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MICROSTRUCTURE, COMPOSITION, AND HARDNESS OF ROCKWELL C HARDNESS BLOCKS

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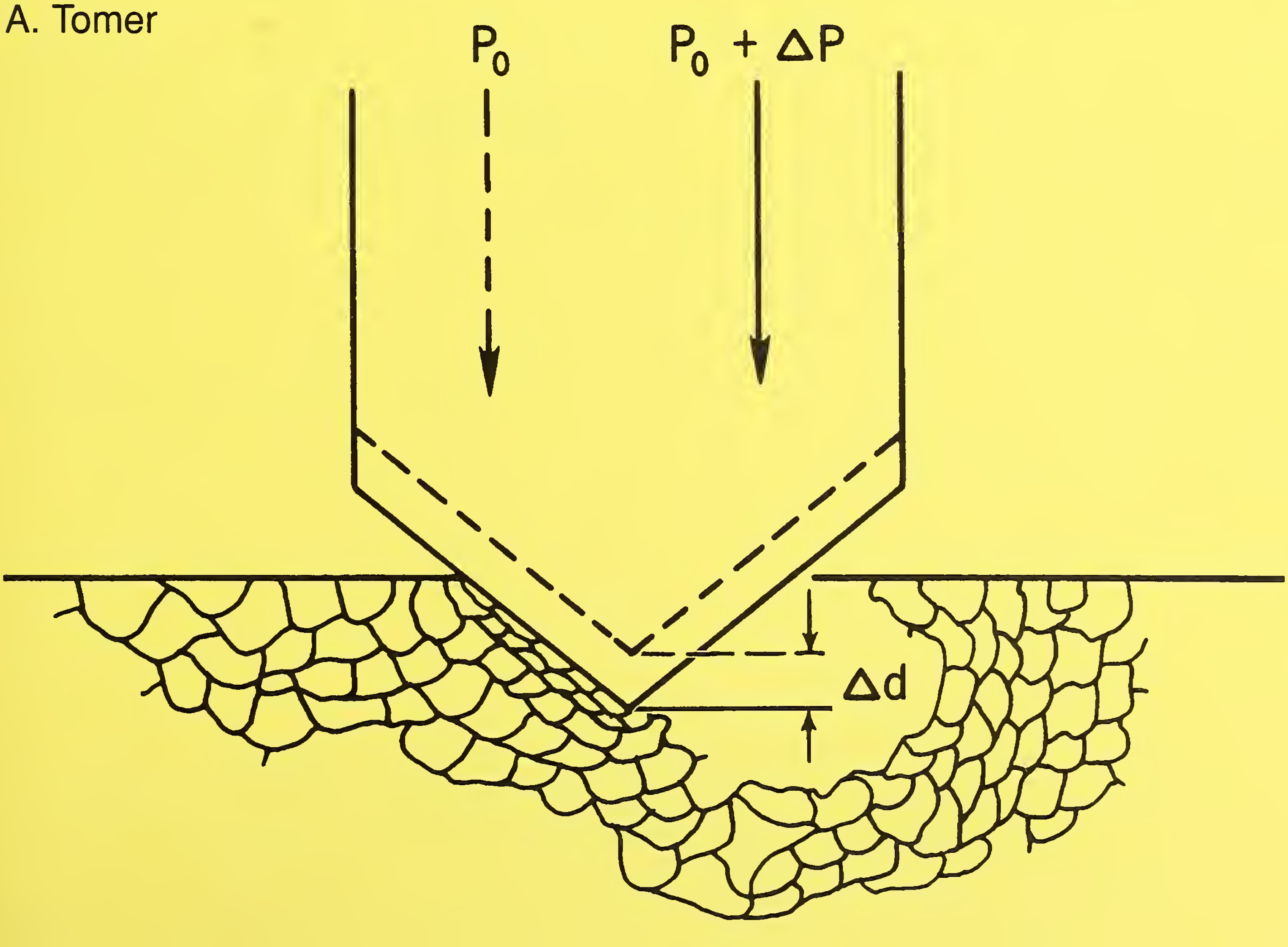

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MICROSTRUCTURE, COMPOSITION, AND HARDNESS OF ROCKWELL C HARDNESS BLOCKS

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CONTENTS

Microstructure, Composition, and Hardness of Rockwell C Hardness Blocks

A Report to the Office of Standard Reference Materials

by T. A. Siewert and A. Tomer

Materials Reliability Division

We examined the microstructure, composition, and hardness of hardness blocks (Rockwell C scale) that are commonly available in this country. Blocks near HRC levels of 25, 45, and 65 were obtained from each of six sources to represent the HRC measurement range. We found that the steels used in these blocks had surprisingly high levels of impurity elements, which are one source of scatter in the hardness values. Other than our concern for the steel purity, we conclude that most of these hardness block producers possess the technology necessary to manufacture a reference hardness block, or series of blocks, if it is found desirable for NIST to distribute a national hardness standard.

