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DEPARTMENT OF MINES AND RESOURCES

BUREAU OF MINES

CANADA

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Ottawa, October 9, 1946.

R E P O R T

of the

ORE DRESSING AND METALLURGICAL LABORATORIES.

Investigation No. 2123.

(Further to Investigation Report)
(No. 1908, dated July 20, 1945.)

Cast Ring Dies Used for
Hot Drawing of Seamless Tubing.

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(Copy No. 5.)

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Origin of Material and Object of Investigation:

Investigation Report No. 1908, dated July 20, 1945, written for Page-Hersey Tubes Limited, Welland, Ontario, contained the results of a metallurgical examination on a cast ring die of British origin which was reported to have given a long service life, and on a die of Canadian origin which performed very poorly. (The British die was removed after 625 tubes had been fabricated as against 22 tubes for the Canadian die.) The investigation revealed a difference in the chemical content of the two dies, and it was recommended that dies of varying chemical content be cast in the Laboratories' foundry and forwarded to Page-Hersey Tubes, Limited, per Mr. C. F. Anderson, General Works Manager. It was also recommended that a study be made of the effect of heat treatment on the serviceability of the dies.

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

A pattern for a 76-mm.-diameter die was forwarded to these Laboratories on March 13, 1945. Two unused dies, one of English and the other of Canadian origin, were forwarded on September 14, 1945, for metallurgical examination.

The object of this report is to briefly cover the work done on the problem over a period dating from July, 1945, to October, 1946.

PROCEDURE:

1. Casting Methods.

Figures 1, 2 and 3 are photographs showing the methods of gating and risering used in casting of ring dies in the Laboratory foundry. Gates, sprues, runners and risers were removed from the casting on an abrasive wheel cut-off machine. The method of gating as illustrated in Figures 1 and 2 was found to produce a sounder casting than that in Figure 3.

A total of 33 dies of various chemical contents were cast in the Laboratories' foundry, of which 31 were forwarded to Page-Hersey Tubes, Limited, for test in service. Of these all but the last 12 dies have been reported on by the company to date.

The results of the test are given in Table I.

(Table I follows,
(on Page 3.)

(Procedure, cont'd) -

TABLE I.

Die No.	Condition	Hardness, Rockwell "C"	Type of Failure	PERFORMANCE, tubes per die		
				Pre-Finishing	Finishing	Total
1	As cast	47	Cracked.	775	660	1,435
2-1	" "	49 $\frac{1}{2}$	Wore out.	380	73	453
2-2	" "	"	" "	700	77	777
3-1	" "	49	" "	310	325	635
3-2	Heat-treated	60	" "	-	25	25
4-1	As cast	49	Broke accidentally.	-	-	-
4-2	Heat-treated	52 $\frac{1}{2}$	Wore out.	-	-	25
5-1	As cast	53	Cracked.	-	-	-
5-2	" "	"	" "	-	-	-
6-1	" "	44	Wore out.	-	110	110
6-2	" "	"	" "	-	35	35
7-1	" "	46	" "	225	-	225
7-2	" "	"	" "	-	38	38
8-1	" "	47	" "	80	-	80
8-2	" "	"	" "	103	-	103
9-1	" "	50 $\frac{1}{2}$	Broke.	-	105	105
9-2	" "	"	" "	-	85	85
10-1	" "	45	Cracked.	-	-	18
10-2	" "	"	Wore out.	-	-	185

Notes:

- 1 Dies Nos. 3-2 and 4-2 were cooled in air from 2000° F. and drawn at 900° F.
- 2 Normal average life of dies in plant service are:
 3" O.D., 12 gauge (dies 1 and 2) -
 British, 660 tubes per die,
 Canadian, 330 " " "
 1" standard pipe, 13 gauge (dies 5 to 10) -
 British, 200 to 250 tubes per die,
 Canadian, 125 tubes per die.

2. Chemical Analysis.

The results of chemical analyses made on the dies cast in the Laboratory foundry and shipped to Page-Hersey Tubes, Limited, are given in Table II.

(Table II follows,
 on Page 4.)

(Procedure, cont'd) -

TABLE II.

