Indian Journal of History of Science, 42.4 (2007) 559-574

PATTERN FORMATION IN WOOTZ DAMASCUS STEEL SWORDS AND BLADES

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(Received 14 February 2007)

Museum quality wootz Damascus Steel blades are famous for their beautiful surface patterns that are produced in the blades during the forging process of the wootz ingot. At an International meeting on wootz Damascus steel held in New York[3] in 1985 it was agreed by the experts attending that the art of making these blades had been lost sometime in nineteenth century or before. Shortly after this time the author began a collaborative study with bladesmith Alfred Pendray to try to discover how to make ingots that could be forged into blades that would match both the surface patterns and the internal carbide banded microstructure of wootz Damascus steels. After carrying out extensive experiments over a period of around 10 years this work succeeded to the point where Pendray is now able to consistently make replicas of museum quality wootz Damascus blades that match both surface and internal structures. It was not until near the end of the study that the key factor in the formation of the surface pattern was discovered. It turned out to be the inclusion of vanadium impurities in the steel at amazingly low levels, as low as 0.004% by weight. This paper reviews the development of the collaborative research effort along with our analysis of how the low level of vanadium produces the surface patterns.

Key words: Damascus steel, Steel, Wootz Damascus steel.

INTRODUCTION

Indian wootz steel was generally produced by melting a charge of bloomery iron along with various reducing materials in small closed crucibles. It produced small ingots of surprisingly high purity steel, the first high quality steel produced by man. The date of the origin of the process is debatable, but it is likely to be sometime before 500 AD. This high quality steel represents

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a triumph of Indian technology. It was not matched in the west until Huntsman developed his similar crucible steel process in England around 1750. The fame of wootz steel is due in part because it was the steel from which Damascus steel swords were forged. These swords will be called wootz Damascus swords in this paper.

The book by Leo Figiel[1] contains the best collection of photographs of wootz Damascus blades that I am aware of. Dr. Figiel made trips to India and the Near and Middle East over many years and acquired a large collection of blades that are illustrated in the book. On one of his last trips, around 1990, he acquired a small heavily corroded blade piece in Rajasthan India that he gave to my collaborator, Alfred Pendray. Fig. 1 presents both faces of the blade after Alfred polished and etched it at all locations except where the gold inlay identifying the bladesmith appears. (Note: we have never been able to obtain a good translation of this inscription.) The pattern on the surface presents a good example of the type of pattern found on the most desirable wootz Damascus blades. In older literature the wavy-like pattern is often called water or watering. Fig. 2 presents a transverse section of the blade. The section has been prepared with standard metallurgical polishing techniques using a boiling picric acid etchant that produces dark carbide (Fe_3C) particles in a white pearlite (steel) matrix. (Note: the etch for Fig. 1 produces the usual reverse contrast on the surfaces of these blades, white carbides in the dark pearlite matrix.) The dark bands of Fig. 2 are sheets of small clustered carbide particles. Longitudinal sections of the blade appears

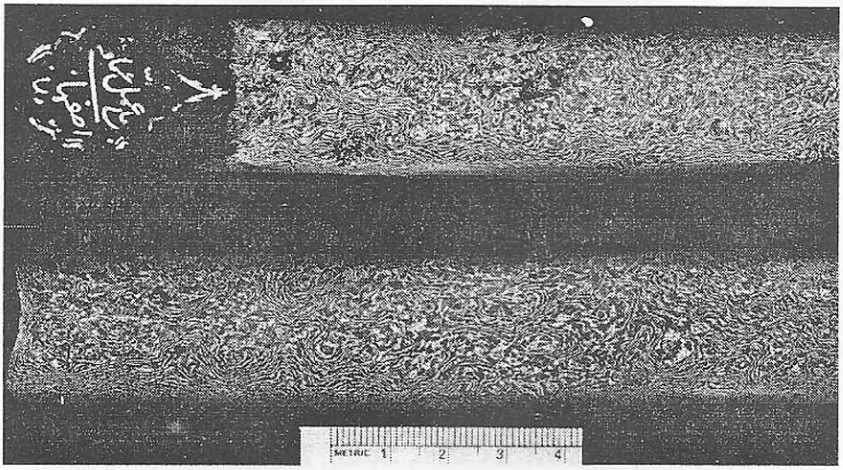

Fig. 1. The Figiel blade purchased in Rajasthan India.

virtually identical to the transverse section of Fig. 2 showing that the bands have a planar geometry. It is the presence of these planar bands of clustered carbide particles lying nearly parallel to the faces of the blades that produces the beautiful surface patterns on wootz Damascus blades.

