NONDESTRUCTIVE METHODS OF ANALYSIS APPLIED TO ORIENTAL SWORDS

MÉTODOS DE ANÁLISIS NO DESTRUCTIVOS APLICADOS A ESPADAS ORIENTALES

POR

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ABSTRACT - RESUMEN

Various neutron techniques were employed at the Budapest Nuclear Centre in an attempt to find the most useful method for analysing the high-carbon steels found in Oriental arms and armour, such as those in the Wallace Collection, London. Neutron diffraction was found to be the most useful in terms of identifying such steels and also indicating the presence of hidden patterns.

En el Centro Nuclear de Budapest se han empleado varias técnicas neutrónicas con el fin de encontrar un método adecuado para analizar las armas y armaduras orientales con un alto contenido en carbono, como algunas de las que se encuentran en la Colección Wallace de Londres. El empleo de la difracción de neutrones resultó ser la técnica más útil de cara a identificar ese tipo de aceros y también para encontrar patrones escondidos.

Keywords - Palabras Clave

Indian swords; "Damascus" steel; "watered" patterns; neutron diffraction; neutron radiography; Prompt Gamma Activation Analysis; Proton-induced Xray-Emission spectroscopy; Wallace Collection; non-destructive analysis.

Espadas indias; acero de Damasco; patrones "diluidos"; difracción de neutrones; radiografía de neutrones; Análisis por Activación de Radiación Gamma rápida; Proton-induced Xray-Emission spectroscopy; Colección Wallace; análisis no destructivos.

INTRODUCTION

The analysis of plate armour is comparatively straightforward. A plate generally has an edge which can, after suitable preparation, be placed upon an inverted microscope and the

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microstructure investigated¹¹. Swords are more problematic, since a section (or at least a half-section) of a sword has to be observed in order to make any deductions about its manufacture. Attempts at investigation from the surface alone have had mixed success² so most results have had to be drawn from excavated, or otherwise damaged swords³.

So it would be enormously valuable to have an entirely non-invasive technique for the analysis of swords. Especially since The Wallace Collection, London, houses a very important collection of Oriental (mostly Indo-Persian) arms and armour, including some 300 swords, as well as over 70 helmets, vambraces and shields, whose analysis presents quite difficult problems. The carbon content of Indian steels is unusually high by European standards, which has been one of the factors leading us to consider other techniques, such as neutron diffraction or gamma-activation.

Accordingly, a series of experiments was planned and carried out at the Budapest Neutron Centre within the Institute of Solid State Physics and Optics, Hungarian Academy of Sciences, Budapest, Hungary. This was made possible by the transnational access program CHA-RISMA (Cultural Heritage Advanced Research Infrastructure Synergy for a Multidisciplinary Approach to conservation/restoration in museums).

DAMASCUS STEEL

The most famous swords in history were those of "Damascus steel" (so-called from their alleged origin in Syria) and a good deal has been written about them. They were made in India or Persia, had a "watered-silk" or "damask" pattern on their surfaces, and no amount of sharpening did ever remove their edge.

In the ancient Middle East a method of making steel developed, entirely different to that of Europe. *Crucible steel* was made by heating lumps of iron with carbon, or a material containing carbon, such as cast iron, in a sealed crucible for many hours, or even days. Eventually enough carbon would have been absorbed for the alloy to melt, and the broken crucible would yield a cake of cast steel, which could be a convenient size for making a sword blade. Since the liquid metal separated from the liquid slag, it was a homogeneous product of high carbon content (1.2 - 1.6%), unequalled in Europe until the 18^{th} century⁴.

An important subdivision of crucible steel was the steel called *wootz*, in South India, *bulad* in Central Asia, and sometimes, misleadingly, "Damascus steel".

Wootz was made, as was all crucible steel, by melting iron with carbonaceous material in a sealed crucible over several days until it wholly or partially melted into a cake of steel, but was then allowed to cool *extremely slowly* (over days, rather than hours). These cakes were exported to centres of arms manufacture, such as Damascus, where they were carefully forged, with considerable difficulty, into sword blades. Since the melting-point of steel falls with increasing carbon content, a lower temperature than usual has to be employed to forge a blade of higher

¹ Williams, A. The Knight and the Blast Furnace. Leiden, 2003, passim.