Die No.:	Carbon, per cent:	Manganese, per cent:	Silicon, per cent:	Chromium, per cent:	Tungsten, per cent:	Nitrogen, per cent:
1 :	2.46	0.60	1.05	25.5	2.54	
2 :	2.52	0.57	0.65	24.9	4.08	
3 :	2.52	0.38	0.33	25.8	2.25	
4 :	2.52	0.94	0.67	25.7	3.26	0.28
5 :	1.54	0.38	0.50	24.8	2.72	
6 :	2.00	0.38	0.35	27.1	2.92	
7 :	2.24	0.40	0.37	27.5	3.03	
8 :	2.59	0.42	0.34	25.2	3.16	
9 :	2.99	0.43	0.33	25.2	3.16	
10 :	2.28	0.35	0.52	24.9	3.16	0.28

Note: Analyses for Dies Nos. 8, 9 and 10 were calculated.

Chemical analyses were made on samples cut from a die of British and of Canadian origin, in order to confirm the results reported in Investigation Report No. 1908. The results are given in Table III.

TABLE III.

	British	Canadian
	- Per Cent -	
Carbon	2.41	2.07
Manganese	0.46	0.18
Silicon	0.30	0.57
Chromium	21.87	21.35
Tungsten	3.04	4.13
Molybdenum	Trace.	1.12
Nickel	"	Trace.
Vanadium	Nil.	Nil.

These results are in substantial agreement with that reported in Investigation Report No. 1908.

The following table (No. IV) shows the results of analyses made on each of two lots of 6 dies cast in the Laboratories' foundry and shipped to Page-Hersey Tubes, Limited for test in actual service. (The results have not been reported to date.) Lot A are identified as 1 to 6, and B as 7 to 12.

(Procedure, cont'd) -

TABLE IV.

	<u>Lot #A(1-6)</u>	<u>Lot #B(7-12)</u>
	Per Cent	
Carbon	2.26	2.28
Manganese	0.39	0.52
Silicon	0.44	0.29
Chromium	24.94	24.36
Tungsten	2.60	2.93
Nitrogen	-	0.28 [Ⓢ]

[Ⓢ] Estimated.

3. Heat-Treating Experiments.

Samples of the alloy from Die No. 1 were heat treated by quenching in oil from 2000° F. and drawing at various temperatures. The results are given in Table V.

TABLE V. - Results of Heat Treating Die No. 1.

<u>Condition</u>	<u>Hardness (Rockwell "C")</u>	<u>Magnetic Properties</u>
As cast	45-49	Magnetic
[Ⓢ] As quenched (2000° F.)	51-52	Non-magnetic
Quench and draw 900° F.	52-53	" "
" " " 1000° F.	60-61 $\frac{1}{2}$	Strongly magnetic
" " " 1100° F.	50-50 $\frac{1}{2}$	" "
" " " 1200° F.	49-51	" "

[Ⓢ] Quenched in oil, resulting in cracking.

Tables VI and VII show the properties of the alloys, Casts Nos. 3 and 4, respectively, resulting from heat treatment consisting of cooling in air from 2000° F. followed by drawing at various temperatures.

TABLE VI. - Properties of Heat-Treated Die No. 4.

<u>Condition</u>	<u>Hardness (Rockwell "C")</u>
As cast	49
Air cooled (2000° F.)	61-63
" " and draw 900° F.	61-62 $\frac{1}{2}$
" " " " 1000° F.	59 $\frac{1}{2}$ -60
" " " " 1100° F.	49-50

(Procedure, cont'd) -

TABLE VII. - Properties of Heat-Treated Die No. 4.

<u>Condition</u>	<u>Hardness (Rockwell "C")</u>	<u>Magnetic Properties</u>
As cast	48-49 $\frac{1}{2}$	Magnetic
Air cooled (2000° F)	50-53 $\frac{1}{2}$	Non-magnetic
" " and draw 900° F.	51 $\frac{1}{2}$ -52 $\frac{1}{2}$	" "
" " " " 1000° F.	51 $\frac{1}{2}$ -53 $\frac{1}{2}$	" "
" " " " 1100° F.	52-52 $\frac{1}{2}$	Slightly "
" " " " 1200° F.	46 $\frac{1}{2}$	Very "

4. Microscopic Examination.

Figures 4 and 5, taken at X100 and X750 respectively, show the microstructure of Cast Ring Die No. 1, which consists of dendrites of primary α solid solution and a eutectic of α and carbides.

Figures 6 and 7, taken at X100 and X750 respectively, show the microstructure of Die No. 1 after heat treatment i.e., heating to 2000° F. and quenching in oil. The α solid solution appears to have precipitated fine carbides.