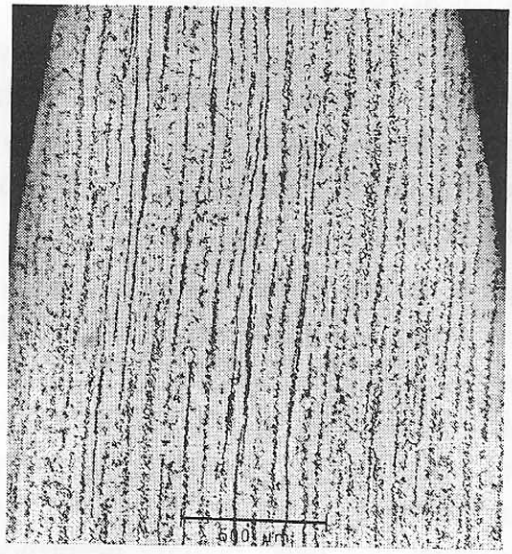

Fig. 2. Transverse section of Fig. 1 blade. Boiling picric etch

The surprising thing about these blades is that the banded structure is not produced by mechanically stacking alternating layers of high and low carbon steel sheets and forge welding them together as is done in so called pattern welded Damascus blades and gun barrels. It is known from historical records that the banded structure of the wootz Damascus blade is produced *in situ* during the forging of the small wootz steel cakes, which were often roughly the size and shape as a hockey puck.

My interest in these wootz Damascus blades was inspired mainly from the writing of C.S. Smith[2]. In his extensive review presented in chapters 3 and 4, Smith presents evidence that the blades were forged from the Indian steel known in the West as "wootz" and states: "there can be little doubt that the water is primarily a result of the structure of the cake of wootz or similar steel from which the blades were forged". Smith documented the evidence of the many, largely unsuccessful, attempts of European scientists to reproduce the patterned blades from forged ingots[2]. Perhaps more intriguingly there was an international conference at New York University in 1985[3] at which it was concluded that there was no known bladesmith who could successfully reproduce a wootz Damascus blade. Concurring in this decision were Smith and G.N. Pant from the National Museum in New Delhi.

Smith had come to the conclusion that the surface patterns resulted from formation of primary carbides (Fe_3C) in the dendritic structure of the wootz ingots from which the blades were forged. Smith had an exchange of letters-to-the Editor of Science magazine in 1983[4,5] with Wadsworth and Sherby who put forth an alternate theory. After reading these papers I thought it likely, based on my research experience in metal alloy solidification, that both might be incorrect and began to think of experiments to examine possible mechanisms of the pattern formation.

A type of banding in steels has been well known since the end of the nineteenth century when reflecting microscopes were first used to examine polished and etched steels. Fig. 3 presents an example of this banding which is usually called pearlite/ferrite banding because the dark component is pearlite (steel of 0.77% C) and the white component is ferrite (essentially pure iron). Virtually all hypoeutectoid (%C less than 0.77%) steels show this pearlite/ferrite banding if the steel has been heavily deformed followed by slow cooling from the austenite range (temperatures usually around 800-900 °C). In rolled or forged plate the bands have been shown to have a planar geometry oriented parallel to the surface of the plate, similar to the carbide bands in wootz Damascus blades. In addition it is known that the bands result from

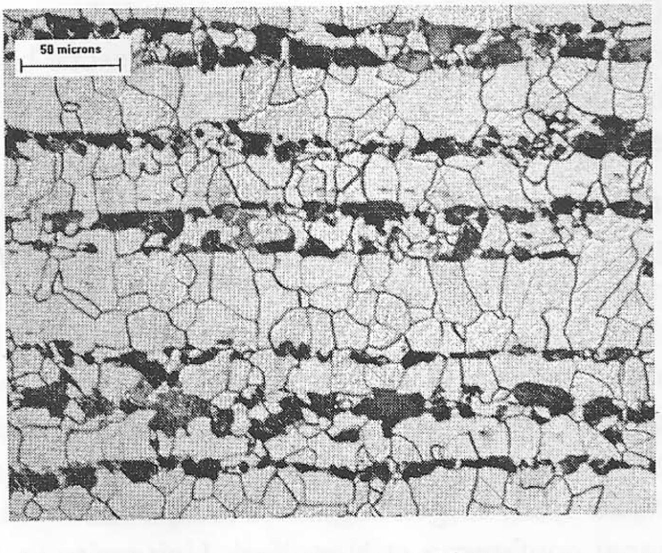

Fig. 3. A wrought 1018 steel slow cooled from 900 °C to room temperature. Deformation direction is horizontal (Nital etch, 240x)

the dendritic microstructure of the solidified metal from which the sheet was formed. The explanation is a bit complicated but is a key to understanding what appears to be forming the carbide bands in wootz Damascus blades.

