The Continuing Study of Damascus Steel: Bars from the Alwar Armory

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The authors published a paper in this journal in 1998¹ titled "The Key Role of Impurities in Ancient Damascus Steel Blades." Because of the continued popularity of the on-line version of this paper,² additional experiments were conducted on some three-century old Damascus bars. The results of those experiments are reported in this paper.

INTRODUCTION

Damascus steel has a long history of interest to metallurgists. C.S. Smith claimed the development of Damascus steel blades as one of the four outstanding accomplishments of early metallurgy,³ and he devoted two chapters of his book4 to Damascus steels. At the end of Chapter 3, in referring to the Damascus blades he states: "It was known that they were forged from Indian steel known as wootz but this was regarded as almost unworkable by European smiths. Porta⁵ says that it can be worked after it has been annealed in calcined gypsum (i.e., lime) and very slowly cooled. Reaumur⁶ deplored the skill of Parisian artisans, none of whom succeeded in forging a tool out of a cake of Indian steel. In Chapter 4 of Smith's book,⁴ he describes the unsuccessful efforts of European metallurgists to reproduce the patterned Damascus steel blades over roughly two centuries. As described in the authors' previous JOM paper,1 research has developed to the point where A.H. Pendray can now routinely produce blades that match the surface patterns and internal microstructure of the best museum-quality Damascus blades.

RIM FORMATION ON INGOTS

In researching how to make blades that matched museum-quality Damascus blades, the authors surveyed the literature on the composition of Damascus steel blades and developed the following average composition (values shown are in weight percent): C-1.60 Mn-0.056 P-0.107 S-0.02 Si-0.043 Ni-0.013 Cu-0.044 and trace Cr. Using this information, small ingots weighing roughly 2.2 kg were made that were similar in size and composition to the wootz ingots. As predicted by Smith's literature citations, the ingots were exceedingly difficult to forge. They were extremely hot short. After many failures, this hot short problem was overcome by packing the ingots in mill scale (iron oxide) and heating them to around 1,200°C for several hours. This treatment decarburized the surface and produced a rim of lower-carbon iron on the ingots that successfully contained the hot short interior during hot forging. The effectiveness of the rim was verified

in an experiment where the rim was locally ground from one edge of an ingot. During subsequent hot forging, the mushy interior of the ingot broke through the surface at the ground region. Metallographic studies revealed that the ingots were loaded with the phosphorous constituent known as steadite. Steadite is a ternary eutectic that forms in ironcarbon ingots at sufficient phosphorous levels. It melts at 970°C, which is below the hot-forging temperature, and it was theorized to be the cause of the extreme hot shortness of the wootz ingots. This theory was verified by producing ingots with lowered phosphorous levels. At phosphorous levels below around 0.03% to 0.04%, the hot shortness was no longer a problem and pattern formation was not affected.

These experiments and the statement by Porta indicate that the old bladesmiths of Damascus utilized some sort of a rim heat treatment to allow forging of the high-phosphorous hot-short wootz ingots. This paper reports on additional experiments that support this idea. In the early 1980s, one of the authors (AP)

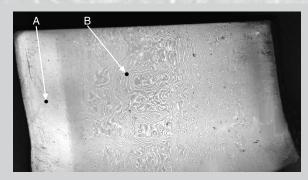


Figure 1. Alwar bar 1 after forging to 4 mm thickness, width = 56 mm.

Over the last 40 years, there has been a discernible increase in the number of scholars who have focused their research on early industrial organizations, a field of study that has come to be known as Archaeotechnology. Archaeologists have conducted fieldwork geared to the study of ancient technologies in a cultural context and have drawn on the laboratory analyses developed by materials scientists as one portion of their interpretive program. Papers for this department are solicited and/or reviewed by Michael Notis, a professor and director of the Archaeometallurgy Laboratory (www.Lehigh.edu/~inarcmet) at Lehigh University.

Element	Bar 1 Loc. A	Bar 1 Loc. B	Bar 1 Piece 2	Bar 3	Kard	Blade 41
C	0.66	1.48	1.47	1.24	1.76	1.56
Mn	<100	<100	<100	500	<100	<100
Р	920	1,040	1,000	660	1,430	370
S	60	70	70	10	110	180
Si	900	900	1,000	400	800	2,300
Ni	500	500	500	200	900	900
Cr	100	100	100	<100	100	900
Мо	<100	<100	<100	<100	100	270
Cu	1,000	1,000	1,200	800	1,000	950
Al	<10	<10	<10	40	<10	<10
V	90	100	100	130	120	47
Nb	<10	<10	10	<10	60	<100
Pb	<5	<5	12	<1	27	30
Sn	<10	<10	<10	20	30	100
Ti	<10	10	10	40	20	20
Zr	10	20	30	_	50	<10
В	4	5	5	<1	8	25

