Genuine Damascus steel: a type of banded microstructure in hypereutectoid steels

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A brief review is given of the evolution of welded Damascus steel and genuine Damascus steel along with the mystery of how the pattern of genuine Damascus steel is produced. The prior studies claiming to have either, reproduced the genuine Damascus steel, or explained the mechanism of pattern formation, are reviewed. None of these studies have allowed modern bladesmiths to reproduce the steel. The author and a bladesmith, Alfred Pendray, have developed a process with which Pendray can produce blades that match the microstructures of the best museum quality genuine Damascus blades. Experimental research is reviewed showing that the microstructure of this steel is produced by an unusual type of banding that requires: (1) the presence of low levels of certain impurity elements in the hypereutectoid steels, with V being most effective, and (2) a thermal cycling heat treatment. A main difference of this type of banding and the ubiquitous banding of hypoeutectoid steels is point (2), the requirement of thermal cycling. A theory for the banding mechanism is presented.

Echter Damaszenerstahl: Ein besonderes Zeilengefüge in übereutektoidischen Stählen. Ein kurzer Überblick über die Entwicklung von feuergeschweißtem und echtem Damaszenerstahl und das Mysterium über die Erzeugung des Musters in echtem Damaszenerstahl wird gegeben. Auch frühere Studien, in denen es um die Reproduktion echten Damaszenerstahls oder Erklärungen, wie die Musterbildung funktioniert, werden einbezogen. Keine von ihnen hat jedoch dazu geführt, dass es einem modernen Klingenschmied gelungen wäre, diesen Stahl zu reproduzieren. Der Autor dieser Untersuchung hat zusammen mit dem Klingenschmied Alfred Pendray ein Verfahren entwickelt, nach dem letzterer Klingen herstellen kann, deren Mikrostrukturen mit denen vergleichbar sind, die die in den Museen existierenden, echten Damaszenerklingen aufweisen. Experimentelle Forschung kommt zu dem Schluss, dass die Mikrostruktur dieses Stahls durch eine ungewöhnliche Art von Zeiligkeit hervorgerufen wird, die entsteht, wenn (1) geringe Anteile an Verunreinigungen in übereutektoiden Stählen vorliegen, wobei Vanadium sehr wirksam ist, und (2) eine zyklische Wärmebehandlung erfolgte. Ein Hauptunterschied zwischen dieser Art von Gefügezeiligkeit und der allgegenwärtigen Zeiligkeit in übereutektoidischen Stählen ist eben diese unter (2) genannte Zeit-Temperatur-Folge. Eine Theorie über diesen Zeilenmechanismus wird vorgeschlagen.

Historical background

It is generally agreed that our ancestors first learned to reduce iron ores to pure iron around 1200 BC [1...3]. The iron formed without melting in a charcoal fired furnace and sintered together with abundant slag inclusions into a mass called a bloom. It had to be hot forged to remove most of the slag inclusions. This bloomery iron was very low in carbon, around 0.06 %[1)], and was full of elongated slag inclusions. It was the main source of iron (except in China) up to the late 14th century when European smiths learned to make their furnaces higher, run them hotter and produce a cast iron which was then processed to a wrought iron that was amazingly similar to bloomery iron [4]. Virtually all early steel was made by carburizing the bloomery iron in charcoal fires with the CO/CO_2 ratio controlled properly to give high carbon potentials in iron at temperatures above around 850 °C where the kinetics of carbon diffusion was appreciable. It was not until the late 18th century that the element C was discovered and found to be the key factor in making hardened steel. Nevertheless early smiths successfully produced martensitic steel as early as 1200 BC [3]. Proper control of temperature and air flow rate to give the CO/CO_2 ratio required in the forge fire to ensure adequate carburization was an art learned only by the more clever bladesmiths.

Swords made from bloomery iron where relatively soft and so steel swords were prized. Most of the steel swords made prior to around 200 AD utilized a process called "piling" [5; 6]. Carburized rods or plates were stacked together and then hot forged to form the blade. Starting around 200 AD the method of pattern welding began to appear [5; 6]. This method, first named by Maryon [7; 8], is similar to piling but a bit more complex. It often involves the addition of folding during the forging process and the use of twisted aggregates of rods in the starting material. Polishing and etching of the surface of the blade generally produces a pattern due to the effect of differences of chemical compositions on etching response in the various starting layers. Modern bladesmiths produce beautiful patterns on blade surfaces using pattern welding, and perhaps the most famous example occurs in pattern welded shotgun barrels. Such steels are now called pattern welded Damascus steel and ref. [9] presents beautiful examples along with the details of how the various patterns are formed by controlling the arrangement and twisting of the starting elements that are hot forged together.

When the Christian warriors of Europe encountered Moslem armies during Crusade times they discovered that the Moslems had steel swords superior to their own steel swords. Historical accounts [10; 11] claim that the swords were encountered by Europeans in Damascus and hence the name, but they were also known in Russia in the same time period under the names "poulad" or "bulat". It is agreed that the swords were forged directly from small ingots of steel formed in crucibles and given the name wootz by English people in 1795 [4]. This steel is widely regarded as a product of India. Bronson [12] has shown that it first appeared around the second century AD, but that it was also produced in the Middle East. More re-

[1)] Except where noted compositions are mass contents in %.