As illustrated in Fig. 4, when a metal alloy like steel solidifies from the liquid the solid/liquid interface appears very similar to a miniature pine forest growing into the air, see view A. The tree-like structures are called dendrites and, as shown in Fig. 4, they grow into the liquid in aligned arrays on each steel grain. The view labeled B in the figure is looking down on the dendrite tops and illustrates that the dendrites are aligned in planar or sheet arrays. Fig. 5 is a blowup of two neighboring dendrites growing upwards. Most impurity atoms cannot dissolve in the solid iron dendrite stalks very much, but have no problem dissolving in the liquid iron between the dendrites. Therefore, as indicated by the arrows, the impurity atoms, such as phosphorus (P), sulfur (S), manganese (Mn), are ejected from the growing solid dendrites into the surrounding interdendritic liquid. They then collect along the bottom of the centerlines of the interdendritic liquid and are arranged in sheets (bands) as illustrated in view B of Fig. 4. The phenomenon is generally called microsegregation[6]. Research has shown that when the austenite dendrites of the steel of Fig. 3 cooled down the segregated impurities of Mn, which

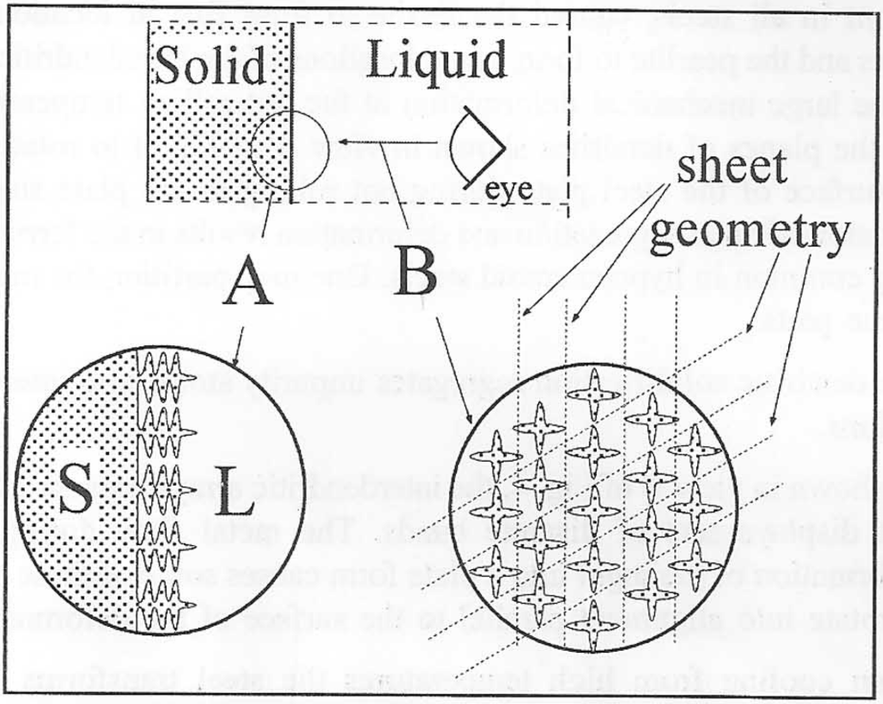

Fig. 4. Transverse and facing views of the solid/liquid interface.