* wt.% for carbon and ppmw other elements

was able to purchase two bars of steel (roughly 1 cm \times 5 cm \times 29 cm) from R. Charlton of Damascus USA that were imported from the Alwar Armory in Rajasthan, India. The bars were examined by G.N. Pant, a professor of museology at the National Museum in New Delhi during the 1985 International Symposium on Damascus Steel held at New York University.7 Pant reported that around two dozen such bars were at the Alwar Armory and they carried a special identification marking. He verified from markings on the two bars that they were from the Alwar Armory and estimated their age at around 300 years old. It was his opinion the bars had been forged from Indian wootz cakes in Persia and then sent back to India. These bars were forged to a blade shape to see if a Damask pattern would develop in them. The forging temperature ranged from roughly 900°C to 680°C. Only one of the bars, called bar 1 here, developed a pattern. Figure 1 is a photomicrograph of the polished surface of bar 1 after forging and then surface grinding to a final thickness of 4 mm. The lack of pattern in bar 2 was the result of graphitization in this bar. Previous work has shown that inadequate reduction on the initial forging cycles of the ingot can lead to graphitization and the loss of pattern formation.8 Figure 1 demonstrates that a well-defined Damask pattern formed in the central region of bar 1 and is surrounded by a non-patterned region at each edge. This is precisely what one would expect to see if the original ingot had been heat treated to produce a ductile rim at its

surface. Surface grinding was required to grind through the rim on the top center of the forged bar, but the rim remains on each edge of the bar. Standard emission spectrographic analysis was carried out on bar 1 at the locations labeled A and B on Figure 1, with A in the rim region and B in the patterned region. The resulting chemical analyses are presented in Table I, where all analyses are given in parts per million by weight (ppmw) except for carbon, which is given in weight percent. The major difference in the two locations is a large reduction in carbon, from 1.48% in the patterned region to 0.66% in the rim. There is also a small reduction in sulfur and phosphorous levels. These results present strong evidence that the Damascus smiths had learned to forge the hot-short wootz ingots by producing a ductile rim on them by some sort of an oxidizing high-temperature treatment.

The authors were recently given another bar from the Alwar armory, which will be called bar 3. The bar came from Oriental-Arms Ltd. of Haifa, Israel, and displayed a characteristic mark that showed some strong similarities to the marks on bars 1 and 2. The as-received bar measured $1.3 \times 3.2 \times 58$ cm. Figure 2 presents a micrograph of the face of the bar after forging the 1.3 cm thickness to 5 mm. As with bar, a Damascus pattern has formed in the center of the bar surrounded by a rim of non-patterned steel on both sides. The rim on this bar was not analyzed, but was assumed to be low carbon since etching reveals the absence of carbides within it. The central patterned region was analyzed, and the results are given in Table I.

Both bars 1 and 3 were sectioned longitudinally and transversely, and both bars revealed aligned bands of clustered cementite particles that are responsible for the patterns on genuine Damascus steel blades. Examples of this microstructure are presented for bar 3 in Figure 3. Figure 3a illustrates the typical characteristic aligned bands of carbide particles in a longitudinal section through the blade. The carbide bands are aligned in the forging plane. Figure 3b illustrates the appearance of a carbide band on the surface shown in Figure 2. The matrix of the bar is a fine pearlite, which was produced by an oil quench from 930°C. The hardenability of these Damascus steels is extremely low. Experiments have found that an oil quench produces martensite only for blade thicknesses less than approximately 1.5 mm.

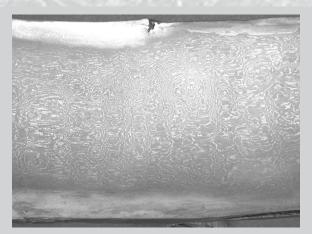


Figure 2. Alwar bar 3 after forging to 5 mm thickness, width = 42 mm.

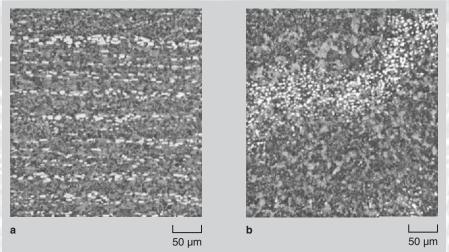


Figure 3. Micrographs of a longitudinal section (a) and surface (b) of bar.