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Table 1. Literature data on chemical composition of genuine Damascus blades

Steel	C	Mn	Si	S	P	Cu	Cr	Ni
range on 12 blades [24]	1.34-1.87	0.005-0.14	0.005-0.11	0.007-0.038	0.05-0.206	0.04-0.06	trace	0.008-0.016
upgrade with data of [23]	1.00-1.87	0.005-0.014	0.005-0.11	0.006-0.038	0.026-0.206	0.03-0.18	<0.01	0.008-0.07
Zshokke #9 [24]	1.41	<0.01	0.05	0.006	0.098	0.09	<0.01	0.04
Zschokke #9 [15]	1.34	0.019	0.062	0.008	0.108	-	-	-

cently, Feuerbach et al. [13] have shown that crucible steel was also produced in Central Asia over 1000 years ago. The crucible steel of Damascus swords is a surprisingly clean hypereutectoid steel with %C in the range of 1.1 to 1.8 %, see top 2 rows of **table 1.** An outstanding collection of high quality Damascus swords was obtained by L.S. Figiel and beautiful pictures of these swords are found in his book [14]. Similar blades may be found in the arms and armor section of most large museums. **Figure 1** presents an example sword, No. Ps4 [14], a sword made in 1691-2 by Asaad Allah, reportedly one of the most famous swordsmiths of the 17th century. The inset shows the details of a typical Damascus blade pattern along with an inscription on the blade identifying the swordsmith. The patterns on Damascus swords and knives appear only on the better quality blades and are commonly referred to as a damascence pattern. It was because of the fact that well made pattern welded swords have surface patterns of a somewhat similar nature that these blades are also called Damascus blades. Because the name Damascus was first used to refer to blades made from wootz steel these blades will be called genuine Damascus blades here and the other blades called pattern welded Damascus blades.

There is a general myth in some of the popular literature that genuine Damascus steel blades possess outstanding mechanical properties, often thought superior to modern steels. This idea was shown to be incorrect as long ago as 1924. A famous Swiss collector, Henri Moser, donated 4 genuine Damascus steel swords, one with a non typical carbon content and microstructure, to B. Zschokke, who performed extensive careful experiments including metallographic and chemical analysis in addition to mechanical testing [15]. A series of bending tests compared samples from the swords to a pattern welded blade and a cast blade from the famous German knife center in Solingen. The 3 good Damascus blades showed significantly inferior bending deflection prior to breakage than the 2 Solingen blades in spite of the fact that the Brinell hardness of the 3 ranged from only 193 to 248, compared to 347 and 463 for the pattern welded and cast Solingen blades, respectively. This is not too surprising in view of the now well known fact that toughness of high carbon steels is inherently low; the Solingen blades had carbon levels of 0.5 to 0.6 % compared to 1.3 to 1.9 % for the 3 Damascus blades. The reputation of Damascus steel blades being superior to European blades was probably established prior to the 17th century when European blades were still being made by forge welding of carburized iron. It is hard to avoid embrittlement of such blades due to imperfect welding during the forging process as well as difficulty with the carburizing process. There is a parallel here with the famous crucible steel of Huntsman in 1740 England. Huntsmen's motivation for the development of crucible steel was to overcome the lack of toughness of blister steel, which was made by the carburizing process descended from the old piling process.

Characterization of genuine Damascus steel blades

In an effort to establish a working definition of what constitutes a genuine Damascus steel a list of 7 characteristics of these blades has been published [16]. To aid in

Blade made by Assad Allah around 1691, 96 cm long by 3.5 cm wide

Figure 1. A genuine Damascus steel blade. Blade No. Ps4 from the Figiel collection [14]

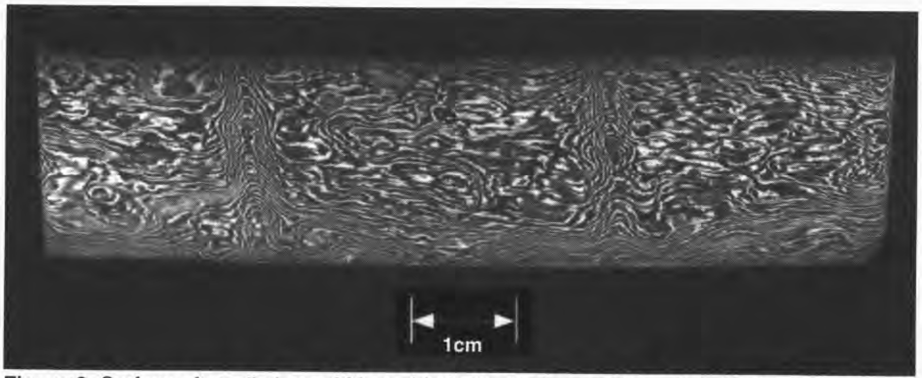

Figure 2. Surface of small piece of Moser blade No. 9 along cutting edge. Ferric choloride etch (magnification: x 1.5)

