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REPORT

of the

ORE DRESSING AND METALLURGICAL LABORATORIES.

Investigation No. 1924.

Metallurgical Examination of Cracked Welds in 35% Ni-15% Cr Steel Retorts Used for Production of Magnesium.

(Copy No. 10)

Bureau of Mines
Division of Metallic
Minerals
-
Physical Metallurgy
Research Laboratories

CANADA
DEPARTMENT
OF
MINES AND RESOURCES

Mines and Geology Branch

OTTAWA August 28, 1945.

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Introduction:

On June 20, 1945, Mr. L. G. Brewer, Mechanical Superintendent, Dominion Magnesium Limited, Haley, Ontario, requested the assistance of these Laboratories in determining the cause of rapid weld failure by cracking of high alloy steel retorts used for the production of magnesium. Mr. Brewer stated that this type of failure was new and began on or around May 6, 1945, and resulted in 45 failures up to July 9. Service life before failure was drastically reduced from that previously obtained and this constituted a serious problem.

On July 4, the writer visited the plant and reviewed the welding procedures and techniques in use. These were those previously recommended by these Laboratories and which

(Introduction, cont'd) -

had proved to be entirely satisfactory up to the initiation of cracking failures. In discussing the problem with Mr. Brewer and other members of the staff, it was brought out that on or around January 15, 1945, a change had been made in the carbon content of the retort material. Since that time a considerable number of the higher-carbon-content retorts had gone into service and were giving service lives in the order of 100 days and were still in service. There is no means of determining the service life to be expected of those still in service.

A check of the retort service life records indicated that of those retorts showing premature failure those above 0.30 per cent carbon were definitely in the majority. It was also stated that all cracks in welds occur at the bottom of a retort and not at the top. A section of retort showing a typical weld failure by cracking was brought back to these Laboratories for examination. Subsequently, a second section was received which was stated to be so brittle that it could be knocked out with a hammer. In addition, a few welding electrodes taken from the stock used to produce the cracking welds were submitted for chemical analysis.

On July 9 and 19, letters were received giving service life, barrel number, carbon content, position in furnace and furnace number of all retorts in service and showing those failing prematurely. A third letter, received on July 7, gave preliminary temperature measurements of the retort barrel surface, obtained by thermocouples, over a range of 16 inches, beginning 2 inches from the inner face of the furnace wall.

Object of Investigation:

(1) To determine the cause of weld cracking.

(2) To offer recommendations to eliminate premature service failures.

PROCEDURE:

(1). From the service life reports the following data were abstracted. These data concern only retorts which failed prematurely in the period of December 1944 to July 9, 1945.

TABLE I.

Retort Barrel No.	Carbon Content, per cent	Service Life, days	Position	
			Furnace	Bank
158	0.18	253	6	B
167	.25	236	6	A
171	.25	255	6	D
174	.21	259	2	A
284	.22	164	3	D
336	.21	162	8	A
339	.24	138	9	D
344	.17	133	7	B
346	.25	150	5	B
354	.19	151	7	B
380	.26	129	9	C
398	.18	128	8	C
404	.18	107	4	A
413	.23	126	8	C
437	.45	47	6	C
472	.38	99	3	C
486	.32	58	2	C
499	.30	65	3	B
501	.30	68	5	A
508	.33	4	4	D
531	.34	64	5	B
537	.34	48	6	C
538	.34	17	5	C
533	.30	57	2	C
536	.34	25	5	B
539	.37	60	4	B
540	.37	64	2	B
541	.29	19	5	B
542	.36	32	8	B
544	.36	21	10	D
545	.30	73	3	B
546	.30	45	3	B
547	.33	73	5	A
548	.33	64	2	B
549	.31	19	10	B
558	.36	9	8	D
563	.37	12	2	A
566	.36	18	2	D
569	.33	22	10	D
586	.32	15	8	A
599	.42	24	3	D
600	.42	6	8	D
611	.31	9	8	C
627	.30	8	8	A
628	.26	11	8	C

(Procedure, cont'd) -

TABLE II.

Bank Frequency	
Bank	Failures, per cent
A	20
B	33.3
C	24.5
D	22.2

Furnace Frequency	
Furnace No.	Failures, per cent
1	2.2
2	17.4
3	13.0
4	6.5
5	15.2
6	10.9
7	4.35
8	22.0
9	4.35
10	4.35

From the data submitted the following information was also obtained. All retort barrels with numbers 530 and higher are shorter than those previously used. All barrels are joined to the condenser by means of an extension piece welded to the condenser. As a condenser is used over and over again, the machining necessary for the preparation of a weld joint eventually compels the use of an extension section even for long barrels. The extension piece varies between 6 inches and 8 inches in length and is usually secured from scrapped retort barrels. The following table shows the percentage failures of long and short barrels in relation to the thickness of the extension pieces used:

TABLE III.

