge of how to find out if, or even how, they were pattern welded. The standard and widely employed method for revealing pattern welded parts is radiography. Unfortunately, radiography provides us with reliable results only when the pattern welded elements are affected by corrosion, and one of the composite constituents (phosphoric iron as a rule) is corroded deeper than the other; this causes a surface relief, which copies the original pattern recognizable on radiograms. When the surface is corroded slightly and/or evenly, the pattern welding is not detectable by X-ray at all, or only in traces. In case the results of radiography are unreliable or difficult to interpret, small objects or fragments can be subjected to SEM-EDAX analysis, by which a distribution of phosphorus (and hence the pattern) can be revealed. As phosphorus remains relatively undisturbed by corrosion *(Salter 2004)*, there is no need for the removal of corrosion layers and the SEM-EDAX analysis is therefore a non-invasive method suitable for "everyday" research and can be successfully applied also to objects entirely corroded, though, as mentioned already, limited in size. When neither radiography nor chemical analyses are helpful or the results obtained require verification, optical metallography can be conducted either on cross-sectional specimens or directly on the object's surface. As samples must be detached from the objects or corrosion layers must be removed and reached metallic surface properly polished and etched, conventional metallography is not used frequently. However, when the information or further details about the pattern welding are essential for the assessment or interpretation of the objects and the above mentioned methods have not provided satisfying results, corrosion layers can still be carefully removed by conservator-restorers on a limited surface area and the denuded metallic surface etched (if still necessary) in order to see the pattern. As the surface cannot be polished, the application of 2% solution of HNO_3 (Nital) has variable success and the use of such etchants as Oberhoffer is avoided because of a risk of contamination by chlorides. In fact, a suitable etchant for the described conditions has not been introduced so far.

2. METHODS AND RESULTS

2.1. Preparing samples

Keeping historical accuracy in mind, we smelted iron blooms as base materials using bog iron ores in smelting experiments in the copies of excavated 10th century Fajszi-type embedded furnaces *(Gömöri 2000)* (about smelting experiments cf. details in Thiele 2012). Phosphoric iron (symbol P) was smelted using phosphoric bog iron ore from Inner-Somogy (South-West Hungary). Wrought iron (symbol I) was smelted using the same ore, charging limestone into the furnace to decrease the phosphorus content of the bloom (about the effect of lime cf. *Török & Thiele 2013*). Steel was produced by the re-smelting and carburizing of pieces of wrought iron (symbol S). The carbon content of the steel bloom was homogenised by multiple plying and forge welding (cf. layers of Sn and Sh materials can be seen in Fig 3).

Pattern welded rods with a cross section of 10 x 10mm and a length of 60 mm combining these base materials were forged. Prior to forging, all the base materials were tested with a portable XRF analyser (which can detect light elements, such as P) to judge if their composition is within the acceptable range. One pattern welded rod was made of the material combinations of phosphoric iron + wrought iron (symbol PIt8) and two of phosphoric iron + steel (symbol PSnt8 and PSht8). All the pattern welded rods consisted of 8 layers, with a 1 : 1 ratio of the base materials and were twisted. Heat treatment was carried out on one of the phosphoric iron + steel rods (symbol PSht8), which was water-quenched from 900°C and tempered in 300 °C for 60 minutes. The other rods were kept in their natural state. Rods made of base materials by pattern welding can be seen in Fig 2.

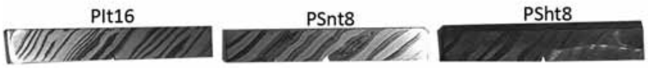

Fig. 2: Pattern welded rods made of base materials. The surface is polished and 2% Nital etched

Metallographic examination under optical microscope was carried out on the cross-sectional specimens cut out of each rod to identify the microstructure and calculate the carbon content (by means of image analysis), cf. Fig. 3. The phosphorus content of phosphoric iron was measured by Energy Dispersive X-ray Spectroscopy under Scanning Electron Microscope (SEM-EDS). The results obtained are summarized in Table 1.