Figures 8 and 9, taken at X100 and X250 respectively, show the microstructure of Cast Ring Die No. 5. The microstructure in Figure 8 is the result of using Vilella's etchant whereas that of Figure 9 is the result of using a special etching reagent, consisting of potassium ferricyanide and potassium hydroxide. The latter etchant causes the carbides to etch up dark.

Figures 10 and 11, taken at X100 and X250 respectively, shows the microstructure of Die No. 10. Here, again, the two contrasting etchants have been used.

Discussion:

In accordance with the recommendations made in Investigation Report No. 1908, dated July 20, 1945, the first

(Discussion, cont'd) -

die cast in the laboratory foundry (Die No. 1, Table II) was intended to approximate the following composition:

	<u>Per Cent</u>
Carbon	- 2.3
Manganese	- 0.20-0.40
Silicon	- 0.20-0.40
Chromium	- 25.0
Tungsten	- 2.75

This composition is similar to that of the British die reported in the previous investigation report and the micro-structure was found to be practically identical (see Figures 4 and 5). The performance of this die was reported to be equivalent to that of the British die and to be over twice that of the Canadian dies. However, it was hoped that further experimentation might result in the development of dies with even greater service life.

Accordingly, cast ring dies No. 2 were cast containing approximately the same chemical analysis as that of Die No. 1, but with higher tungsten content (see Table II). These dies were reported to have given service considerably inferior to that of Die No. 1 (see Table I).

It was then decided to cast another set of 2 dies (3-1 and 3-2) similar in chemical composition to that of Die No. 1 in order to check the performance of Die No. 1. Die No. 3-1 was in the "as cast" condition and had a hardness of 49 Rockwell "C" whereas Die No. 3-2 had been heat-treated to 60 Rockwell "C". The former produced 325 tubes as a finishing die and 310 more as a pre-finishing die. Since the final size of the tubing so produced was 2" O.D. x 11 gauge as compared with 3" O.D. x 12 gauge for Die No. 1, it is not possible to make a fair comparison. However, the heat-

(Discussion, cont'd) -

treated die, in spite of its greater hardness, was reported to have given very poor results and the comment was that "it was too soft."

Another set of two dies, 4-1 and 4-2, containing an analysis similar to that of Die No. 1 but with an addition of approximately 0.28 per cent nitrogen, was forwarded for test purposes. Die No. 4-1 was in the "as cast" condition, whereas Die No. 4-2 was heat treated to a slightly higher hardness. Unfortunately Die No. 4-1 was broken accidentally in service. However, the comment was that "it appeared to be of good quality." The heat-treated die wore out very rapidly and was found to be quite unsatisfactory.

It was then decided to cast a series of dies having the same chemical analysis but with varying carbon contents, in order to determine the effect of the carbon content on the service life. Accordingly a series of dies numbered from 5 to 9 containing 1.5, 2.00, 2.25, 2.50 and 3.0 per cent carbon were forwarded to the plant for testing. In addition, a set containing 2.25 per cent carbon but with 0.28 per cent nitrogen (cast No. 10) was also forwarded. These dies were used for tubing of 1" final diameter (13 gauge), and therefore the service conditions were much more severe than for Die No. 1. The results of this series showed the following:

1. The dies which gave the best results were Nos. 7 and 10 (see Table I). This is thought to be of considerable significance since the compositions of these dies approximate that of Die No. 1, the difference being in the nitrogen content of No. 10.

(Continued on next page)

(Discussion, cont'd) -

2. Considerable variation in the performance of 2 dies cast from the same heat was noted. This is most disconcerting and indicates variable service conditions.

Because of the latter observation, it was decided to cast one dozen dies consisting of two lots of 6 dies per lot. These dies were intended to approximate Dies Nos. 7 and 10 respectively. It was hoped that the average results so obtained would be of greater reliability than that obtained from one or two dies. (The results have not been reported to date.) The analyses of these dies are reported in Table IV.

Heat Treatment

The heat treating experiments indicated that greater hardness could be obtained by heating to 2000° F., followed by cooling in air. More severe quenching, such as oil quenching, invariably resulted in cracking. The air-hardened alloy was non-magnetic, thus indicating an austenitic structure. Drawing at 900° F. resulted in greater hardness (probably due to the formation of martensite) but further drawing resulted in decreased hardness and increased magnetic properties. Microscopic examination of the heat-treated alloy showed the presence of finely divided carbides which must have been precipitated from the α solid solution during heat treatment (see Figures 6 and 7). Although greater hardness was obtained by heat treating, the wear resistance was found to be definitely less than in the "as cast" condition. This is undoubtedly the reason why these alloys are used in the "as cast" condition.

