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THE MATELLOGRAPHIC EXAMINATION OF IRON IMPLEMENTS FROM MEDIAEVAL VILLAGE IN DRACY, BOURGOGNE (FRANCE)

In 1965–1981, the Polish archaeological expedition from Institute of Material Culture of Polish Academy of Science in Lodz carried out excavations in Dracy, Bourgogne (France). The Author of this paper acquired some iron implements found there for matallographic examinations, namely: 9 knives, 7 horseshoes, a pair of scissors, one arrow, one fibula and two fragments.

METHODS OF EXAMINATION AND PRESENTATION OF THE RESULTS OBTAINED

The methods of the examinations were „standardized", i.e. the same as in all other Author's researches[1].

a) the quantitative chemical analyses of phosphorus, and – if possible – nickel, copper and manganese content were carried out; the phosphorus content was determined using the weight method, nickel and copper content – using photometric method.
b) the structure of metal was observed using the matallographic microscope with estimation of grain size of structural constituents using ISO standard 643-83.

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c) the microhardness of particular structural constituents was measured with the Hanamann's tester using 50 gms loading for the iron and steel not heat-treated or 100 gms loading for quenched steel; each result is the arithmetic mean of 5 measurements,
d) Vickers hardness of metal was determined using 10 kgs loading for iron and steel without heat treatment and 30 kgs for quenched steel. Each result is the arithmetic mean of 2–4 measurements.

The phosphorus content represents the quality of the metal and the type of ore used for smelting, i.e. the origin and provenience of implement examined, the scope of the determination of nickel and cooper content is similar. The manganese content was used to estimate the technology of the iron smelting process; indirect smelting may have appeared in fourteenth century France (the blast furnace + the finery); such a metal contains more manganese (and silicon).

Other examinations reveal the characteristics of metal and technology.

To estimate the accuracy of microhardness measurements the mean range was calculated as follows: for ferrite – 5,7 kg/mm^2 (i.e. standard deviation – 2,3 kg/mm^2), for pearlite and sorbite 9,2 kg/mm^2 (i.e. standard deviation – 4,3 kg/mm^2). So the microhardness determination accuracy for ferrite was about +- 5 kg/mm^2, and for pearlite or sorbite – about +-18,8 kg/mm^2.

The technology of examined implements was determined basing on objective criteria, described elsewhere[2]. The procedure of the presentation of the results obtained was the same as presented in another paper of the Author[3].

THE RESULTS OF THE EXAMINATIONS

The examined iron implements from Dracy are presented in Fig. 1, and the technology of its fabrication – in Fig. 2.

The results of chemical analyses are given in Table 1 (the knives and scissors) and Table 2 (other implements). The result of matallographic examinations, with grain determination, measurements of microhardness of structure constituents and Vickers hardness are summerized in Table 3 (the knives and scissors) and Table 4 (other implements).

The examined knives present very different techniques of fabrication, and – probably –origin. The most complicated technique was used for making the knife No 1, welded of tree straight bands of steel and three bands of iron. (fig. 4 and 4).

In the steel band, near the cutting edge of the knife, a sorbitic structure and a fine pearlite in other two were observed. The microhardness of the former was higher (303 kg/mm^2) than in the two other (about 250 kg/mm^2).

The structure of the iron bands was ferritic. Carbon diffusion from steel parts into iron parts was observed.

Slag inclusions in the metal were of a uniform dark colour (type A according to the Author's classification[4]), some of them – especially in the iron parts – contained rounded precipitations of light phase on dark background (type D2).

The knife No 8, forged of one piece of iron, contained bands of steel, on the edge and on the back of the instrument. In the steel bands a tempered martensite (fig. 5) and in the iron part a ferritic structure were found. The slag inclusions were mostly of a dark colour (type A), only some inclusions were light (type C).

The knife No 6 was made of one band of steel (the edge) and one band of iron (the back). In the steel band a tempered martensite with trace of ferrite (fig. 6), and in the iron back – a fine grained ferrite were observed. The slag inclusions were of a uniform dark colour (type A), although some of them contained a rounded light phase precipitation on dark background (type D1).

Three knives, Nos 2, 3 and 4 were forged of irregularly carburized steel, containing 0,3–0,7% C and 0,075–0,19% P (fig. 7). In the structure of knives Nos 2 and 4, sorbite and ferrite were observed. Apart from the slag inclusions of a dark colour (type A0), in the knives Nos 3 and 4 some inclusions containing rounded precipitations of light phase on dark background (type D1) were observed.

The three described knives Nos 2, 3 and 4 were heat-treated. The knives Nos 2 and 4, after having been quenched, were probably tempered at a higher temperature than the knife No 3, which was revealed in the fine structure of the tempered martensite (fig. 8).

All knives, made of steel or welded of iron and steel were heat-treated.

The knives Nos 5, 7 and 9 were made of iron. The knife No 5 presented a fine grained ferritic structure of high phosphorous iron, with dark slag inclusions (type A). In the knife No 7 forged of low phosphorus fine grained iron, very small traces of pearlite were observed (fig. 9). Apart from the dark slag inclusions (type A), some other inclusions containing rounded precipitations of light phase on dark background were observed (type B and D1).

In the ferritic structure of the knife No 9 forged of low phosphorous iron, small precipitations (lenght up to 0,03mm) of nitride α"– $Fe_{16}N_2$ could be seen. The indentification of these precipitatons were published by G.R. Booker, J. Norbury and A.L. Sutton[5]. The slag inclusions on the knife No 9 were generally of a dark colour (type A), but some of the inclusions contained some rounded phases on black background (type B and D1).