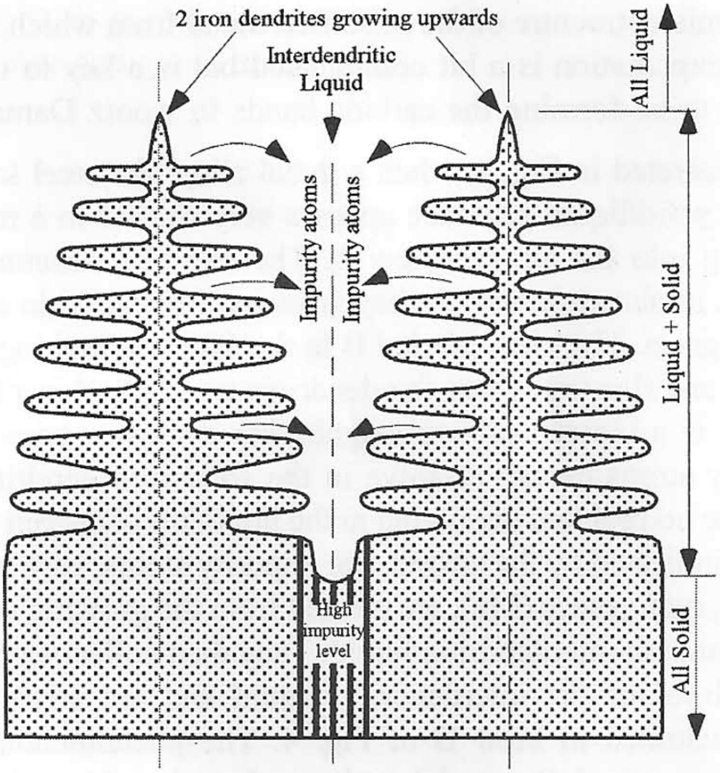

Fig. 5. Neighboring iron dendrites growing into the liquid.

is present in all steels, caused the ferrite to form first at locations of the dendrites and the pearlite to form last at locations of the interdendritic regions. Also, the large mechanical deformation at the hot rolling temperatures has caused the planes of dendrites shown in view B of Fig. 4 to rotate parallel to the surface of the steel plate during hot rolling to the plate shape. This combination of microsegregation and deformation results in the ferrite/pearlite banding common in hypoeutectoid steels. One may partition the mechanism into three parts:

A. The dendritic solidification segregates impurity atoms into interdendritic regions.

B. As shown in view B of Fig. 4, the interdendritic arrays possess a geometry that displays sets of discrete bands. The metal flow during the hot deformation of the ingot into a plate form causes some of these band sets to rotate into alignment parallel to the surface of the deformation.

C. Upon cooling from high temperatures the steel transforms into two components, pearlite and ferrite. During the final cooling the segregated impurities cause the pearlite to form preferentially in the interdendritic regions of the steel and ferrite in the dendritic regions, thereby producing the banded structure.

Smith thought that the banded microstructures of wootz Damascus steels resulted somehow from the dendritic solidification structure of the wootz ingot. He hypothesized that the carbides were forming directly from the liquid in the interdendritic regions. However, recent research[7] had shown that this does not happen in high purity iron-carbon alloys. Wootz Damascus steel is a relatively pure iron-carbon alloy and an average chemical composition of 8 blades showing excellent surface patterns is given in Table 1. Our initial experiments[8] on alloys matching these compositions confirmed that carbides do not form in the interdendritic liquid in small ingots. Wootz Damascus steels are hypereutectoid steels (%C greater than 0.77%) which means that on cooling from high temperatures the structure transforms to pearlite and carbides rather than to pearlite and ferrite as in the hypoeutectoid steels that display the banding of Fig. 3. I thought it likely that the carbides were forming along the interdendritic regions when the structure transformed to pearlite and carbide on the final cool-down of the blade, similar to how the ferrite forms first along dendrite sheets in pearlite/ferrite banding. My initial research[8] showed this hypothesis to be incorrect. However, as a result of this research I met Alfred Pendray, a practicing bladesmith in Florida, who had set up a gas fired furnace in his shop which produced small steel ingots of the same size and shape as some of the Indian wootz ingots.

Table 1. Average composition of 8 wootz Damascus blades[8]

Element	C	Si	Mn	S	P
Weight %	1.6	0.043	0.056	0.02	0.11

Alfred had also read C.S. Smith's book and was aware of the literature showing how the Indian smiths had made their ingots. He used small clay graphite crucibles charged with iron and steels plus a slag forming mixture of broken glass and oyster shells packed down with green leaves. I started examining Pendray blade pieces for chemical composition and structure and found that occasionally the blades were remarkably similar to patterns like those of Figs 1 and 2, which we came to call wootz Damascus patterns.