(Discussion, cont'd) -

In reply to an enquiry regarding the use of Carboloy dies for hot drawing seamless tubing, the Canadian General Electric Company, Toronto, Ontario, advised against its use at the present time.

The Nitralloy Corporation, 230 Park Avenue, New York, N.Y., suggests the use of the following alloy hardened by quenching and tempering at 950° F., followed by nitriding for 24 hours at 950° F.:

Carbon	-	1.50	per cent
Chromium	-	12.0	"
Molybdenum	-	1.0	"
Vanadium	-	1.0	"

The Molybdenum Corporation of America, Pittsburgh, Pa., suggests the use of $2\frac{1}{2}$ per cent of molybdenum as an addition to the alloys cast in these Laboratories. They also recommend heating to 400° F. each time before placing the dies in service as a measure for reducing breakage.

A die of German origin,^① which was reported to have given excellent results, is as follows:

<u>C</u>	<u>Si</u>	<u>Mn</u>	<u>Cr</u>	<u>Ni</u>	<u>W</u>	<u>V</u>
0.44	1.80	0.85	14.0	6.0	2.5	0.77

CONCLUSIONS:

1. The results of the experiments indicated that an alloy of chemical composition approximating that of the British dies gave highest service life.

2. Heat-treated dies, although having a greater hardness, gave a greatly inferior service life as compared with dies in the "as cast" condition.

3. Because of the inconsistency of results

^① Iron Age, May 30, 1946.

(Conclusions, cont'd) -

obtained from alloys cast from the same heat, it was concluded that there must be a considerable number of variables in the service conditions. Hence, reliable results can only be obtained by using the average figure for many tests.

RECOMMENDATIONS:

1. The casting of dies of the following analysis is recommended:

	<u>Die A</u>	<u>Die B</u>	<u>Die C</u>
	-	Per Cent	-
Carbon	- 2.25	2.25	2.25
Chromium	- 25.00	25.00	25.00
Tungsten	- 2.75	2.75	4.75
Nitrogen	- -	0.28	-
Silicon	- 0.2-0.4	0.2-0.4	0.2-0.4
Manganese	- "	"	"

2. A statistical analysis of the results obtained by casting dies of the above analyses should be made. Only by maintaining such a record can a rational comparison be made of the performance of dies of various chemical contents.

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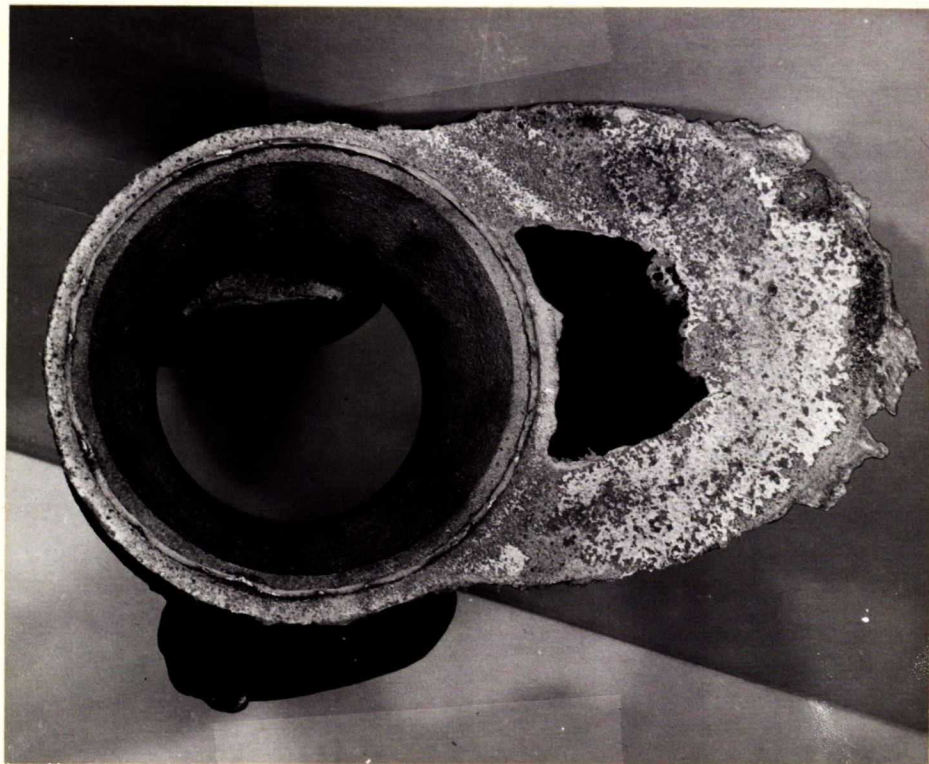
AF:LB.

