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SUB-CRITICAL ANNEALING OF WOOTZ INGOTS FOR CORRECT FORGING OF DAMASCUS STEEL ACCORDING TO MEDIEVAL CHRONICLES

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ABSTRACT
A closer look at the manufacturing process of Damascus steels (al hindi), based on archaeological and historical sources to develop a hypothesis that leads us to the technology required to forge this steel correctly.

KEYWORDS: wootz, Damascus steel, metallography, annealing, forging.

INTRODUCTION
Never before has it been possible to manufacture bladed knives from Damascus steel due to the complex series of heat treatments that the steel must undergo before forging. Furthermore, temperature is highly critical throughout the heat-forging process; black red or blood red (705ºC) must never be exceeded. This did not occur to Western blacksmiths, or even scientists, who always began the forging stages when the metal was white hot, which, as will be shown below, makes the steel crack like clay.

The best 17th, 18th and 19th century scientists and engineers attempted to determine the characteristics of this type of steel, without ever managing to clear up its mystery [1, 2]. In the second half of the 20th century some scientists and engineers did manage to forge these steels correctly[3-29], but they did not look like historical Damascus swords on the outside.

In most cases, we think that the research has taken the wrong approach. These hypereutectoid steels (with a very high carbon content, from 1.4 to 2% in mass) are always forged with the necessary precautions to avoid brittleness during the heating stages, and continuous proeutectoid cementite is not allowed to form on grain boundaries. They even discovered the initial secret of forging at the start, hitting or laminating with small blows, which crushes the continuous proeutectoid cementite on the grain boundaries of the surfaces of the piece; the same process used on present-day hyper-eutectoid alloy tool steels [30]. Subsequently, forging is done conventionally without surpassing 750ºC to avoid continuous cementite reappearing on the grain boundaries.

However, the majority of the authors, if not all, have not considered the transcendental importance of how these steels are handled before the ingot or flat plate is ready for forging. This prior handling includes: the method for obtaining ingots and their size, the cooling cycle, initial annealing process to soften the ingot before forging, and the manipulation of the ingot so that it has the correct initial flat shape before forging.

These stages are essential to get a forged piece of Damascus steel with all its aesthetic qualities.
All these secrets can only be extracted from the meticulous study of antique weapons forged by master blacksmiths; it also requires an exhaustive approach to the written sources which talk about these operations with varying degrees of accuracy. In the same way, there are very interesting tales from travelling witnesses and scientists from the 19th and early 20th centuries when there were still a few blacksmiths who knew how to forge these steels [31-42].

A closer look at the manufacturing process is needed and archaeological and historical sources must be consulted to be able to develop a hypothesis that leads us to the technology required to make this steel. If this prior research is skipped, and time is not taken to study actual samples or review the vast bibliography on manufacturing from the medieval period, these steels can be correctly forged, but pieces that demonstrate all the mechanical and aesthetic elements that made them famous will never be manufactured.

EXPERIMENTAL TECHNIQUE

This stage is crucial for perfect heat forging of Damascus steel. This is just another of the secrets in the rite of forging these steels. It is correctly described in the chronicles and it is equivalent to the heat treatment that is currently performed with hypereutectoid steels for tools before heat forging. In addition, this is the treatment that is carried out before forging hypercarburized alloy steels for tools. We have thus known for many centuries how to forge steels with ultra-high carbon content.

A small ingot (wootz) of Damascus steel (al-hindi) is sealed with dampened refractory clay. This simple method of coating a piece of steel with clay before heat treatment is the same one that master blacksmiths used and continue to use in Japan to anneal and temper katana swords [1, 43].

The steel ingot (wootz) is wrapped in damp refractory clay to stop it from oxidising during the annealing or softening process (Figure 1).

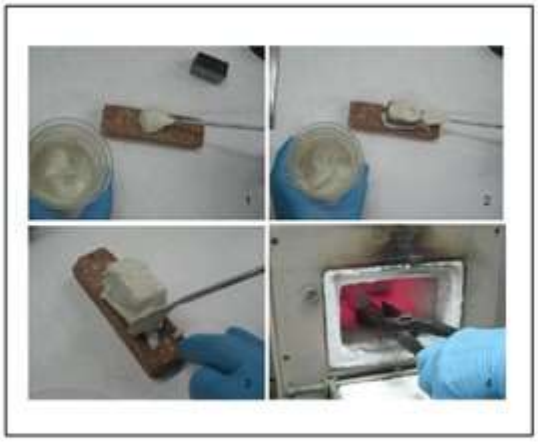

Figure 1: Sealing a chunk of Indian steel with a mixture of sodium silicate and kaolin before putting it in the oven, as an annealing treatment to soften it.