Key words: hardness; impurity elements; inclusions; indentation hardness; microstructure; Rockwell hardness

Background

Measurement of indentation hardness is a simple, yet widely used technique for characterizing the microstructure of a material. Although the technique is based on measuring the penetration of a loaded indentor into a material, the very small displacement requires a high level of sophistication from the equipment. When the highest accuracy is required from such an engineering test, many procedural variables must also be controlled. These variables can be within any of the three components in a hardness measurement system: hardness measurement device, indentor, and hardness block. The Metallurgy Division has been investigating the first two, and the Materials Reliability Division studied the third.

We obtained samples of the various Rockwell C scale (HRC) hardness blocks sold within the U.S. We examined their composition, microstructure, and hardness. From this catalog of data on these products, we developed an understanding of the technology in this industry. We were also able to estimate whether this technology would be able to meet the expected requirements for the production of a national standard hardness reference block.

Procedure

We found six sources for hardness reference blocks in the U.S and purchased hardness blocks from their inventory (without any special accuracy requirements or description of the program). From each source, we obtained three blocks, one each in the hardness ranges near 25, 45, and 65 HRC. These values span the HRC hardness range and match the ranges used by the Metallurgy Division in their evaluation of bias and reproducibility. They also fit the three hardness ranges specified in ASTM E 18-89a, Standard Test Method for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials, for the measurement of repeatability of hardness machines.

The evaluations were arranged in the order of increasing destructiveness. The first step in the evaluation was examination of the microstructure at the surface used for the hardness measurement. A common nital etch was suitable for etching all the blocks. We were able to view the microstructure at the measurement surface with virtually no effect on the hardness block. The etch was followed by optical emission spectroscopy to determine the composition. This technique involved establishing an electrical arc between an electrode and the surface, and determining the composition from the optical spectrum. Finally, we sectioned the blocks to examine a surface normal to the surface used for hardness measurement. Comparison of the microstructure and texture on the hardness measurement surface and second surface provided information on the steel processing procedures. It indicated the shape of the intermediate steel product from which the blocks were produced (for example, plate or round bar stock) and the orientation of the hardness measurement surface with respect to the rolling direction. This transverse section also permitted comparison of the microstructure at the center of the block with that at the surface.

For reference, we also took five readings of the block hardness, before etching the surface. The readings were taken in a line that went from one edge to the other through the center of the block, but the measurements were never closer than 3 mm to the edge. These measurements were made on our generic laboratory hardness machine (perhaps 20 years old), without the special calibrations performed by the Metallurgy Division in their testing of bias and reproducibility. However, we followed the measurement procedures specified in E 18 section 7 (that is, disregarding the first two measurements) and the machine verification procedures specified in E 18 sections 11 and 13 (that is, verification by the standardized test blocks furnished by the machine producer within the past 6 months). We were careful to take the hardness readings before using the other evaluation techniques. The data represent those which might be obtained by an average user on an average machine. We compared these data to the calibration data reported on the certificate shipped with the blocks, representing evaluation on the manufacturer's reference machine.

For the hardness readings on each block, we computed the mean and root mean square deviation. The standard deviation is a statistical measure of the spread in the data, based on the square of the difference between each value and the mean. Therefore, it is a single parameter that includes a weighting factor for the spread in the data. We also report the spread in the data (total range between maximum and minimum values), defined as the repeatability in E18. We consider this to be an inferior measure of the distribution of the measurements, since it does not distinguish between situations when all the values are at the extremes or only two values are at the extremes with all the remaining values clustered around the mean.

We did not use the same blocks as those used by the Metallurgy Division, because they wished to archive their specimens for future comparisons. Since we could not be certain that the compositions, heats of steel, or processing procedures had not changed during the interval between the two block procurements, we chose a different (numerical) system of identification.

Our Division has a program to monitor the production of Charpy V-notch reference specimens for the Office of Standard Reference Materials. These specimens are fabricated from high purity (vacuum induction melt - vacuum arc remelt) steel alloy grades. We included two of these, a 18.5Ni-3Mo-0.7Ti maraging steel and a 4340 low alloy steel, in our evaluation of the microstructure.

Results and Discussion

1. Composition

Tables 1 to 3 list the compositions for the 18 blocks which we examined, with each hardness range in a separate Table. Below each composition, we have identified the commercial alloy grade that appears to match the composition. Several of the blocks seemed to be made from unusual grades or had compositions that were at the extremes of the ranges. This is surprising in that most manufacturers would be expected to use a commonly available steel, but the requirements are only for hardness, and the manufacturers are free to use any material that they wish.

All the commercial blocks had surprisingly high levels of impurities. The P values ranged from 0.004 to 0.020 wt.% and the S values ranged from 0.005 to 0.026 wt.%. Current steelmaking technology can easily restrict the impurity levels to the lower half of this range and can reach below the lower end of these ranges, when very tight controls are imposed. For example, Aerospace Materials Specification 6414E (a 4340 alloy steel processed by vacuum remelting) restricts the P and S values to less than 0.015 wt.%, with the typical values being much lower. Seven of the blocks do not meet this purity criterion. During the bidding procedure for a heat of steel in the Charpy program, we found various steelmakers willing to accept an upper limit of 0.010 wt.% for these elements. A strict double vacuum melt procedure can limit these elements to still lower ranges. For example, the 4340 heat from our Charpy program had a P value of 0.006 wt.% and a S value of 0.0003 wt.%. Since these elements can form second-phase particles with substantially different hardnesses from that of the matrix, their presence should be minimized. We suggest that the quality of the hardness blocks could be improved substantially by stricter control of the steel.