² Williams, A. "Science and fakery: the limitations of Science in the Analysis of Arms and Armour" *Journal of the Arms & Armour Society*, 18 (London, 2006) 249-254. Biborski, M. Stepinski, J. & Zabinski, G "The Coronation sword of the Kings of Poland" *Gladius* 31, 2011, 93-148.

³ Williams, A. The Sword and the Crucible (Leiden, 2012) passim.

⁴ Craddock, P. "New light on the production of crucible steel in Asia". *Bulletin of Metals Museum of the Japan Institute of Metals* 29 (Sendai, 1998) 41-66.

carbon content than usual, notwithstanding its hardness. This forging broke up the cementite network left over from the casting, reducing brittleness, and producing the characteristic pattern ("watered silk") visible after polishing and etching on the surface of the blade.

It is not simply of interest to describe their metallurgy, fascinating though that is, but to reconstruct their original appearance. Many of the swords in the Wallace Collection would originally have had the surface finish resembling "watered-silk" but they have been polished by over-zealous 19th century collectors, thus destroying their intended appearance, which illustrated their metallurgy. Detection of such hidden patterns will be needed to precede, and inform, any ethical decisions to be made about their conservation and display.

These patterns are the results of the segregation of massive cementite particles on slowly cooling cast steels and their careful subsequent forging. Since this segregation is of the order of magnitude to generate a visible pattern (after polishing and etching) it was hoped to apply neutron diffraction to its detection.

It is important to remember that many crucible (hypereutectoid) steels may not have had any pattern⁵. It is also the case that many swords which appear to have some sort of pattern on the surface may in fact be showing the results of etching a layered structure.

PREVIOUS ANALYSES

A relatively modest number have been analysed, since informative analysis has involved taking a sample from the metal, and examining it microscopically. Excavated swords generally have ragged or broken edges which can make this acceptable, but "Damascus" blades are generally in such a good state of preservation that this method is ruled out.

After the pioneering articles by Belaiew (1921⁶ and Zschokke (1924)⁷ another by Panseri (1965)⁸ showed the microstructure of Damascus steel, and they were followed by France-Lanord (1969)⁹ and Piaskowski (1978)¹⁰. Extensive efforts by Verhoeven and Pendray¹¹ to recreate this process have led to a series of very detailed papers on its metallurgy. They have suggested that traces of carbide-forming elements were essential to the banding responsible for the visible pattern, and indeed, Panseri had earlier pointed out that traces of chromium would stabilise cementite, Fe₂C.

⁵ See, for example, Craddock, P. & Lang, J. 2003 "*Mining & Metal production through the ages*" especially "Cast iron, Fined iron, Crucible steel" 231-257, and "Early Islamic Crucible steel production at Merv" 258-266.

⁶ Belaiew, N.T. "Damascene steel" Journal of the Iron & Steel Institute (London, 1918) 100, I, 417-439; and ibid. (1921) 104, II, 181-4.

Zschokke, B. "Du damassé et des lames de Damas" Revue de Métallurgie, 21 (Paris, 1924) 635.

Panseri, C., 1965. "Damascus steel in legend and reality". *Gladius*, 4, 5-66.
 France-Lanord, A., 1969. "Le fer en Iran au premier millenaire avant J-C".

Revue d'histoire des mines et la métallurgie, Jarville, 1 (1): 75-126.

¹⁰ Piaskowski, J., 1978. Metallographic examination of two Damascene steel blades. Journal for the History of Arabic Science, 2, Aleppo: 3-30.

¹¹ Verhoeven, J.D., Pendray, A.H. and Peterson, D., 1992. Studies of Damascus steel blades. Materials Characterisation, 29: 335-41 and ibíd. (1993) 30:175 and 187. Also Idem. and Danksch, W.E., 1998. "The key role of impurities in ancient Damascus steel blades". Journal of the Minerals, Metals and Materials Society 50 (9): 58-64.

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The Moser collection (in Bern) of over 200 swords was cataloged by Zeller (1955)¹² over a number of years. Zeller initially distinguished ten types of "Damascus" patterns, but later reduced that number to five and eventually three¹³.

From this collection, Zschokke was allowed to select six swords which he studied in destructive detail, and this pioneering effort remains the most extensive study of their metallurgy and mechanical properties. He said "The Oriental and especially the Persian weaponsmiths understood how to bring out from the raw Indian steels, (so-called *wootz*) a whole series of different damask patterns, that become visible on the finished blade only after corroding with weak acids. Out of the approximately ten damask types in Persia, however, only a few usually occur frequently.We will therefore restrict ourselves also to three easily distinguishable Basic Forms, that partially agree with the Persian categories".