(Figures 1 to 11 follow,
(on Pages 12 to 17.)

Figure 1.



Figure 2.



PHOTOGRAPHS SHOWING GATING AND RISERING METHODS
USED IN THE CASTING OF RING DIES IN THE LABORATORIES'
FOUNDRY.

Figure 3.



ALTERNATE METHOD OF CASTING RING DIES (DISCARDED).

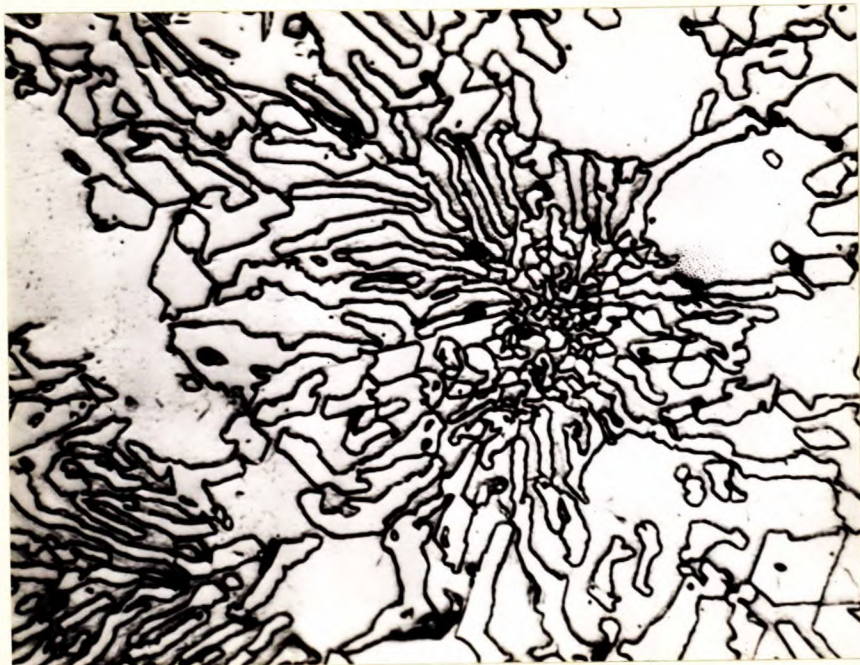
(Approximately 1/3 actual size.)

Figure 4.



X100. (Etchant: Vilella's reagent.)

Figure 5.



X750. (Etchant: Vilella's reagent.)

RING DIE NO. 1 (AS CAST CONDITION).

Microstructure consists of dendrites of primary alpha solid solution in a background of eutectic (α + carbides).

Figure 6.



X100. (Etchant: Vilella's reagent.)

Figure 7.

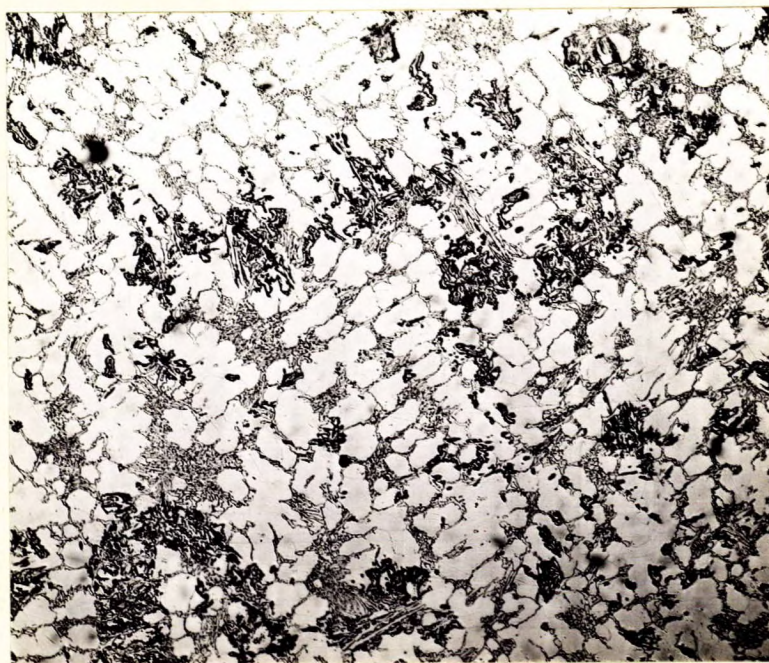


X750. (Etchant: Vilella's reagent.)