The scissors were made of irregularly carburized steel, containing 0,3 to 0,8% C. In the structure, tempered martensite was observed. The slag inclusions were of a dark colour (type A), the scissors were heat treated, probably quenched and tempered (fig. 10).

Some differences in metals and technologies were observed in the examined horse-shoes. Horse-shoes Nos 2, 5, 6 and 7 were made of high phosphorous iron containing 0,20–0,46% P (arithmetic mean – 0,27% – fig. 11). In the horse-shoe

No 2, some slag inclusions of a uniform dark colour (type A) and two-phase inclusions type D1 and D2 were observed.

In the ferritic trains of horse-shoe No 2, shape precipitations (lenght 0,02mm) of nitrides γ'– Fe_4N, and small ones (lenght below 0,01mm) of nitride α''– $Fe_{16}N_2$ were observed.

In the horse-shoe No 5, apart from dark colour slag inclusions (type A), there existed also a few light ones (type C), and some two-phase ones, type D2.

The slag inclusions were also present in similar structures as observed in the horse-shoe Nos 6 and 7, only the slag inclusions type D2 were replaced by inclusions type B.

The horse-shoe No 4 presented a ferritic structure with traces of pearlite. It was made of a low phosphorous steel, containing about 0,05% C; the mechanical properties of such a steel are very similar to these of iron with its low concentration of phosphorus. Apart from numerous dark slag inclusions (type A), inclusions with numerous precipitations of light phase (type D22), sometimes in dendritic form (type D32) were observed.

A more complicated structures was observed in the horse-shoe No 3. It was forged of low phosphorous iron (very similar to that used for making the horse-shoe No 4), and hardened by secondary carburization (the cementation).

The cementation was quite deep. Near the cemented surface of the horse-shoe the structure was pearlitic i.e. carbon content reached about 0,8% C (fig. 12). No heat treatment was applied. The slag inclusions in the metal contained some rounded precipitations of light phase on dark background (type D). Some inclusions revealed a uniform dark colour structure (type A).

The horse-shoe No 1 was made of two pieces of metal: one was a fine-grained, high phosphorous iron, the other – low carbon steel containing about 0,15% C (fig. 13). The welding of both fragments was rather poor (fig. 14). Slag inclusions contained bright „glittering" points on dark background.

DISCUSSION AND COMMENTS

All examined iron implements from the mediaeval village in Dracy (14th cent.) were made of metal smelted in bloomery (direct process), not using the indirect process which may have appeared in France at that time.

The classification of this metal should be grounded on the carbon and the phosphorus content, the most important constituents of the early iron[6], as follows:

1. metal, smelted using low phosphorous iron ores (hematite, siderite, etc.)[7]:
   a. steel, containing below 0,05–0,06% P,
   b. Iron, containing below 0,15–0,18% P;
2. metal, smelted using high phosphorous iron ores (bog ore etc.):

a. steel, containing over 0,06% P (up to 0,4%),

b. iron, containing 0,18–0,5% P (may be – to 1,0% P).

The other elements (Ni, Cu, Mn) exist as traces and have no essential influence on the properties of the metal. Thus, the low phosphorous steel was used probably only for the fabrication of the fibula. The knives Nos 1 and 9, and the horse-shoes Nos 3 and 4 were forged of low phosphorous iron.

More implements (76%) were made of high phosphorous metal of a rather poor quality: the knives Nos 2, 3, 4 and scissors – of steel, and the knife No 5, the horse-shoes Nos 1, 2, 5, 6, 7 the arrow and both fragments Nos 1 and 2 – of iron. The knives Nos 1, 6 and 8 welded of iron and steel belong to this group. Similar irons were used in mediaeval Poland[8].

The inhabitants of the mediaeval village in Dracy, made mostly use of the metal which was smelted in this region using high phosphorous ores. The origin of the implements made of low phosphorous metal is not determined.

The techniques used for making the examined implements were different. The cutting tools, the knives and scissors were made of steel or welded of iron and steel. However, the knives Nos 1, 5 and 9 were forged of iron, these different techniques could be used in the same production centre.

The knife No 6 welded of iron and steel, represents technique of welding iron with the most frequently used method since the 14^{th} cent A.D. (type IV 1A1 according to the Author's classification)[9].

The techniques used for the fabrication of the knives Nos 1 and 8 were not present in the mediaeval Poland of that time.

On the territory of Poland in the early Middle Ages the cutting tools were mostly welded of iron and steel (51,8%), other were forged of iron (20,2%) or steel (11,8%); 16,2% of the cutting tools was made of iron and carburized (cemented).

The horse-shoes, the arrow and the (other?) fragments were forged of iron. The horse-shoe No 3 was carburized (cemented), but not heat-treated as were all the other examined knives and scissors.

The horse-shoes from Rougemont le Château (neighbourhood of Belfort), in the 13^{th}–14^{th} cent. were forged of iron; in some of them a carburized band (cemented?) was observed[10].

Similarly, mediaeval horse-shoes and arrows found on the territory of Poland were made of iron. One horse-shoe (from Sieradz, 13^{th} cent.) and some arrows (from Tum, near Łęczyca, 12^{th}–13^{th} cent.) were cemented after forging (but not heat-treated).

In the horse-shoe No 7 and the knife No 9 the nitrides were observed. However, these precipitations in the early iron implements were not a result of a special nitriding process as thought by R. Marechal[11]. Bloomery iron can be soaked in nitrogen during smelting and – when the cooling rate is suitable – the nitrides are precipitated.