These three Basic Forms are, (although there are numerous intermediate transitional ones):

1. VEIN DAMASK "MASERDAMAST"

Its design resembles a wood grain ...while some probably appeared in the Orient as the "Kara-Khorassan" and "Kara-Taban" damasks, which are marked with a conspicuous brownish or blackish colouring. (kara = black).

The essential features about this damask is its irregularity in the design, only when when viewed alongside - or crosswise - it appears as a bundle of fibres.

2. Stairway damask "Treppendamast"

The Persian "Kirk ner deban" and the Turkish "Kirk ner Derven" both mean 40 steps. Nerdeban also means "the ladder", and the characteristic of this damask is the crosswise strips with which it is marked. It is called also the "ladder of Mohammed" or "Jacob's ladder" and appears as a ladder that can have up to 40 rungs.

3. WAVE DAMASK "WELLENDAMAST"

The distinct longitudinal direction of the wave design is characteristic of this. This is also the type, apparently after which the English term for damask "watered steel" was devised."

It seems likely that these different forms of pattern are the results of different forging and heat-treatment techniques and so might be identified with different workshops. Accordingly, any analytical techniques must not merely identify the chemical elements and the crystalline phases present but also their distribution within the volume of each blade.

ANALYSIS OF SOME INDO-PERSIAN AND EUROPEAN BLADES BY MEANS OF PGAA

To determine the bulk elemental composition of various objects, one possible technique is Prompt Gamma Activation Analysis (PGAA); PGAA is a nuclear analytical technique for nondestructive quantitative determination of elemental compositions. The sample is irradiated in a beam of slow (i.e. low energy) neutrons and the gamma-rays from the radiative capture are

¹² Zeller R. & Rohrer, E.F. "Orientalische Sammlung Henri Moser; Katalog der Waffensammlung" (Bern, *Historisches Museum*, 1955).

¹³ Zeller, R. "Über damaststahl in der Orientalische Sammlung Henri Moser" *Jahrbuch des Bernisches Historisches Museum* (1924) and also (ibid. 1914, 1928, 1931-5).

detected. Since neutrons can go as deep as a few centimetres underneath the surface, PGAA provides bulk composition characteristic for the irradiated volume. Contrary to the conventional neutron activation analysis (NAA), the irradiation and the detection are simultaneous. The energies and intensities of the peaks in the gamma spectra are independent of the chemical state of the material; hence the analytical result is free of matrix effects. In most cases, major components and a few significant trace elements can be quantified. PGAA has been successfully applied to characterize archaeological objects made of various rocks, glass, as well as metals.

The present PGAA measurements were carried out at the PGAA and at the NIPS experimental stations of the Budapest Neutron Centre¹⁴. The intensities of the neutron beam, characterized by the thermal equivalent flux, were about 8×10^7 cm⁻²s⁻¹ and 2×10^7 cm⁻²s⁻¹, respectively. The Compton-suppressed HPGe detectors have been precisely calibrated for energy and coun-



Figure 1. Specimen A. European small-sword of 18th century.



Figure 2. Specimen B. "Cut-steel" hilt from a European small sword of 18th century.

¹⁴ Szentmiklósi, L., Kis, Z., Belgya T. & Berlizov A. N. 2013, "On the design and installation of a Compton-suppressed HPGe spectrometer at the Budapest neutron-induced prompt gamma spectroscopy (NIPS) facility", *Journal of Radioanalytical and Nuclear Chemistry*, 298, 1605-1611.

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ting efficiency. The gamma-ray spectra were evaluated using the Hypermet-PC program. The spectroscopic data library used in the analysis was established earlier at the Centre for Energy Research. The composition was determined using the k_0 -method, while the uncertainties of the concentrations were calculated according to the rules of error propagation¹⁵.

Three blades (2 swords and 1 dagger), both European and Indian, katar, a kukri, an Indian steel bow, and a collar from a "Maximilian" harness were analysed with PGAA.

The two parts of the bow (which were made to be screwed together) were studied separately.



Figure 3. Specimen C. Indian sword of 17th or 18th century -a broken blade of length 21cm and hilt 21cm (12 cm width).



Figure 4. Specimen D. Indian dagger of 19th century.



Figure 5. Specimen E. Indian kukri (knife) of 19th century.

