Reducing grain size in pattern welded damascus steel

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Abstract—During pattern welding, the used steels are exposed to high temperatures resulting in coarse grains. During this research, the method of normalizing inside a heat treating oven by an air quench is used to refine the steel's grains. Results show this method is suitable to refine very coarse grained steels to fine grained steel, but it is not possible to obtain very fine or ultra fine grains, salt pot furnaces are preferred for this application.

I. INTRODUCTION

Pattern welded damascus knife blades are exposed to very high temperatures during production. Pattern welding is a process of forge welding two different pieces of steel together creating an alternating stack of approximately 8 layers, called a billet. Pattern welding is primarily done because of its beautiful appearance. A completed pattern welded knife has a beautiful pattern consisting of many random lines and shapes on its surface.

Forge welding occurs at 1250°C. At this temperature the austenite grain is growing at very high rates. Another process of pattern welding is elongating the billet and reducing its thickness thus reducing the steels grain size. These two phenomena create an interaction. Fine grained steel has advantages over course grained steel, it has higher toughness, making it a more suitable material in knife applications. Toughness increases edge retention.

Since different types of pattern welding can give different outcomes in terms of grain size, the end result is unknown. When a knife blade needs to be produced to the highest quality standards, one should be aware of the steels grain size. In this research the effects of forging at high temperatures are taken into consideration and the known heat treatment of normalizing to refine the grain size is investigated.

II. MATERIALS AND METHODS

A. The steels

O2 and 75Ni8 are the chosen tool steels for this application. Their composition can be found in appendix III, Table 4. These tool steels both have very good characteristics regarding cutting ability and are both used in industry in knife, saw, tool and die operations. In the pattern welding application these steels are favorite because of their austenitizing temperatures being quite similar resulting in similar hardenability. Both steels are oil hardening. During the pattern welding process, the carbon content of the richer O2 will diffuse into the leaner 75Ni8 creating an overall more balanced carbon content of +/- 0,82%C, a hypereutectoid steel. Pure nickel plates in a pattern welded steel are known to form a barrier preventing carbon to migrate to another layer. The 2% Ni added to 75Ni8 is negligible, so carbon is still able to pass through to the adjacent layer. The amount of carbon diffusion to the air is negligible and is not taken into consideration.

Another reason why these steels are chosen is to reveal the damascus pattern, the knife as an end result is etched in ferric chloride after it has been polished with 800 grit sandpaper. Due to the high manganese content in the O2, black oxides are formed and the steel is etched relatively deep. Because of the 2% nickel in the 75Ni8, the steel of this layer resists the etch better resulting in a less deep etch and it does not form the black oxides, the oxides are colored grey. This phenomenon gives the contrast between the layers, making the typical damascus pattern visible (Fig. 1, appendix II). After etching the higher located layers of the 75Ni8, these layers are polished again with 800 grit sandpaper, making them shiny, giving even more contrast.

B. The damascus billet

A stack of 30 alternating layers was made, 82x40x66mm in size. The billet contained 15 pieces of 2,4mm thick 1.2842 tool steel and 15 pieces of 2mm thick 75Ni8 tool steel (Fig. 2, appendix II). The billet was heated in a gas fired forge to approximately 1250°C, called a welding heat (Fig. 3, appendix II). The forge takes about 10 minutes to heat the billet up to welding heat. When this temperature is achieved the billet is soaked for another 3 minutes. In the meanwhile the billet is fluxed in anhydrous borax to create a protective layer on the steel's surface to prevent it from oxidizing which inhibits a successful weld.

C. The forge welding process

When the billet has a homogenous heat and has soaked for 3 minutes the whole piece is rapidly removed from the forge and is compressed under the hydraulic forging press. The main goal at this point is to fuse the two steels together. This welding process is done by pressure and temperature in a solid state. Good welding success requires a homogenous heat and clean steels to begin with. The oxide layer known as scale has to be removed from the steels prior to stacking them in the billet. Care is taken to minimize deformation when welding. The used steels have a working range from 1050 - 850°C. At 1250°C those steels easily fall apart when too much pressure is applied.

D. Grain growth due to high temperature

When forging at high temperatures, the steel is in the austenitic phase. Grain growth occurs in the austenitic phase and depends on time and temperature, temperature being the most important factor [1]. The higher the temperature, the faster the grains grow and the bigger the grains become.

E. Grain refinement due to forging

The thickness of the billet is reduced in height by a factor 8. This reduction forces the grains to flatten to elongated grains. The heat in the steel initiates dynamic crystallization resulting in finer equiaxed crystals [2].

F. Samples

A small piece of the forged damascus knife has been cut off and has been separated in 8 equal pieces, 10x10x2,4mm (Fig. 4, appendix II). Care was taken to not overheat the small pieces during this operation. All the samples were marked by a unique symbol to identify them.

Table 1. Sample properties

Sample	Operation
1	1 normalizing cycle
2	2 normalizing cycle
3	3 normalizing cycle
4	5 normalizing cycle
5	7 normalizing cycle
6	No normalizing; as forged
A	O2 as produced by steel mill
B	75Ni8 as produced by steel mill

G. Furnace fixtures

A metal hook was fixed on the samples and a magnet on a stick was used to get the samples in and out the oven rapidly. The samples were placed on a steel rack inside the oven (Fig. 5,6,7, appendix II).

H. Refining grains by phase transformations

"For simplicity consider first a pearlitic steel. At room temperature there will be some average pearlite grain size. When this steel is heated above its Ac1 temperature austenite grains will begin to form. The new austenite grains start to form on the old pearlite grain boundaries as shown schematically in Fig. 1. After a short time all the old pearlite grains are replaced with a completely new set of austenite grains. The new austenite grains have their smallest size immediately after the pearlite is consumed, before significant grain growth occurs as the temperature rises and time proceeds. Following are two factors that enhance the formation of the smallest possible initial austenite grain size.

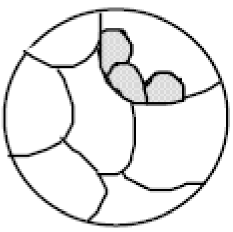


Fig. 1. Formation of new grains

- Faster heating rates cause the austenite grains to nucleate closer together and enhance small grain size.
- Smaller original pearlite grains produce smaller austenite grains.

When the austenite is cooled back down below the Ar1 temperature a whole new set of pearlite grains is formed and the same two factors: rate of transformation and size of the prior grains, control the size of the new grains. Hence, on simply heating and cooling through the transformation temperature three different sets of grains are involved. When dealing with hypoeutectoid steels the same ideas apply only now one must heat above the Ac3 temperature before 100 % austenite is formed."[3] This grain refining method is known as normalizing.

I. Normalizing by air quench

Normalizing can be done by varying methods. To obtain accurate temperature control during both heating and cooling of the samples, an electric heat treating furnace has been modified with an air quench system.

Copper tubes were fit on the bottom of the furnace and holes were drilled through the bricks, ending up in the middle of the oven. The copper tubes were connected to hoses of equal length, double "T-pieces" were used to connect the hoses to an air compressor. A valve was connected to regulate the air quench (Fig. 8, appendix II).

Special air diffusing nozzles were created to fit in the end of the air holes. The nozzles make sure the air does not flow in a straight line up. By using these nozzles the air is introduced to the furnace interior evenly (Fig. 9,10, appendix II). Additional holes were drilled in the top bricks of the furnace, to let the air out.

J. Furnace heating and cooling ability

The following table shows the maximum heating and cooling ability of the furnace and the isothermal values used for the phase transformations to complete.

Table 2. Heating and cooling ability of the oven

	°C	**Minutes**
Heating	550-810	4:00
Austenitizing soak	810	2:00
Cooling	810-550	2:45
Additional pearlite formation time	550	7:00

One normalizing cycle is graphed in the O2 CCT diagram. A 75Ni8 CCT diagram is not available.