RING DIE NO. 1 (HEAT TREATED).

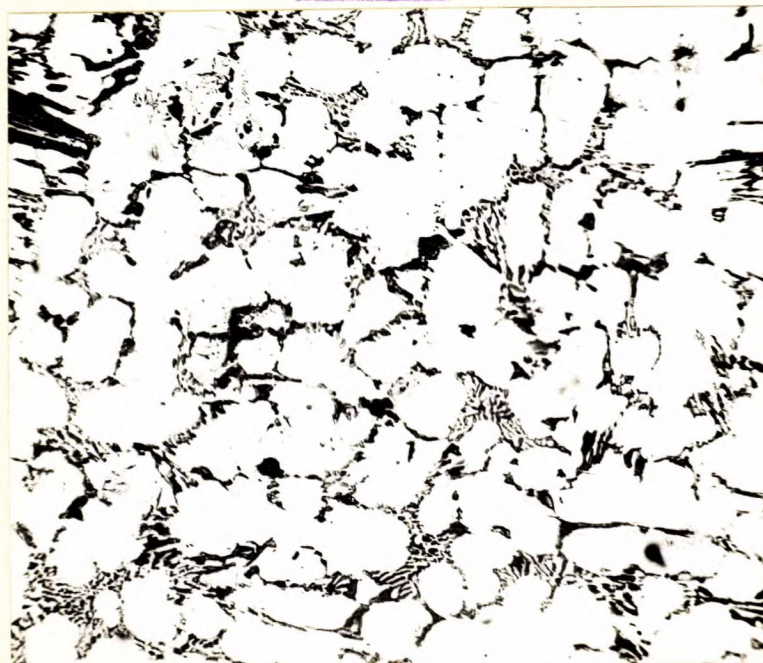
Heated to 2000° F. and quenched in oil.
Microstructure probably consists of primary alpha
solid solution and precipitated carbides.

Figure 8.



X100. (Etchant: Vilella's reagent.)

Figure 9.

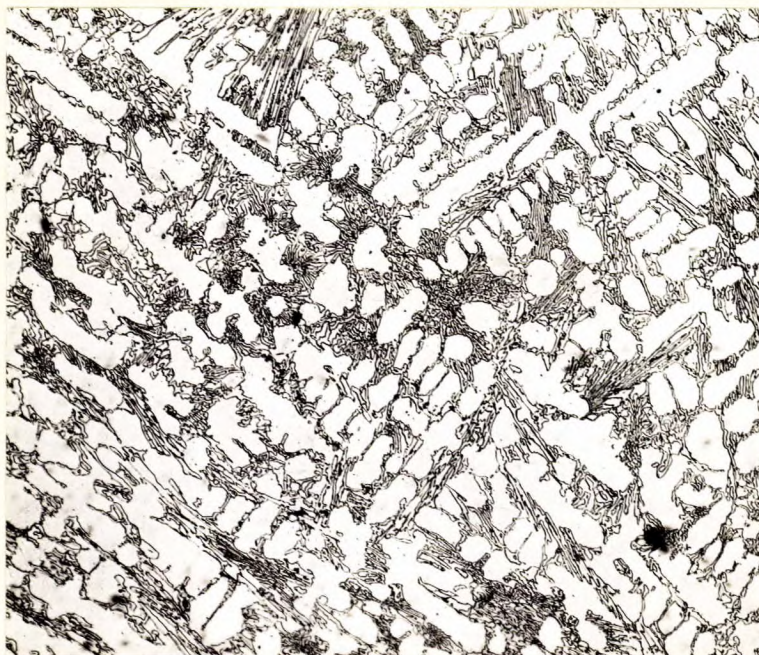


X250. (Etchant: potassium ferricyanide and potassium hydroxide.)

CAST RING DIE NO. 5 (AS CAST).