illustrating these characteristics, micrographs of a genuine Damascus blade are presented in **figures 2** and **3.** This blade is a small piece, 18 mm · 88 mm, of blade number 9 of the original 1923 study of Zschokke [15]. The small piece was donated to the author by E. Klaey of the Berne Historical Museum in Switzerland where the Islamic collection of H. Moser is now displayed. Figure 2 presents the surface pattern on this blade showing the famous "Mohammed's Ladder" pattern comprised of the parallel spaced features. This pattern is found on many high quality genuine Damascus blades as illustrated in Figiel's book [14]. Transverse and longitudinal sections of the blade are shown in figure 3. A reverse contrast has been obtained by etching in boiling picric acid which stains the cementite black in a white pearlite matrix. This reversal of contrast from the usual etchant shows the carbide distribution more clearly. The composition of this steel as measured by both modern emission spectroscopy and combustion analysis [24] is given in table 1 along with Zschokke's [15] analysis. **Table 2** presents the list of 7 characteristics that were compiled based on literature data of the Zschokke blades and other genuine blades both in the literature and in the author's collection [16]. The 3 Zschokke blades have since been metallographically analyzed [23] and the results of blade #9 shown in figures 2, 3 will be reviewed to illustrate the 7 characteristics of table 2. (1) The matrix is fully pearlitic. (2) The band spacing was 50 μm and the range of particle diameters on the face, longitudinal and transverse sections ranged from 5.7 - 11 μm, 5.6 - 12 μm and 3.9 - 11 μm, respectively. Careful comparison of figures 3a and 3b shows that the continuity of the cementite bands is somewhat less on the transverse section than on the longitudinal section. This means that the bands in this blade are more like wide ribbons than fully planar sheets. Examination of transverse and longitudinal sections of several genuine Damascus blades has found that this is sometimes the case. Therefore, points 3 and 4 have been modified from the original reference which had required totally planar bands. Points 5 and 6 fall in the range of table 2, as seen from figure 3 and table 1. Point 7 could not be evaluated as the 18 mm wide piece supplied from Zschokke blade #9 was sectioned at the cutting edge and was not wide enough to include the back edge of the 30 mm wide original blade.

There has been a mystery about Damascus blades that western scientists have periodically studied since the late 18th century. Initial studies in the west were unable to reproduce the famous patterned blades. Smith [11] documents early reports that western smiths were even unable to forge the brittle wootz ingots. For example, a 1722 report by Réaumur deplored the skill of Parisian artisans, none of whom succeeded in forging a tool out of a cake of Indian steel. It was known that the bands of cementite particles responsible for the surface patterns were produced in some manner by simply forging the crucible steel ingots into blade shapes without any special folding operation. Following a study by the famous English scientist Faraday [25], there were two extensive studies, first in France by Brèant [26] and then in Russia by Anassoff [27]. Smith [11; 28] has presented reviews of these studies showing that both lead to production of blades that apparently produced excellent reproductions of the surface patterns of genuine Damascus blades. Unfortunately, documentation of the methods used by Brèant and Anassoff was not sufficient to allow later smiths to reproduce the famous blades.

Figure 3a. Transverse section of figure 2 blade. Etched in boiling picric to produce dark cementite in white pearlite matrix (x 100)

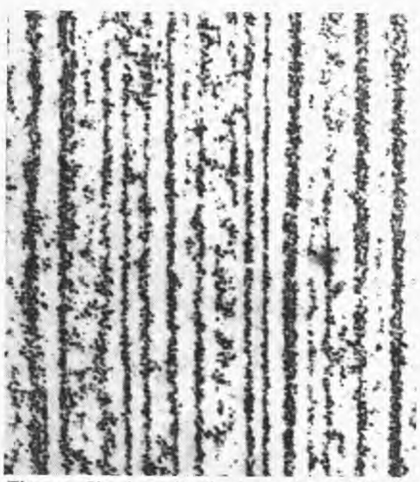

Figure 3b. Longitudinal section of figure 2 blade. Etched in boiling picric to produce dark cementite in white pearlite matrix (x 100)

Theories of the mechanism for cementite band formation

The mechanism by which the alignment of the cementite bands occurred remained uncertain. Three main theories evolved which all required 2 steps: (1) the generation of an array of cementite particles formed in the ingot and (2) forging below the A_{cm} temperature to

Table 2. Seven characteristics of genuine Damascus blades [16]

No.	characteristics
1	the matrix may be pearlitic, bainitic, or tempered martensite depending upon the final heat treatment of the blade, but is generally pearlitic [10; 15; 17; 20]
2	the cementite particles are well rounded as opposed to faceted. They have a shape that can vary from equiaxed to oblong with aspect ratios as high as 3 to 4 [18]. The equiaxed particles range in diameter from 3 to 20 μm 10; 15; 17; 19; 20]. The spacing of the linear arrays of cementite particles ranges from 30 to 100 μm [21]
3	the 3 dimensional geometry of the arrays is best approximated as nearly planar. There are only a few interconnections between sheets in a field of view at 50x
4	there are no significant differences in particle size or shape on transverse versus longitudinal sections [21; 22]. However, the distribution is sometimes more linear on longitudinal sections
5	the number of particles across the thickness of a linear array on a transverse or longitudinal section is defined as, *n*. The value of *n* is virtually always larger than 1, and will generally average around 4 - 6
6	the composition range for 10 genuine Damascus blades is summarized in the top row of table 1 [24], and it has been upgraded with newer data from [23] in row 2
7	a seam or fold is often, but not always, present along the back edge of the blade. Transverse sections through the fold show a decarburized layer on its surfaces [21; 22]

avoid dissolving the carbides, break them into smaller particles, and align them into bands as a result of the forging flow. **Table 3** shows that two of the theories [28; 10] located the array in the interdendritic regions of the ingot and the third theory [29] had the array located on the grain boundaries of large austenite grains. Belaiew's theory [10] is a bit difficult to follow because he refers to a "structure of large crystals". Careful reading of the paper reveals that this means a dendritic structure with cementite formed between the dendrite arms as a result of cooling below the A_{cm} line. Belaiew claims that this structure was present in his ingots as well as those of his Russian countryman, Anassoff.