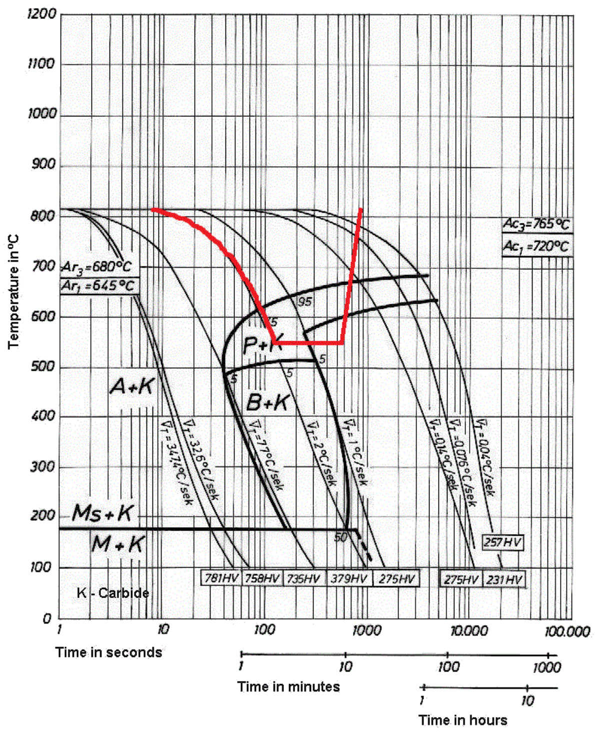


Fig. 2. The O2 CCT diagram with cooling and heating lines in red.

Each sample was air cooled from 550°C to 15°C after the last normalizing cycle resulting in a pearlitic structure.

K. Metallographic research

Each sample was embedded and ground to 2000 grit (Fig. 11, appendix II). After grinding on the flat disc's the samples were polished, first with 3 microns diamond polishing compound and later with 1 micron polishing compound. The samples were etched in 1% Nital for 4 seconds.

L. ASTM grain size number

To calculate the ASTM grain size number the following formula was used:

$$N = 2^{n-1}$$

The capital "N" is defined as the number of grains per square inch at 100x magnification. The lower case "n" is defined as the ASTM grain size number.

A square inch was too big for most of the photographs due to the inconsistency of the shapes of the damascus layers. Half a square inch was used instead. Some layers were too small-grained, therefore graincounting was sometimes done at higher magnificated pictures. A conversion factor was calculated for both the use of the half inch and for the magnifacation differences.

The surface 0,5in.^2 fits four times in the surface of 1in.^2 so the conversion factor 4 is used at 100x magnification. The factor 16 was used for the 200x magnification and the factor 100 was used for the 500x magnification.

To find the ASTM grain size number the logaritm has to be taken from both sides of the formula:

$$N = 2^{n-1}$$

giving us:

$$\log(N) = (n-1)\log(2)$$

<->

$$n = (\log(N) + \log(2))/\log(2)$$

Grain size number 0 is defined as a very coarse grain. Grain size number 15 is defined as ultra fine.

III. RESULTS

The "as forged" sample number 6 was compared to sample number 5 which had 7 normalizing cycles. On both samples, three counting's were performed on the O2 and three counting's were performed on the 75Ni8. In the photographs of the "as forged" sample, one of both steel's grains were considerably smaller than the other. O2 has vanadium alloys, so we assume this smaller grained and darker appearing layer is O2. From now on we will refer to the darker layers as O2.

As forged sample averages

O2:
Counting's: 28,5 + 33 + 34 = 95,5
Average: 95,5 / 3 = ~ **32**

75Ni8:
Counting's: 30 + 39 + 51 = 120
Average: 120 / 3 = **40**

7 normalizing cycles sample averages

O2:
Counting's: 41,5 + 56,5 + 66,5 = 164,5
Average: 164,5 / 3 = ~ **55**

75Ni8:
Counting's: 62 + 68 + 71,5 = 201,5
Average: 201,5 / 3 = ~ **67**

Table 3. Calculated ASTM grain size numbers

	ASTM GRAIN SIZE NUMBER	
	As forged	7 normalizing cycles
O2	12,5	9
75Ni8	10,5	9

IV. Discussion

The grain size after the forge welding and forging processes was better than expected. Both steels had a "very fine" relative grain size after the forging, which is considered a very good result in terms of edge retention.

Unfortunately the normalizing process used in this research had a contrary outcome. Instead of refining, the grains of both steels have grown to bigger sizes and equaled out evenly at ASTM grain size 9.

Earlier testing with a very coarse grained test specimen showed the normalizing method by air quench inside the heat treating oven had positive results on the specimen. The specimen went from an estimated ASTM grain size 1 to an estimated ASTM grain size 7.

The normalizing method by air quenching is lucrative, but has a maximum grain size that can be obtained at ASTM grain size 9. To get even finer grains, the steels have to be heated and cooled faster, which is not possible with this method. When heating up, it is critical to austenitize as short as possible and heating should be performed as fast as possible. In order to get more nucleation points for grains to grow at the cooling stage, the cooling has to be performed faster.

From the results it can be seen that the initially finer grained O2 levels out at ASTM grain size 9, and the initially coarser 75Ni8 levels out at the same final size. This phenomenon underpins my conclusion that ASTM grain size 9 is the finest that can be achieved with this method and that the method is working. From this point the growth initiated by time and temperature when austenitizing can't compensate the refinement caused by the phase transformations.

In order to get ultra fine grains I suggest the use of molten salt bath furnaces. A high and a low temperature salt bath for respectively austenitizing and quenching has to be used in order to heat and cool within seconds. Other benefits of salt baths are the absence of air which results in virtually no scaling, oxidation and decarburization.

V. ACKNOWLEDGEMENTS

I want to thank the following persons for greatly helping me.

- This document is awaiting approval. -

The metallographic procedures were done at the TU Delft University, Materials Science and Engineering (MSE) department, The Netherlands. Sander Van Asperen taught me how to perform the embedding, grinding, polishing, etching and microscopic procedures.

VI. REFERENCES

[1] John D. Verhoeven, Metallurgy of Steel for Bladesmiths & Others who Heat Treat and Forge Steel (2005) Iowa State University, 2005, pp.67–68.

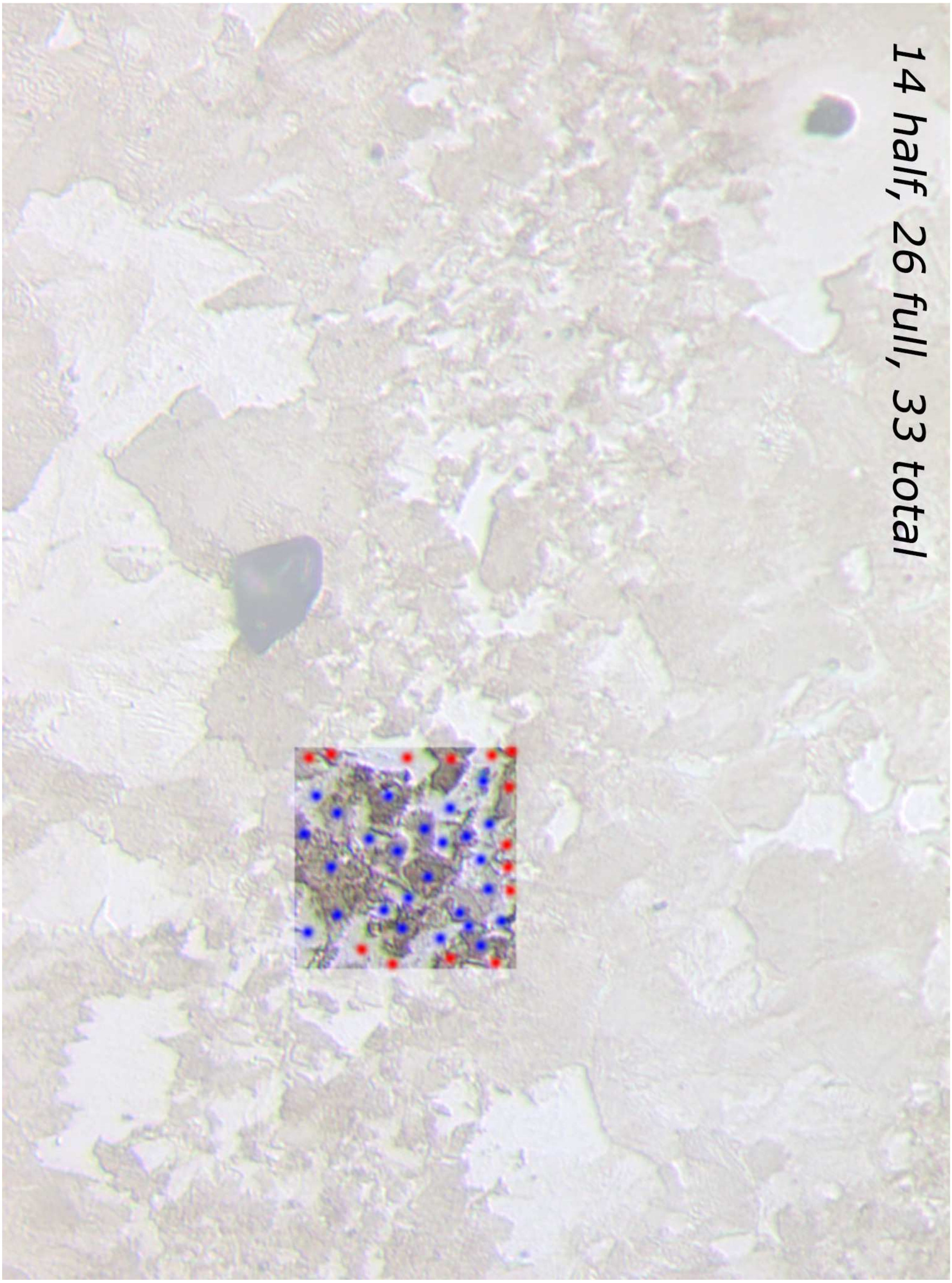
[2] John D. Verhoeven, Metallurgy of Steel for Bladesmiths & Others who Heat Treat and Forge Steel (2005) Iowa State University, 2005, pp.70–71.

