This study is focused on the nails found at Inchtuthil, Perthshire (UK) dated back to 87 A.D.. The investigated nails were analyzed to characterize these objects. After the sectioning of the sample, an accurate optical microscopy examination has been performed in order to study the different structural constituents composing the microstructure. SEM-EDS analysis allowed to quantitatively characterize the chemical composition of non-metallic inclusions, while the SEM-EBSD examination revealed the crystallographic textures featuring the examined alloy. This information, coupled with the measurements of the micro-hardness suggests a new hypothesis on the plastic deformation process adopted for the realization of the observed nail.

**KEY WORDS:** Inchtuthil, Roman nails, carburising, non-metallic inclusions, metallography, SEM-EDS-EBSD

**EXPERIMENTAL PROCEDURE**

In this experimental investigation, a nail belonging to class E has been analysed (Fig. 1). The nail has been sectioned along the shank axis and transversally cut in two coupons. After grinding and polishing the coupons have been etched by immersion in the Nital metallographic etching (0.5%HNO$_3$ for 100ml of ethanol) for 25s.

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**INTRODUCTION**

The nails found at Inchtuthil, Perthshire, are one of the most interesting and consistent findings performed in the 20th century about the Roman steelmaking production$^{1,2,3}$. The Roman fortress at Inchtuthil covered around 20.000m$^2$ and was probably capable of holding some 5.500 men. The nail have been found in a pit 3.6m deep and covered under 1.8m of clean beaten earth. This operation has probably been performed on 87 A.D. in order to avoid that the nails can fall into hands of the Scot tribes, which prized the iron based products more than the silver and gold ones for the intrinsic potential of this material in the production of weapons and structural devices.

The total number of nails found is between 875.000 and 900.000 pieces. These are featured by different sizes as a function of the different applications and of stresses which had to be faced. Considering the large amount of the Roman nails found, these manufactures have not a large interest in the research on archeometallurgy because of their poor artistic and aesthetic value. The deep study performed by N.S. Angus et al.$^4$ allowed to classify the nails into six well defined classes, each identified by the overall length, the area of the shank section at the middle point of the shank, the shape of the head and its thickness.

The analysis developed in this study has been performed on a nail belonging to the class E according to the classification defined by Angus and co-workers, featured by an overall length of 38-63mm, by a circular disk head characterized by 9-16mm diameter and a square shank section. This type of nail is the one found with the highest frequencies, because 763.840 pieces have been counted and the observation has been integrated with the ones performed on another type of nail belonging to the class B as defined by Angus (overall length of 171-241mm, circular disk head shape, square shank section) carried out on 1992$^5$. 

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The macroscopic multi-layered aspect observed on the nail found at Inchtuthil. The clearer is the layer aspect, the higher is the perlite content of the region revealed by etching.

Struttura a strati evidenziata dal macro-attacco in un chiodo rinvenuto presso Inchtuthil. Gli strati chiari sono quelli con maggiore presenza di perlite.
allowed to observe and distinguish the different phases by optical microscopy and the distribution of the structural constituents contained in the nail. The chemical composition of the non-metallic inclusions has been measured by SEM-EDS technique and all the different phases revealed within them by EBS (Electron-Back-Scattering) have been chemically analysed. The SEM-EBSD (Electron-Back-Scattering-Diffraction) analysis has been performed on two areas of the nail sections along the shank axis. The analysed areas are prevalently constituted by ferrite and are located at 10mm far from the head side and at 10mm from the nail tip. Each observed area featured by an extension of 10mm² has been scanned by a pixel resolution of 2.5μm² associated to an accelerating voltage of 20kV. On each identified region characterized by a particular presence and distribution of the structural constituents, a microhardness Vickers test has been performed to point out the trend of the micro-hardness values. The microhardness value related to each region is the result of an averaging procedure performed on three different measurements performed with the application of a 25g load for 15s.

RESULTS

The performed metallographic etching has pointed out also the macroscopic characterization of the alternated phases (Fig. 2, Fig. 3, Fig. 4). There is an evident presence of a layered structure indicating the phases characterised by a different carbon content. The optical microscope observations have confirmed the presence of heterogeneous structure with the presence of ferrite and ferrite-perlite structural constituents, clearly indicating significant gradient in the carbon content of the different layers (Fig. 3, Fig. 4). This particular distribution of the structural constituents has been revealed also in the nail belonging to group B in which also a more significant presence of acicular ferrite takes place. The morphology of acicular ferrite suggests that it has been formed at the perlitic boundaries as the result of pro-eutectoid formation (Fig. 5).