Table 3. Source of the arrays of cementite particles for step 1 of the three theories

Author	source of cementite array in ingot
Smith [28]	primary cementite formed between dendrites during solidification. the geometry of the array matched that of the interdendritic dendrite regions
Belaiew [10]	secondary cementite formed between dendrites as ingot cooled below the A_{cm} temperature. The array geometry was the same as above
Wadsworth and Sherby [29]	grain boundary allotriomorph cementite formed by a 2-step heat treatment of the ingot: (1) the ingot was heated above A_{cm} for a long time to produce very large austenite grains (2) it was then cooled slowly to produce the plate-like grain boundary allotriomorph cementite arrayed on the austenite grain boundaries

The author began research efforts trying to reproduce the structure of genuine Damascus steel blade in the mid 1980s [24] using vacuum induction melting (VIM) to produce ingots in a controlled laboratory environment followed by hot rolling to blade shapes. After this work proved unsuccessful a cooperative research was carried out with practicing bladesmith, A. H. Pendray, in which ingots were produced both by VIM and by gas fired melting in closed clay graphite crucibles followed by hot forging with an air hammer. Initial work, utilizing charges that yielded ingots matching the C, Si, Mn, S and P levels of line 1 of table 1, produced only sporadic success. The work did however show why the old wootz ingots were so difficult to forge and Pendray developed a method to overcome the problem. At the high P level of these ingots the ternary eutectic steadite forms throughout the interdendritic regions of the ingot. Steadite melts at 950 °C and causes the ingot to become hot short above this temperature. Packing the ingots in iron oxide mill scale and holding at around 1200 °C for several hours causes a thin rim of purified iron to form on the ingot surface which successfully contains the hot short interior during forging. It seems likely that the oriental smiths had learned this trick needed to forge the high P wootz ingots. This conclusion is supported by experiments on a bar of steel obtained from the Alwar Armory located in the state of Rajasthan, India [18]. The bar was thought to have been forged from a wootz ingot. When it was forged down from the as-received thickness of 10 mm to a final thickness of 2-3 mm at temperatures between 680 and 900 °C it exhibited the characteristic surface pattern of genuine Damascus steel with similar internal bands of clustered cementite particles. The surface regions of the bar contained a rim of nearly pure iron which had to be ground away to reveal the characteristic surface damascene pattern.

Initial experiments indicated that none of the 3 theories discussed with table 3 appeared to be correct. In ingots without S or P additions no primary cementite appeared between the austenite dendrites. This was also true in ingots with small S and P additions, both in the 0.01-0.02 % range. This result was not surprising because it had been shown [30] that the high diffusion coefficient of C in austenite produces very nearly equilibrium freezing conditions in pure Fe-C alloys, which means that interdendritic cementite will only occur for %C values above the maximum solubility in austenite at the eutectic temperature, a value of 2.01 %C. These results present experimental evidence that theory 1 is not operative. For ingots with %P of 0.1 %, cementite was found in the interdendritic regions [24]. It was present as large chunky particles that had nucleated around small pools of the steadite that formed in those locations. This cementite was secondary cementite forming on the steadite as the temperature fell below the A_{cm} temperature, which corresponds to the mechanism of theory 2.

Hot rolling or forging of these ingots did not produce the characteristic aligned cementite bands of genuine Damascus steel. The cementite was more or less randomly distributed in the final blade. The results present experimental evidence that theory 2 is incorrect. Two series of experiments were done to see if genuine Damascus microstructures could be obtained with theory 3. The original experiments of Wadsworth and Sherby (W-S) [29] had produced some microstructures showing bands of cementite particles utilizing hot rolling for the deformation mode. If genuine Damascus blades where made using the method of this theory the deformation mode would have been hot forging. Therefore, experiments which generated the cementite arrays using this method, see table 3, were done with hot forging by A.H. Pendray [18; 31] and bladesmiths, H.F. Clark and W. F. Moran Jr. [32]. The experiments were not able to generate the genuine Damascus steel microstructures. Several problems occurred. First, the cementite particles did not break up and spheroidize adequately and produced larger and faceted particles than in the genuine blades illustrated in figure. 3. Second, the surface pattern appeared as an elongated grain structure in contrast to point 3 of table 2. Third, the distribution of the particles was considerably different on a transverse section than on a longitudinal section. As previously discussed [18; 31] the shape change that occurs on forging to a blade with cementite arranged on equiaxed grain boundaries is not adequate to produce the same planar distribution of cementite on both the transverse and longitudinal section of a blade, as is found in genuine Damascus blades, see points 3 and 4 of table 2. Wadsworth and Sherby continue to feel that their method might have been used to produce genuine Damascus blades [33; 34]. To the author's knowledge, however, no bladesmith has produced blades with the W-S method showing surface patterns and microstructures that match genuine Damascus blades. The basic method of W-S was actually utilized prior to their work by Harnecker [35] at the Henckels company in Solingen. She prepared high carbon steel by carburizing both a fairly pure Swedish iron and an ingot steel at 1000 - 1100 °C for 10 - 12 days followed by slow cooling, which would have produced the cementite array for step 1 of the W-S method. She was careful to do subsequent forging below the A_{cm} temperature. The microstructures obtained [35] display coarse blocky cementite particles with an elongated grain structure similar to those found in our work [18; 31; 32].

Figure 4. A reconstructed Damascus blade containing a combined Mohammed ladder + rose pattern. Ferric chloride etch (full size)

Figure 5. Longitudinal section of figure 4 blade showing bands of cementite particles that the surface pattern. Nital etch

Development of a working method to reproduce genuine Damascus blades

The cooperative research with Pendray has advanced to the point that he can produce blades that match the microstructures of the best museum quality genuine Damascus blades. **Figure 4** presents a recent blade and **figure 5** shows the transverse microstructure of the blade. Both the surface pattern of the blades and the microstructures on transverse and longitudinal sections are a close match to the genuine Damascus blades. The Mohammed's ladder pattern and the circular rose pattern were produced using the forging trick that is discussed in [23]. The remainder of the paper will present evidence showing that the microstructure of this steel is produced by an unusual type of banding that requires: (1) a thermal cycling heat treatment, and (2) the presence of low levels of certain impurity elements in the hypereutectoid steels. Following is the main experimental evidence for both of these requirements.

