FILE COPY

file

O T T A W A July 19th, 1943.

R E P O R T

of the

ORE DRESSING AND METALLURGICAL LABORATORIES.

Investigation No. 1442.

Examination of Samples from Airscrew Retaining Mechanism Thrust Washers.

(Copy No. 10.)

Bureau of Mines
Division of Metallic
Minerals
-
Ore Dressing
and Metallurgical
Laboratories

CANADA

DEPARTMENT
OF
MINES AND RESOURCES

Mines and Geology Branch

O T T A W A July 19th, 1943.

R E P O R T

of the

ORE DRESSING AND METALLURGICAL LABORATORIES.

Investigation No. 1442.

Examination of Samples from Airscrew Retaining
Mechanism Thrust Washers.

Origin of Request and Object of Investigation:

On June 14th, 1943, thirteen samples of fully heat-treated chromium-molybdenum-tungsten-vanadium steels were received from the Canadian Propellers Limited, 5400 Hochelaga Street, Montreal, Quebec, for examination. An accompanying letter, signed by G. E. Green, Chief Inspector, and subsequent letters of June 22nd and July 8th, stated that the samples were from two heats of steel, gave details on heat treatments used, and described the members from which the samples were taken. It was stated that the latter were thrust washers installed in an airscrew retaining device. These washers, formerly made in the U. S. A., were now being

(Origin of Request and Object of Investigation, cont'd) -

obtained in Canada.

The previous American supplier had examined the Canadian-made product and reported that (1) the material had been heat-treated and forged so as to develop a carbide network in the annealed state and a weak and brittle structure with very large grain in the hardened state; (2) the material fractured coarsely; (3) hardness tests revealed soft spots; and (4) Magnaflux tests showed heavy patterns, indicating the presence of retained austenite. The U. S. A. manufacturer attributed these alleged defects to poor forging and heat-treating practice.

A microscopic examination was requested in order to check on the above criticisms and to determine the metallurgical quality of the steel.

Nature of Samples Received:

All samples received were quite small, so it was impossible to conduct a full physical examination. The samples were numbered 1 to 13. Samples 1 to 10 were from the same batch heat. Samples 11 to 13 were from another.

Specification on Chemical Composition:

The chemical analysis specified for this steel was as follows:

		Per cent
Carbon	-	0.50-0.60
Manganese	-	0.20-0.40
Silicon	-	0.15-0.45
Chromium	-	7.00-8.00
Molybdenum	-	2.50-3.25
Tungsten	-	1.30-1.80
Sulphur	-	0.025 max.
Phosphorus	-	0.025 "
Vanadium	-	0.20 "
Copper	-	0.20 "

Dendritic Structure:

A deep etch on the "as received" material showed that the ingotism of the cast steel was not completely eliminated by forging.

Fracture Tests:

The "as received" specimens were notched and then fractured. Marked differences in the appearance of fractures of different samples were observed. Samples 1 and 2 showed excellent glassy fractures. Samples 3 and 12 exhibited good fractures. The other samples fractured badly, Samples 5 to 9 inclusive showing particularly poor breaks. A comparison between the fractures of Samples 2 and 9 is given in Figure 1.

Although the small size of the samples prohibited actual measurement of the impact strength, a rough estimate made from the severeness of the blow needed to fracture the different materials showed that the samples exhibiting poor fractures were more brittle than those which broke with fine silky fractures.

Retained Austenite:

A hardness survey on the material as received was conducted by testing etched specimens in a Vickers machine; it was possible to see that the hardness variations were, in most cases, associated either with the non-uniform distribution of carbides or with the dendritic condition of the steel. The readings obtained varied from 695 to 775 V.H.N., the average being 730 V.H.N.

X-ray diffraction patterns, using the Debye-Scherrer method, were obtained from the material in the following conditions:

1) As received,
2) As quenched from 2000° F., and
3) As quenched from 2200° F.

The specimens were cut to a sharp angle and electro-polished. The

(Retained Austenite, cont'd) -

sharp-angled corner was exposed to the X-ray beam and a complete half-pattern was obtained.

No γ-iron lines were obtained on the spectrograms from the specimens as received or from the specimen as quenched from 2000° F. The spectrogram obtained from the steel quenched from 2200° F. revealed the presence of the α and γ lattices, both phases being coarse-grained. These results are in agreement with the findings of the microscopic examination of the material.