Many manufacturers use the same composition, which raises the possibility that several of the manufacturers may have obtained their blocks from the same source (private labeling). All the steels fit within the category of tool steels and have the potential to reach a high hardness with common heat treatments.

2. Block Macroscopic Characteristics

Tables 1 to 3 list the hardness block form (shape) and orientation of the measurement surface with respect to the rolling direction. The blocks were round or square, with length/width dimensions between 45 and 60 mm and thicknesses between 8 and 14 mm.

In the Tables, we use RB to represent blocks whose internal structure indicated they were cut from a round bar and P to represent those cut from a plate. We use T to represent the situation when the block surface is transverse to the rolling direction and P to represent the situation when the block surface is parallel to the rolling direction and parallel to the original plate surface. Thus, P-P indicates a block that was cut from a plate with the measurement surface oriented parallel to the rolling direction. Four of the sources cut their blocks (for all three hardness ranges) from round bars, and two cut their blocks from plates.

A possible disadvantage of the T orientation is that the measurement surface includes the entire cross section of the original ingot, so any segregation within the ingot cross section could lead to variations in hardness. (Visible banding that indicates a nonuniform distribution of alloying elements in the ingot was observed in several of the structures.) A possible disadvantage of the P orientation is that MnS inclusions are oriented parallel to the surface. Surface preparation could end with a large MnS inclusion at, or just under, a substantial portion of the surface. If contacted by the indenter, this region would indicate an erroneous hardness reading. These disadvantages are of less importance as the quality of the steel is improved, since additional refinement will reduce the impurities, producing a more uniform structure.

3. Block Microstructure

Tables 1 to 3 also list the measurements of the characteristic dimension of microstructural features such as grain size, inclusion size (greatest dimension of elongated inclusions such as MnS), prior (austenite) grain size, and (final) grain size. Since the hardness measurement is a mechanical deformation which samples a volume of the steel microstructure, the best material would have the smallest dimensions for all these features. Small feature dimensions and a low percentage of different phases (such as inclusions) would result in a more uniform deformation during the indentation, and so a more uniform hardness measurement. A unit increase in the Rockwell hardness value corresponds to a 2μm decrease in the penetration of the indentor, a value that gives a perspective on the effect of the inclusion sizes listed in Tables 1 to 3.

Figures 1 to 3 show typical microstructures found in these blocks. Figure 1 shows the acicular structure (large length-to-width ratio) features found in the HRC hardness block from manufacturer 1. Figure 2 shows the spheroidal structure of the HRC block for Manufacturer 2. Figure 3 shows a more complex structure (combination of acicular and spheroidal structures) found in the O tool steel composition. The structure varied from location to location within a block, making it difficult to draw general conclusions from the structure.

Figures 4 and 5 show examples of the banding that appears when a microstructure with compositional variations is etched. Figure 4 shows light etching bands that are oriented with long Mns inclusions in the HRC 45 block from Manufacturer 1. These bands are the result of regions of difference composition that form during the solidification of the steel ingot, and are elongated into bands during the subsequent rolling processes. Figure 5 shows dark etching bands at the surface of the HRC 65 block of Manufacturer 4.

Figures 6 to 9 show examples of the various inclusions shapes found within the structures. Figure 6 shows a side view of a typical elongated MnS inclusion in the HRC 65 block from Manufacturer 4. Since such an inclusion has properties different than the steel matrix, a large inclusion can affect the measurement. Figure 7 shows a less elongated MnS inclusion and some blocky inclusions in the HRC 65 block from Manufacturer 1. Figure 8 shows circular MnS inclusions indicative of less reduction in the steel processing in the HRC 65 block from Manufacturer 3. Figure 9 shows a rectangular inclusion in the HRC 65 block from Manufacturer 2.

Some blocks have combinations of these features or other features. Figure 10 shows a combination of elongated inclusions and porosity in the HRC 65 block from Manufacturer 1.

Figure 11 shows the scale at the edge of the measurement surface of the HRC 25 block from Manufacturer 4.

4. Hardness Measurements

Tables 4 to 6 list the five hardness measurements taken on each block in our laboratory and the five measurements (the first five if more than five were listed) that were reported by the manufacturer.

In general, the NIST measurements had a wider spread (larger standard deviation and reproducibility) than the values reported by the block manufacturers. Much of this might be attributed to the age and low sophistication of the hardness machine used to make our measurements. However, this does not explain why some of our measurements were consistent, especially with the measurements of Manufacturers 2, 4, and 5. Our differences were greatest for the softer blocks (25 and 45 HRC) of Manufacturers 1, 3, and 6. One interesting observation is the measurements for the HRC 25 block from Manufacturer 6. We measured two low values in our linear trace across the specimen, each at a location about halfway between the center and the edge. The identical location of the two low values (as a distance from the edge) caused us to repeat the measurements on another line. Again, we measured much lower values (about 1.8 HRC lower) at these locations. The manufacturer's data were quite consistent, but this might be because they did not check these locations. We postulate that this change in hardness is real and is due to nonuniform distribution of alloying elements within the plate from which the block was cut. This finding supports the need for thorough quality control during block production and acceptance testing.