[3] John D. Verhoeven, Metallurgy of Steel for Bladesmiths & Others who Heat Treat and Forge Steel (2005) Iowa State University, 2005, pp.68–69.

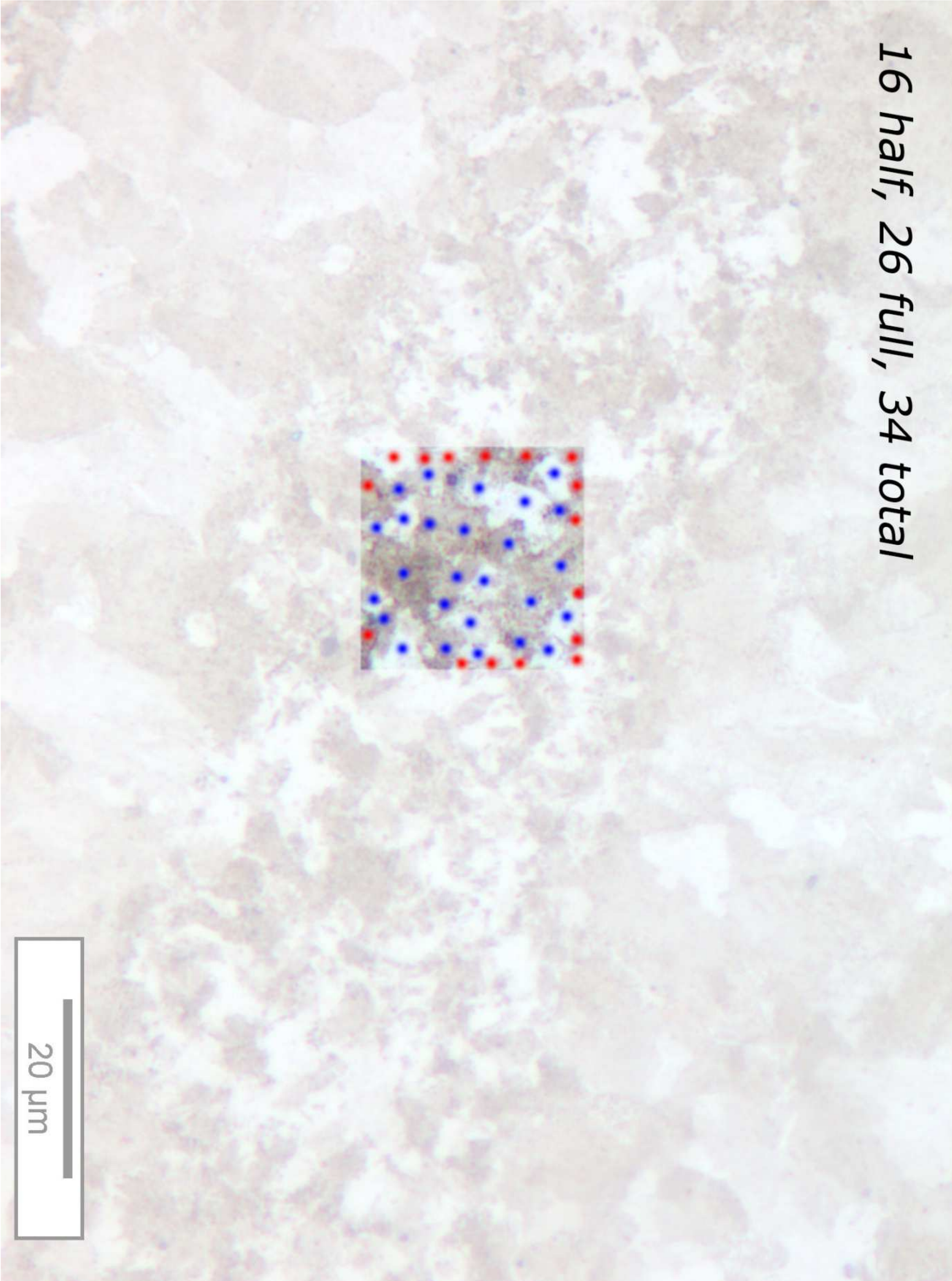
VII. APPENDICES

Appendix I

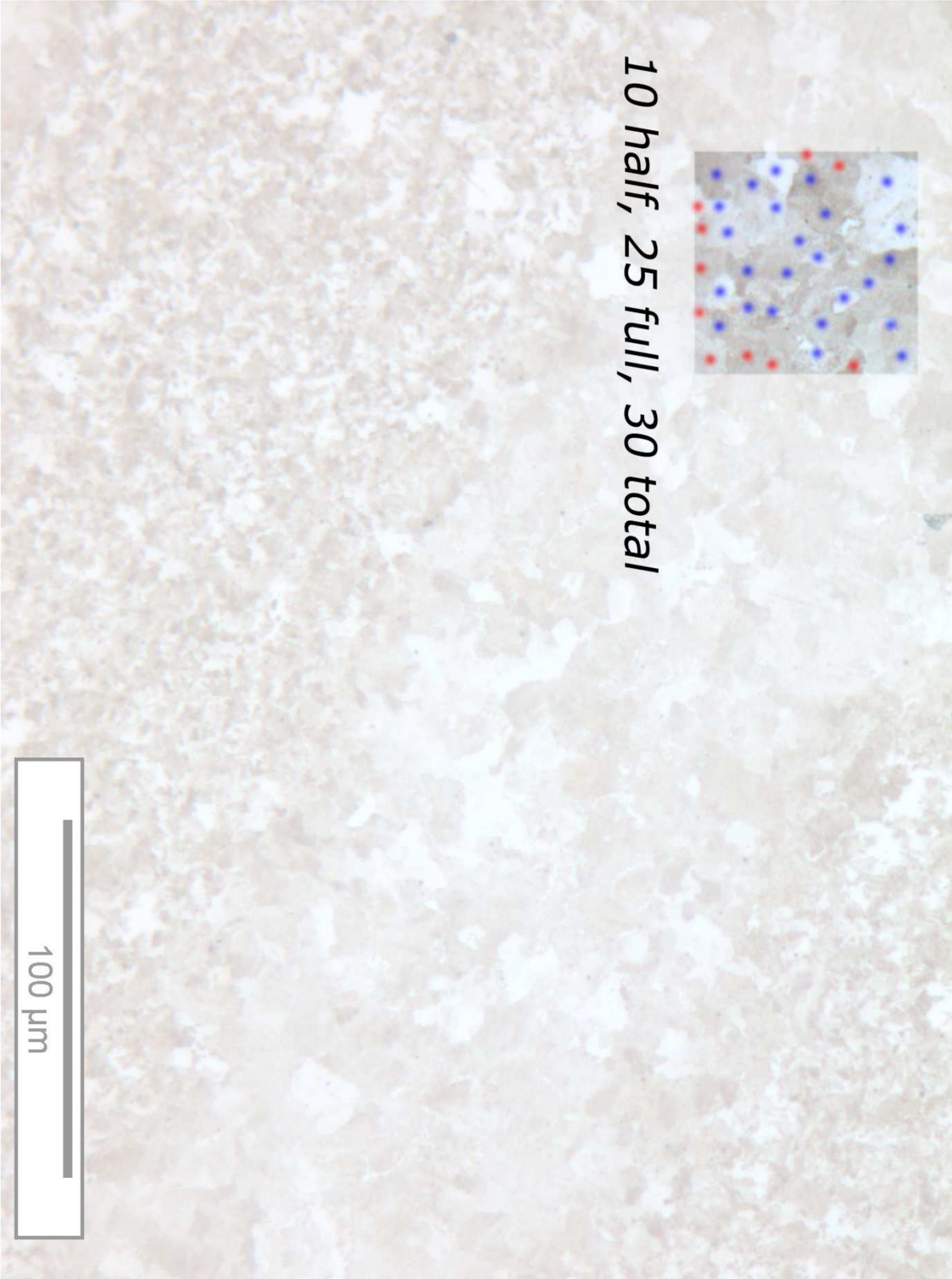
Steel	Condition	Area number	Area surface	Magnification	Conversion factor	Counted grains	ASTM g.s. number
O2	As forged	5a	0,5in.^2	500x	100	28,5	12,5