Cold-temperature treatments were also given to the same set of samples, as this treatment is known to produce a breakdown of the unstable austenite. A temperature of -95° F. was obtained by an immersion in a solution of dry ice in alcohol. Hardness before and after immersion showed a difference only in the case of the sample quenched from 2200° F.; in this material the hardness jumped from 56 to 59 Rockwell "C". This points to a breakdown of austenite and supports the results of the X-ray analysis, which indicated retained austenite in samples quenched from too high a temperature.

<u>Microscopic Examination of the "As Received" Samples:</u>

In the unetched condition the steel looked clean.

The grain size and the carbide patterns of all samples were checked under the microscope. The grain size, as revealed by the carbide envelopes, is shown herein in photomicrographs taken at X200 magnification. This fact makes it necessary to divide the size of the grains by 4 in order to have a comparison with the standard test. The samples were hand-polished and then etched in a 20 per cent nital solution. Photomicrographs of all the samples were taken at magnifications of X200 and X1,000.

The samples can be divided into the following three

(Microscopic Examination of the "As Received Samples, cont'd) -

groups, according to the presence of carbide envelopes:

<u>Group 1</u>: Structures believed satisfactory (including Samples 1 and 2).
<u>Group 2</u>: Structures believed probably satisfactory (including Samples 3 and 12).
<u>Group 3</u>: Structures believed definitely bad (including the remaining specimens).

Reproduction of two photomicrographs of each of these three groups is given in Figures 2 to 7. The representative samples of the three groups are, respectively, Nos. 1, 12, and 9. Within a same group, the structures of all samples are quite similar.

One sample (Sample 1) was given a $K_3Fe(CN)_6$ + KOH etch in the hope that the carbide distribution might be better developed. However, it is believed that the carbide pattern is shown quite as well with the nital etch (see Figure 12).

Microstructures shown in Figures 2 to 7 are all finely troosto-martensitic, containing free embedded carbide. Carbide envelopes were found in the nine samples of the third group. In the first and second groups these carbide envelopes can be detected only by a close examination.

<u>Microscopic Examination of the Heat-Treated Samples</u>:

Mr. G. E. Green, the chief inspector of Canadian Propellers Limited, supplied the following data on the recommended heat treatments:

A. <u>FORGING</u>: Forging should never be performed over 2050° F. or below 1800° F. After forging, the forgings should be air cooled. They should then be reheated uniformly to 1500°-1550° F., and air cooled.

B. <u>ANNEALING</u>: Heat slowly to 1650° F., allowing sufficient time for charge to come to heat, hold at heat 8 hours, and then slowly cool for 32 hours. Forging should never be removed from the furnace at a temperature above 1000° F.

C. <u>HARDENING</u>: Preheat to 1550° F., then transfer quickly to high heat furnace at 2000° F. Allow to remain in furnace from five to ten minutes, depending on size of washer. Heating for quenching should never be over 2090° F. and never below 2000° F. Follow by oil quench

(Microscopic Examination of the Heat-Treated Samples, cont'd) -

> for eighteen to twenty-five seconds, and bring up to air cool by an air stream. Follow with a second drawing treatment at 1000° to 1020° F. for one to one and three-quarter hours, and cool as before.

There was not sufficient material available to check on whether the recommended forging practice would remove the carbide envelopes. However, in order to check on whether the heat treatment of the "as received" material was as recommended, the samples were given the specified hardening heat treatment, examined under the microscope, and checked for hardness. No differences were found between the reheat-treated and the "as received" specimens.

Figure 8 is a photomicrograph of the structure of Sample 9 after quenching from 2000° F. Note the remaining carbide envelopes and the marked coring from the first solidified product.

To check on the effect of improper heat treatment, samples were overheated at 2200° F. and underheated at 1800° F. In the first case (Figure 9) the specimen revealed a coarse structure of retained austenite and martensite. In the second case (Figure 10), the structure was martensitic, containing free carbide.

Another sample was given the recommended annealing treatment. A very fine distribution of spheroidized carbides in a matrix of ferrite was found. Figure 11 shows this microstructure. Note the slight evidence of carbide envelopes at "A".