The thermal cycling heat treatment. The ingots are made by charging a mixture of pure iron, a high purity Fe-4 %C alloy called Sorel iron that is used in the U.S. ductile iron industry, and some graphite into the bottom of a small clay graphite crucible. The total carbon is calculated to end up with the desired % C of around 1.5 %C in the ingot. The charge is covered by a mat of green leaves which holds up a layer of broken glass used to form the slag. The leaves, along with a chunk of graphite on top of the glass layer ensure reducing conditions in the crucible, which is closed by a top sealed with a ceramic cement. The crucible temperature is monitored and held at temperatures increasing slowly from

1440 up to 1480 °C in about 30 to 40 minutes and then furnace cooled. The final ingots have a mass of around 3 kg, and are around 45 mm high and 120 mm in diameter. The ingots are flat across the top displaying large radially directed surface dendrites. Their microstructure displays a combination of grain boundary and Widmanstatten cementite with no apparent relationship to the prior dendrite arrays, which can be revealed by etching in either Stead's, or Oberhoffer's etch. Also, they contain significant amounts of microporosity in the interdendritic regions, which accounts for the flat top with no shrink cavity. The very slow cooling rates recommended by Anossoff were found detrimental, as they produced excessive cavity porosity, apparently due to CO evolution.

Initial experiments [18] showed that good patterns where sometimes obtained in ingots with low P levels (0.02-0.04 %) and most subsequent experiments utilized low P because such ingots were not hot short and did not need the laborious rim heat treatment described above. Initial experiments also found that often the final blades would be heavily graphitized with the bands of cementite particles being replaced by bands of graphite. This problem was found to be due to the microporosity of the ingots [36]. By employing a heavy deformation at high temperatures in the initial stages of the forging to close the microporosity the problem was eliminated. After the ingots are forged to bars of around 1 cm thickness the maximum forging temperature is maintained about 50 to 100 °C below the A_{cm} temperature. A systematic study of longitudinal microstructures versus the extent of forging revealed that the alignment of the cementite particles occurred gradually during the forging process [37]. Initially the particles were found as heavy clusters at prior austenite grain boundaries and tri-junctions, as randomly arrayed clusters, and as individual particles. As forging progressed there was a gradual replacement of these structures by the aligned bands of clustered cementite particles. This result showed that the bands could not have been forming by any of the 3 theories of table 2 because all of these require the cementite particles of the final bands to be remnants of the original arrays postulated by the theories.

The discovery of the gradual formation of the bands of clustered cementite particles led to a series of very significant experiments [38]. Blade pieces showing a distinct set of bands were heated above the A_{cm} temperature for 3 min to dissolve all the cementite particles and then quenched to room temperature. This produced a martensite + retained austenite structure with complete destruction of the surface pattern. The blade pieces were then thermal cycled 6 times from a high temperature of 50 - 80 °C below the A_{cm} temperature down to 500 °C. This type of experiment, which will hereafter be termed TC (thermal cycling) experiments, found that after 6 cycles the cementite bands had returned with the particle diameters somewhat reduced as illustrated in **figure 6,** where (a) shows a longitudinal section of the as-received blade and (b) a longitudinal section after the TC treatment. As in the original forging, the alignment of the cementite particles into bands occurred progressively, from very little alignment after the first cycle to successively better alignment on subsequent cycles up to around 6 cycles. These TC experiments were also done on 2 genuine Damascus steel blade pieces [38] as well as the Alwar bar blade [18] and the same results were obtained. The blade of figure 6 is one of the genuine Damascus blades, a blade donated to us by L.S. Figiel.

These results suggested that the bands might be forming as a type of banding in both the genuine Damascus blades and the reconstructed blades. Banding is well known and widely studied in hypoeutectoid steels. Numerous studies [39] have shown that it occurs as a result of microsegregation of third elements in Fe-C-*X* alloys, where *X* is the third element. Virtually all wrought hypoeutectoid steels that are slow cooled from the austenite region will display a pronounced ferrite/pearlite banding, consisting of alternating ferrite and pearlite bands. In sheet material the bands are planar, having the same appearance on both transverse and longitudinal sections. It is well established that the bands are produced by a kinetic effect upon the austenite-ferrite transformation. The microsegregated element causes the ferrite to form first along either the purer dendrite centerlines (e.g. X = Mn) or the impurity segregated interdendritic centerlines (e.g. X = P). The effect can also be produced by selective nucleation of the ferrite on manganese sulfide compounds which microsegregate to interdendritic regions. Relatively slow cooling is required so that the ferrite will grow as blocky allotriomorph particles that push the carbon ahead of their growth fronts, eventually forming pearlite bands between the advancing ferrite bands. Perhaps the strongest evidence that this phenomenon is due to microsegregation of the *X* element is the following type of experiment. If a banded steel is fully austenitized and cooled fairly rapidly it is found that the