Because the process only worked occasionally we set up a cooperative research program to systematically study all of the variables, such as composition of the charging metals, time and temperature variations of melting and cooling and variations of the forging process. After roughly 10 years of work Alfred was able to produce wootz Damascus patterned blades consistently as described in the *Scientific American* article[9] and more completely detailed in ref[10]. We discovered several important factors which will be described in a sort of relative order of importance in the following:

1. Role of Impurity Atoms

Previous research had established the chemical composition of wootz Damascus blades as shown in Table 1 and this became the target composition. Initially[11] the metal charge was made up of various mixtures of 1010 steel, Sorel metal, wrought iron and Armco iron (a quite pure electrolytic iron). Some of the ingots produced good banding and some did not. Eventually we noticed that the good ingots usually employed Sorel iron as one of the iron ingredients and we were able to consistently produce good banding by using a mixture of the Sorel iron and Armco iron. By doing extensive chemical analysis on these ingots we found that they contained very low levels of the carbide forming elements, V, Cr and Ti[12]. This led to a study[13] where the metal ingredient was the purest form of iron that we had, Armco iron, doped with individual pure carbide forming elements V, Cr, Nb, Mn, Mo or non carbide forming elements, Cu, P and S. The results confirmed that the banding was produced by quite low levels of the carbide forming elements, with the strongest banding formed in ingots doped to 0.01 to 0.03 % V or 0.03 % Mo. These were the lowest levels of V and Mo studied[13]. The previous study[12] had established that V levels as low as 0.004 % produced good banding. It was because of this extremely low level that it took us so many years to discover the importance of the V and other carbide forming elements for the successful pattern formation of wootz Damascus blades.

The second study[13] utilized electron probe microanalysis in the scanning electron microscope which showed that the carbide forming elements had become concentrated to much higher concentrations in the carbide bands of the blades. We knew that the carbide bands responsible for the pattern were forming in the interdendritic regions of the ingots because all the sulfide particles where aligned with the carbide bands and sulfides are known to form in the interdendritic regions. Hence, these results provided strong evidence that the banding mechanism of the wootz Damascus blades must have strong similarities to pearlite/ferrite banding of hypoeutectoid steels.

Subsequent to this work we did chemical analyses on as many high quality ancient wootz Damascus steel blades available to us that could be sectioned. In the first study of 7 blades[14] (which included the Fig. 1 blade) we found that 6 of the blades contained V at levels ranging from 0.004 to 0.027 %. In the only blade with low V (less than 0.001 %) the carbide forming element Mn was present at 0.05 % and the %C was unusually low, 1.0%. It seems likely that this combination accounts for the good banding of this particular blade. In a continuing study of 3 additional wootz Damascus blades[15], the V levels was found to vary from 0.01 to 0.013 %. Interestingly, our success in tracking down the importance of low level carbide elements involved a bit of luck. The Sorel metal, that eventually allowed us to generate good patterns, consistently contains a low level of V. Sorel metal, which is widely used to make ductile cast iron, comes from an ilmenite ore in Canada that contains a low level of V impurities, around 0.04%. The source of this V is the ore deposit, which has suggested to us that the ore deposits used to make Indian wootz cakes that successfully produced wootz Damascus patterned blades probably also contained low levels of V. It would be interesting to study some of the Indian ore bodies that could be identified as possible source of the ancient Indian wootz ingots.

2. Forging Wootz Ingots

In Smith's review of wootz Damascus steel[2] he points out that after Europeans began studying wootz cakes from India they had great difficulty in forging them. For example, "Reaumur[16] deplored the skill of Parisian artisans, none of whom succeeded in forging a tool out of a cake of Indian steel." In Pendray's initial experiments with steel compositions similar to those of Table 1 it was found that the ingots became extremely brittle at the initial hot forging temperatures, a phenomena known as "hot shortness". Alfred devised a trick to overcome this problem. He packed the ingots in mill scale (iron oxide) and heated them very hot (around 1100-1200 °C) for several hours and was then able to forge them. This treatment was shown to produce a rim of lower %C iron on the ingot surface. The oxidizing treatment essentially purified the outer rim of the ingot, lowering the carbon and phosphorus levels there. We found that the source of the embrittlement in the wootz cakes was the high level of P. Phosphorous forms a eutectic component with Fe in iron-carbon alloys called Steadite that melts around 970 °C. On hot forging, which is done at temperatures above 970 °C, the Steadite melts and leads to the embrittlement called hot shortness. The lowered %C in the oxidized rim allows it to be ductile at hot forging temperatures and it acts as a container which eliminates the hot shortness. We found that if the P was reduced to around 0.03% the hot shortness of non-oxidized ingots went away but the carbide banding remained. Experiments from then on utilized low P levels.