	Short Barrels	Long Barrels	Total	Percentage.
Extension piece 7/8 inch thick	1	0	1	2.3
" " 1 " "	15	14	29	67.5
" " 1-1/8 " "	6	4	10	23.3
" " 1¼ " "	3	0	3	7.0

The data recorded in Table I were charted on graph paper and the results obtained are shown in Figure 1.

The data from the letter giving temperature measure-

(Procedure, cont'd) -

ments of retort barrel was plotted on graph paper and the results obtained are shown in Figure 2.

(2). The section of retort showing typical failure by weld cracking was cleaned and examined visually. Figure 3 shows the appearance of the bottom of the retort weld and its severe cracking. Figure 4 shows the appearance of the top of the retort weld and its less severe cracking. No cracks were detected in the side positions of the weld. Figure 5 shows cracks at the root of the welds and running at right angles to the welds and apparently originating in casting flaws. For convenience, this sample will henceforth be referred to as Sample No. 1.

(3). The weld section stated to be very brittle and removable by hammering was cleaned and visually examined. Figure 6 shows the peculiar woody structure of the fractured weld. This sample will be subsequently referred to as Sample No. 2.

(4). The sample of welding electrode, said to be from the stock used to produce the cracked welds, was used to deposit a chemical analysis pad of metal as per the American Welding Society's standard method. The analysis of this deposited weld metal is as follows:

		Per Cent
Carbon	-	0.04
Manganese	-	1.57
Silicon	-	0.65
Chromium	-	13.67
Nickel	-	35.50
Molybdenum	-	Trace.

(5). Chemical analyses of retort and condenser sections were made, the sample being obtained by milling transverse to

(Procedure, cont'd) -

to the length of the sections. This ensures obtaining a general analysis, including any decarburized metal on the surfaces. The following table lists the results secured.

	Sample No. 1.		Sample No. 2.	
	Retort	Condenser	Retort	Condenser
	- Per cent -			
Carbon -	0.27	0.22	0.41	0.26
Manganese -	0.82	0.45	0.48	0.71
Silicon -	1.29	0.86	0.82	0.68
Chromium -	20.24	18.68	16.08	17.66
Nickel -	35.75	37.50	37.56	37.95
Molybdenum -	0.17	0.07	0.15	0.07

(6). Sections were removed from the top and the bottom of Sample No. 1. These samples were polished and etched in aqua regia. Figures 7 and 8, respectively, show the samples in this stage of preparation.

(7). A section was removed from Sample No. 2 and prepared as above. Figure 9 shows this sample in this stage of preparation.

(8). Sections of top and bottom welds of Sample No. 1 and a section of Sample No. 2 were subjected to a microscopic examination. Figure 10 shows the structure of the condenser material and Figure 11 the structure of the retort material. Figure 12 shows the structure around cracks in the retort material adjacent to the weld. Figures 13 and 14 show two types of structures existing around weld metal cracks in both Sample No. 1 and Sample No. 2. Figure 15 shows the structure of the weld metal in the vicinity of the root where no cracks were found.

(9). The large numbers of what appear to be carbides around the cracked areas of the welds seem to be in excess of what would normally be encountered in the ordinary weld. To

(Procedure, cont'd) -

check the carbon content in the cracked weld areas, a groove was milled from the bottom of weld of Sample No. 1 in the cracked area and analysed for carbon content only. This was found to be 0.08 per cent.

(10). An attempt was made to identify the precipitate by means of its x-ray diffraction pattern. A sample was machined from the weld metal that was badly cracked and put into an electrolytic bath containing dilute HCl. Since carbides are not soluble in the electrolyte they can be collected as a sludge as the surrounding metal is dissolved. The sludge was dried and its diffraction pattern obtained.

It can be definitely stated that no sigma-phase material was found in the sludge. The pattern obtained does not agree with sigma-phase material or any of the known carbides as described in the literature. The lattice type is possibly similar to some carbides but the dimensions are different. It is probable that iron in the carbide is responsible for the difference. The only possible formula that can be applied to the carbides would be $(CrFe)_7C_3$.