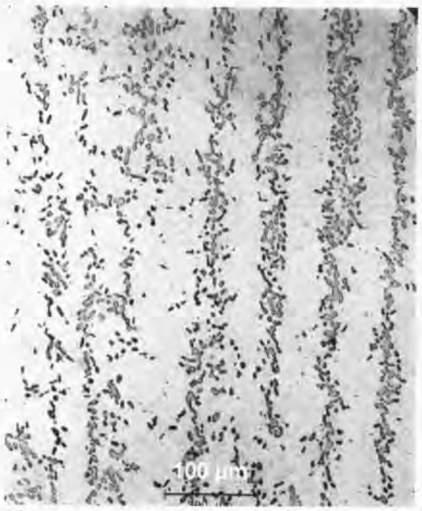

Figure 6a. Longitudinal section of genuine Damascus blade from Figiel. Boiling picric etch

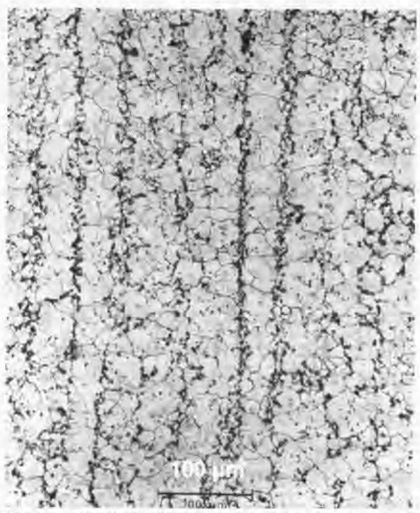

Figure 6b. Longitudinal section of blade of figure 6a after TC treatment employing 6 thermal cycles. Boiling picric etch

Table 4. Chemical analysis of ingots with and without Sorel iron. Units are ppmm (parts per million referring to mass, 100 ppmm equals a mass content of 0.01 %)

Ingots	P	S	Co	V	Ti	Cr	W
Sorel added	130-380	100-250	36	60-90	17-34	73	22
no Sorel	30-60	70-90	10	<10	9-14	22	<3

banded structure disappears. When the same steel is fully austenitized again at the normal low austenitization temperature and slow cooled the banding returns. If, however, the steel is austenitized for an extended time just below its melting point the steel can no longer be made to form a banded microstructure. The extended high temperature heat treatment has caused the microsegregated *X* element to homogenize by diffusion and, hence, the loss of the banded structure.

A similar set of high temperature experiments was performed on the steels which had been shown to have the cementite bands return by the TC treatment [38]. They were heated to 1200 °C for 18 hours, a time calculated to be adequate to homogenize a typical substitutional *X* element in austenite, such as Mn. After this treatment it was found that the TC treatment would no longer produce a banded microstructure. The cementite particles were now distributed randomly following the TC treatment, compare **figure 7** to figure 6b. These experiments presented strong

Table 5. Analyzed level of added element [41]

Banding	level of added element in ppmm
strong bands	V: 350 and 110, Mo: 300
weak bands	Cr: 400, Nb: 300, Mn: 200, P: 380, S: 420
no bands	Cr: 100, P: 100, S: 90

evidence that the cementite bands in the genuine Damascus steel blades and in the reconstructed blades is produced by the microsegregation of some third element. They did not however determine what *X* element addition was responsible for the banding.

The impurities causing the banding. The initial experiments were often not successful in producing aligned clusters of cementite bands in the forged blades. However, after Sorel metal was used as part of the starting crucible charge the success rate went way up. Chemical analyses revealed no impurity elements other than Mn, Si, S or P at levels above around 0.05 % and it was initially assumed incorrectly that levels this low were not significant. Using the Sorel metal as a large fraction of the charge, a series of 8 experiments were run which all produced excellent band formation. Chemical analysis revealed that these ingots and the Sorel metal all contained low levels of the element V [40]. Three successive experiments utilizing only electrolytic iron in the charge showed no bands. Careful emission and mass spectroscopy analyses showed that the ingots that banded were higher in the impurity elements shown in **table 4.** Subsequent experiments were done in which the electrolytic charge was doped with low levels of P + S and with low levels of V + Cr + Ti. Ingots with only P and S added at levels of 90 to 290 ppmm (parts per million referring to mass) did not show banding. Ingots without P and S additions (P < 20 ppmm and S < 90 ppmm) and with Cr: 100-200 ppmm, V: 60-130 ppmm, Ti: 11-19 ppmm, showed distinct banding. This result lead to a careful study in which ingots were prepared from electrolytic iron doped only with the individual carbide forming elements, V, Mo, Cr, Nb, and Mn as well as ingots doped with only P or S [41]. This work also included a study of microsegregation using electron probe microanalysis, (EPMA). The effectiveness of the various elements in causing bands to form in the final blades is given in **table 5.** It is seen that V and Mo were the most effective alloy additions for causing formation of strong bands.

Because of the very low level of the added impurity elements the EPMA work required some means of locating the interdendritic regions (IRs) where the added elements might be microsegregated. Fortunately, even at the lowest level of S in these ingots, 30 ppmm, FeS inclusions were easily located in the optical microscope after etching and served as markers for the IR locations. EPMA was done at around 4 - 5 IRs on each ingot. Primary carbides were found in the IRs of the V, Nb and Mo ingots. The cemen-

Figure 7. Longitudinal section of blade of figure 6a after 18 h anneal at 1200 °C followed by 6 cycle TC treatment. Boiling picric etch

Table 6. Analysis of 8 well patterned genuine Damascus blades (all in ppmm, except carbon)