Conclusions

1. Many tool steel compositions can be processed into the hardness ranges suitable for hardness reference blocks.

2. The combination of the similarity of the block characteristics measured in this study and the narrow hardness range (precision) found in the Metallurgy Division study indicates that many sources have the processing technology necessary to produce primary reference hardness blocks.

3. There were relatively high levels of impurity elements (P and S that form undesirable second-phase particles like inclusions and precipitates) in all the commercial hardness blocks. An improvement in reproducibility of the hardness values is possible if cleaner steels are specified.

4. Combining the existing processing technology with cleaner steels should result in hardness reference blocks with greater precision than those available now. Such blocks should be suitable for use as national reference blocks.

5. A thorough test procedure must be established before the block consistency can be guaranteed.

Table 1. Composition Data (and Tentative Identification) for the Blocks near HRC 25

	[---------------Manufacturer---------------]					
Element	1	2	3	4	5	6
C	0.41	1.04	0.98	0.99	0.96	0.93
Mn	0.74	0.70	1.16	0.42	1.09	1.19
Si	0.28	0.58	0.25	0.12	0.30	0.24
P	0.008	0.018	0.008	0.013	0.019	0.004
S	0.010	0.026	0.010	0.012	0.010	0.018
Cr	0.85	4.73	0.48	0.09	0.50	0.56
Ni	1.84	0.16	0.10	0.04	0.07	0.13
Mo	0.25	5.65	1.16	0.02	0.18	0.09
Cu	0.03	0.11	0.13	0.00	0.14	0.13
Al	0.04	0.02	0.01	0.01	0.02	0.003
V	0.01	2.21	0.20	0.00	0.15	0.15
Co	0.02	0.30	0.01	0.00	0.02	0.02
W	0.00	8.22	0.05	0.00	0.50	0.041
Fe	Bal.	Bal.	Bal.	Bal.	Bal.	Bal.
ID	4340	M	O-1	1095	O-1	O-1
Product form	*RB-T	RB-T	RB-T	P-P	RB-T	P-P
Banding	no	no	no	no	no	no
MnS inclusion size (μm)	none	few	25	none	40	none
Prior grain size (μm)	40	10	20	40	10	20
Grain size (μm)	20 X 1	2.5	<2	17 X 2	<2	<2

* RB-T indicates round bar/transverse slice, P-P indicates plate/in plane

Table 2. Composition Data (and Tentative Identification) for the Blocks near HRC 45

	[	-------	Manufacturer	-------	]	
Element	1	2	3	4	5	6
C	0.42	1.03	0.91	0.40	0.93	0.96
Mn	0.85	0.65	1.19	0.91	1.51	1.18
Si	0.20	0.46	0.34	0.27	0.34	0.24
P	0.013	0.020	0.009	0.008	0.010	0.004
S	0.023	0.026	0.013	0.013	0.011	0.008
Cr	1.03	4.75	0.51	0.24	0.24	0.53
Ni	0.06	0.18	0.17	0.02	0.01	0.12
Mo	0.19	5.47	0.08	0.08	0.32	0.09
Cu	0.17	0.12	0.04	0.00	0.03	0.12
Al	0.01	0.01	0.00	0.00	0.02	0.003
V	0.00	2.25	0.07	0.00	0.04	0.16
Co	0.01	0.63	0.01	0.00	0.01	0.02
W	0.00	8.32	0.53	0.00	0.00	0.40
Fe	Bal.	Bal.	Bal.	Bal.	Bal.	Bal.
ID	4140	M	O-1	1040	O-2	O-1
Product form	*RB-T	RB-T	RB-T	P-P	RB-T	P-P
Banding	yes	no	no	no	no	no
MnS inclusion size (μm)	70	60	100	20	few	none
Prior grain size (μm)	25	25	30	50	40	10
Grain size (μm)	20 x 2	2	2x17	12	20	?

Table 3. Composition Data (and Tentative Identification) for the Blocks near HRC 65

Element	[---	---	Manufacturer ---	---	---	---]
	1	2	3	4	5	6
C	0.90	0.96	0.93	1.03	0.90	0.92
Mn	1.18	1.23	1.21	1.08	1.48	1.21
Si	0.19	0.23	0.26	0.32	0.33	0.33
P	0.017	0.011	0.009	0.009	0.004	0.004
S	0.022	0.017	0.005	0.009	0.009	0.009
Cr	0.55	0.74	0.47	0.86	0.28	0.62
Ni	0.09	0.20	0.16	0.06	0.06	0.22
Mo	0.22	0.12	0.09	0.07	0.16	0.11
Cu	0.12	0.06	0.13	0.03	0.04	0.11
Al	0.01	0.01	0.00	0.01	0.008	0.005
V	0.20	0.12	0.19	0.01	0.06	0.05
Co	0.01	0.02	0.01	0.01	0.01	0.02
W	0.48	0.52	0.55	0.88	0.01	0.44
Fe	Bal.	Bal.	Bal.	Bal.	Bal.	Bal.
ID	O-1	O-1	O-1	O-1	O-2	O-1
Product form	RB-T	RB-T	RB-T	P-P	RB-T	P-P
Banding	no	yes	no	yes	no	no
MnS inclusion size (μm)	200	70	500	15	>10	none
Prior grain size (μm)	10	10	10	15	10	10
Grain size (μm)	7	?	10	?	10	10