¹⁵ Révay, Zs. 2009, "Determining Elemental Composition Using Prompt Gamma Activation Analysis". *Analytical Chemistry*, 81, 6851-9.

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Figure 6. Specimen F. Indian katar (dagger) of 18th century.



Figure 7. Specimen G. European (German) collar from an armour of the 16th century.



Figure 8. Specimen H. Indian steel bow made in two parts of the 18th century.

With the help of PGAA, we were able to quantify traces of Mn in every objects; occasionally H, B, Cl, Co, Ni, Cu, Ag and Au were also measured. P, S and Ca were identified in only one instance. Unfortunately, the main objective of the PGAA experiments was not fulfilled: the carbon content of the steel objects was below the detection limit of PGAA - since PGAA is quite insensitive for carbon.

The detailed elemental compositions with the detection limits are listed in Table 1 and Table 2.

			Н	В	Ρ	s	CL	CA	MN	FE	Co	N	Cu	AG	AU	С
			0,005	0,0001	I	0,05	0,001	0,05	0,005	0,2	0,005	0,01	0,02	0,003	0,003	ŝ
31	6 WE1A	Indian Steel Bow 1A, blued surface	0,0112	0,00021	n.d.	n.d.	n.d.	n.d.	0,377	9,66	n.d.	n.d.	0,021	0,025	0,004	n.d.
31	7 WE1A	Indian Steel Bow 1A, not blued surface	0,0082	0,00024	n.d.	n.d.	0,0052	n.d.	0,270	7,66	0,006	n.d.	0,050	n.d.	0,005	n.d.
31	8 WE1B	Indian Steel Bow 1B, blued surface	0,0071	0,00023	n.d.	n.d.	0,0033	n.d.	0,283	9,66	0,007	n.d.	0,051	n.d.	0,003	n.d.
31	9 WE4D	Indian Blade, crucible steel	n.d.	n.d.	1,0	n.d.	n.d.	n.d.	0,025	98,9	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
32	0 WE7G	Rear-plate of collar	0,0903	0,00024	n.d.	n.d.	0,0240	n.d.	0,024	99,5	n.d.	n.d.	0,120	0,109	0,084	n.d.
32	1 WE1A	Sword	0,0187	n.d.	n.d.	n.d.	n.d.	n.d.	0,186	97,8	0,039	n.d.	2,0	n.d.	n.d.	n.d.
32	2 WE1B	Sword	n.d.	0,00026	n.d.	n.d.	0,0047	n.d.	0,124	99,5	0,018	0,045	0,29	n.d.	n.d.	n.d.
32.	3 WESE	Knife	0,0068	n.d.	n.d.	n.d.	n.d.	n.d.	0,014	99,95	n.d.	n.d.	n.d.	0,025	n.d.	n.d.
77	9 WE3C	Indian sword	n.d.	0,00023	n.d.	n.d.	0,0109	n.d.	0,008	9,66	0,022	n.d.	0,15	n.d.	n.d.	n.d.
78	0 WE3C	Indian sword	n.d.	0,00021	n.d.	n.d.	0,0098	0,06	0,019	99,7	0,018	0,027	0,18	n.d.	n.d.	n.d.
78	1 WE9	pendray sample (see ref. 11)	n.d.	n.d.	n.d.	n.d.	0,0046	n.d.	0,156	99,8	0,005	n.d.	n.d.	n.d.	n.d.	n.d.
78.	2 WE6F	iron dagger	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,014	96,99	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
78	7 WE2 (from F)	guard	n.d.	n.d.	n.d.	n.d.	0,0042	n.d.	0,085	96,96	n.d.	n.d.	0,042	n.d.	0,009	n.d.
79,	0 WE4D	steel dagger	n.d.	0,00065	n.d.	n.d.	0,0018	n.d.	0,005	96,99	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
62	1 WE9	pendray sample (see ref. 11)	0,021	n.d.	n.d.	0,06	0,0048	n.d.	0,118	99,8	0,0057	n.d.	n.d.	n.d.	0,022	n.d.

Table 1. PGAA results.