KEY IMPURITY ELEMENTS IN THE INGOTS

The initial efforts to produce Damask patterns in small forged ingots were successful only a fraction of the time. However, when Sorel iron was used in combination with electrolytic iron and graphite to make up the ingots, the success rate went way up. In retrospect, it seems obvious that there must have been some element in the Sorel iron that caused the success. The successful ingots were chemically analyzed to check this possibility. The initial studies9 only analyzed for the elements shown in the average composition developed by the authors. Subsequent analyses were eventually extended to other elements and found small levels of vanadium present in the ingots and also in the Sorel iron. The levels, however, were very small, only around 0.01% (100 ppmw) or less. Because there were so many variables in the overall processing that might have controlled the pattern formation of the final blades, we continued to disregard the low levels of vanadium for an embarrassingly long time. After several years of work, we finally did critical experiments on ingots made only from pure electrolytic iron and those doped with low levels of carbide-forming elements: vanadium, chromium, and titanium.¹⁰ This work, along with a more systematic study,11 established that low levels of the carbide-forming elements-V, Mo, Cr, Nb, and Mn-were effective in causing the pattern to form during hot forging, with V and Mo being most effective.

The previous *JOM* paper¹ presented data on seven museum-quality Damascus blades, and all but one contained vanadium at levels of 40 to 270 ppmw, which overlapped the 60 to 110 ppmw level that control experiments found adequate to cause Damask pattern formation.^{10,11} The one exception contained manganese at levels above those the control experiments required for band formation.¹¹ One of the main purposes of this work was to do chemical analyses on the Alwar bars and two other Damascus blades to see if they also contained low levels of carbide-forming elements.

Several chemical analyses of the Alwar bars are given in Table I. A second piece of bar 1 was forged to a blade shape and it also displayed a good Damask pattern. The column of Table I labeled Bar 1–Piece 2 is the analysis taken on the patterned region of this piece. It is seen that the composition is very close to that found on position B of bar 1. The excellent agreement between these two analyses taken at different locations along bar 1 indicates that the composition is uniform in the bar and that the precision of the analysis is quite good.

Gene Beall provided us with an oriental kard blade from his collection which exhibited a fine Damask pattern. As shown in Figure 4, the pattern contained the famous Mohammed's ladder structure found on the higherquality blades. The figure shows a 6 cm length out of the total blade length of 21 cm. The blade was analyzed and the results are presented in Table I. The kard blade and both Alwar bars are seen to contain vanadium impurities in the 100 ppmw to 130 ppmw range. These data offer strong supporting evidence that the element vanadium was present at very low levels in most genuine Damascus steels and played a key role in the formation of the Damask pattern during the hot forging of the ingots.

Additional support for the key role of vanadium and other carbide-forming elements for pattern formation comes from a recent chemical analysis of a rare early experiment in which we were able to produce a good Damask pattern without using Sorel iron. The ingot, labeled 41 in previous publications,9,12 was made from a mixture of electrolytic iron (sometimes called Armco iron), an iron powder from Allied Metals Inc., and an available bar of wrought iron. This blade produced excellent Damask patterns (see Figure 6b of Reference 1), but had not previously been analyzed for low levels of carbide-forming elements. The recent analysis of this blade, shown in the rightmost column of Table I, reveals that it has levels of vanadium, molybdenum, and chromium in the range previously found¹¹ adequate to produce cementite band formation.

MECHANISM OF PATTERN FORMATION

The surface pattern in Damascus blades results from bands of cementite

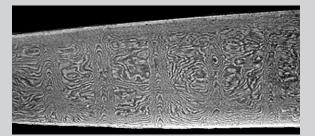


Figure 4. The kard blade; analysis given in Table I.

particles lying nearly parallel to the forged surface of the blade. If the vanadium is removed from the ingot, the cementite particles form in a random manner and no surface pattern occurs. So, somehow the minute amount of the vanadium in the ingots is causing the cementite particles to become arranged in parallel bands during the hot forging. Some early research¹² revealed an experimental technique that sheds light on the mechanism by which the Damask pattern forms during the hot forging of the ingots. The experiment, called TCT here (for thermal cycling treatment), consists of heating a Damascus blade with a well-formed surface pattern above its A_{cm} temperature to dissolve the cementite particles and then quenching the blade in water. The quenched sample is then thermally cycled at least six times from roughly 940°C to 500°C. After quenching, the blade consists of a mixture of martensite and retained austenite; all the cementite particles are gone and no surface pattern persists. After the first thermal cycle the cementite particles had returned, but with a nearly random alignment. However, after additional cycles an alignment began to occur and after six or more cycles distinct bands of cementite, again lying in the forging plain, became clear. Experiments on both museum-quality blades and the reconstructed blades¹² showed similar results. This same type of gradual transition of randomly arrayed cementite particles into aligned bands was also found to occur during the hot forging of the small vanadiumcontaining ingots into blades.9