Element	no. 7	no. 9	no. 10	old b.	Figiel	Voigt	Kard
C mass contents in %	1.71	1.41	1.79	1.51	1.64	1.00	1.49
P	1010	980	1330	950	1630	260	1440
S	95	60	160	53	85	115	90
Cr	<100	<100	<100	<100	<100	<100	<100
Mo	<100	<100	<100	<100	<100	<100	<100
Nb	<100	<100	<100	<100	<100	<100	<100
Nb	<100	<100	<100	<100	<100	<100	<100
Mn	150	<100	300	100	200	500	100
V	145	50	270	40	40	<10	60

tite surrounding the sulfides showed a pickup in 3 of the ingots (atomic fractions): V: 0.5-1.2 %, Mo: 1.2-1.6 % and Cr: 0.1-0.2 %. The FeS of the V and Cr ingots showed a pickup of two elements (atomic fractions): V: 8-19 % and Cr: 2.8-2.9 %. In the Mn ingot the FeS picked up an atomic fraction of only 0.2-0.7 % Mn, but the ingot also contained the usual sulfide of commercial steels, (MnFe)S, with Mn atomic fractions of 22 - 24 %. The 380 ppmm P ingot displayed steadite in the IRs. Hence, the EPMA study shows that all of the added elements display significant amounts of microsegregation, which is a requirement for a banding mechanism. And perhaps most significantly, the elements V, Mo and Cr were all found to segregate into the cementite located in the IR regions but not to cementite located away from those regions. Such a result is significant in that it could lead to a large effect on the kinetics of cementite growth in the IRs of these ingots.

These results suggested that carbide forming elements, in particular V and Mo, might have played a critical role in allowing the old bladesmiths of the Islamic world to produce the banded microstructures required for the patterns on genuine Damascus blades. To evaluate this possibility, the 3 swords of the Zschokke study, blades 7, 9 and 10 along with 4 genuine Damascus blades obtained by the author were analyzed. The full analyses are given in [23] and **table 6** presents the analysis for P, S and the carbide forming elements. The very high P content of the blades suggest that perhaps this element might be responsible for the patterns in these blades. However, we have prepared several VIM (vacuum induction melted) ingots from electrolytic iron with high P additions along with low levels of Mn and S [40] and, after learning how to forge these ingots, found that they did not produce a banded microstructure. These results show that the high P level of the Damascus blades, by itself, is not the key to their ability to form bands on forging. The data of table 6 presents evidence that the key impurity element responsible for band formation in the old genuine Damascus blades might have been V, and in some cases Mn, see the Voigt blade.

Additional evidence for a banding mechanism. Two additional pieces of experimental support for a banding mechanism were found. First, ingots were made from both (1) electrolytic iron + Sorel iron in the usual manner and (2) electrolytic iron doped with Mn, P, Si and S and produced by VIM in high purity alumina crucibles [40]. The first ingot produced aligned cementite bands on forging but the second did not. Ingot pieces were then given the TC treatment and afterwards the VIM ingot pieces displayed a random distribution of cementite particles. However, the Sorel ingot pieces displayed clustered arrays of spheroidized cementite particles concentrated in the IRs of the ingot. **Figure 8a** shows this interdendritic distribution of the clustered cementite particles and **figure 8b** is a higher magnification micrograph illustrating the spheroidized and clustered microstructure of the particles. These experiments show two important results: (1) Some microsegregated element, most probably V from the Sorel iron, is causing the cementite to form preferentially in the IRs of the ingot. (2) The thermal cycling treatment is effective in producing a geometry of clustered cementite particles. Both of the these results support the theory that the bands of clustered cementite particles in the reconstructed blades is formed by a type of banding requiring low levels of carbide forming impurity elements and a thermal cycling heat treatment.

If a banding mechanism is responsible for the bands of clustered cementite particles, then the spacing of the bands must correspond to the dendrite spacing in the original ingot reduced by an amount corresponding to the extent of reduction in the forging process. The second set of experiments measured the primary and secondary dendrite spacing in original ingots by etching polished samples with Stead's etch [37]. A good correlation was found between the band spacing and the primary dendrite spacing reduced by an amount corresponding to the reduction on forging. There is a study available on classical ferrite/pearlite banding that provides micrographs which allow measurement of spacing in both the ingots and the hot rolled banded steels. Comparing these data shows that the final band spacing also correlates with the primary dendrite spacing [42].

A theory for band formation

The gradual alignment of the cementite particles into aligned bands of cluster particles as the number of forging steps increased was discussed above. Also, the TC treatment showed that the alignment of the cementite particles into bands could be made to occur in the same gradual process so long as the lower temperature of the cycling process was below A_{r1}. A systematic study of the evolution of the aligned bands [38] during the TC technique employed lower temperatures of the thermal cycling process both below and above the A_{r1} temperature. If the lower temperature was below A_{r1} it was found that the band re-formation was faint after the first cycle and rapidly became strong as the number of cycles increased to the maximum

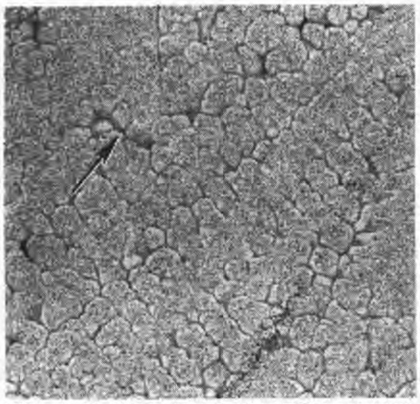

Figure 8a. Sorel ingot after TC treatment. Boiling picric etch (x 25)