Table 4. Hardness Measurements on the Blocks near HRC 25

Manufacturer	1	2	3	4	5	6
Five Hardness Readings Made by NIST	28.0	26.0	23.8	23.8	25.0	24.2
	27.2	26.0	24.0	23.7	25.0	22.8
	27.5	26.0	24.4	23.8	24.8	24.5
	27.0	26.0	25.0	23.8	24.8	24.4
	27.4	26.0	24.4	23.5	25.0	22.5
Average	27.4	26.0	24.3	23.7	24.9	23.7
Standard Deviation	0.38	0.00	0.46	0.13	0.11	0.95
Reproducibility	1.0	0.0	1.2	0.3	0.2	2.0
Five Hardness Readings, Reported by Block Mfg.	28.0	26.2	24.5	24.8	25.6	24.6
	27.9	26.5	24.7	24.7	25.5	24.6
	27.8	26.7	24.4	24.7	25.7	24.6
	27.8	26.4	24.6	24.5	25.4	24.7
	27.9	26.7	24.4	24.7	25.6	24.9
Average	27.9	26.5	24.5	24.7	25.6	24.7
Standard Deviation	0.08	0.21	0.13	0.11	0.11	0.13
Reproducibility	0.2	0.5	0.3	0.3	0.3	0.3

Table 5. Hardness Measurements on the Blocks near HRC 45

Manufacturer	1	2	3	4	5	6
Five Hardness Readings Made by NIST	46.5	44.5	45.0	44.8	44.0	44.0
	46.3	44.4	43.8	45.0	44.0	43.8
	45.9	44.5	44.6	45.2	43.8	43.9
	46.2	44.2	44.6	45.0	43.5	43.8
	46.0	44.0	43.8	45.0	43.8	43.6
Average	46.2	44.3	44.4	45.0	43.8	43.8
Standard Deviation	0.24	0.22	0.54	0.14	0.20	0.15
Reproducibility	0.6	0.5	1.2	0.4	0.5	0.4
Five Hardness Readings, Reported by Block Mfg.	47.2	44.4	44.1	46.0	44.5	44.3
	47.2	45.2	44.0	46.0	44.7	44.4
	47.2	44.4	44.2	46.1	44.4	44.4
	46.9	44.8	44.1	46.0	44.4	44.4
	47.5	44.6	44.0	46.0	44.7	44.4
Average	47.2	44.7	44.1	46.0	44.5	44.4
Standard Deviation	0.21	0.33	0.08	0.04	0.15	0.04
Reproducibility	0.6	0.8	0.2	0.1	0.3	0.1

Table 6. Hardness Measurements on the Blocks near HRC 65

Manufacturer	1	2	3	4	5	6
Five Hardness Readings Made by NIST	62.5	64.0	65.0	63.2	64.8	64.2
	62.9	63.8	65.0	63.0	64.8	64.0
	62.8	63.8	65.0	63.2	65.0	64.2
	62.8	64.0	65.0	63.3	65.0	64.2
	62.8	64.0	65.0	63.0	65.0	64.2
Average	62.8	63.9	65.0	63.1	64.9	64.2
Standard Deviation	0.15	0.11	0.00	0.13	0.11	0.09
Reproducibility	0.4	0.2	0.0	0.3	0.2	0.2
Five Hardness Readings, Reported by Block Mfg.	62.3	63.9	64.4	63.7	65.1	64.0
	62.2	63.9	64.4	63.8	65.0	64.1
	62.2	63.7	64.3	63.8	65.1	64.0
	62.0	63.8	64.5	63.8	65.1	63.9
	62.2	63.7	64.4	63.8	65.0	64.1
Average	62.2	63.8	64.4	63.8	65.1	64.0
Standard Deviation	0.11	0.1	0.07	0.04	0.05	0.08
Reproducibility	0.3	0.2	0.2	0.1	0.1	0.2

Figure 1. Microstructure of HRC 45 block from manufacturer 1 showing acicular structure of a martensitic phase steel (large length to width ratio). 1000X

Figure 2. Microstructure of HRC 25 block from manufacturer 2 showing a spheroidal structure. 1000X

Figure 3. Microstructure of HRC 65 block from manufacturer 4 showing the more complex structure (acicular and spheroidal) of O tool steel grade. 1000X