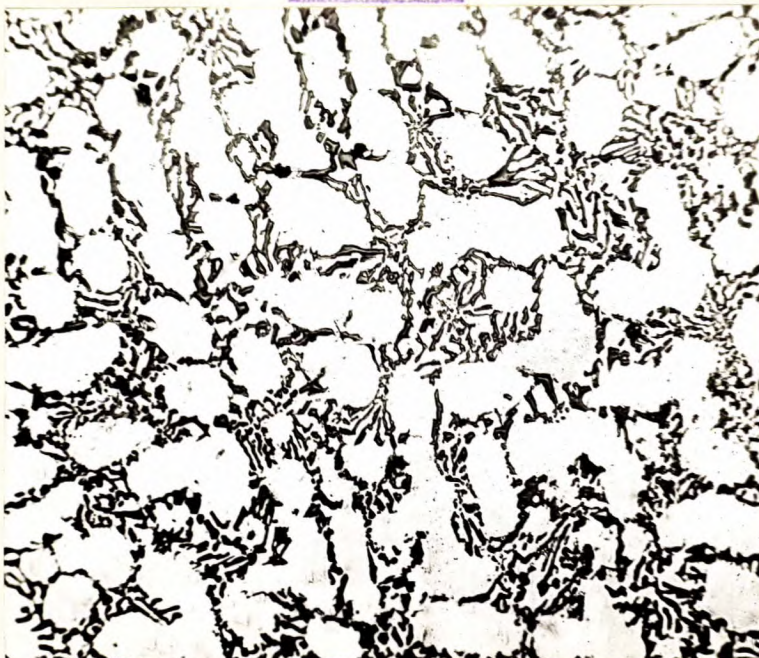
Potassium ferricyanide and potassium hydroxide etchant causes carbides to etch up dark.

Figure 10.



X100. (Etchant: Vilella's reagent.)

Figure 11.



X250. (Etchant: potassium ferricyanide and potassium hydroxide.)

CAST RING DIE NO. 10.

Microstructure consists of dendrites of primary alpha solution and eutectic of carbides + austenite.

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AF:LB.

APPENDIX TO REPORT OF INVESTIGATION NO. 2123,
DATED OCTOBER 9,
1946.

The following letter was received at the
Physical Metallurgy Research Laboratories on October 30, 1946:

(Copy)

PAGE-HERSEY TUBES, LIMITED
Welland, Canada.

Welland Works,
October 29th, 1946.

Mr. S. L. Gertsman,
Department of Mines and Resources,
568 Booth Street,
Ottawa.

Dear Sir:

Due to the steel billet shortage brought on by recent strikes, we were unable to schedule an operation to use the twelve dies you sent us, per your letter of June 28th, until last week.

The twelve dies were all used in the same position in our Ringbed and all were used to produce the same size and gauge of tubes. In general, they were the best wearing dies we have had in our bench to date and the only fault to be found with them was that when trouble was encountered in the bench and it became necessary to knock the die out of its holder in order to clear the bench, several of them broke while being removed. The results of the twelve dies are as follows:

First Die used	finished	1115	Tubes
Second " "	" "	855	"
Third " "	" "	103	" and cracked while being removed from its holder in order to clear the bench.
Fourth " "	finished	1400	Tubes
Fifth " "	" "	625	" and cracked while being removed from its holder in order to clear the bench.
Sixth " "	finished	1050	Tubes
Seventh " "	" "	310	" and cracked while in use.
Eighth " "	" "	425	" and cracked while being removed from its holder in order to clear the bench.
Ninth Die used	finished	235	Tubes
Tenth " "	" "	1105	"
Eleventh " "	" "	1250	"
Twelfth " "	" "	483	"

We are forwarding to you by Express ten of the

(Appendix to Report of Investigation No. 2123, cont'd) -

dies. The fifth and eighth dies listed above were lost, due to their having been broken up and proper care not taken to collect the pieces.

We are sorry to state that we had to grind these dies on the outside in order to make them fit our adapters, so that the original markings from you were lost. Further, eight of the dies being returned to you could be ground to a larger size and used as pre-finishing dies.

In general, this lot of dies is above the average we have been using during the past years. We feel that your work has definitely pointed the way to longer life dies for us and we will be glad to hear further from you.

Yours very truly,

PAGE-LERSEY TUBES, LIMITED

(sgd) C. F. Anderson,

General Works Manager.

CFA/KG.

Copied at
Ottawa, Ontario,
October 31, 1946.
SLG:LB.