On the territory of central Poland in the Antiquity, the nitrides are very frequent in the iron implements found in cremation cemeteries, but are not observed in irons found in settlements.

The iron and steel technology observed in examined implements from Dracy (Bourgogne) may probably be typical of mediaeval villages in France.

Notes

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[2] J. Piaskowski: *Proposals for a standardisation of the criteria for determining technological process in early iron implements*. In: *The Crafts of Blacksmith*. Belfast 1984 p. 157–168.

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[4] J. Piaskowski: *Classification of the structure of slag inclusions in early objects made of bloomery iron*. „Archaeologia Polonia" 1976 Vol. 17 p. 139.

[5] G.R. Booker, J. Norbury, A.L. Sutton: *Investigation of nitride precipitation in pure iron and mild steel*. „Journal of the Iron and Steel Institute" 1957 Vol. 187 p. 205–215.

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Jerzy Piaskowski

METALOZNAWCZE BADANIA PRZEDMIOTÓW ŻELAZNYCH ZE ŚREDNIOWIECZNEJ WSI W DRACY, BURGUNDIA (FRANCJA)

Przedstawiono wyniki badań metaloznawczych 21 przedmiotów żelaznych z XIV wieku z wczesnośredniowiecznej wsi w Dracy, Burgundia (Francja), pochodzących z prowadzonych tam prac archeologicznych Instytutu Kultury Materialnej PAN w Łodzi (Polska). Zbadano skład chemiczny i twardość badanych przedmiotów (na zawartość P, Ni, Cu, Mn), określono strukturę metalu i twardość Vickers'a, mikrotwardość składników struktury, określono sposób ich wykonania (technologię).

Najbardziej skomplikowaną technologię wykazał nóż nr 1, zgrzany z 3 prętów żelaznych i 3 prętów stalowych. Z jednego z tych ostatnich zostało ukształtowane ostrze noża. Nóż nr 6, wykuty z żelaza, posiadał nakładkę stalową, stanowiącą ostrze (był to najczęściej stosowany sposób wykonywania noży w Polsce, oznaczony symbolem IV.1.A.2). Nóż nr 8 posiadał także taką nakładkę, z tym, że drugą taką nakładkę umieszczono na jego grzbiecie (typ IV.1.B.2).

Natomiast noże nr 2,3,i 4 oraz nożyce, wykute ze stali dymarskiej o nierównomiernym nawęgleniu, zawierały 0,075–0,15% P. Wszystkie opisane wyżej narzędzia były poddane prawidłowo przeprowadzonej obróbce cieplnej.

Natomiast z żelaza dymarskiego wykuto noże nr 5,7 i 9 (typ 1); ten ostatni, wykuty z żelaza o bardzo nikłej zawartości fosforu (0,015%P), zawierał dużą domieszkę niklu (0,31%Ni). Z żelaza wykuto także 6 szt. podków (niektóre z nich wykazały śladowe nawęglenie pierwotne; podkowa nr 3 wykazała przy powierzchni nawęglenie, prawdopodobnie wtórne), grot strzały, zapinka oraz nieokreślone fragmenty nr 1. W nożu nr 9 i w podkowie nr 2 wystąpiły wydzielenia azotków – $Fe_{16}N_2$ i Fe_4N.

Zbadane przedmioty żelazne ze wsi Dracy wykonane zostały z żelaza i stali, przy zastosowaniu takich samych technik, jakie występowały w średniowieczu na ziemiach Polski.

Table 1. Results of chemical analyses of knives and scissors from Dracy, Bourgogne

No.	Object	Inv. No.	Weight gms	Content, %			
				P	Ni	Cu	Mn
1.	Knife No 1	154	11,8	0,188	0,08	0,00	0,00
2.	Knife No 2	225	5,2	0,075	0,03		0,00
3.	Knife No 3	165	16,7	0,15	0,00	0,00	
4.	Knife No 4	161	11,5	0,075	0,09		0,02
5.	Knife (?) No 5	102	7,8	0,216	0,11	0,00	
6.	Knife No 6	308/67	12,9	0,18	0,00		
7.	Knife No 7	324/67	5,0	0,04	0,09		
8.	Knife No 8	331/67	4,5	0,15	0,12		
9.	Knife No 9	335/67	10,7	0,013	0,31		
10.	Scissors	390/67	9,0	0,15			

Table 2. Results of chemical analyses of horse-shoes and other implements from Dracy, Bourgogne

No.	Object	Inv. No.	Weight gms	Content, %			
				P	Ni	Cu	Mn
11.	Horse-shoe No 1	212	37,0	0,216	0,05	0,17	0,08
12.	Horse-shoe No 2	124	28,0	0,460	0,07	0,00	
13.	Horse-shoe No 3	330/67	9,8	0,01	0,08		
14.	Horse-shoe No 4	357/67	27,6	0,05	0,06	tr.	
15.	Horse-shoe No 5	365/67	13,4	0,20	0,11	tr.	
16.	Horse-shoe No 6	368/67	98,6	0,20	0,10	0,00	
17.	Horse-shoe No 7	381/67	14,8	0,24	0,00		
18.	Arrow	227	12,0	0,267	0,00		0,018
19.	Fibula	222	6,7	0,075	0,00		0,00
20.	Fragment No 1	156	4,0	0,341	0,09		
21.	Fragment No 2	102	4,9	0,244	0,07		

Table 3. Results of metallographic observations, grain size estimation and microhardness and hardness measurements of knives and scissors from Dracy, Bourgogne