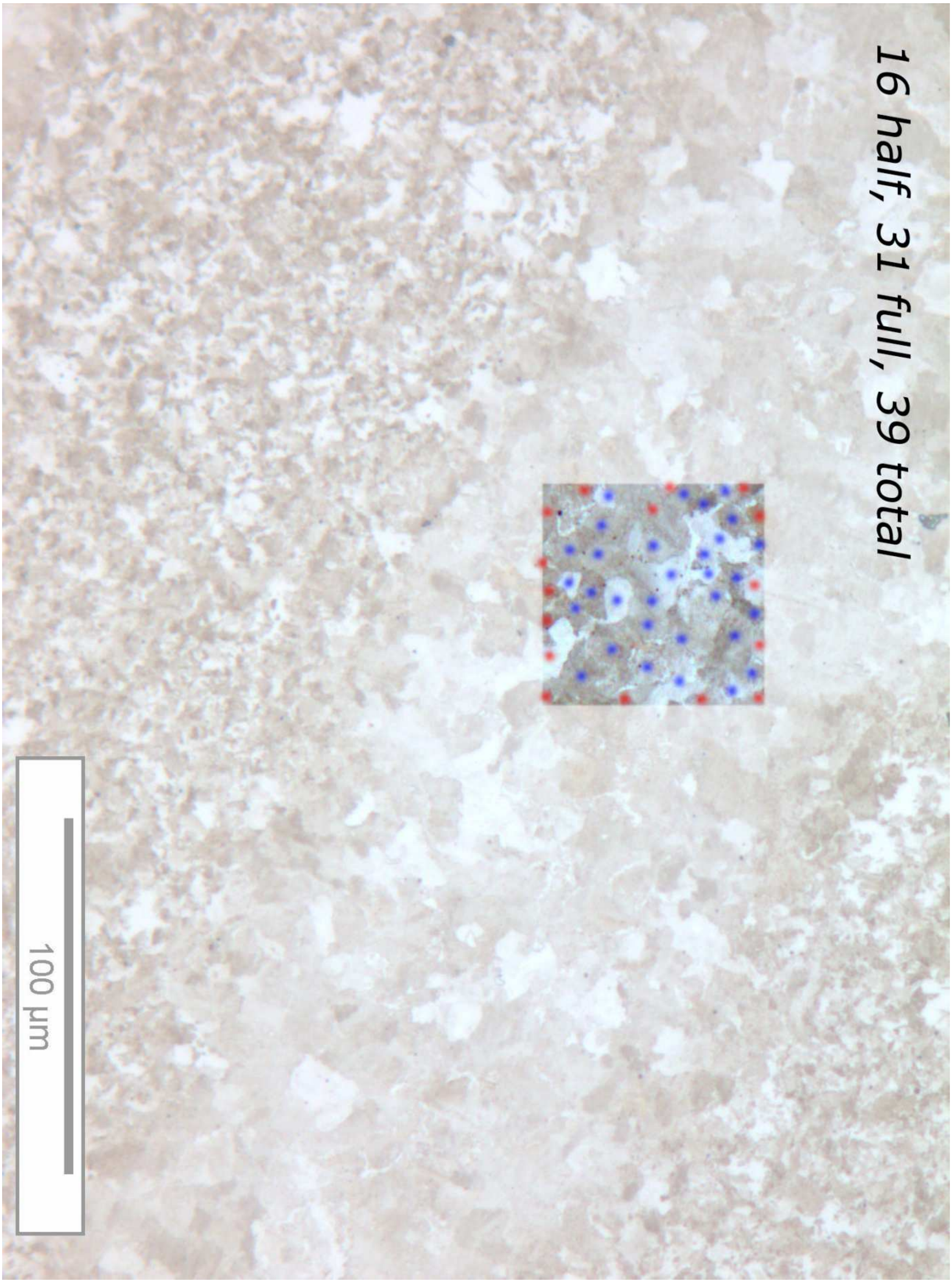
Steel	Condition	Area number	Area surface	Magnification	Conversion factor	Counted grains	ASTM g.s. number
O2	As forged	5b	0,5in.^2	500x	100	33	12,5



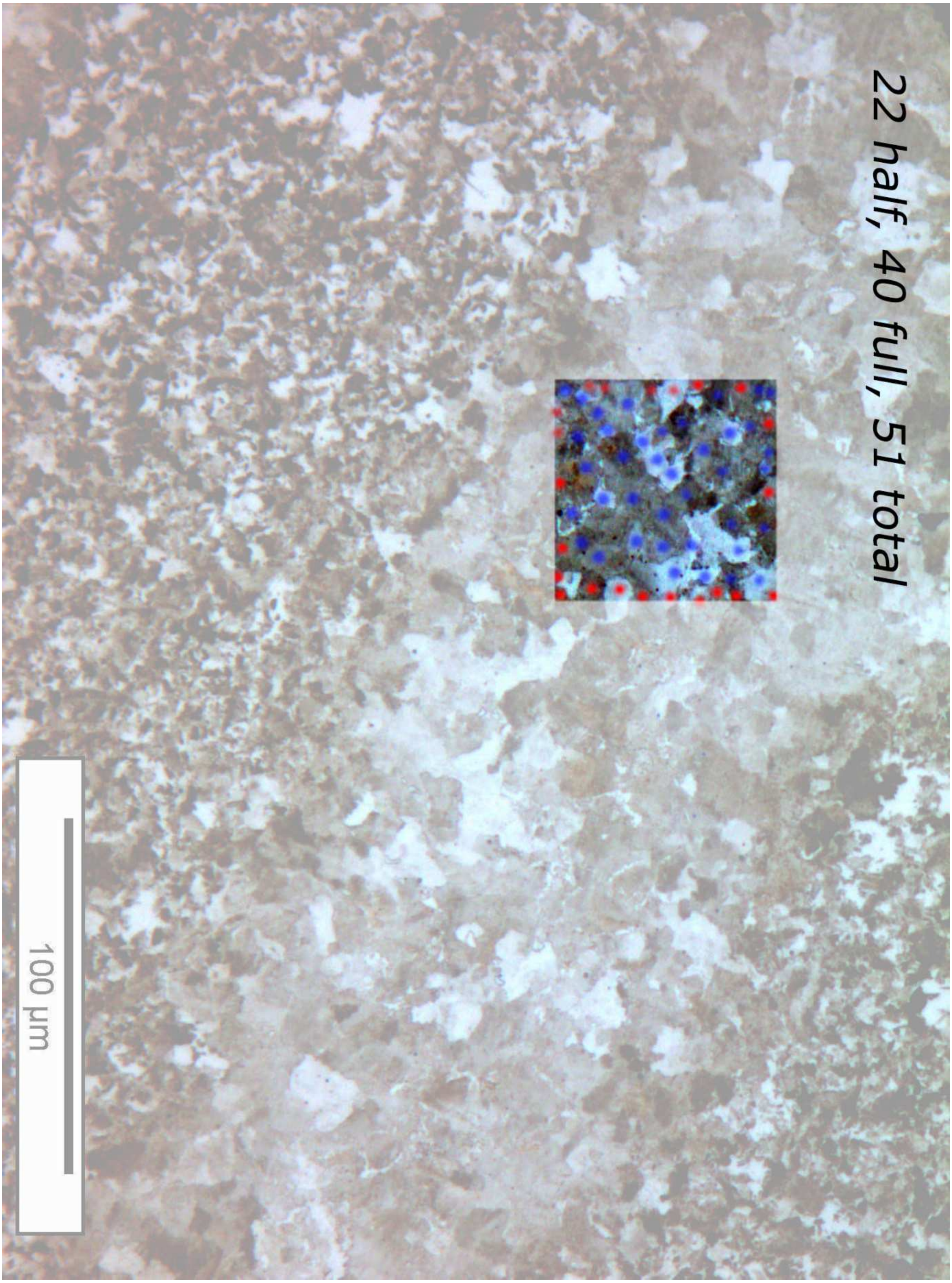
Steel	Condition	Area number	Area surface	Magnification	Conversion factor	Counted grains	ASTM g.s. number
O2	As forged	5c	0,5in.^2	500x	100	34	12,5