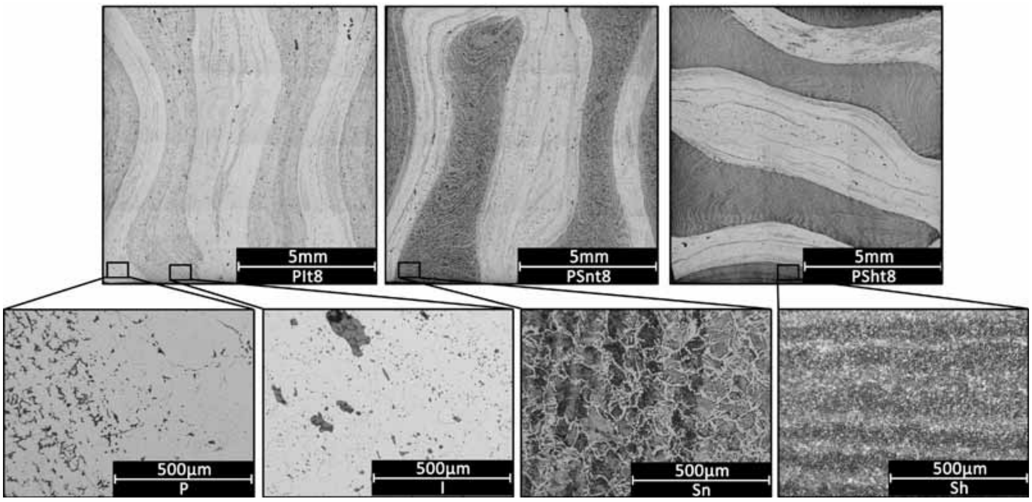

Fig. 3: Top: metallographic macro-photographs of cross sectional specimens from pattern welded rods; Bottom: metallographic pictures of base material layers

Pattern welded rods	Symbol	Chemical composition and microstructure of base material layers
Phosphoric iron + wrought iron, twisted, 8 layers	PIt8	Phosphoric iron: ferritic, C = 0wt%, P = 0.9-1.1wt%, Wrought iron: ferritic with little pearlite, C = 0.05wt%, P = 0wt%
Phosphoric iron + steel in natural state, twisted, 8 layers	PSnt8	Phosphoric iron: ferritic, C = 0wt%, P = 0.9-1.1wt%, Steel: pearlite with proeutectoid ferrite, C = ca. 0.6wt%, P = 0wt%
Phosphoric iron + hardened and tempered steel, twisted, 8 layers	PSht8	Phosphoric iron: ferritic, C = 0wt%, P = 0.9-1.1wt%, Steel: tempered martensite, C = ca. 0.6wt%, P = 0wt%

Table 1: Microstructure and chemical composition of specimens from rods made of base materials and by pattern welding

Regarding the conducted analyses it can be stated that all the tested materials correspond with the basic types of material used in the past and all of them can be considered suitable from the perspective of historical accuracy.

2.2. Etching tests

The existence of the works on chemistry presented in part 1.2 (which are primarily Oriental in the examined period) do not correspond to the contemporary metal etchers' empirical knowledge on suitable acids. Nevertheless, acetic acid, citric acid and tartaric acid might have been commonly known due to the fermentation of fruits as well as tannic acid from nutgalls. Also, sulphuric acid, nitric acid and hydrochloric acid could have been used without being defined. Contemporary sword-makers must have used these relatively weak acids easily available in a natural state. In our experiments, we focused on the study of a wide range of contemporary technology besides the possible professional practices of the period, thus we also tried the stronger acids that were also available in the period.

Etching tests were carried out using distilled water, diluted citric acid, acetic acid (vinegar), phosphoric acid, sulphuric acid, nitric acid and hydrochloric acid at a temperature of 20 °C and 70 °C, on a concentration of 2vol% and 10vol% and on an etching time of 10sec and 60sec. Etching tests were carried out in a total of 114 cases on the cross-sectional specimens cut out of the pattern welded rods. The polished surface of the specimens were roughed with abrasive paper P320 to reconstruct the surface roughness that could be created in the period examined. After each etching test a macro image was taken of the revealed surface pattern using SRL camera under the same external conditions and camera settings. Macro photos can be seen in figure 4 arranged in table.