No.	Objects	Structure constituents	Grain size	Micro-hardness kg/mm^2	Vickers hardness kg/mm^2
1.	Knife no 1				
	Edge				
	steel edge	sorbite		303	
	iron layer I	ferrite	7	191	
	steel layer I	fine pearlite		258	221
	iron layer II	ferrite	7	191	
	steel layer II	fine pearlite		243	245
	Back iron	ferrite	6	151	110,3
2.	Knife No 2	sorbite		408	264–
		ferrite	8	191	–181
3.	Knife No 3	martensite		530	
		martensite		473	242–
		sorbite		347	–245
		ferrite	8	232	
4.	Knife No 4	sorbite		435	270–
		ferrite	5	172	–163
5.	Knife (?) No 5	ferrite	7	205	202
6.	Knife No 6	martensite		389	309
		ferrite	7	205	173
7.	Knife No 7	ferrite	8	186	147,1
		pearlite	tr.		
8.	Knife No 8				
	steel egde	martensite		562	
	iron back	ferrite	6	135	178
9.	Knife No 9	ferrite*	6	126	95,8
		ferrite*	2	110	
10.	Scissors	martensite		455	249
		ferrite	8	210	

* including phase A (γ'Fe_4N?) and phase B (α'$Fe_{16}N_2$?)

Table 3. Results of metallographic observations, grain size estimation and microhardness and hardness measurements of horse-shoes and other implements investigated from Dracy, Bourgogne

No.	Objects	Structure constituents	Grain size	Micro-hardness kg/mm^2	Vickers hardness kg/mm^2
11.	Horse-shoe No 1	ferrite	7	210	179
		pearlite	6	270	
		ferrite	8	251	236
12.	Horse-shoe No 2	ferrite	3	194	206
13.	Horse-shoe No 3	ferrite fine	4	156	181
		pearlite	4	239	
		ferrite	tr.	175	
14.	Horse-shoe No 4	ferrite	8	168	148,8
		pearlite	tr.		
15.	Horse-shoe No 5	ferrite	3	152	137,7
		ferrite	7	158	
16.	Horse-shoe No 6	ferrite	6	186	170
17.	Horse-shoe No 7	ferrite	4	170	135,5
		ferrite	6	151	
18.	Arrow	ferrite	5	174	123
19.	Fibula	ferrite	6	137	120,7
		pearlite	7	210	
20.	Fragment No 1	ferrite	4	255	225
		ferrite	7	216	
21.	Fragment No 2	ferrite	7	170	193

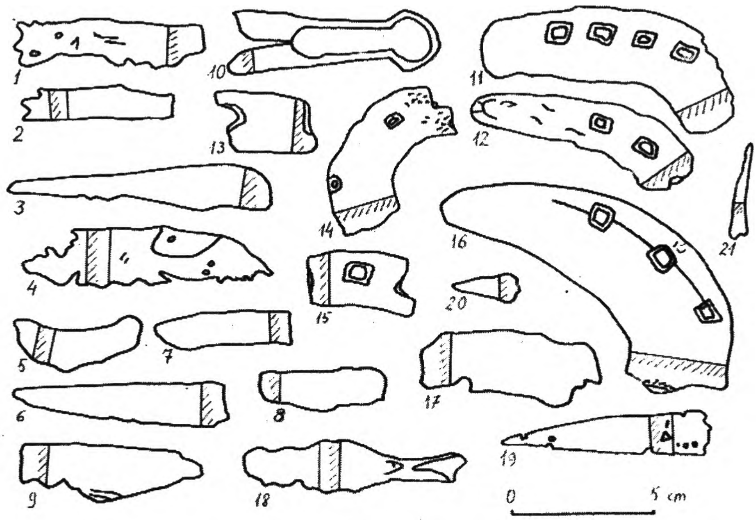

Fig. 1. Skizze of examined implements from Dracy, Bourgogne:
1 – Knife No 1, 2 – Knife No 2, 3 – Knife No 3, 4 – Knife No 4, 5 – Knife No 5, 6 – Knife No 6, 7 – Knife No 7, 8 – Knife No 8, 9 – Knife No 9, 10 – Scissors; 11 – horse-shoe No 1, 12 – horse-shoe No 2, 13 – horse-shoe No 3, 14 – horse-shoe No 4, 15 – horse-shoe No 5, 16 – horse-shoe No 6, 17 – horse-shoe No 7, 18 – arrow; 19 – fibula; 20 – fragment No 1; 21 – fragment No 2 (with position of the specimen cut out).