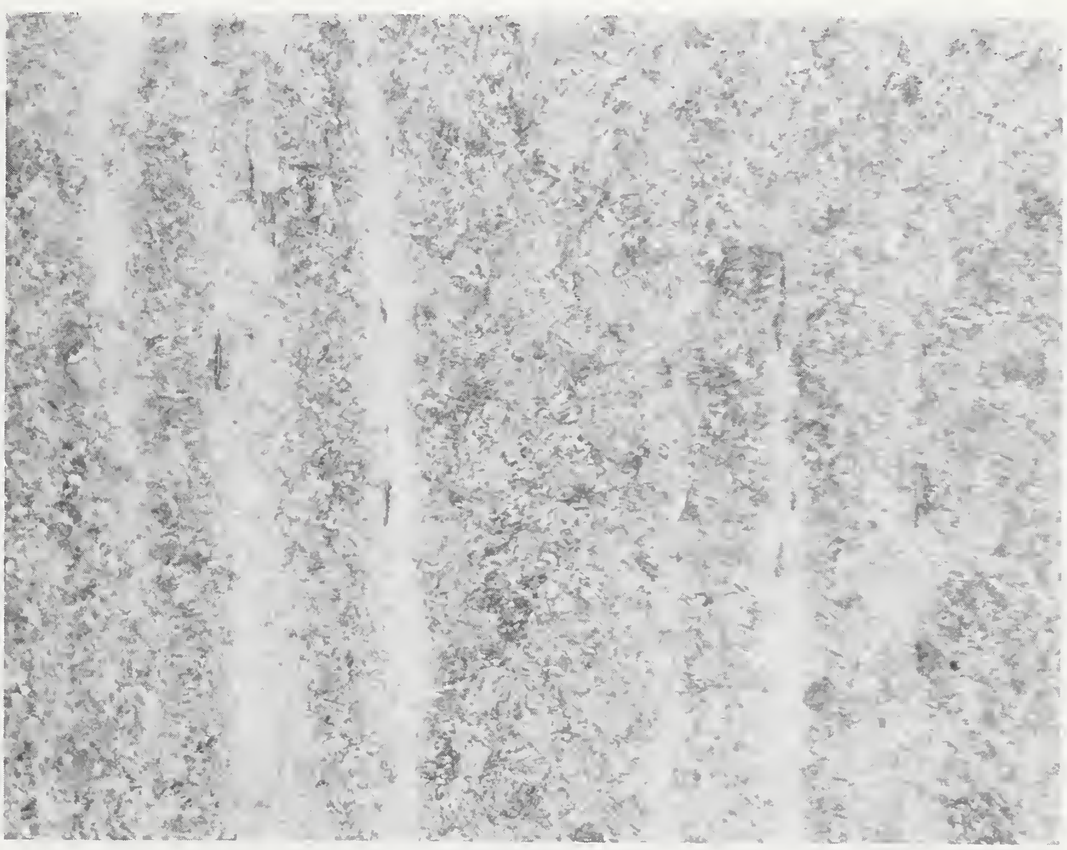

Figure 4. Light etching bands (with elongated inclusions) in the microstructure of the HRC 45 block from manufacturer 1. 200X

Figure 5. Dark etching bands at the surface of the HRC 65 block from manufacturer 4. 50X

Figure 6. Elongated MnS inclusion found in HRC 65 block from manufacturer 4. 1000X