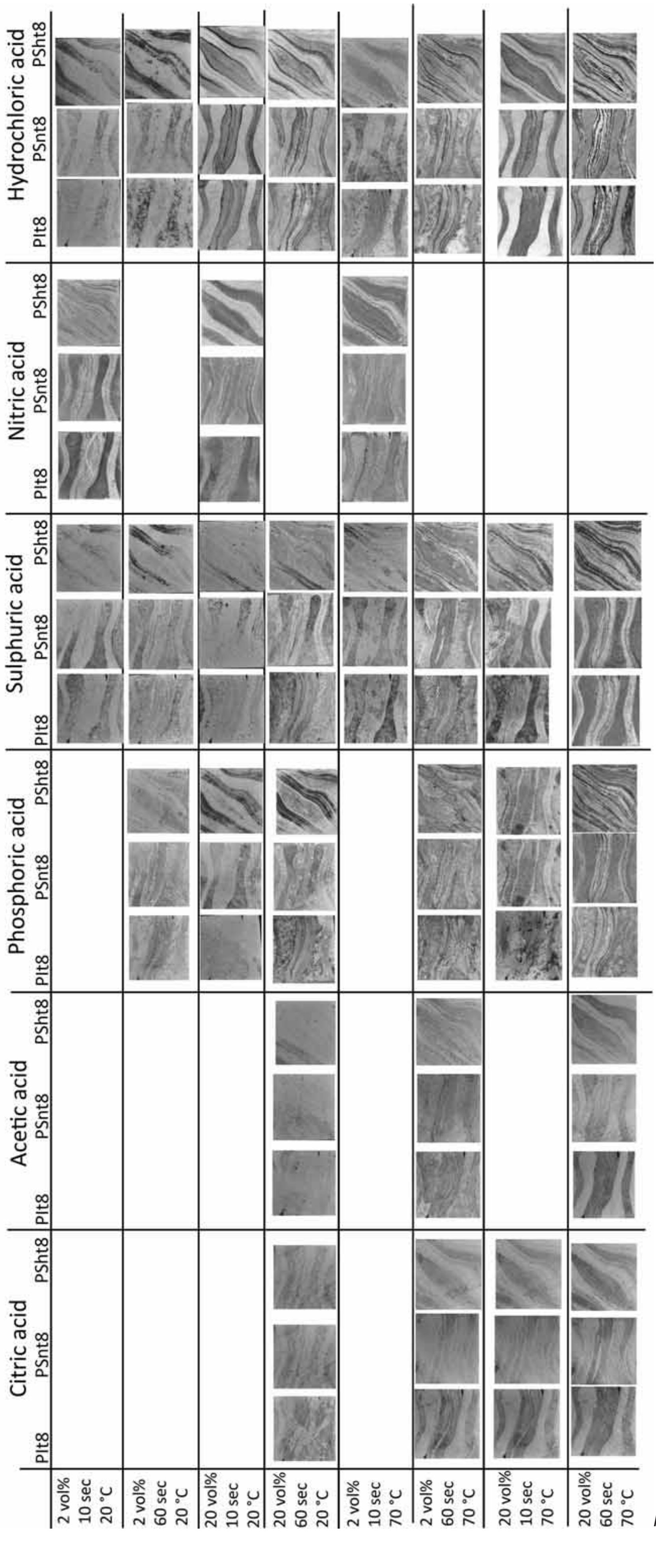

Fig. 4: Macro image of etched specimens

3. DISCUSSION

A preference list of the material combinations and etching parameters can be made based on the macro images of etched specimens to characterise the visibility of the surface pattern. We defined two parameters to make an objective preference list:
Parameter A: Brightness difference between two layers
Parameter B: Brightness homogeneity of each layer
These parameters were calculated using a photo editing software (Photoshop CS3). First, the macro images were converted to grayscale, then the mean and standard deviation of pixel brightness values (about 150 000 pixels) for two layers next to each other was measured (cf. fig 5).

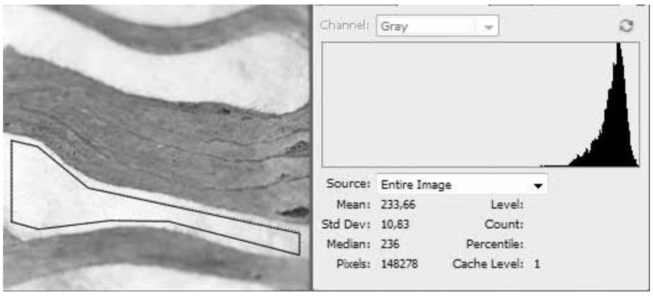

Fig. 5: The brightness of the pixels (histogram on the right) on the analysed area (poligon on the left)

This way, two values could be assigned to each macro image. Parameter A was calculated as the difference of the mean of pixel brightness values for two layers next to each other, while parameter B was calculated as the mean of the standard deviation of pixel brightness values for two layers next to each other. Values of parameter A and B are summarized in table 2. We considered the visibility of the surface pattern acceptable if the absolute value of parameter A was higher than 40 and if the value of parameter B was lower than 20. According this rule, the surface patterns with acceptable visibility are marked with a rectangle in table 2.