(11). To substantiate a theory regarding the orientation of the precipitate shown in Figure 14, the following test was conducted. A tensile test bar of 35/15 material containing 0.10 per cent carbon was pulled to failure in a tensile machine. Samples were machined from the test bar at the area of greatest deformation close to the fracture and also from the threaded end of the bar where no plastic deformation has taken place. Both samples were heated to 1400° F. for 18 days and then examined under the microscope. Figure 16 shows the structure of the test bar at the area of greatest deformation, while Figure 17 shows the structure of the same test bar where no deformation had taken place.

(Continued on next page)

Discussion:

A mathematical test of the significance of the furnace and bank failure frequency figures shown in Table II indicates that there is no preferred furnace or bank in which failures are more liable to occur. In other words, in spite of the apparently greater chance of failure in furnace 2 or bank B, the failures occurring at these stations are due to chance and not to an assignable cause.

In Table III it is shown that 67.5 per cent of the failures occur with both long and short barrels when the condenser extension piece is 1 inch in thickness. It would appear at first glance that this is highly significant, but without knowing the percentage of all extension pieces which are of this thickness it is impossible to properly assess its importance. It is apparent that if the largest percentage of all extension pieces are of this thickness the largest number of failures would be expected to occur in this group.

Using the data of Table I and the chart Figure 1, it can be shown mathematically that the chances of such a distribution of failures can be due to chance in only two out of 100 such samples. That is, it can be considered definitely proven that the high carbon content (above 0.27 per cent), or some property of the metal associated with it, is having an adverse effect on service life, under the conditions of operation to which the retorts in this sample were subjected. This being the case, it is natural to suspect that carbide precipitation may be in part responsible. With alloys of this composition it is known that carbide precipitation takes place when the metal is heated within the range 800-1600° F. An examination of Figure 2 reveals that this temperature range exists between 20.6 and 26 inches from the open end of the retort. Retort metal and condenser metal within this distance range will be subject to those conditions conducive to severe carbide

(Discussion, cont'd) -

precipitation. The original practice of bricking up the furnace port after installing a retort has been changed to the use of asbestos rope to plug the port. Also, some of the retorts with short service life, included in the failure sample, have been used in magnesium distillation and the production of calcium. In these latter cases insulation is applied to the condenser from the furnace wall to the water jacket. This would have the effect of increasing the critical length in which the temperature conditions favour carbide precipitation.

The variables of retort barrel length, differences in port sealing, and variations in total area outside the furnace which is insulated, all are probably having some effect on service life. Since the retort metal at operating temperature has a low yield point, the mechanical support of the retort can also be very important with regard to its effect on service life. The frequent appearance of cracks at the bottom of a weld indicates that this area is subject to tensile stresses and/or fatigue as would be encountered in a simple beam. The conditions would exist where the retort is not being supported by all saddles and furnace wall rollers. Bottom weld failures might be attributable to lack of support at the furnace port where the condenser end attachments and the first saddle within the furnace walls are the two closest support areas.

Figures 3 and 4 show that cracks in the welds occur in both the top and the bottom and are not entirely confined to the bottom area, at least in this particular sample. Figure 5 shows that cracks on the inside of the barrel have originated from casting defects. Figures 7 and 8 show that the cracking in the bottom areas of the weld are much more pronounced than the top areas of the weld. This lends weight to the theory of severe stress concentration in that area. It will be noted

(Discussion, cont'd) -

that the heaviest crack in the bottom of the weld originates in an unfused root. This fact, together with cracking in the top area of the weld, indicates alternating stresses since when the weld at the bottom of the retort is in tension its root and the top areas of the weld at the top of the retort are in compression. These conditions would not cause cracking in these areas. It will also be noted from Figure 8 that there is a sharp change of section in the weld area and that cracking has occurred in the retort barrel material adjacent to the weld. Figure 9 shows a similar condition with regard to Sample No. 2.

Chemical analysis reveals that the welding electrode deposits low carbon metal of the proper alloy content and that the condenser and retort barrel materials of both samples are of the proper alloy composition. It is interesting to note that the retort barrel material of Sample No. 2 contains 0.41 per cent carbon and this is the sample said to be quite brittle. However, the data supplied indicate that retorts containing as high as 0.58 per cent carbon are still in service after over 100 days, so that it is apparent the high carbon content alone is not responsible for premature service failures. The chemical analysis of a badly cracked weld area of Sample No. 1 showed a carbon content of 0.08 per cent, which indicates a satisfactory welding technique in that carbon pick-up is quite low.