Following recipes from the chronicles, the clay-sealed ingot is put in the oven at 780ºC for 48 hours. At this sub-critical annealing temperature, the proeutectoid cementite that has precipitated ledeburitically in the grain boundaries does not dissolve in the austenite [30]. In this way, it undergoes a highly effective globulisation process, similar to what occurs with hypereutectoid tool steels in present-day industrial processes. After 48 hours it is left to cool slowly in the furnace, which will cause a more or less complete globulisation of the eutectoid cementite (Figure 2).

Figure 2: Clay sealing of a flat piece of Indian steel, annealing treatment for softening and later release from clay wrapping.

Both medieval and modern chronicles describe in detail how Damascus steel or Indian steel was forged. The crucial information is the maximum forging temperature; which can never be exceeded or the proeutectoid cementite would reform in the grain boundary during the cooling, resulting in a brittle piece of Damascus steel.

The micro-structures were obtained using a conventional optical microscope and a scanning electron microscope.

RESULTS AND DISCUSSION

The sweetening treatment of the steel by decarburisation or partial globulisation of the primary cementite is a heat treatment prior to forging hyper-carburized steel for tools. This means both the decarburisation and globulisation of sub-critical annealing above the eutectic temperature and within the stability limits of the primary cementite. This produces a globulisation process of this cementite. If the Damascus steel is insulated with refractory clay, temperatures of 760ºC to 780ºC could be used over a period of two or three days and cause intense globulisation of primary cementite. If the steel was not protected with refractory clay, the same temperatures were used, but the heating time did not exceed two or three hours, with superficial decarburisation taking place during this time.

The decarburisation caused a 0.5 to 1 mm deep decarburised surface, where the resulting structure was a eutectoid or hypereutectoid steel. This layer acted as a shield for the proeutectoid cementite to emerge to the surface on grain boundaries. In this way, it was possible to start the forging without causing cracking and fragile fracturing that would be catastrophic. Of course, once the weapon or the piece of Damascus steel was forged a fine layer must be removed from the surface using a whetstone to reach the heart of the steel (Figure 3).

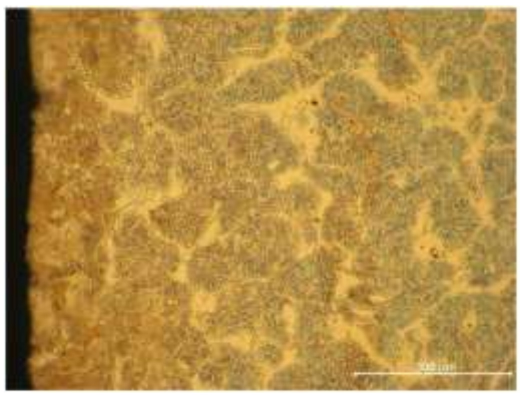

Figure 3 Image obtained by conventional optical microscopy where the decarburised layer, which has been reduced to an eutectoid, can be seen on the left, and on the right, the hypereutectoid Damascus steel with the globulised perlite cementite and the proeutectoid ledeburite. The decarburisation treatment involved heating to 800ºC for 2 hours and cooling in the furnace.

The other heat treatment prior to forging, described in medieval Muslim chronicles and also by Western chroniclers in modern times, is globulisation. This treatment came centuries ahead of modern metallurgy in terms of forging steel for tools. It requires sub-critical temperatures and long treatment times. To avoid carburisation, the treated pieces should be coated with refractory sand, as mentioned in the chronicles we consulted. The sub-critical annealing temperature for globulisation is between 760ºC and 780ºC for times that vary between 48 and 72 hours, followed by slow cooling in the furnace.

The resulting structures, as long as the cooling is slow in the oven, tend towards more or less intense staggering and partial globulisation of the primary ledeburite cementite or on grain boundaries; as well as total globulisation of the perlite cementite (Figures 4 and 5).

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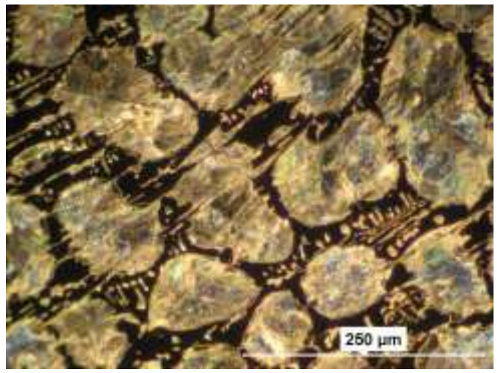

Figure 4: Image obtained by conventional dark field microscopy of an area of Damascus steel, with a sub-critical annealing softening treatment lasting 3 days at 760ºC and slow cooling in the furnace. The ledeburite cementite mesh, which is created due to the effect of the treatment, can be clearly seen.