Steel	Condition	Area number	Area surface	Magnification	Conversion factor	Counted grains	ASTM g.s. number
75Ni8	As forged	5d	0,5in.^2	200x	16	30	10

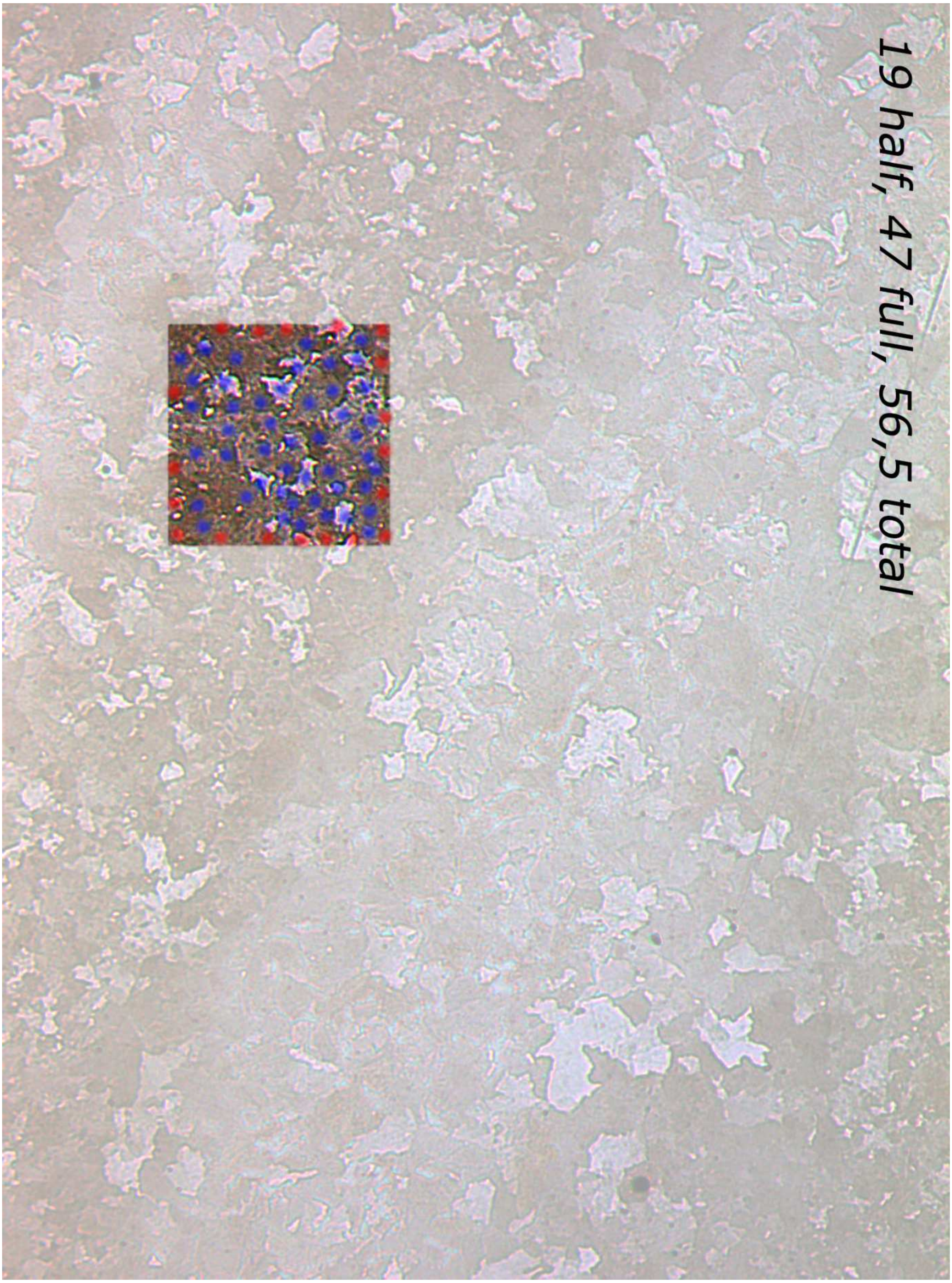


Steel	Condition	Area number	Area surface	Magnification	Conversion factor	Counted grains	ASTM g.s. number
75Ni8	As forged	5e	0,5in.^2	200x	16	39	10

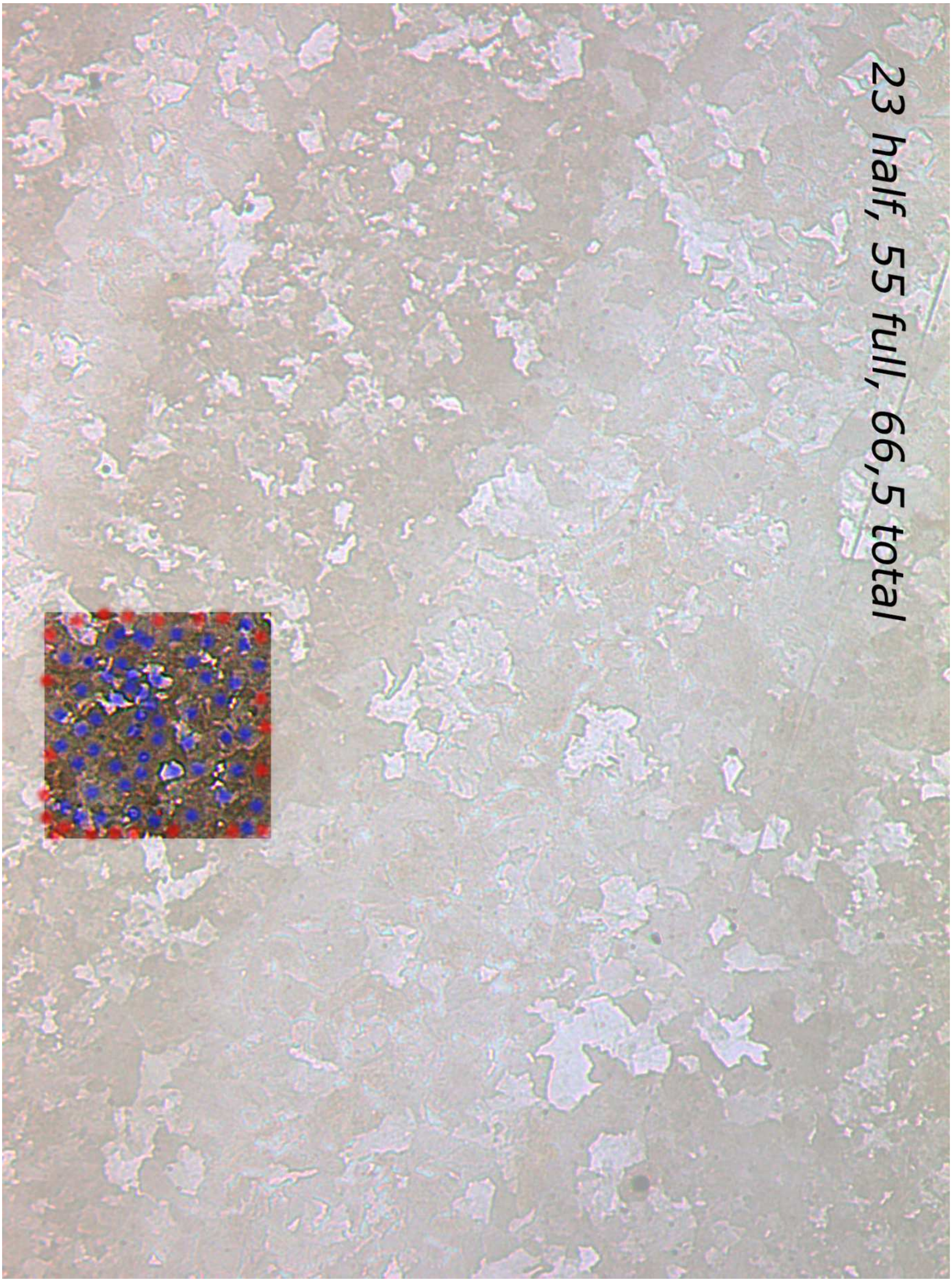


Steel	Condition	Area number	Area surface	Magnification	Conversion factor	Counted grains	ASTM g.s. number
75Ni8	As forged	5f	0,5in.^2	200x	16	51	10,5