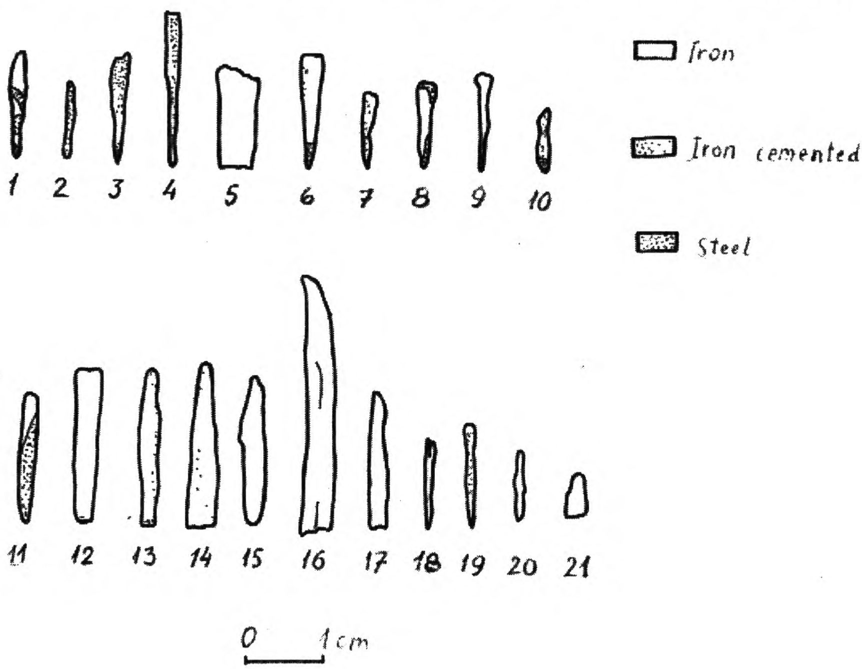

Fig. 2. Technology of examined implements from Dracy, Bourgogne (as fig. 1).

Fig. 3. Macrostructure on the cross-section of knife No 1 (dark-steel, white iron). Nital etched, 6x.

Fig. 4. Fragment of the polished surface of knife No 1, Nital etched, 6x.

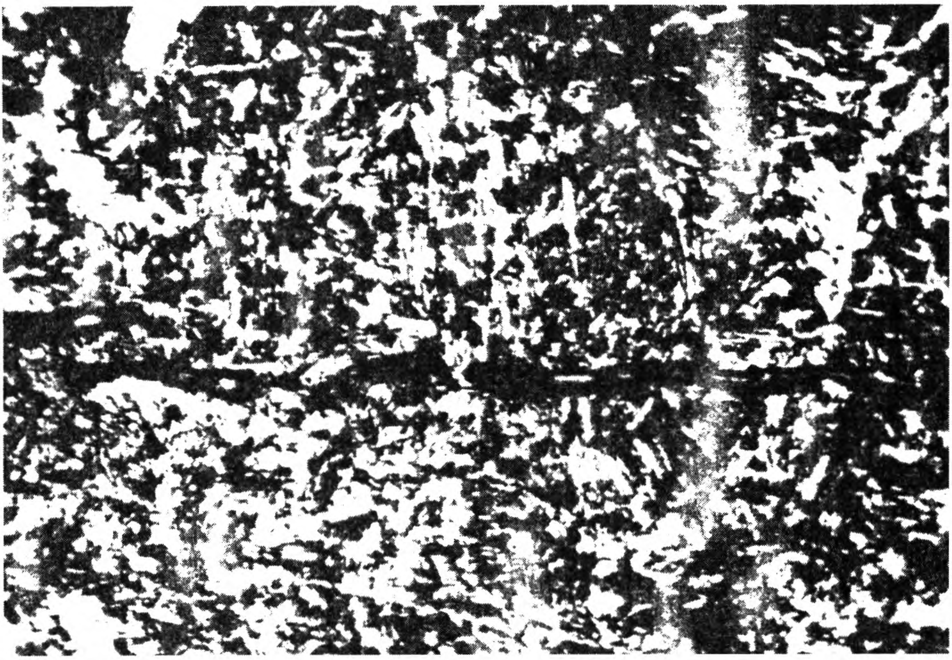

Fig. 5. Structure of steel edge of knife No 8. Nital etched, 500x.

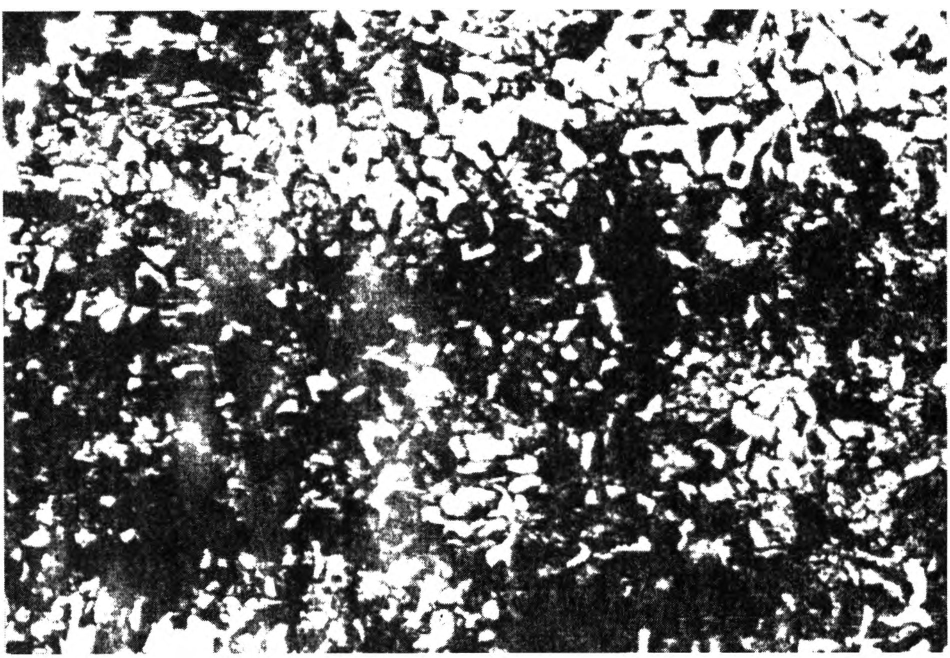

Fig. 6. Structure of steel edge of knife No 6. Nital etched, 500x.