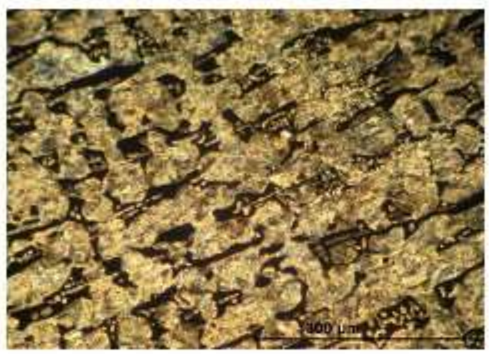

Figure 5: Micrograph obtained by means of conventional dark field microscopy, where the structure observed is the result of the sub-critical annealing used for softening. Look at the fragmentation caused in the ledeburite cementite. The matrix is perlite because it was cooled in the open air after the sub-critical annealing treatment.

Once the steel is treated by decarburisation or sub-critical annealing, the steel can be heat forged to give it the chosen shape: daggers, mirrors, swords, arrow tips, compasses, etc.

The ideal forging temperature for Damascus steel should be maintained in the range between 650ºC and 750ºC. This should never be exceeded as it would become highly critical above 800ºC. This would cause the formation of much of the continuous cementite on the grain boundary and make the steel more fragile. Short forging intervals are needed, which require many intervals of heating in the forge. Any lack of control over the upper temperature would ruin all the tedious work that had been put into the heat and thermochemical decarburisation treatments. The broken piece would not be usable (Figure 6).

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Figure 6: Macrograph of chunks of Damascus steel that broke during forging due to incorrect heat treatment.

The structure obtained, by heat forging (650-750ºC), consists of bands or threads of large primary cementite crystals, cut up and significantly globulised and, among them, intensely globulised proeutectoid cementite in a fine grain ferrite matrix (Figures 7 and 8).

Figure 7: Detailed view of the ingot structure after sub-critical annealing followed by heat forging. Image obtained by means of conventional dark field microscopy. The image shows the structure of different sizes of carbides in alternate parallel sheets.

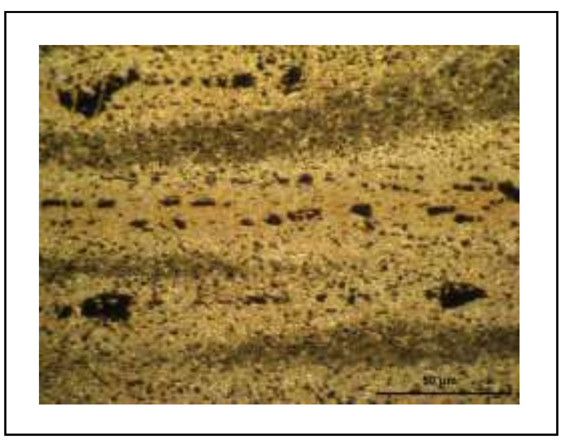

Figure 8: Detail, using greater magnification, of the structure of the previous image.

CONCLUSIONS

Based on research into the medieval chronicles, it has been possible to reveal one of the secrets to correctly forge Damascus steel (al hindi). This secret consists of a heat treatment aiming at destroying the continuous mesh of proeutectoid or ledeburite cementite that surrounds the perlite grains, preventing correct forging of these steels.

It is true that many scientists studying the issue, such as R.A.F. de Reàmur lament that forging Damascus steel is impossible [43, 44] in the West; more specifically, forging the wootz ingots that were imported from India. Here, medieval Muslim armourers were several centuries ahead of the west in forging hyper-carburized steels for tools. The first disadvantage of hyper-carburized steel is that the grain boundaries for the original austenite appear in the form of a mesh or a continuous network of cementite. The cementite is very fragile when cold or at yellow and white temperatures and, furthermore, this network of cementite crystals comes to the surface. A reasonably strong hammer blow causes a fragile crack or many simultaneous cracks that can break the steel as if it were clay.

Medieval and modern Muslim blacksmiths knew that it was necessary to prepare the ingot prior to forging. They globulised and segmented the continuous cementite on grain boundaries using a heat treatment that is identical to what is performed today with tool steels. They called it sweetening or softening the steel, we call it sub-critical annealing. This treatment of heating the metal to temperatures where partial fragmentation and globulisation of the cementite occurs was done at slightly higher temperatures than the eutectoid and, of course, far from the austenite region. This heating, identical to what is done nowadays, used ingots or chunks of Indian steel, coated with refractory clay to prevent decarburisation. Nowadays, the sub-critical annealing of hyper-carburised steels, alloyed or not, is performed in controlled atmosphere furnaces.
This operation managed to segment, cut up and globulise the dense network of proeutectoid cementite. After that, similar to the case of present-day tool steels, it was a question of heat forging without reforming the feared continuous network that makes the proeutectoid cementite more fragile. This is achieved by not exceeding forging temperatures over 760-780ºC, to avoiding reaching the austenite region in the heating which would reform the fragile network of proeutectoid cementite and would ruin the weapon when it was struck during forging. This was the secret or the key that western blacksmiths did not unlock until the 20th century.

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