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Wet 31 3				Rel. unc %		2,2			63,		18,	0,001						
Spect: Spect: Spect: Spect: Spect: TP3	790	4	steel dagger	Conc. / wt%	n.d.	0,00065	n.d.	n.d.	0,0018	n.d.	0,005	99,99	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
New 317 318 318 319 319 310 317 <td></td> <td></td> <td></td> <td>Rel. unc %</td> <td></td> <td></td> <td></td> <td></td> <td>12,</td> <td></td> <td>4,</td> <td>0,01</td> <td></td> <td></td> <td>24,</td> <td></td> <td>33,</td> <td></td>				Rel. unc %					12,		4,	0,01			24,		33,	
	787	2	guard	Conc. / wt%	n.d.	n.d.	n.d.	n.d.	0,0042	n.d.	0,085	99,9	n.d.	n.d.	0,042	n.d.	0,009	n.d.
				Rel. unc %							12,	0,002						
	782	6	iron dagger	Conc. / wt%	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,014	99,99	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
				Rel. unc %					19,		7,	0,01	18,					
				Rel. unc %		3,0			4,	17,	5,	0,02	4,	Ш,	8,			
	780	3	iron sword	Conc. / wt%	n.d.	0,00021	n.d.	n.d.	0,0098	0,06	0,019	7,00	0,018	0,027	0,18	n.d.	n.d.	n.d.
				Rel. unc %		3,4			6,		22,	0,01	7,		7,			
	617	3	iron sword	Conc. / wt%	n.d.	0,00023	n.d.	n.d.	0,0109	n.d.	0,008	9,66	0,022	n.d.	0,15	n.d.	n.d.	n.d.
				Rel. unc %	16,						6,	0,003				12,		
	323	5	Knife	Conc. / wt%	0,0068	n.d.	n.d.	n.d.	n.d.	n.d.	0,014	99,95	n.d.	n.d.	n.d.	0,025	n.d.	n.d.
				Rel. unc %		3,9			40,		3,1	0,02	6,	24,	5,			
	322	-	Sword	Conc. / wt%	n.d.	0,00026	n.d.	n.d.	0,0047	n.d.	0,124	99,5	0,018	0,045	0,29	n.d.	n.d.	n.d.
				Rel. unc %	4,						3,7	0,1	4,		4,			
	321	1	Sword	Conc. / wt%	0,0187	n.d.	n.d.	n.d.	n.d.	n.d.	0,186	97,8	0,039	n.d.	2,0	n.d.	n.d.	n.d.
				Rel. unc %	4,0	5,			6,		3,4	0,02			Ш,	5,	8,	
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				Rel. unc %			15,				8,	0,2						
Spectr. 316 317 318 318 Nr. Code 8 1 317 318 1 Nor. Code 8 1 8 1 318 1 Indian Indian Indian Indian Seel $Seel$	319	4	Indian Blade, crucible steel	Conc. / wt%	n.d.	n.d.	1,0	n.d.	n.d.	n.d.	0,025	98,9	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Spectr. 316 317 318 317 318 Nr. Code 8 3 3 317 318 r Code 8 r 8 8 8 r Code 8 r 8 31661 31661 31661 Name Bow 1A, Bow 1A, 30001 30001 30001 30001 Et Det Conc. 800 31400 30001 30001 H $0,0052$ Surface 800023 300012 300012 300012 H $0,0052$ 300023 300023 300023 300023 H $0,0062$ 300023 300023 300023 300023 H $0,00023$ 300023 300023 300023 300023 H $0,00023$ 300023 300023 300023 300023 H $0,00023$ 1000023 10003 10013				Rel. unc %	10,	3,2			34,		3,5	0,01	б,		θ,		14,	
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Spectr. 316 317 Nr. 316 317 None 88 8 None 800^{14} $1ndian$ Sweld $Syeld$ $Syeld$ Name $Bow 1A_1$ Bow Bow 1A_1 $Bow 1A_1$ Bow Sweld $Syeld$ $Syeld$ Sweld $Symed$ Bow Bow 1A_1 $Bow 1A_1$ Bow Sweld $Symed$ $Symed$ Bow 1A_1 $Bow 1A_1$ Bow Sweld $Symed$ $Symed$ Bow 1A_1 $Bow 1A_1$ Bow Bow 1A_1 $Bow 1A_1$ Bow $Wydoc$ $Wydoc$ $Symed$ $Wydoc$				Rel. unc %	14,	3,6			14,		3,2	0,01	13,		12,		Ш,	
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Spectr. 316 Nr. 316 None 8 Name $9xeel$ Sweel $8xeel$ Name $9xeel$ Burdan $8xeel$ Name $9xeel$ PL ywe Burdan $8xeel$ PL ywe Burdan ywe PL ywe P				Rel. unc %	10,	5,					4,	0,02			18,	7,	16,	
Spectr. Nr. Nr. Code Name EL bet. EL wt%6 H 0,005 P I Q	316	8	Indian Steel Bow 1A, blued surface	Conc. / wt%	0,0112	0,00021	n.d.	n.d.	n.d.	n.d.	0,377	9,66	n.d.	n.d.	0,021	0,025	0,004	n.d.
EL H H B B B B B B B B B B B B B B C C C C	Spectr. Nr.	Code	Name	Det. Lim./ wt%	0,005	0,0001	I	0,05	0,001	0,05	0,005	0,2	0,005	0,01	0,02	0,003	0,003	3
				EL	Н	в	Р	s	СГ	СА	MN	FE	Co	ī	Cu	AG	Au	С