A TCT experiment was carried out on a piece of Alwar bar 1. After holding at 1,100°C for 3 min., the sample was water quenched and then cycled six times under an inert atmosphere between 940°C and 500°C. The bands of clustered cementite particles aligned in the forging plane reappeared after the six cycles, but with a finer size and also smaller spacing. Hence, the Alwar bar gives the same results as obtained on other genuine Damascus blades: The cementite bands can be destroyed by quenching from just above the Acm temperature, but they will reform with a smaller particle size by thermal cycling at least six times.

These and previous TCT experiments provide some evidence for how the carbide banding arises during the cyclic forging of genuine Damascus blades. As discussed in more detail elsewhere,^{10,13} the mechanism is similar to the ferrite/pearlite banding mechanism of hypoeutectoid steels in at least two aspects. First, during solidification, the vanadium microsegregates into arrays lying in the interdendritic regions and then the forging causes the interdendritic arrays to become aligned into planar bands lying parallel to the forging plane. Second, the presence of the microsegregated vanadium along the planar bands causes the carbides to form as clustered arrays along the bands rather than as random arrays during the forging process. A major difference with pearlite/ferrite banding is that the cementite bands do not form on a single cool-down from the austenitizing temperature. Rather, these bands of clustered cementite particles form slowly, over several thermal cycles. The mechanism that causes the cementite particles to form or coarsen preferentially along the microsegregated vanadium bands is not well established.

CONCLUSIONS

These experiments lend strong support to the idea that two factors inhibited western bladesmiths from reproducing the patterned Damask steel blades for so many years:

- The wootz steel ingots from which the blades were made were extremely hot short due to the high phosphorous content. Successful forging requires either a rim heat treatment of the ingot or a reduction of phosphorous to levels on the order of 0.03–0.04% or less.
- In addition to the high carbon level of Damascus steels, it is necessary to have a low level of

carbide-forming elements present in the precursor ingots. The most effective of these is vanadium, with experiments^{10,11} showing levels of 60 ppmw to 110 ppmw being adequate to cause pattern formation during hot forging.

ACKNOWLEDGEMENTS

The Nucor Steel Co. has supported this work by providing all of the chemical analyses reported here. All metallographic work was done at the Ames Laboratory, which is supported by the U.S. Department of Energy, Office of Basic Energy Research, Iowa State University, contract W-7405-ENG-82.

References

- 1.J.D.Verhoeven, A.H. Pendray, and W.E. Dauksch, JOM, 50 (9) (1998), pp.58–64; www.tms.org/pubs/journals/JOM/ 9809/Verhoeven-9809.html.
- 2 J.J. Robinson, JOM, 53 (10) (2001), p. 1.

3. C.S. Smith, *Four Outstanding Researches in Metallurgical History* (Philadelphia, PA: ASTM, 1963), pp. 17–23.

- 4. C.S. Smith, A History of Metallography (Cambridge, MA: MIT Press, 1988).
- 5. G.B. della Porta, *Magiae naturalis libri XX* (Naples, 1588). (English trans. London, 1658; reprinted, New York, 1958.)
- 6. R.A.F. de reaumur, *L'Art de convertir le fer fogge en acier* ... (Paris, 1722; English trans. Chicago, 1956).

7. D.W. Ecker and G.N. Pant, *The Damascus Blade: Legends and Realities*, Proc. Symp. on the Blacksmith Art (New York: New York University, 1985).

8. P.M. Berge et al., *Iron and Steelmaker*, 22 (3) (1995), pp. 67–72.

9. J.D. Verhoeven and A.H. Pendray, *Materials Characterization*, 29 (1992), pp. 195–212.

10. J.D. Verhoeven, A.H. Pendray, and E.D. Gibson, Materials Characterization, 37 (1996), pp. 9–22.

11. J.D. Verhoeven et al., ISS Transactions, Iron and Steelmaker, 25 (11) (1998), pp. 65–74.

 J.D. Verhoeven, A.H. Pendray, and P.M. Burge, Materials Characterizations, 30 (1993), pp. 187–200.
J.D. Verhoeven, Steels Research, 73 (2002), pp. 347–355; www.mse.iastate.edu/files/verhoeven/ steelresearchsize2.pdf.

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