In order to determine whether the carbide envelopes could be removed by the final high-temperature treatment, Sample 9 was given a new hardening treatment at the recommended temperature. It was allowed to remain in the furnace at 2000° F. for fifteen minutes. The result is shown in Figure 13, where it can be seen that carbide envelopes still

(Microscopic Examination of the Heat-Treated Samples, cont'd) -

remain, although smaller in size and less continuous.

Further on, a wrong annealing treatment was suspected to be a cause of the remaining of the carbide envelopes in the final product. Sample 5 "as received" (Figure 14) was annealed properly. Figure 15 shows the result of the proper anneal. Note the lack of continuity of carbide envelopes, which are now spheroidized. The final hardening treatment was given also. Note the final disappearance of carbide envelopes (Figure 16) after the full heat treatment.

DISCUSSION OF RESULTS:

It was impossible to check for the chemical analysis of the different samples, so that any variations depending on composition cannot be taken into account in the present report. The samples were also too small to check on the variations in the forging procedure. Crude fracture tests definitely indicate low impact strength, however.

The presence of dendritic patterns in the finished product shows that forging had not completely eliminated the "as cast" structure. It must be noted that even the samples which contained only traces of carbide envelopes have shown the presence of these dendrites. It is thought that all the material has been reduced to the same extent by forging. It is, in fact, a reasonable assumption that there is a uniform reduction practice in a given mill for a given type of steel.

The U. S. A. manufacturer of the washer reported that the Canadian washer showed soft spots, which, in their opinion, indicated the presence of retained austenite. From the results of this investigation, however, it is thought that both the lack of uniformity in structure, due to the presence of dendrites, and the uneven distribution of carbides are more

(Discussion of Results, cont'd) -

possible causes of these variations in hardness.

The Vickers hardness testing machine provides a good means of checking on the cause of hardness variations on an etched specimen, as it makes possible the testing of selected areas of the microstructure. The hardness survey carried out for the present investigation has shown that the variations were, for the most part, associated with the dendritic structure and carbide segregation, rather than with retained austenite. In fact, all the experiments made to detect retained austenite gave negative results. Nevertheless, it must be remarked that small amounts, undetectable with the methods herein used, may be present.

In the opinion of the U. S. A. manufacturer of the product, the poor properties of the Canadian-made washers were due to the presence of carbide networks which, in turn, were present as a result of the forging and annealing treatment. They also thought that the steel was also injured by a grain coarsening produced in the final heat treatment. In detail, they believed that overheating in forging had caused segregation of material high in carbon at the grain boundaries and that this material was not sufficiently distributed in the subsequent annealing operation. Grain coarsening was assumed to be due to overheating in the hardening operation. They further reported that the original ingot structure had not been removed in the forging operation and considered this to be an evidence of insufficient hot-working.

The results of the microscopic examination confirmed the presence of carbide envelopes and the incomplete removal of ingot structure. In the absence of an equilibrium diagram for this steel, explaining structure variations must involve

(Discussion of Results, cont'd) -

a certain amount of guesswork. The U. S. A. manufacturing firm are probably right in attributing the presence of the carbide envelopes to overheating in forging. Steel pouring temperature and amount of reduction of the ingot could affect the coarseness of the material and the persistence of the coarse ingot structure. The carbides appearing as envelopes doubtless were in solution in the austenite prior to cooling from the forging. The amount of these carbides in solution would be a function of the finishing forging temperature and would increase definitely with the temperature. As a consequence, an ingot that has been overheated in forging would, being of a higher carbon austenite, have a greater tendency to precipitate carbides on cooling or on a subsequent reheating operation. These carbides, once precipitated, are difficult to get into solution again, because complex carbides of chromium, tungsten, molybdenum and vanadium lack mobility and consequently are not subject to the effect of simple heating. Indeed, the even distribution of carbides (as distinguished from the carbide envelopes) and the removal of the cast ingot structure are largely governed by the forging itself; a 90 per cent reduction is usually recommended for such a type of steel. An ingot that has been overheated in forging is more likely to show carbide network prior to annealing.

This investigation has shown that the recommended annealing operation spheroidizes the carbides uniformly although the steel still shows slight evidence of carbide envelopes. If the annealing operation had been properly carried out on the forgings which had been finished at high temperature, these would not show carbide networks such as the one exhibited by Sample 9 (see Figures 6 and 8) unless

(Discussion of Results, cont'd) -

the carbide network originally present in the "as cast" structure was unusually coarse.