Table 2. PGAA results.

DETECTION LIMITS ARE QUOTED IN MG

H 0.3 C 460 Fe 9 P 100 O 10 000 N 60

Although the elements of most interest were difficult to detect, an interesting observation was made on the blued surface of the Indian steel bow.

It is known that heating *in air* to around 200 - 250°C produces a blued surface on steel, important historically for decoration as well as rustproofing. It has generally been assumed that this coating was magnetic iron oxide, Fe_3O_4 .

It was surprising to find wustite (FeO) also present as a component, and this will mean reevaluating some of our theories about armour decoration¹⁶.

NEUTRON RADIOGRAPHY

Neutron radiography as a direct imaging technique has been used to reveal the inner details of the objects, where the visual representation of an object is obtained non-destructively by detecting the modification of an incident beam as it passes through the matter. The interactions between the radiation and the object determine the contrast, revealing the internal structure of the sample.

A setup called NORMA was recently installed as a part of the NIPS experimental station, where the thermal equivalent flux of the guided cold neutron beam is about 2×10^7 cm⁻²s⁻¹ and the cross-sectional area of the neutron beam is 40×40 mm².

The objects were positioned in the sample chamber using ad hoc sample support. Their positions were controlled by Gd-markers attached to the object under investigation. The transmitted neutrons create signals in a two-dimensional position sensitive detector (a ⁶Li-doped ZnS scintillator coupled to a Peltier-cooled Andor CCD camera) located behind the sample. The spatial resolution (about 500 μ m) of the imaging system at the 100 mm object-detector distance was limited by the divergence of the neutron beam (L/D=233). The raw two-dimensional digital image was corrected with the open beam profile recorded in the absence of the sample (to compensate for the spatial inhomogeneity of the beam) and also with the dark image recorded with closed neutron beam.

RADIOGRAPHS

Those of the Indian sword (# C) and Indian knife (# E) show some discontinuity in the blades which *might* be due to segregation of the cementite in a hypereutectoid steel.

¹⁶ See for example Thomas, B.& Gamber, O. "Katalog der Leibrüstkammer" (Vienna, 1976) 26.



Radiograph 1.

Radiograph 2.

ANALYSIS OF SOME INDO-PERSIAN BLADES BY MEANS OF PIXE AND TOF-ND

List of the Oriental blades used for these experiments: none of these had been investigated by PGAA

- 1. A finely watered blade of a sossun pata form (reverse edge).
- 3. A blade of a sossun pata form (reverse edge) but with watering unknown.
- 4. An Arabian (?) blade with a gilded hilt with an etched inscription showing it to have been presented to a Commander Williams by the Sultan of Oman in 1840.
- 5. An Indian pattern-welded blade with a *koftgari* inscription on the back of blade done by counterfeit damascening.
- 6. A late 19th century military blade with in inscription showing it to have been sold by Monton, bright plated, with a surface decorated by etching.
- 8. An Indian (?) watered blade with a long-stranded pattern, and an inlaid inscription in gold along the forte closest to hilt.
- 9. A finely watered Indian blade with a with an umbrella inlaid in gold (*chatri*) and an inscription in a cartouche. A closely-woven pattern with a "ladder".
- 11. A shamshir blade with "open" dark watering.
- 12. A finely watered Indian blade; a shamshir with an inscription in a cartouche inlaid in gold, and bearing a closely-woven pattern on its surface.



Figure 9. Blade 1. A finely watered blade of a sossun pata form (reverse edge).