Fig. 7. Macrostructure on the cross-section of knife No 2. Nital etched, 11x.

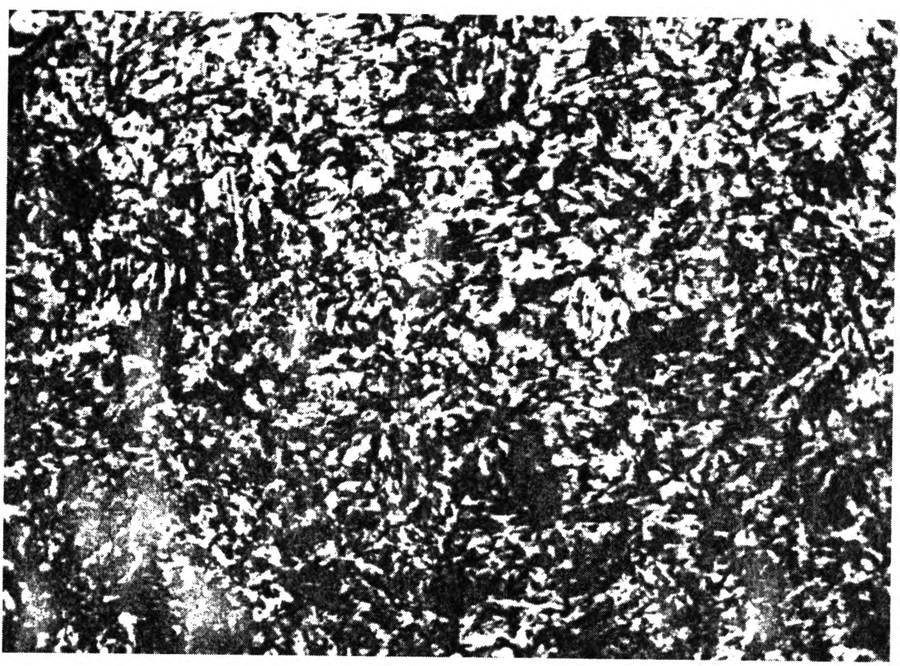

Fig. 8. Structure of knife No 3 made of steel, Nital etched, 500x.

Fig. 9. Structure of knife No 7 made of fine grained iron, Nital etched, 500x.

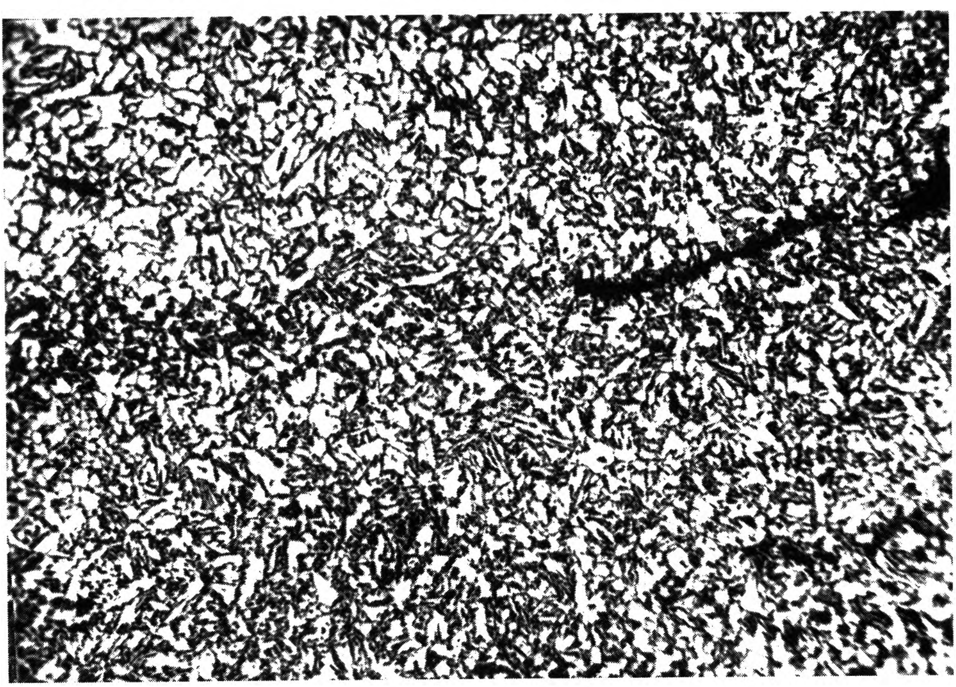

Fig. 10. Structure of scissors, Nital etched, 100x.