On the assumption that carbide envelopes are formed in the cooling from forging or in the reheating of the forging prior to annealing, the grain size is, if the carbides are precipitated in the first slow cooling of the forging, the austenitic grain size characteristic of the finishing forging temperature; on the other hand, if the carbide network is produced in the reheating operation, the austenitic grain size is then characteristic of the temperature at which this operation is carried out. As carbide segregation is slow in this steel, and as the recommended heat treatment involves an air cooling for forging and subsequent reheating at 1500°-1550° F., and as the grain size revealed by the carbide network is small, the assumption is that the grain size shown by the carbide network is more characteristic of the reheating temperature than of the finishing forging temperature.

It should be pointed out strongly that, on the assumption that carbides precipitated prior to the annealing operation, the grain size revealed by the carbide envelopes is not that which the steel reached on being heated for hardening. Unfortunately, it was found impossible to obtain an etching reagent which would reveal the real grain size of the troosto-martensite, so the claim of the U. S. A. manufacturer that the steel is coarse-grained cannot be confirmed or denied. Fracture tests would indicate that coarseness in fracture was associated with the presence of carbide networks, as material containing only small amounts of the latter exhibited good fractures. Definitely, the examination showed that the recommended hardening heat-treatment will not remove carbide envelopes

(Discussion of Results, cont'd) -

of the type exhibited by some of the "as received" samples.

Differences shown by the three groups of samples are thought, therefore, to be present as a result of the differences in the finishing forging temperatures and, possibly, in the annealing operation. Those forged at too high a temperature, or subjected to too short an annealing operation, showed more carbide envelopes and have poor physical properties. Overheating in hardening may have an influence in lowering the physical properties by inducing grain coarseness but no evidence was found to support or deny this contention. Indeed, the continuous rings on the X-ray spectrogram and the fineness of the troosto-martensite, found in the first and second groups, at least indicate that this condition was not universal.

A comparison of Figures 14, 15, and 16 gives a very definite answer to the question of whether the carbide envelopes can be removed by proper annealing followed by hardening. The treatment responsible for the removal of carbide envelopes is really the annealing treatment which definitely spheroidizes the carbides and then takes off the continuity. This treatment is quite critical and it should be pointed out strongly that the 32-hour-cooling specification should be thoroughly observed.

CONCLUSIONS:

1. Examination of the structure of the steel segregates the steel into three groups, one with only small traces of carbide envelopes, one with more distinct traces of carbide envelopes, and one with definitely marked carbide envelopes.

2. Fracture tests show that coarseness of fracture,

(Conclusions, cont'd) -

and probably low impact strength, is associated with the presence of these carbide envelopes.

3. All samples showed traces of original dendritic structure, indicating that forging had not been severe enough.

4. There was no evidence of retained austenite in the "as received" material. Rather, hardness variations were associated with inhomogeneity due to the presence of uneven distribution of carbides, and with dendritic structure.

5. The grain size, as estimated from the carbide envelopes, varied from 4 to 8. This was considered to be the grain size of the austenite in the reheating operation that followed the forging and preceded the annealing.

6. It was not found possible to develop the grain size of the troosto-martensite. Consequently, it was not possible to determine whether the forgings had been overheated in the hardening operation.

7. The recommended hardening procedure will not completely eliminate the carbide envelopes, especially if these are coarse.

8. It is thought that the carbides are present as a result of overheating in forging and as a result of incomplete annealing.

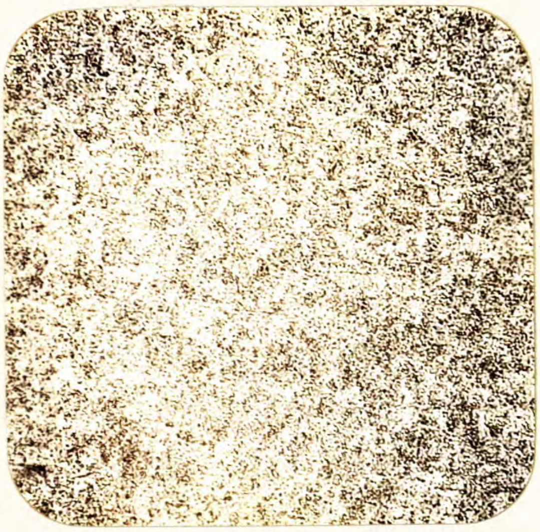
0000000000000
0000000
000

AD:GSF:PES.