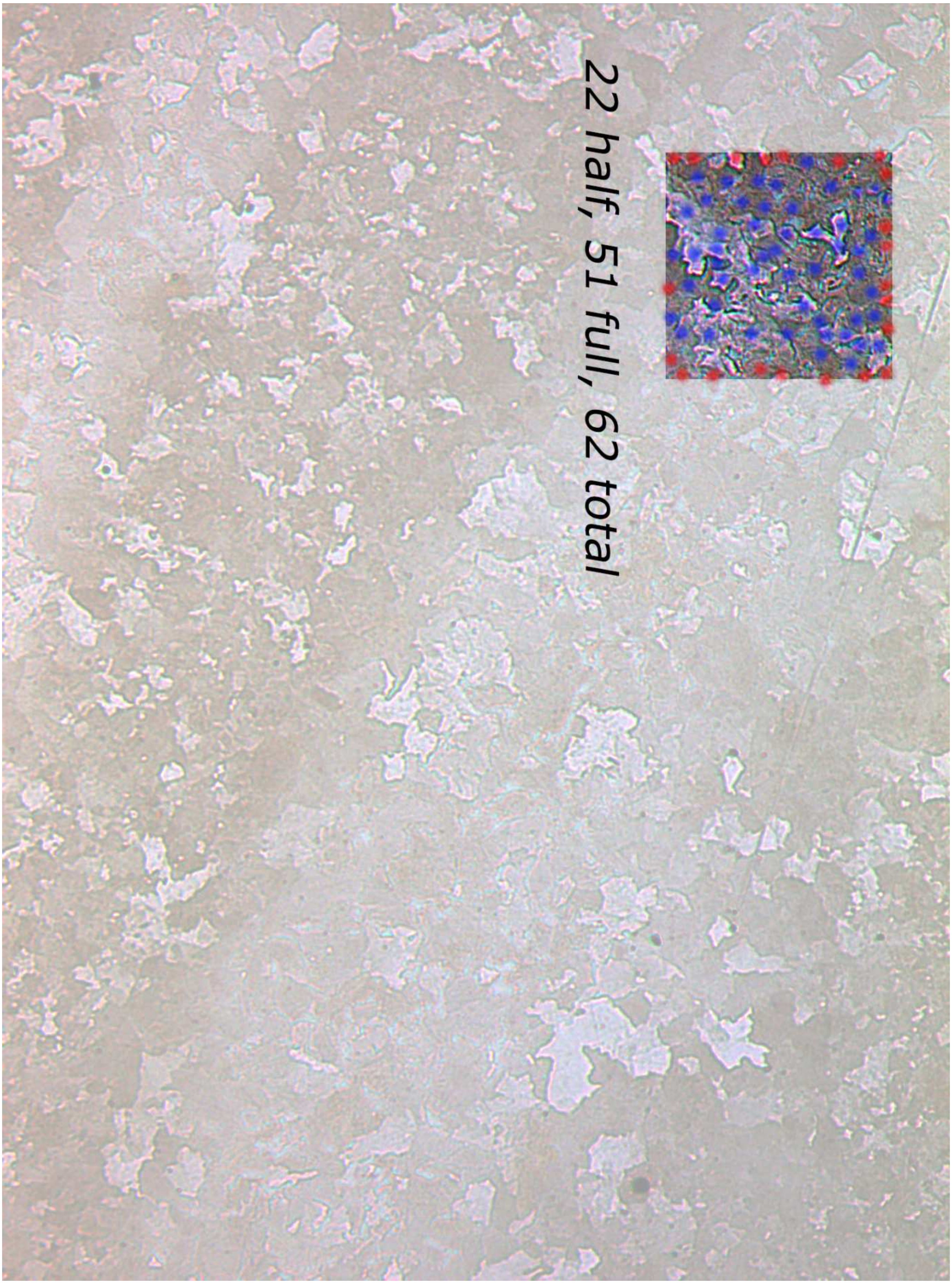
Steel	Condition	Area number	Area surface	Magnification	Conversion factor	Counted grains	ASTM g.s. number
O2	7 cycles	6a	0,5in.^2	100x	4	41,5	8,5



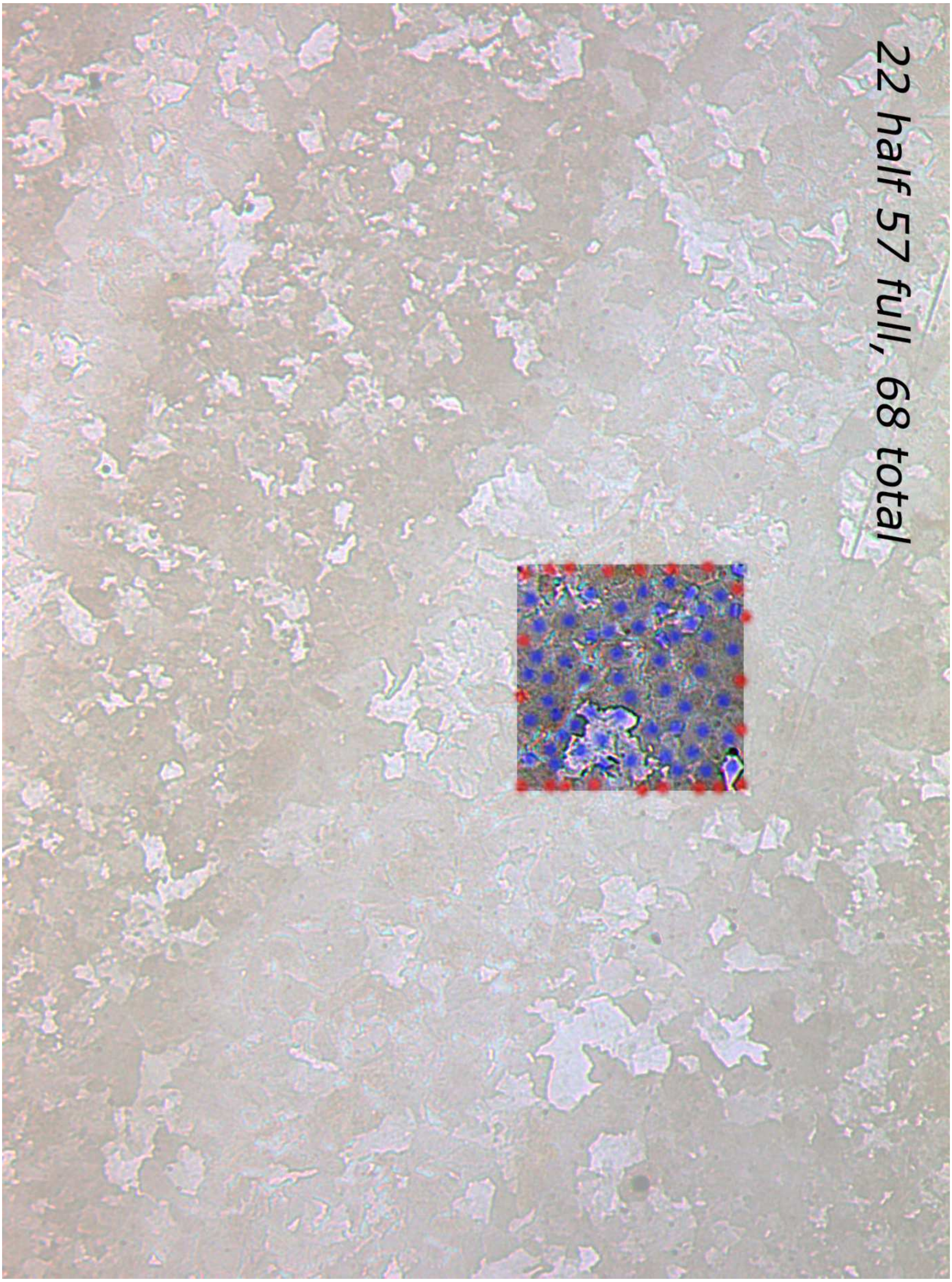
Steel	Condition	Area number	Area surface	Magnification	Conversion factor	Counted grains	ASTM g.s. number
O2	7 cycles	6b	0,5in.^2	100x	4	56,5	9