All the non-metallic inclusions revealed by the SEM-EBSD analysis have pointed out a two-phase structure constituted by a dark matrix of fayalite (2FeO·SiO₂) with the presence of the clear wustite (FeO) phase in globular or dendritic form (Fig. 6, Fig. 7). The textures pointed out by the SEM-EBSD showed a sharp anisotropy pattern (Fig. 8, Fig. 9) characterized by the induction of strong and clear texture revealed on the ODF diagram:

- in the shank traces of γ-fiber (from {111}<100> to {111}<110> the induction of strong and clear texture revealed on the ODF diagram:

<table>
<thead>
<tr>
<th>Test</th>
<th>MgO</th>
<th>Al₂O₃</th>
<th>SiO₂</th>
<th>P₂O₅</th>
<th>CaO</th>
<th>TiO₂</th>
<th>MnO</th>
<th>FeO</th>
</tr>
</thead>
<tbody>
<tr>
<td>spot A</td>
<td>-</td>
<td>1.7</td>
<td>2</td>
<td>-</td>
<td>-</td>
<td>3.9</td>
<td>12.3</td>
<td>-</td>
</tr>
</tbody>
</table>

Typical elongated non-metallic inclusion found in the nail and the chemical composition related to point A and point B.

Tipiche inclusioni non metalliche allungate riscontrate all’interno del chiodo. Le analisi chimiche si riferiscono ai punti indicati con A e B.
The CSL recognized 10mm far from head. 
Diagramma CSL misurato a 10mm dalla testa.

The grain boundary misorientation recognized at 10mm far from tip. 
Misure di microdurezza realizzate in diversi punti di un chiodo appartenente al gruppo E.

<table>
<thead>
<tr>
<th>Zone</th>
<th>HV</th>
<th>Microstructure</th>
</tr>
</thead>
<tbody>
<tr>
<td>Head</td>
<td>112</td>
<td>Ferrite-Perlite</td>
</tr>
<tr>
<td>Connection Head-Shank</td>
<td>104</td>
<td>Ferrite-Perlite</td>
</tr>
<tr>
<td>Upper side Shank</td>
<td>114</td>
<td>Ferrite-Perlite</td>
</tr>
<tr>
<td>Lower side Shank</td>
<td>135</td>
<td>Prevalently perlite</td>
</tr>
<tr>
<td>Middle central region</td>
<td>108</td>
<td>Ferrite</td>
</tr>
<tr>
<td>Tip region</td>
<td>117</td>
<td>Ferrite-Perlite</td>
</tr>
</tbody>
</table>

Tab. 1
Micro-hardness measurements featuring the different zones of the nail belonging to group E. 
Misure di microdurezza realizzate in diversi punti di un chiodo appartenente al gruppo E.

<table>
<thead>
<tr>
<th>Zone</th>
<th>HV</th>
<th>Microstructure</th>
</tr>
</thead>
<tbody>
<tr>
<td>Head</td>
<td>208</td>
<td>Ferrite-Perlite</td>
</tr>
<tr>
<td>Connection Head-Shank</td>
<td>143</td>
<td>Ferrite</td>
</tr>
<tr>
<td>Upper side Shank</td>
<td>298</td>
<td>Ferrite-Perlite</td>
</tr>
<tr>
<td>Lower side Shank</td>
<td>343</td>
<td>Perlite</td>
</tr>
<tr>
<td>Middle central region</td>
<td>389</td>
<td>Perlite</td>
</tr>
<tr>
<td>Tip region</td>
<td>272</td>
<td>Ferrite-Perlite</td>
</tr>
</tbody>
</table>

Tab. 2
Micro-hardness measurements featuring the different zones of the nail belonging to group B. 
Misure di microdurezza realizzate in diversi punti di un chiodo appartenente al gruppo B.

The ODF diagram of the texture revealed at 10mm far from tip. 
Diagramma ODF della tessitura misurato a 10mm dalla punta.

The ODF diagram of the texture revealed at 10mm far from head. 
Diagramma ODF della tessitura misurato a 10mm dalla testa.

The CSL recognized 10mm far from tip. 
Diagramma CSL misurato a 10mm dalla punta.

The CSL recognized 10mm far from head. 
Diagramma CSL misurato a 10mm dalla testa.

The multi-layered structure by which the nail is made is confirmed by the observation of the different sections along the nail shank (Fig. 1, Fig. 2).

The layers characterized by high perlite content are present also in the core of nail and near the head, the perlite colonies assumed a significant deflection due to the plastic deformation, carried out by Roman blacksmiths to shape the head. One of the main point to be clarified for a correct interpretation of the observed structure is the comprehension of the technique followed to realize such an heterogeneous structure due to the superimposition of Byzantine and Roman blacksmiths.
6). The realization of such a slag with these characteristics is not possible in the previously analysed nail of Class B, which clearly shows the higher carbon concentration of the perlite grain (also proving that Fe:C) can act as a slowing factor for the recovery and of the following recrystallization process of the grains themselves. The misorientation of the boundaries seems consistent with this hypothesis and the lower concentration of the boundaries on the smallest angles in the head region, appears to be due to the lower boundary extension associated with the low-melting point slag used in these grains. Such a phenomenon can be associated to the heat developed by the plastic deformation due to the forging of the head which has probably taken place. According to the recrystallizations, the micro-hardness measurements have shown that these values are fundamentally ruled by the carbon content and then by the presence of the perlite region, which provides the highest strength of the steel. The maximum micro-hardness reached in the nail of Class B, studied in 1992\(^\), is much higher, because in the perlite region a maximum value of 389HV has been detected. This is certainly due to the presence of completely perlite regions intentionally formed in order to increase the strength of the nail featured by a greater size, because the larger nails were generally dedicated to undergo applications implying higher stresses (Fig. 15). This observation confirms that the ancient artisans were able to modulate the microstructure and the related properties as a function of the size of the nails and of the final applications.