By another bit of luck we were able to obtain experimental evidence that the ancient Indian smiths also used Alfred's oxidation trick to overcome the hot short problem in forging their wootz ingots. In the early 1980s Alfred was able to purchase 2 bars of steel (roughly 1cm x 5 cm x 29 cm) from R. Charlton of Damascus USA that were imported from the Alwar Armory in Rajasthan India. Alfred had Pant examine these bars at the 1985 meeting in New York. From the special markings punched into the surface of the bars, Pant verified that they were from the Alwar Armory and estimated their age at roughly 300 years old. Fig. 6 presents a micrograph of bar 1 after Alfred forged it to a 4 mm thickness, heavily ground the surface and polished and etched it. One sees that a good wootz Damascus pattern has developed in the

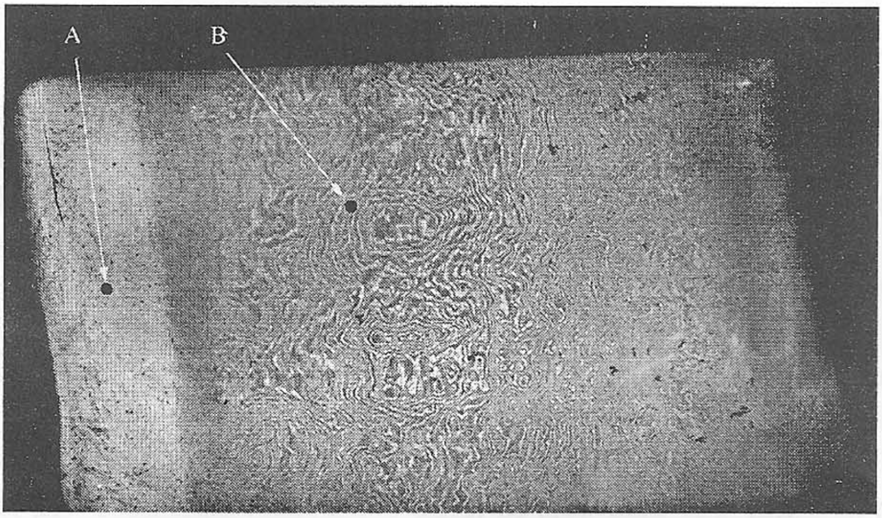

Fig. 6. The ground and polished Alwar bar.

center of blade face and that a light colored rim is present on the edges. Analysis of the center and rim, at A and B on Fig. 6, found that both C and P were lowered in the rim, from 1.48 to 0.66% and 1.04 to 0.92% respectively. The V analyses were 0.01% in the center and 0.009% in the rim. These results on this and a similar bar[15] present strong evidence that the ancient Indian smiths were producing a ductile low carbon rim by oxidizing the surface of their ingots to avoid the hot short problems, a trick not discovered by most nineteenth century western smiths.

3. Destruction and Reformation of the Pattern

In the course of our experiments we discovered an important experimental technique we call the TC (thermal cycle) treatment[17]. If a wootz Damascus steel blade is heated above its A_{cm} temperature, which is around 1000°C for most blades, all of the carbide particles will dissolve into the steel matrix, and upon cooling back to room temperature the pattern will be gone. The TC treatment consists of heating a blade to around 1050-1100°C for a short while and then quenching it in water. At this point there are no carbides in the blade and the pattern is destroyed. The blade is then thermally cycled between a low temperature of around 500 °C and a high temperature of around 950 °C. After the first thermal cycle it is found that the carbides have reformed in a random distribution. However, on subsequent cycles it was found that the carbides gradually began to form into the banded arrays parallel to those present in the original blade. After around 6 cycles the banding quality was similar to that in the original blade. The same result was found on blades made by Alfred Pendray and on several ancient blades we were able to study. Fig. 7 displays the results found with the Figiel blade of Fig. 1. Fig. 7(a) presents a longitudinal section of the blade before the TC treatment, and Fig. 7(b) after a 6 cycle TC treatment. Note that the bands of clustered carbides have returned in roughly the same geometry but with smaller particle diameters The forging operation used to produce the original blade is known to have required a few dozen thermal cycles which would lead to larger particle sizes than the simple 6 cycle TC treatment. These experiments offer strong evidence that the hypothesis of both Smith[4] and Wadsworth-Sherby[5] cannot explain the banding formation in wootz Damascus steels. In both hypotheses the carbide particles present in the final blade must be those particles, or pieces of those particles, formed prior to the deformation