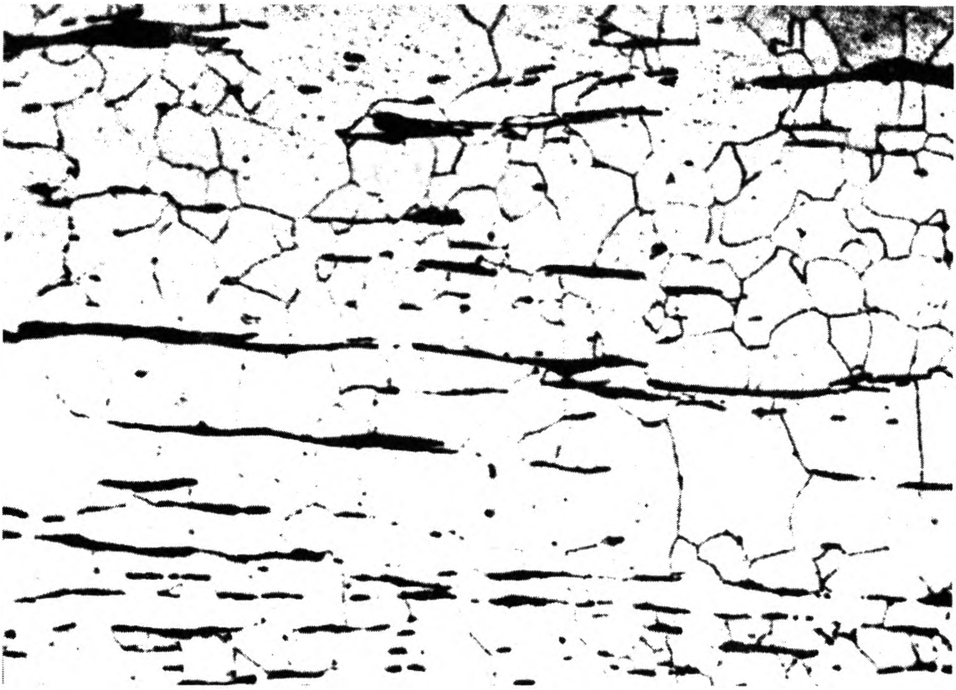

Fig. 11. Structure of horse-shoe No 7, made of the iron. Nital etched, 100x.

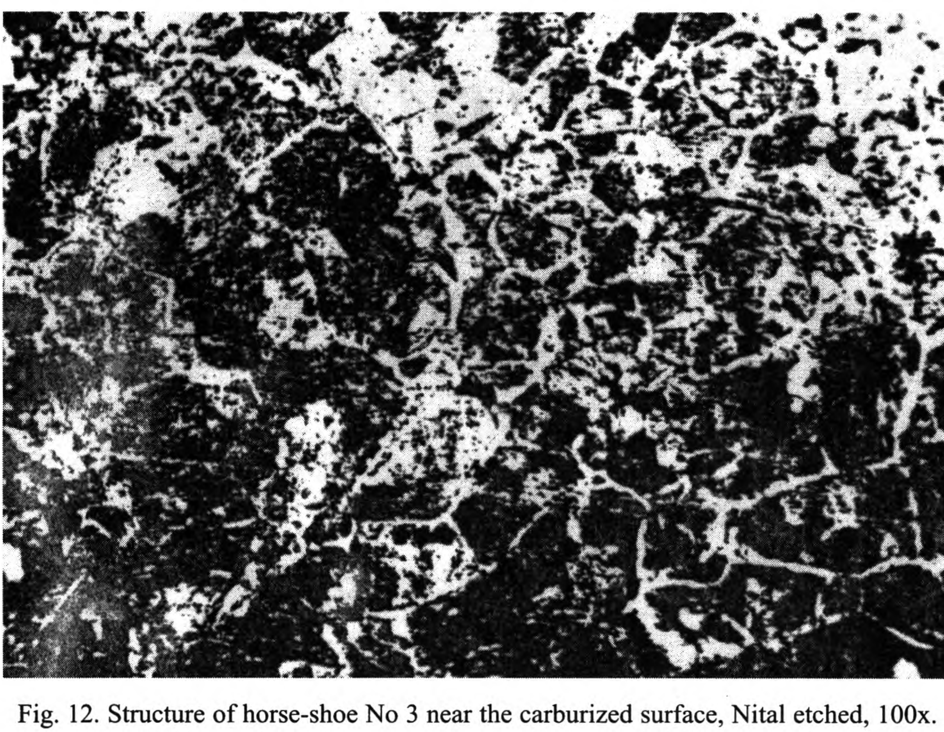

Fig. 12. Structure of horse-shoe No 3 near the carburized surface, Nital etched, 100x.

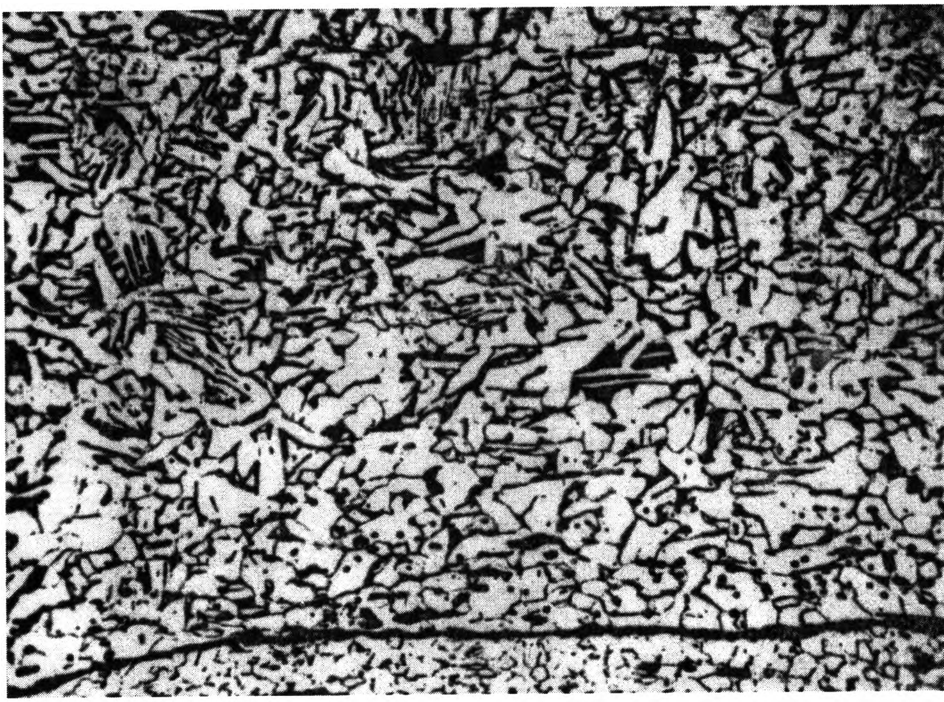

Fig. 13. Macrostructure on the cross-section of horse-shoe No 1. Nital etched, 5,5x.