Figure 10. Blade 3. A blade of a sossun pata form (reverse edge) but with watering unknown.



Figure 11. Blade 4. An Arabian (?) blade with a gilded hilt with an etched inscription showing it to have been presented to a Commander Williams by the Sultan of Oman in 1840. Blade 4. Close up



Figure 12. Blade 5. An Indian pattern-welded blade with a *koftgari* inscription on the back of blade done by counterfeit damascening.



Figure 13. Blade 6. A late 19th century military blade with in inscription showing it to have been sold by Monton, bright plated, with a surface decorated by etching.



Figure 14. Blade 8. An Indian (?) watered blade with a long-stranded pattern, and an inlaid inscription in gold along the forte closest to hilt. Blade 8 Close up.



Figure 15. Blade 9. A finely watered Indian blade with a with an umbrella inlaid in gold (*chatri*) and an inscription in a cartouche. A closely-woven pattern with a "ladder". Blade 9. Close up 1. Blade 9. Close up 2.



Figure 16. Blade 11. A shamshir blade with "open" dark watering.



Figure 17. Blade 12. A finely watered Indian blade; a shamshir with an inscription in a cartouche inlaid in gold, and bearing a closely-woven pattern on its surface. Blade 12. Close up.

PROTON-INDUCED XRAY-EMISSION SPECTROSCOPY (PIXE)

The PIXE measurements were performed at the 5MV Van de Graaff accelerator of the Institute of Particle and Nuclear Physics, Wigner Research Centre, Hungarian Academy of Sciences. A Proton beam of 2.5 MeV energy was extracted from the evacuated beam pipe to air through a 7.5 µm thick Kapton foil. Target-window distance of 10 mm was chosen at which distance the beam diameter was found to be about 1 mm. External beam currents in the range of 1-5 nA were used. Characteristic X-ray spectra were taken by an AMPTEK X-123 X-ray spectrometer. The energy resolution of the 25mm² x 500µm SDD detector was 125 eV for the Mn Kα line. The detector was positioned at 135° with respect to the beam direction. To reduce the low energy X-ray counts an Al absorber of 100 µm thickness was applied in front of the detector. The net X-ray peak intensities and the concentration calculations were made by the off-line GUPIX program package¹⁷.

¹⁷ J.L. Campbell, T.L. Hopman, J.A. Maxwell, Z. Nejedly, "The Guelph PIXE software package III: alternative proton database", *Nucl.Instr.Meth.* B 170193-204 (2000).

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The beam of protons is directed onto the surface of the target specimen, in this case, a sword. The Xrays generated by the protons' collision with atoms in the target are collected and analysed. Since the frequency of the Xrays is related to the atomic number of the target atoms, the Xray spectra can yield the elemental composition of the target specimen.

Compared with Xray-Fluorescence, this technique can offer a more closely focussed beam, although elements lighter than fluorine cannot be determined.

Seven of the swords listed above swords were analysed by PIXE - Numbers 4, 5, 6, 8, 9, 11 and 12. The results (in ppm) are tabulated below.

				1	1		1			
SWORD NUMBER	Mn	Fe	NI	Tı	V	Cr	Cu	Zn	AG	Au
4	2792	996525	0		trace		292.8	170.1	0	0
5	280.6	996714				trace	2040		0	191.3
inscription	792.7	697141					755.6		0	300989
6	577.6	403662	579228				8106	8119	0	287.5
	418.5	995090	356.4				3814	260.8	0	0
	473.6	994738	165.7				4157	481	0	0
8	2057	994100				trace	1300		2342	71.6
	1472	997997					415.9		0	0
	1622	995227					1849		1167	19.2
9	38.9	999150					702.4		0	0
umbrella	1011	696154					814.1			302021
cartouche	742.1	233914					1919		6781	756530
11	313	998395					1130		0	
	281.9	999060					566.3		0	
	416	350937		0	0		8994		0	639608
12	380.3	426136			trace		8323		14597	550620

Table 3.

Blades number 8, 9, 11 and 12 have watered patterns (the so-called "Damascus steel") while number 4 *might* have such a pattern, but the surface is in such a corroded condition that no pattern is visible.

It is interesting that only blades 4 and 12 seem to contain any traces of vanadium in their steel. It has been suggested that traces of vanadium were essential for the segregation of the cementite in order to form a pattern on the surface, but this does not seem to be the case here.