The most visible surface pattern appeared after the etching of the pattern welded sample made of the combination of phosphoric iron + tempered steel using 20vol% hydrochloric acid for 10sec on 70°C (parameter A has an extremely high value of 118.9). Etching with 20vol% hydrochloric acid resulted in a clearly visible surface pattern for all material combinations both on 20 °C and 70 °C. Although hydrochloric acid was not an easily available acid in the Middle Ages, it can be stated that the most suitable acid for revealing the surface pattern for pattern welded objects was hydrochloric acid among the tested acids.
Regarding the archaeological iron objects, when revealing pattern welded parts on a selected surface area is essential, applying a weak (2vol%) solution of nitric acid at room temperature or (if necessary) heated on high (70 °C) temperature seems to be one of the convenient methods for the particular purpose. The objects, however, must be in the care of conservator-restorers who can adequately treat them to avoid a risk of their future corrosion (at least, the etched surface must be properly washed by ethanol and distilled water). Hydrochloric acid is not appropriate for the risk of contamination of the treated surface by chlorides, which can initiate or support the development of chloride corrosion causing serious problems to archaeological iron objects.

4. CONCLUSIONS

The etching tests conducted on reconstructed composites allow the following conclusions to be drawn:
The most visible pattern appears in the case of composites combining phosphoric iron and tempered steel, when 20vol% hydrochloric acid is applied as etchant for 10 sec on 70 °C.
Applying a weak (2vol%) solution of nitric acid at room and increased temperature seems to be one of the convenient methods for revealing the surface pattern of excavated archaeological objects. Of course, these must subsequently undergo appropriate conservation treatment.

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Parameters		Citric acid			Acetic acid			Phosphoric acid			Sulphuric acid			Nitric acid			Hydrochloric acid		
		PSh	PSn	PI	PSh	PSn	PI	PSh	PSn	PI	PSh	PSn	PI	PSh	PSn	PI	PSh	PSn	PI
2 vol% 10 sec 20°C	A										11.8	34.9	6.8	**64.3**	**46.0**	3.0	2.7	16.1	22.8
	B										13.3	17.7	11.2	14.3	19.0	14.8	11.3	13.8	20.1
2 vol% 60 sec 20°C	A							-38.0	-9.1	-22.9	1.9	13.0	21.3				27.0	14.3	41.1
	B							18.2	25.8	13.7	19.6	17.4	22.5				24.6	17.1	25.9
20 vol% 10 sec 20°C	A	-	-	-	-	-	-	-6.4	31.5	**40.1**	-19.9	8.1	-1.9	32.0	-2.6	**-69.6**	**-47.1**	**-60.4**	**-48.6**
	B	-	-	-	-	-	-	11.8	19.7	19.3	10.6	16.6	10.6	12.7	18.3	12.6	14.3	18.0	16.1
20 vol% 60 sec 20°C	A							-12.1	0.7	45.9	-28.2	31.3	1.3				4.0	-31.1	-33.1
	B							27.2	24.5	32.7	22.4	26.4	14.3				24.9	22.4	19.2
2 vol% 10 sec 70°C	A										38.6	20.5	11.5	9.9	6.2	**-48.9**	-31.0	17.1	-28.4
	B										23.7	20.9	24.7	12.5	13.7	17.3	18.7	25.8	11.3
2 vol% 60 sec 70°C	A	**-45.9**	-17.9	-33.4	-26.2	-26.8	-12.7	-8.2	-2.6	3.2	4.6	-34.6	-27.2				-46.9	-17.0	10.0
	B	18.6	19.7	12.8	17.7	19.4	15.2	27.7	18.4	24.0	22.0	22.8	24.9				30.8	28.2	20.5
20 vol% 10 sec 70°C	A	**-52.6**	-14.8	-34.1				-41.3	-33.7	-34.3	**46.4**	26.4	18.4				**-118.9**	**-58.3**	**-49.3**
	B	18.1	20.5	12.5				32.1	22.9	16.5	18.4	21.7	28.2				11.4	16.9	17.8
20 vol% 60 sec 70°C	A	**-48.1**	-26.1	**-49.2**	**-57.3**	-14.1	-23.4	21.2	14.9	-3.6	**47.3**	49.9	45.3				-19.0	5.6	-23.9
	B	15.1	23.2	15.0	10.5	18.0	14.7	23.8	21.4	31.5	15.9	20.6	34.0				28.1	34.1	32.8

Table 2: Parameter A and B for each macro image of etched specimens (surface patterns with acceptable visibility are marked with a rectangle)

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