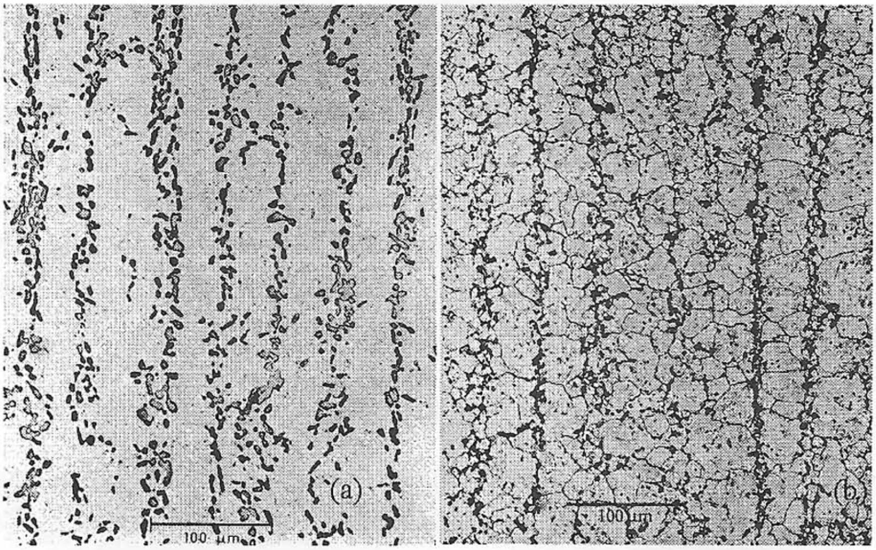

Fig. 7. (a) Longitudinal section of Figiel blade. (b) Long. section after TC treatment of 6 thermal cycles. Boiling picric etch.

on forging. The complete destruction of the original banded carbide particles in the TC treatment followed by the reappearance of the aligned carbide bands on thermal cycling similar to that of the forging process shows that both of these hypothesis are incorrect.

Several research studies[18] on pearlite/ferrite banding in hypoeutectoid steels proved that the banding was caused by microsegregation of impurities between dendrites by the following experimental technique. Bars that showed strong banding when simply cooled from around 850 °C were heated for long times, 6-24 hours, at very high temperatures, around 1200°C. After this treatment pearlite/ferrite banding no longer occurred on cooling from 850°C. The high temperature treatment was shown to have homogenized the impurity atoms in the bars by the process known as diffusion. Following this lead we subjected wootz Damascus blades that displayed good patterns to a temperature of 1200°C for around 18 hours. In both the Pendray blades and the ancient blades after this high temperature anneal, a TC treatment of the blades no longer produced bands of carbides, the carbide distribution was random. Fig. 8 illustrates the results for the Figiel blade. These results present very strong experimental evidence that the carbide band formation is caused by the microsegregation of some impurity element or elements during solidification of the wootz ingots. Interestingly, at the time of the TC work[17]

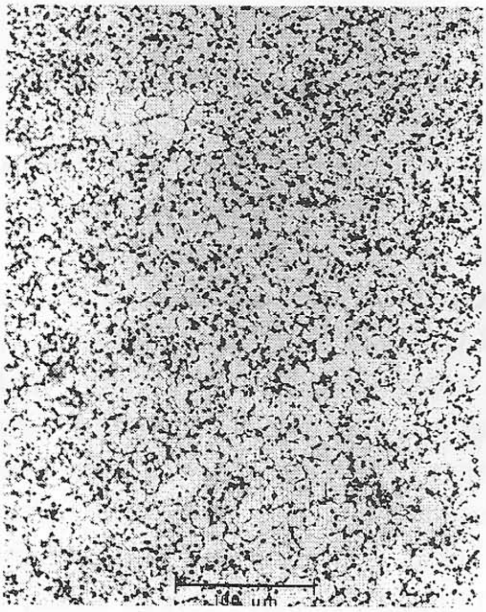

Fig. 8. Long. section of Figiel blade after 18 h anneal plus 6 cycle TC treatment.

we thought the responsible impurity was probably one of the 4 well known impurity elements in wootz Damascus steels shown in Table 1, Si, Mn, P or S. It was only later that we discovered that the responsible element in the blades we were studying was V, present at much lower concentrations than these four elements.