CONCLUSIONS

- The analysed nail belonging to the Group E shows a composite structure featured by the simultaneous presence of ferrite colonies and perelite grains, which characterize also the nail belonging to group B featured by more extended perlite regions which provide the highest hardness value;
- the ancient blacksmiths seem to have modulated the mechanical properties as a function of the nail size which is certainly related to different applications and so to different loads;
- the non-metallic inclusions are mainly constituted by fayalite (2FeO.SiO\(_2\)) saturated by Fe\(_3\)O\(_4\). Probably, this particular formulation has been intentionally applied, because it is the characteristic of the low melting point EDS, and this certainly favours the evacuation of the slags trapped during the forging process in solid state;
- the iron oxide often feature the bulk of a layer characterized by particular structural constituents but are always present also in the region dividing two different perlite regions, where the layers are also characterized by the presence of very small recrystallized ferrite grains which can be produced by the jointing of the surface realized by welding operated among the different layers;
- the performed observations are plausible and consistent with hypothesis that the method followed to produce the multi-layered structure is the welding realized by the friction developed between adjacent layers, but it is not possible at this stage to completely exclude that these results have been obtained by the surface decarburization of a steel initially enriched by carbon, although this last hypothesis does not well clarify some observed aspects of the microstructure;
- the performed observations are plausible and consistent with the hypothesis that the presence of Y-fiber components associated to the cube one are probably produced by a partial recrystallization of the deformed austenite;
- the micro-hardness profile of the nail belonging to group E has shown values included in the range between 104HV and 135 HV, with the maximum values associated with the presence of carbon enriched structural constituents. The comparison among the values measured in this study and the ones formerly performed on the nail of group B (revealing higher value of hardness) confirms that the ancient artisans were able to modulate the microstructure and the related properties as a function of the size of the nails and of their final applications.

ACKNOWLEDGEMENT

The authors desire to thank very much Mr. Piero Pellin for the performed SEM-EDS analysis and Ing. Sebastiano Zorzi for his efforts in the preparation of the analysed samples.

REFERENCES

ABSTRACT

CHIODI DEI LEGIONARI ROMANI RINVENUTI PRESSO INCHTUTHIL

Parole chiave: storia della metallurgia

Il presente articolo è focalizzato sullo studio di alcuni reperti provenienti da uno dei più famosi ed eclatanti ritrovamenti di componenti strutturali di età romana. I chiodi sono stati rinvenuti nel Pertshire, presso Inchtuthil dove i legionari romani li abbandonarono nel 87 d.C. per evitare che cadessero nelle mani dei nemici. I chiodi presentano una struttura multistrato, con alternanza di regioni perlitiche e ferritiche (Fig. 1, Fig. 2, Fig. 3, Fig. 4, Fig. 5). Tale eterogeneità microstrutturale si riflette nella significativa variazione delle durezze misurate nelle diverse regioni del chiodo (Tab. 1, Tab. 2). Le inclusioni riscontrate all’interno dei chiodi sono di natura esogena e sono senza dubbio inclusioni di natura fayalitica saturate in ossido di ferro (Fig. 6, Fig. 7), che sono bassodondenti e furono con ogni probabilità elaborate per consentire di rivestire le superfici da forgiare a caldo, evitando l’ossidazione che avrebbe impedito di saldare tra loro le superfici dal blumo da cui i chiodi sono stati ricavati. L’analisi cristallografica delle tessiture presenti all’interno del chiodo (Fig. 8, Fig. 9, Fig. 10, Fig. 11, Fig. 12, Fig. 13), indica che vi è una rilevante presenza di fibra-γ. Questo fenomeno sembra suggerire che, seppure su basi empiriche, fu messo a punto un ciclo termo-mecanico in cui si eseguiva una deformazione plastica a caldo dove la trasformazione fra fase γ e fase α doveva aver luogo prima della ricristallizzazione della fase γ. D’altra parte la presenza di grani con crescita abnorme (Fig. 15) e la presenza intensa di bordi grano di tipo Σ3 e Σ13 sembra indicare che il materiale è stato mantenuto ad alta temperatura in fase ferritica, tanto da portare ad un accrescimento significativo dei grani ferritici.