The Inscriptions on swords 5, 9 and 11 seem to have been done in virtually pure gold, while that on 12 is slightly less pure (but still around 22 carat !).

NEUTRON DIFFRACTION

The different phases which may be present in steels include:

- Ferrite (α-Fe or pure iron)
- Cementite (Fe₃C) which may be present as massive cementite, or as a component of pearlite, or bainite, or tempered martensite.
- Martensite, which is seldom found entirely untempered.

Austenite (γ -Fe) which is not usually found in historic steels, unless a very high-carbon steel has been quenched, but not tempered, in which case retained austenite could be a micro-constituent.

These phases have different lattice parameters, and so may be distinguished by neutron diffraction. The carbon content of the steel may be derived from the proportion (atomic %) of cementite present. It is also possible that irregularities in the distribution of cementite (due to a "watered" pattern) may be revealed.

Of course, for revealing such a pattern, only two phases (ferrite and cementite) need to be detected. The cementite spacing may be from 1 to 10 μ but is mostly 2 - 5 μ .

If two spectra are obtained from the same blade, measured by rotating the blades around their longitudinal axes, the preferred orientation of both the ferrite and cementite can be determined as well.

Six of these swords were analysed by TOF ND; they were numbers 1, 3, 4, 5, 8, 9.



RESULTS - GRAPHS FOR BLADES 1, 4, 8 AND 9

Graph for blade 1.

Graph for blade 4.



The diffraction patterns are roughly normalized to 10g ferrite content (the typical weight of the illuminated volume) using the intensity of the indexed ferrite peaks. The "pure" cementite is an artificial specimen, containing some ferrite, martensite and graphite.

RESULTS OF NEUTRON DIFFRACTION

- 1. A finely watered blade; "High cementite", (perhaps 1.8%C) and it shows marked "anisotropy".
- 3. No visible pattern; "No cementite".
- 4. An Arabian blade; a "little cementite" (around 0.45%C) with a slight difference in different orientations, so "slight anisotropy".
- 5. A "pattern-welded" blade; "No cementite at all".
- 8. A watered blade with a long-stranded pattern, this was "slightly anisotropic", and had a much lower C% than #4 (around 0.1%).
- 9. A finely watered blade; an overnight run showed clear segregation of cementite, but "little or no anisotropy".

Sword Number	A "WATERED" PATTERN VISIBLE	CEMENTITE PRESENT	ANISOTROPIC DISTRIBUTION OF CEMENTITE				
1	yes	yes	yes				
3	no	no	no				
4	obscured	little	slight				
5	no	no	no				
8	yes (long)	little	slight				
9	yes	yes	no				

Table 4. Table of results

COMMENTS

As expected, most of the watered blades gave diffraction patterns high in cementite. But not all of the high-carbon blades showed anisotropy.

The approximate carbon content may be deduced from the relative heights of the diffraction peaks for cementite and ferrite, on the linear scale, but more calibration work will be needed to make this a reliable quantitative method of analysis.

Anisotropy is evident when different positions of the blade appear to indicate different amounts of cementite. The difficulty is that while the segregation of cementite may lead to a pattern where the rows of cementite particles intersect the surface, different forging techniques (such as those employed to form the "Ladder" patterns) may involve redistributing the rows of cementite particles in two directions, both parallel to and perpendicular to, the longitudinal axis of the blade and so cancel out the apparent anisotropy.

CONCLUSIONS

The most useful techniques for the non-destructive analysis of swords seem to be, to some extent PIXE, but especially, ND. PIXE analysis on seven Indo-Persian blades was carried out. The trace elements found in some blades did not include vanadium, which has been claimed by some workers to be necessary for the formation of "Damascus" patterns. Perhaps other

mechanisms are involved in their formation. Analysis of the gold used to inlay inscriptions showed that there was sometimes variation in its composition between blades. But if the gold alloy used in any one workshop or group of workshops was constant in composition (which seems likely) then we might have a potential method of identifying workshops in the Bactrian-Persian-Indian cultural area.

Six Indo-Persian blades were analysed by ND which has proved to be *the most useful method of identifying high-carbon steels* (such as are to be found in "Damascus" blades) but the detection of anisotropy in patterned blades has proved to be more complex. Some blades with clearly visible patterns show less anisotropy that might have been predicted. This could be due to forging methods which have led to a less asymmetrical distribution of carbides than expected. However if this were to be found regularly, then this possible insight into different forging techniques could be extremely interesting.

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