Figure 1.

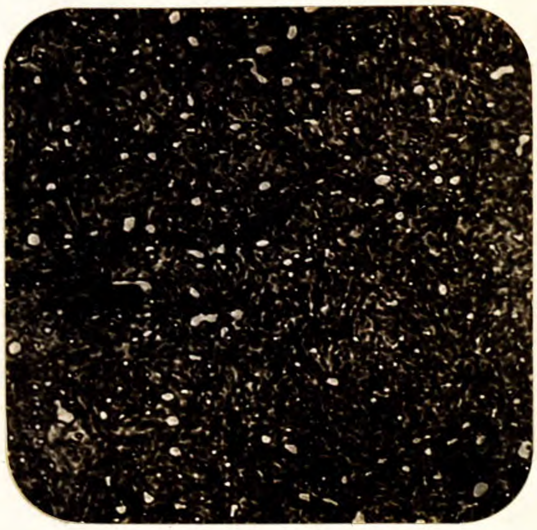
COMPARING THE FRACTURES OF
SAMPLES 2 and 9.

Figure 2.



X200, nital etch.

Figure 3.



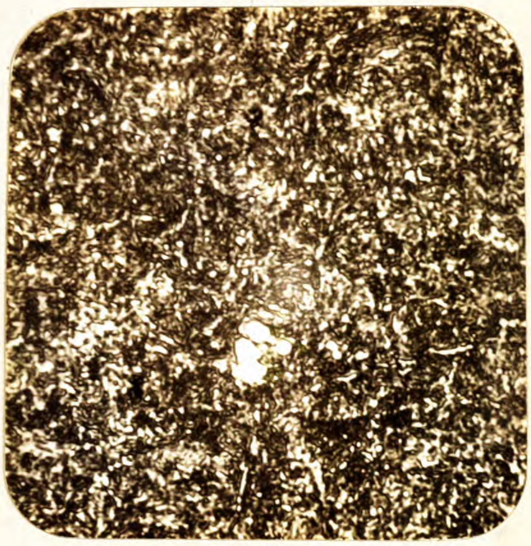
X1500, nital etch.

MICROSTRUCTURE OF SAMPLE 1, REPRESENTATIVE
OF GROUP 1.

Figure 4.

X200, nital etch.

Figure 5.



X1500, nital etch.

MICROSTRUCTURE OF SAMPLE 12, REPRESENTATIVE
OF GROUP 2.

Figure 6.

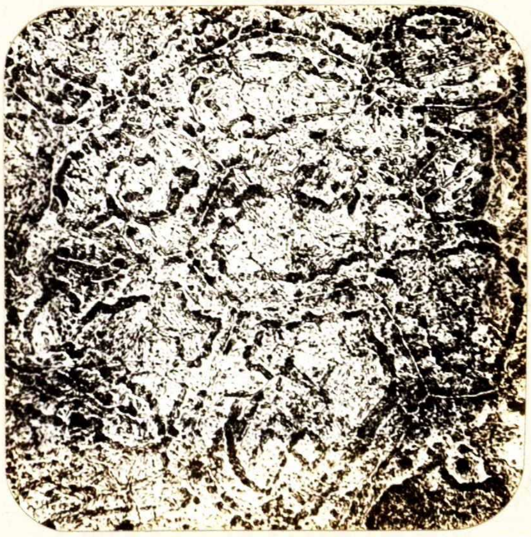
X200, nital etch.

Figure 7.

X1500, nital etch.

MICROSTRUCTURE OF SAMPLE 9, REPRESENTATIVE OF GROUP 3.

Figure 8.

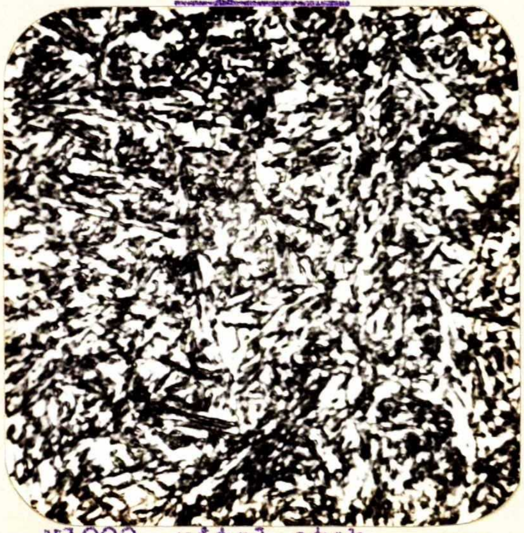


X200, nital etch.
SAMPLE 9, AS QUENCHED FROM 2000° F.
Note presence of coring.