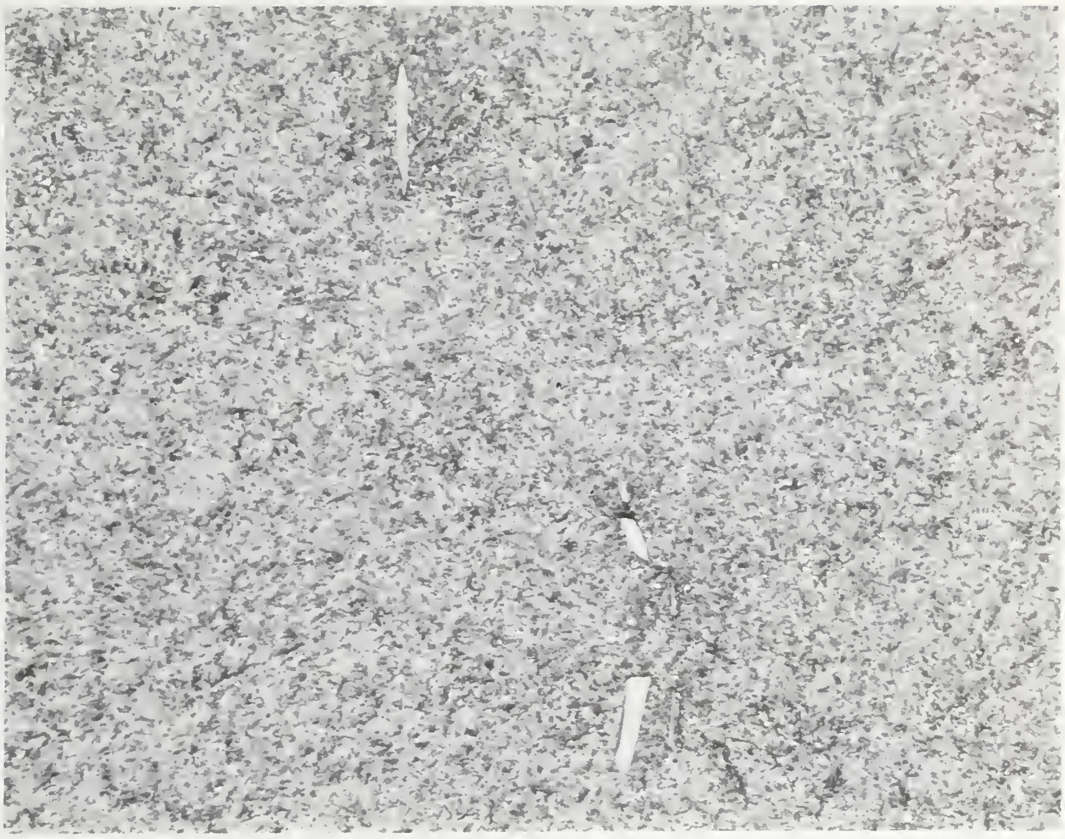

Figure 7. Slightly elongated MnS inclusion (near top) and block inclusions (near bottom) of HRC 65 block from manufacturer 1. 400X