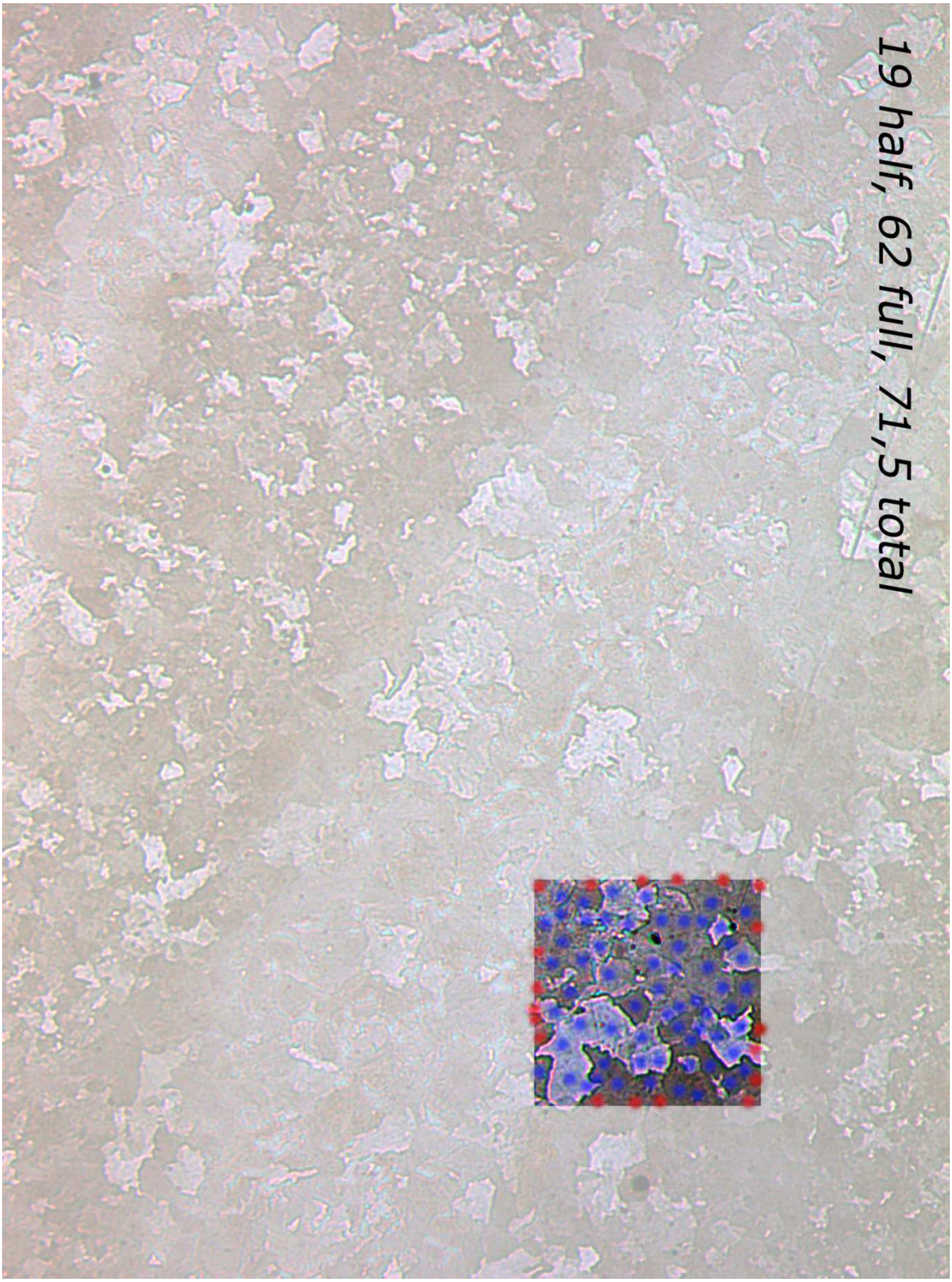
Steel	Condition	Area number	Area surface	Magnification	Conversion factor	Counted grains	ASTM g.s. number
O2	7 cycles	6c	0,5in.^2	100x	4	66,5	9



Steel	Condition	Area number	Area surface	Magnification	Conversion factor	Counted grains	ASTM g.s. number
75Ni8	7 cycles	6d	0,5in.^2	100x	4	62	9



Steel	Condition	Area number	Area surface	Magnification	Conversion factor	Counted grains	ASTM g.s. number
75Ni8	7 cycles	6e	0,5in.^2	100x	4	68	9



Steel	Condition	Area number	Area surface	Magnification	Conversion factor	Counted grains	ASTM g.s. number
75Ni8	7 cycles	6f	0,5in.^2	100x	4	71,5	9