Figure 9.

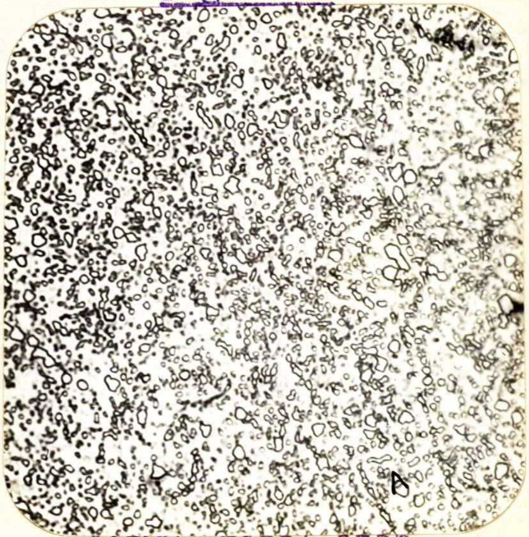
X500, nital etch.
AFTER QUENCHING FROM 2200° F.

Figure 10.



X1000, nital etch.
MICROSTRUCTURE OF THE STEEL AS QUENCHED FROM 1800° F.

Figure 11.

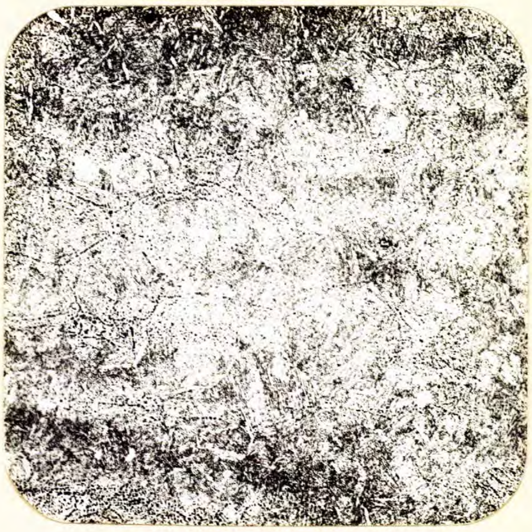


X1500, nital etch.
MICROSTRUCTURE OF THE STEEL ANNEALED.

Figure 12.

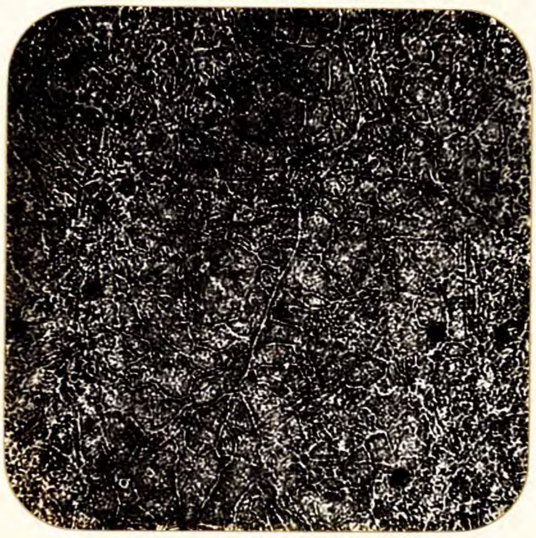
X500.
CARBIDE PATTERN IN SAMPLE 1
AFTER ETCHING IN BOILINGG
$K_3Fe(CN)_6$ + KOH solution.

Figure 13.



X200.
CARBIDE ENVELOPES IN SAMPLE 9
AFTER REMAINING IN FURNACE FOR
15 MINUTES AT 2000° F. AND THEN
QUENCHING.

Figure 14.



X500.
SAMPLE 5, AS RECEIVED.

Figure 15.

X500.
SAMPLE 5, ANNEALED.
Showing discontinuity of carbide
envelopes.

Figure 16.

X500.
SAMPLE 5, AFTER ANNEALING
AND HARDENING.
Note disappearance of carbide
envelopes.

AD:GSF:PES.