Our present theory is that the banding formation in wootz Damascus steel blades is due to microsegregation of carbide forming impurities, usually V. The general mechanism for the alignment into bands is the same as that for pearlite/ferrite banding in hypoeutectoid steels for steps A and B listed above, but differs for step C. Whereas the band formation in pearlite/ferrite banding is complete after a single cool down, in the wootz Damascus case several thermal cycles are required to produce the banding. During the repeated heating and cooling cycles of the forging process that forms the blade shape, the carbides present in the original ingot are continually being partially dissolved and reformed. The microsegregated V impurities cause the reforming carbides to slowly become positioned primarily along their banded locations as the number of forging cycles becomes large, thereby producing the bands of clustered carbides responsible for the beautiful surface patterns of the wootz Damascus blade. We have presented an hypothesis for the mechanism by which the V causes this effect[12,13].

4. The Mohamed's Ladder Pattern

Many of the better museum quality wootz Damascus blades that have been preserved show distinct surface patterns. One is a ladder pattern that is often called Mohammed's ladder. As shown at the arrows in Fig. 9 it consists of a series of transverse doublet surface patterns spaced at regular intervals along the blade length similar to the spaced rungs of a ladder. The second distinct pattern is a circular formation similar to that shown between the ladder rungs in Fig. 9 which is often called a rose pattern. As discussed in[14] there had been some controversy in the literature as to how these patterns were formed. Smith[2] argued that the patterns were produced by cutting grooves into the surface of a nearly finished blade and then forging them out in the final blade. The blade of Fig. 9 was produced by Pendray (blade 41) using Smith's method. The ladder pattern was made by machining shallow grooves transversely across the nearly finished forged blade, while the rose pattern was made by drilling shallow holes. A more detailed discussion of these results may be found in[14]. Fig. 9 has been presented to also illustrate the excellent quality of the patterns formed on Pendray's reconstructed wootz Damascus blades. In addition, this particular blade has an interesting history in our long effort to learn how to reproduce the famous wootz Damascus blades. The blade, called no. 41, was made in April, 1991 using a metal charge of Armco iron plus wrought iron[11]. It was one of the very few blades

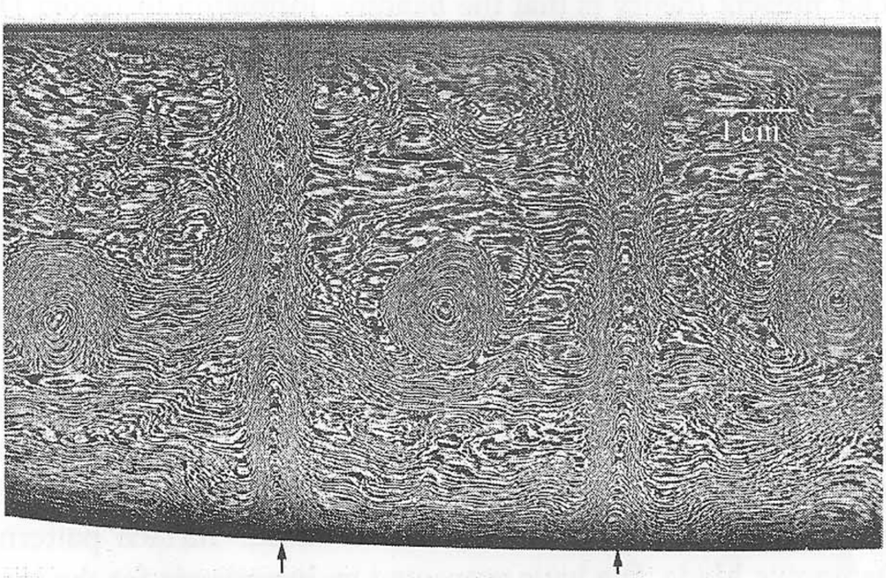

Fig. 9. Mohammed ladder and rose pattern on Pendray blade 41.

displaying a good pattern that had not included some Sorel iron in the metal charge. After we finally discovered the importance of the very low levels of V impurities for pattern formation we went back and analyzed blade 41 for low levels of impurity atoms. The full analysis, given in[15], shows that the blade contained 0.005% of V and 0.09% of Cr. Apparently these critical carbide forming elements happened to be present in the wrought iron charge and the result is further evidence for the importance of low levels of carbide forming elements for carbide band formation in wootz Damascus steel.

ACKNOWLEDGEMENTS

It should be clear from the text that master bladesmith Alfred Pendray made major contributions to this work. I would also like to acknowledge many useful discussions with Bill Dauksch, a fellow metallurgist retired from Nucor Steel who also helped us obtain funding and chemical analysis support from Nucor Steel. At Iowa State University the Department of Materials Science and the Ames Laboratory of the USDOE provided me with laboratory facilities and support throughout this study.

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