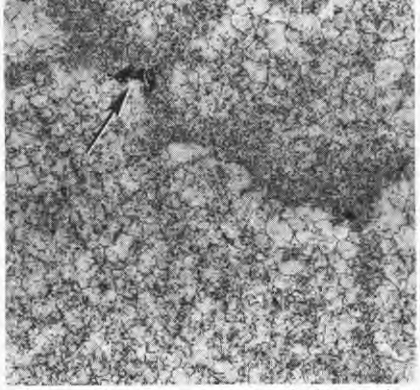

Figure 8b. Enlargement of the region located at arrow in figure 8b (x 200)

employed, 6. If, however, the lower temperature was never allowed to go below A_{r1} the cementite remained dominantly as grain boundary cementite and the bands of cluster cementite particles did not reform. By cycling through the A_{r1} temperature on each cycle a new set of austenite grain boundaries was created on each heat-up and the presence of these boundaries would provide additional locations for the cementite to reform during the cool-down step of a cycle. Other experiments [40] showed that the reformed cementite bands were collinear with FeS inclusions, thereby proving that they were forming on the deformed IRs of the ingot. The collinear arrangement of the cementite bands with FeS particles was also consistently found in forged blades. These results show that during the thermal cycling the microsegregated impurities are causing the cementite particles to gradually increase in size and density along the remnant IRs of the forged ingot as the number of thermal cycles increases; and it suggests that any nucleation of particles occurs dominantly on austenite grain boundaries.

A possible mechanism for the selective coarsening of cementite particle along the IRs during thermal cycling has been proposed [40]. During the heat-up part of each thermal cycle the smaller cementite particles will be removed by dissolution while the larger particles will remain at a reduced size. During the cool-down portion of the cycles the larger particles will grow. It is not likely, however, that the smaller particles will reform at adequate rates to replace themselves during cool-down because this requires nucleation, and the presence of the nearby larger particles provides sites for cementite growth without need of nucleation. Hence if bands of larger particles once form, the cycling process will cause them to grow at the expense of the smaller particles. Therefore, any mechanism reducing the mobility of the cementite/austenite interfaces selectively in the IRs should cause larger particles to be partially retained on the heat-up part of the thermal cycles and lead to the band formation. The EPMA study showed that all of the elements that lead to some level of band formation are microsegregated to the IRs, which is a necessary requirement of a banding mechanism. It also found that the cementite in the IRs contained a significant concentration of the two most effective carbide forming elements, V and Mo. This would lead to reduced mobility of cementite/austenite boundaries in the IRs because of either diffusion constraints or para-equilibrium effects of these elements. A possible mechanism for the effectiveness of S and P in producing moderate banding is complicated by the fact that their presence only seems to enhance banding at intermediate concentrations without the presence of the carbide forming elements. Both P and S are surface active elements and it may be that the expected segregation to cementite/austenite interfaces and consequent reduction of their mobility occurs only at the intermediate concentration levels.

An alternate possible mechanism for the formation of the aligned bands of clustered cementite particles is that submicroscopic particles or defects are present in the IRs which serve as preferred nucleation sites for the cementite particles during the thermal cycling. Possibilities include small particles, such as carbide, nitrides, oxides or sulfides, or submicroscopic voids. Both SEM studies [43] and TEM studies [44] were done, searching for submicroscopic particles or voids displaying a planar array. Small voids were not found with careful imaging in the TEM. Small particles were often found in both SEM and TEM, but they were randomly arranged showing no evidence of alignment with prior cementite bands. Hence, it seems most likely that the cementite is simply nucleating on austenite/austenite grain boundaries during the thermal cycling and then forming into aligned bands of clustered particles by the coarsening processes such as that discussed above along with some coarsening due to the classical Ostwald ripening mechanism.

The banding mechanism that seems to be operating here differs from the banding which occurs in the classical pearlite/ferrite banding of hypoeutectoid steels in that thermal cycling is required in the present case. The pearlite/ferrite bands form after a single cool down from the austenite region. However in these hypereutectoid steels no significant carbide banding is observed after the first thermal cycle. It only develops slowly with repeated thermal cycles. A recent study [39] of banding in the hypereutectoid bearing steel AISI 52100 (1.0 C, 0.35 Mn, 1.45 Cr) found that it will not form bands of cementite/pearlite on a single cool down from the austenite region. However, when heat treated to the standard spheroidized form it does produce alternating bands of high and low density regions of very fine spheroidized cementite particles in a ferrite matrix. And, this banding occurs in a single cool-down from the sub A_{cm} temperature employed in the standard spheroidizing heat treatment procedure. The mechanism involves a divorced eutectoid transformation [39]. Hence, the banding in these Damascus type steels appears to be a unique type of banding in hypereutectoid steels requiring thermal cycling.

Conclusion

A review is presented of the famous genuine Damascus steel blades describing attempts of western scientists to reproduce these blades and their various theories to explain the origin of the unique microstructure responsible for the attractive surface patterns of the blades. The art of making these famous blades was lost in the early 19th century and none of the theories or techniques have allowed modern bladesmiths to reproduce these blades. In a cooperative research program between the author and a practicing bladesmith, A.H. Pendray, a method has been developed which successfully matches the internal and surface microstructure of the genuine Damascus blades. The experimental technique is described and the results of a research program are presented which comes to the following conclusions. The unique microstructure of this steel is produced by an unusual type of banding that requires: (1) the presence of a surprisingly low level of certain impurity elements in the hypereutectoid steels, with V being most effective, and (2) a thermal cycling heat treatment. A main difference of this type of banding and the ubiquitous pearlite/ferrite banding of hypoeutectoid steels is point (2), the requirement of thermal cycling. A theory for the banding mechanism is presented.

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