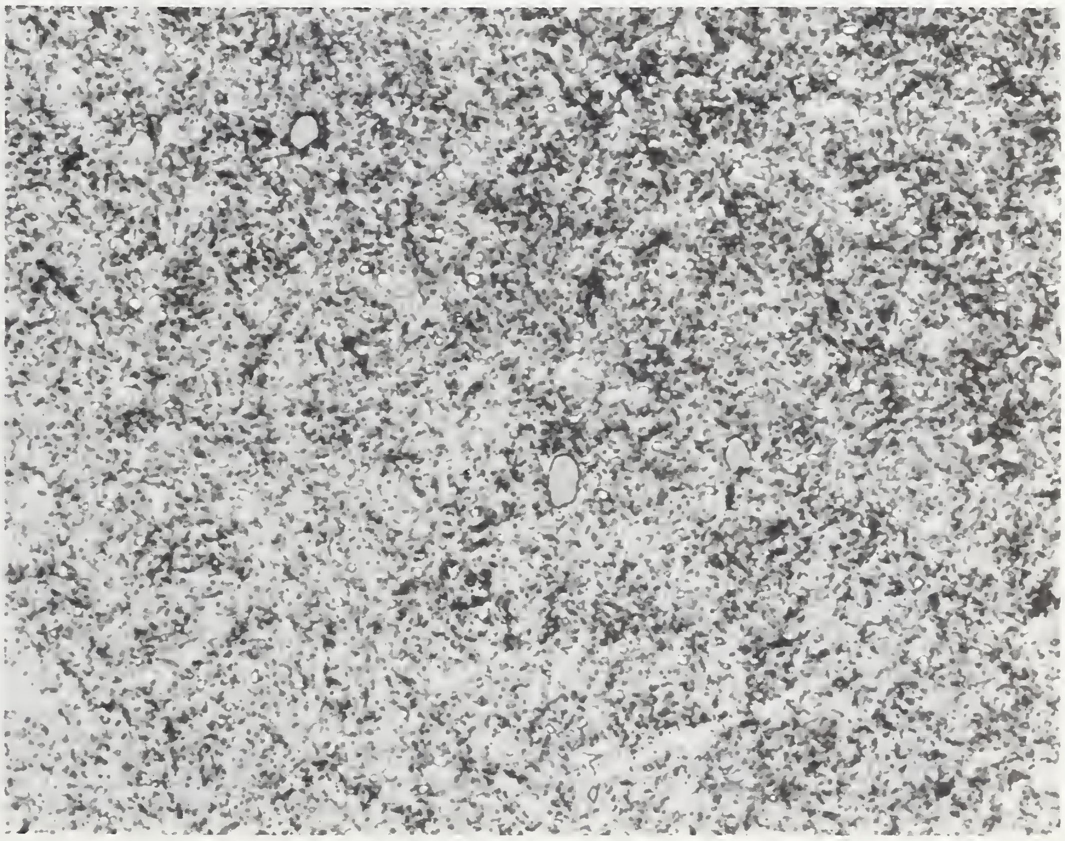

Figure 8. Circular inclusions in HRC 65 block from manufacturer 3. 1000X

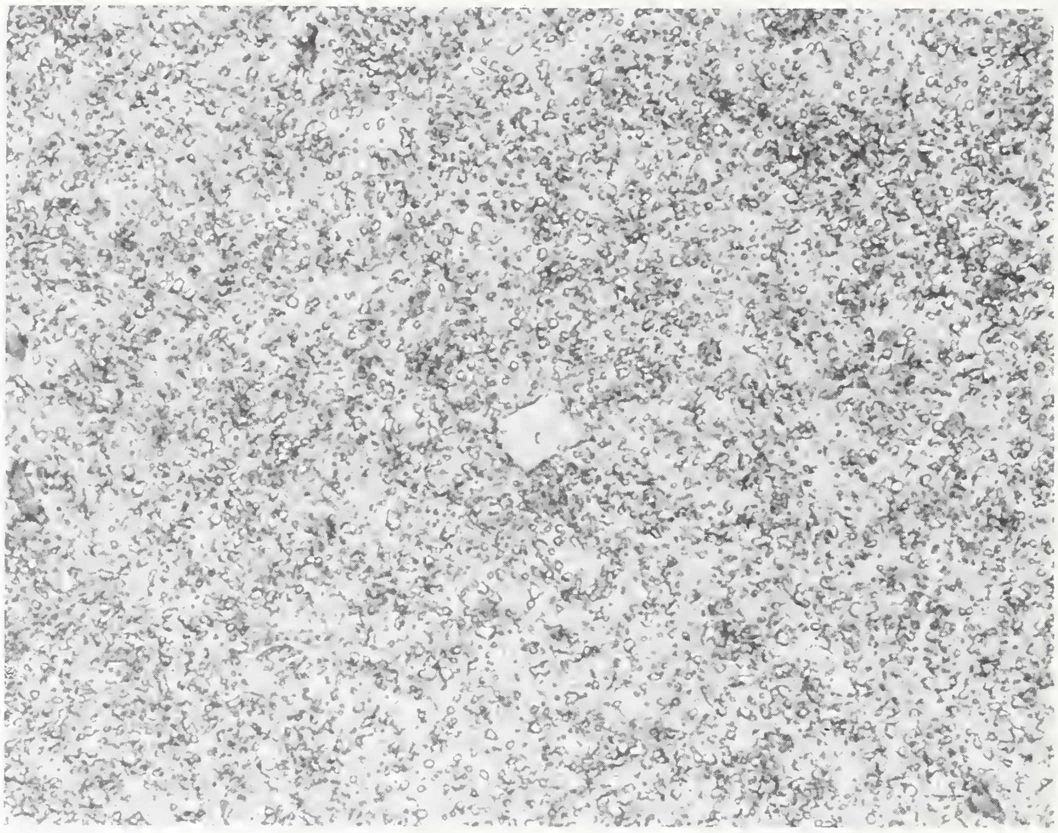

Figure 9. Rectangular inclusion in HRC 65 block from manufacturer 2. 1000X

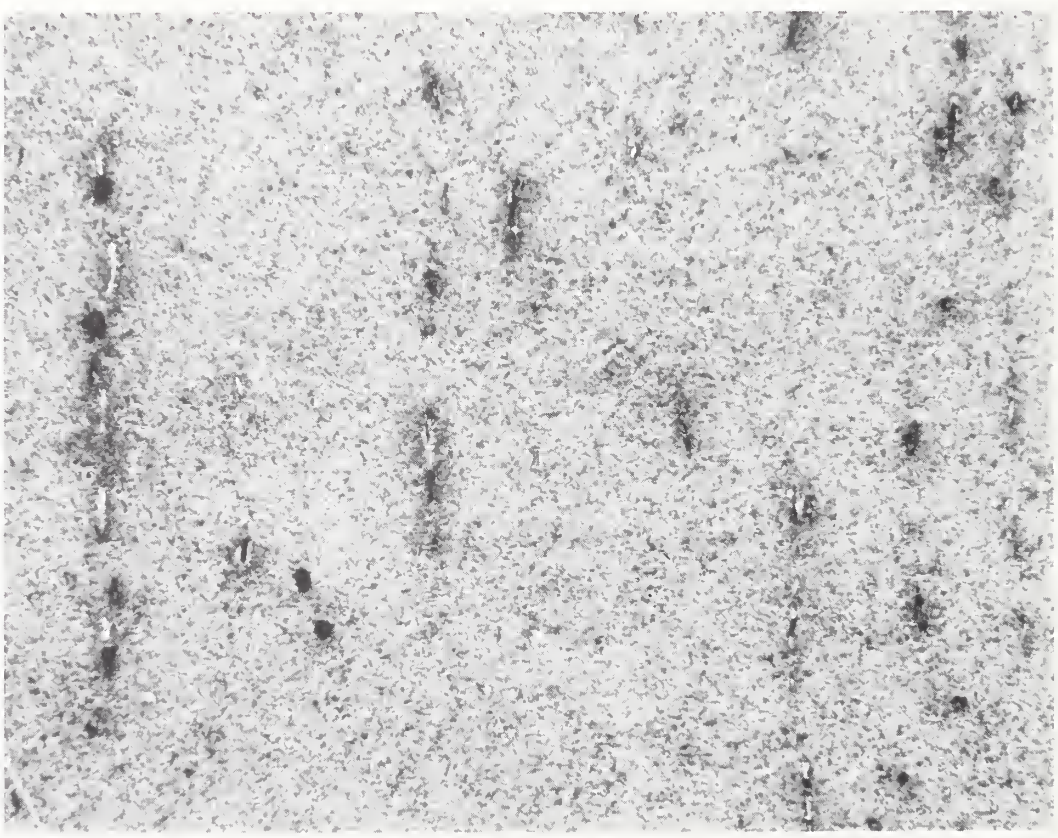

Figure 10. Elongated inclusions and porosity in HRC 65 block from manufacturer 1. 100X

Figure 11. Scale (oxide) at the edge of the measurement surface of the HRC 25 block from manufacturer 4. 1000X

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hardness; impurity elements; inclusions; indentation hardness; microstructure; Rockwell hardness

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