Appendix II

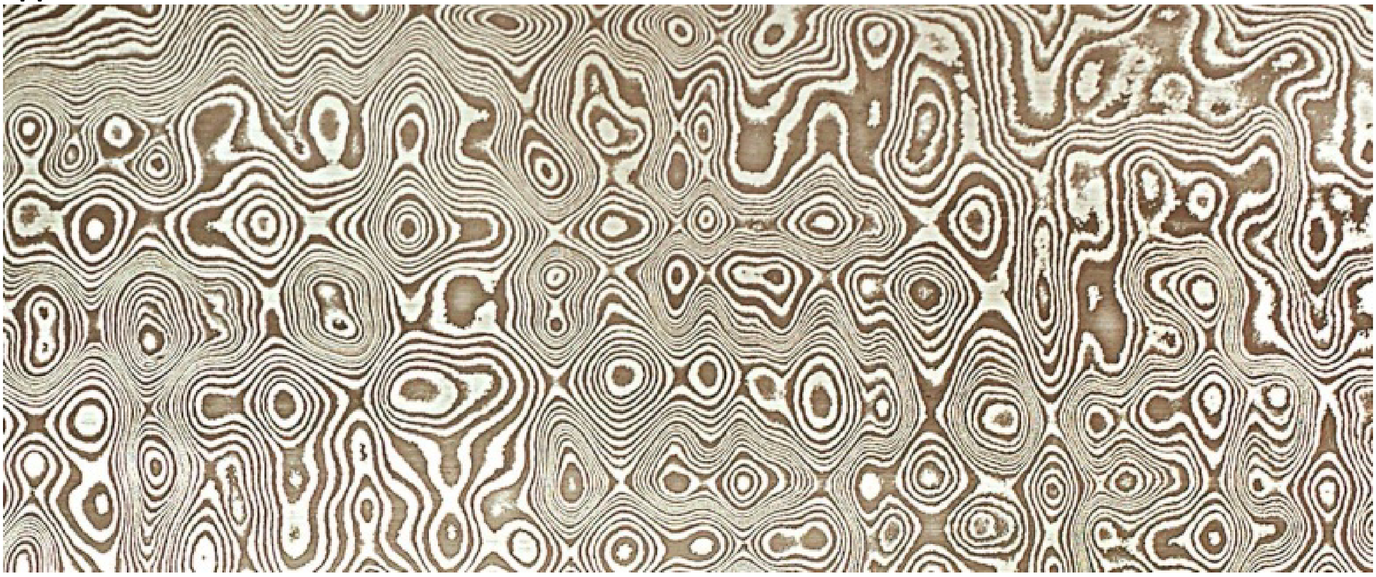


Fig. 1. Visual example of pattern welded damascus steel

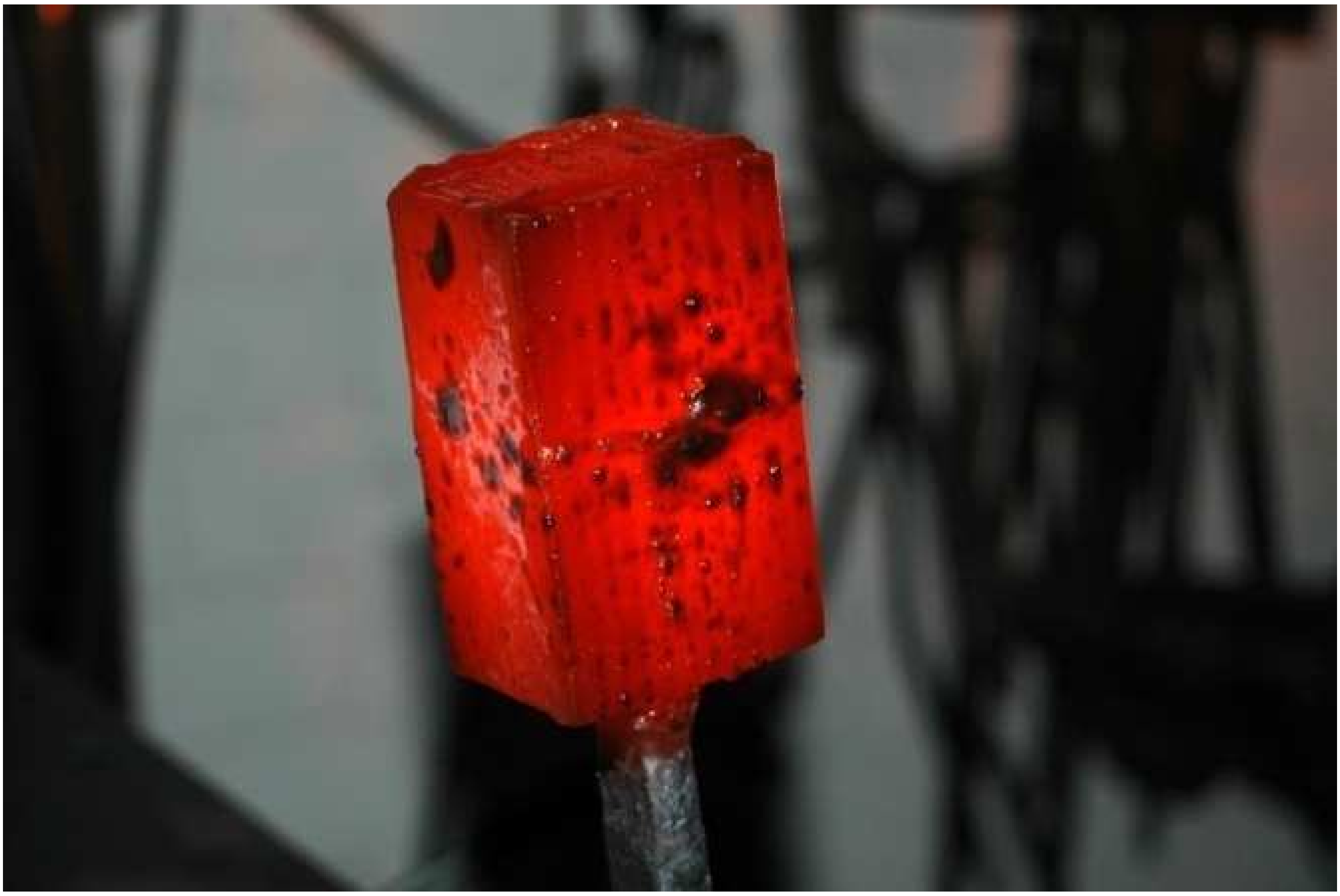


Fig. 2. The damascus billet


Fig. 3. The propane-butane fired forge at welding temperature


Fig. 4. The samples being cut on a bandsaw

Fig. 5. A sample with a metal hook placed on a magnet

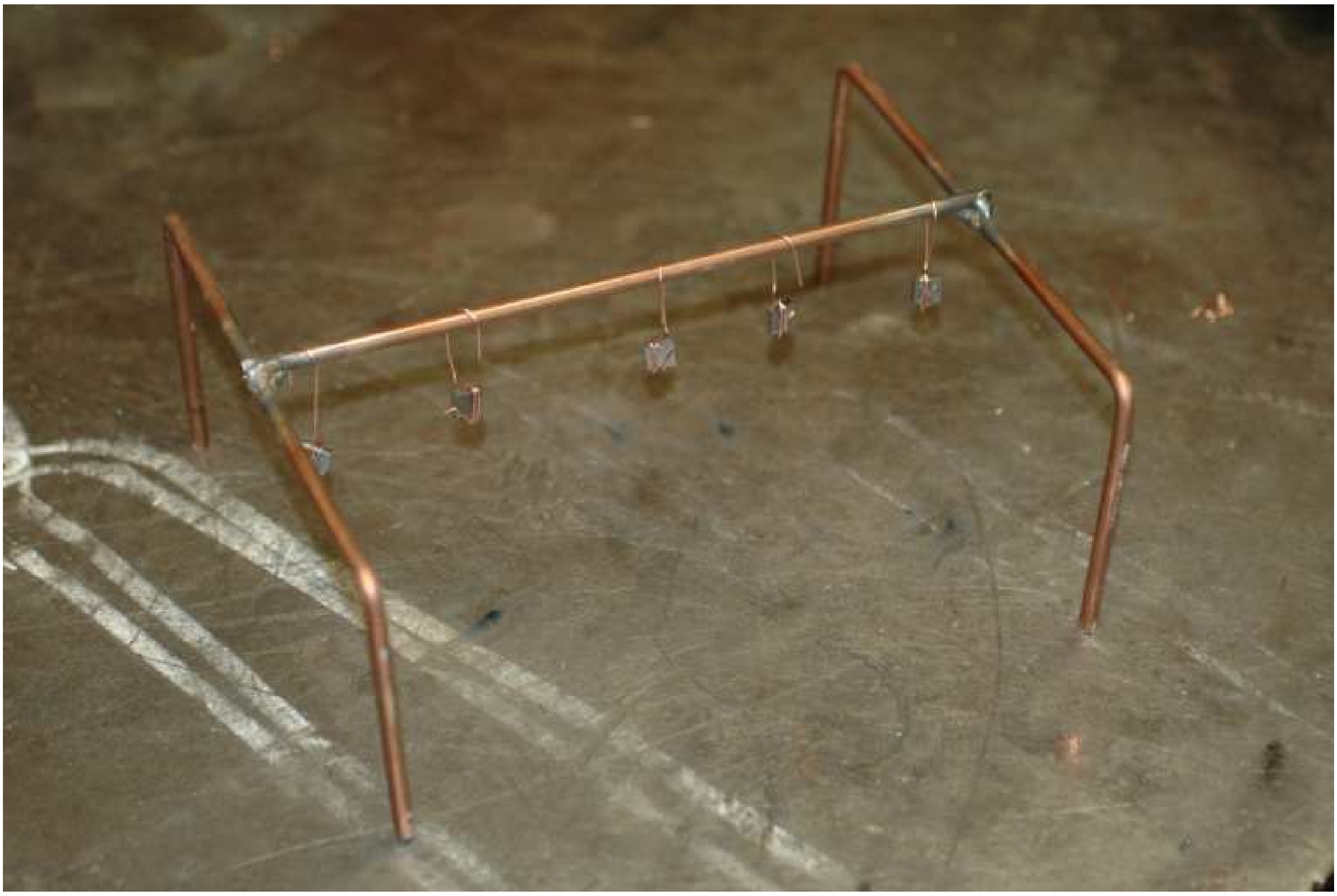


Fig. 6. The samples on a rack

Fig. 7. The rack inside the heat treating oven

Fig. 8. The heat treating oven with air tubing

Fig. 9. One of the air diffusing nozzles

Fig. 10. The nozzles inside the oven

Fig. 11. An embedded sample on a grinding disc.

Appendix III

Table 4. O2 and 75Ni8 composition.

Material number	In this research referred to as	Very similar to	% C	%Si	%Mn	%P	%S	%Cr	%Mo	%V	%Ni	
1.2842	O2		0,85	0,10	1,80			0,20	/	0,05	/	**min**
			0,95	0,40	2,20	0,03	0,03	0,50	/	0,20	/	**max**
1.5634	75Ni8	15N20	0,72	0,15	0,30					/	1,80	**min**
			0,78	0,35	0,50	0,025	0,025	0,15	0,10	/	2,10	**max**