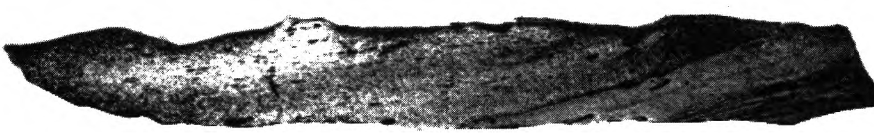

Fig. 14. Structure of horse-shoe No 1: above: iron. below: mild steel. Nital etched, 100x.

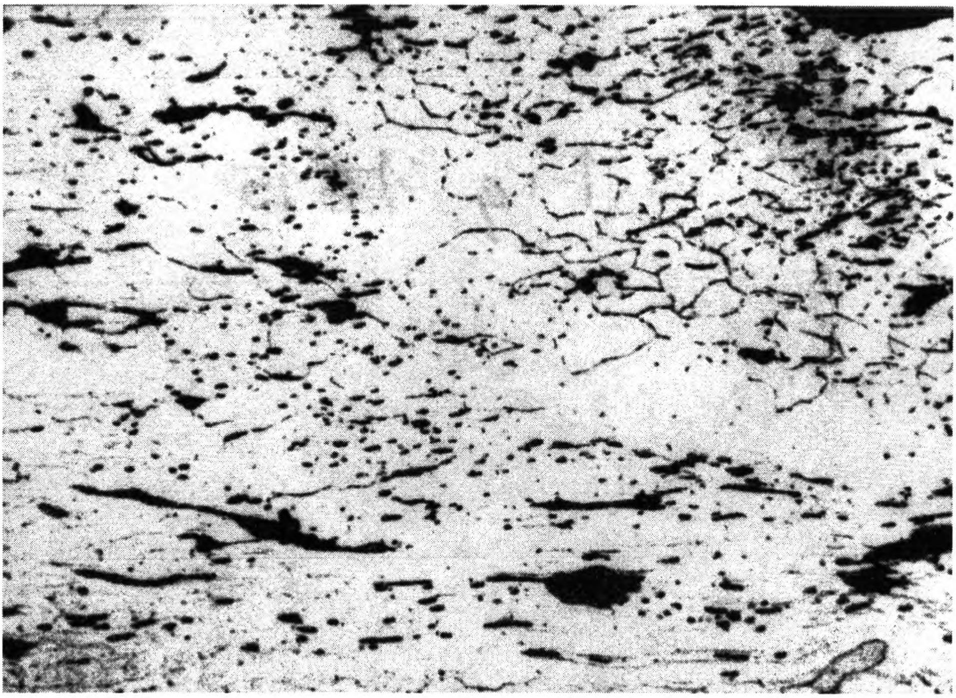

Fig. 15. Structure of fragment No 1. Nital etched, 100x.

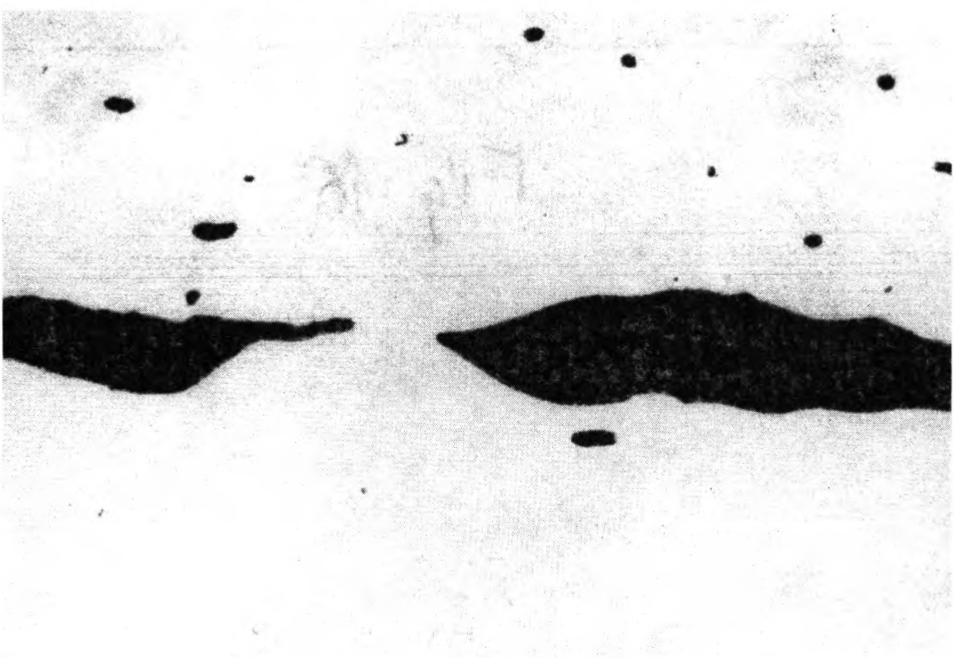

Fig. 16. Slag inclusions of type D1 in of fragment No 1, No etched, 500x.