Figures 10 and 11 show respectively the structures of the condenser and barrel materials. Both show fine carbides in the grains and fairly heavy grain boundary carbides. Figure 12 shows cracks in the barrel material adjacent to the weld and reveals that they follow grain boundaries containing precipitated carbides. Figures 13 and 14 show two types of carbides found in the badly cracked welds of both samples. These carbides are fine nodules in one case and somewhat acicular in the

(Discussion, cont'd) -

other. In the latter case it will be noted that the orientation is 90° to one another. Where welds are not badly cracked they have the structure shown in Figure 15, in which there are some fine carbides in the grains and very light grain boundary carbides. The 90° orientation of the precipitate, shown in Figure 14, raised some doubts as to the nature of the precipitate. It is known that such orientation is the result of either hot or cold work and deformation of the material. In alloys of this nature the possibility of the precipitation of sigma-phase material cannot be ignored. Carbides may also exhibit the same orientation under the same conditions. X-ray diffraction patterns definitely fail to reveal the presence of sigma-phase material and give a general confirmation of the presence of carbides. The lack of precise identification of particular carbides is attributable to iron in the carbide.

Knowing the precipitate to be carbides, it should be possible to reproduce the structure showing the 90° orientation of carbide particles. This was done by the procedure outlined under "Procedure". Figure 16 shows this carbide particle orientation and was obtained by severely deforming material and then heating to the upper end of the carbide precipitation range of 18 days. Figure 16 shows the effect of the same heat treatment on undeformed material. It will be noted that the carbides are beginning to migrate to the grain boundaries but that there is no preferred orientation.

The phenomenon of slip plane carbide precipitation is readily explained. Material strained is not in equilibrium, and in an effort to regain equilibrium conditions carbides are precipitated along the crystal boundaries and along slip planes. These carbides are at right angles to one another due to the cubic shape of the crystals. The carbide precipitation is not necessarily accompanied by the formation of a crack but the general stiffening effect can highly stress the material and cause a crack to follow a grain boundary containing carbides. All material in the vicinity of a crack is strained and that material in the immediate vicinity has been plastically strained prior to the formation of the crack. The material permanently deformed will exhibit the slip-plane type of carbide precipitation. This explains the profusion of carbides in the cracked areas of the welds.

It would, then, appear from the above that the welds are being stressed in some manner, probably as a result of the mechanical support given to the retort. The lower-carbon retorts previously used were sufficiently ductile at their operating temperature to prevent any stress concentration, by readily yielding to the stresses over the entire stressed area. The higher carbon retorts, being stiffer, naturally force the deformation to take place at the more ductile area--that is, the weld. However, it is recognized that the higher carbon content alone is not the sole factor contributing to premature service failure. The temperature of the area in which the weld falls will determine the rate of carbide precipitation. The range of distance over which carbide precipitation can take place is, in turn, determined by the type of insulation used and the area insulated.

None of the above remarks should be construed as condemnation of the higher-carbon retorts. There is good reason to believe that their higher creep resistance should result in better service life. In the light of the information now obtained it should be possible to adjust the service conditions so that the present difficulties are overcome. It is believed that the above gives a sound explanation of the causes of early failure of the retorts examined and that the reason for better service life of other high-carbon retorts lies in their conditions of mechanical support and/or the location of the weld with regard to its operating temperature.

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Conclusions:

1. There is no preferred furnace or bank in which failure occurs more frequently than others.

2. It is impossible to state the significance of the higher percentage failures when 1-inch-thick extension pieces are used, without knowing the percentage of all extension pieces which are of this thickness.

3. With regard to the failures taking place between May 6 and July 19, 1945, it is definitely proven that high carbon content (above 0.27 per cent), or some property of the metal associated with it, is having an adverse effect on service life. It should be stressed that this applies only to these retorts and the service conditions to which they were subjected.

4. Weld location within the temperature zone 800° to 1600° F. would result in severe carbide precipitation and consequent embrittlement.

5. Methods and extent of insulation would affect service life, inasmuch as they affect the distance range over which the temperature conditions are conducive to carbide precipitation.

6. The location of cracks in welds (top and bottom) and the greater profusion of cracks in the bottom of welds, both point to stress concentration in welds, probably resulting from inadequate mechanical support and also some kind of alternating stress.

7. Stress concentration can be caused by improper machining for preparation of weld joints in permitting sharp changes of section (as shown by Sample No. 1).

8. Chemical analysis shows that welding electrodes, retort and condenser materials are of the proper alloy contents and that carbon pick-up in welds is very slight.

9. It has been shown that the 90° orientation of carbides found in the badly cracked areas is produced by permanent

(Conclusion, cont'd) -

deformation of the material. This confirms the presence of mechanical stresses in the welds.

10. It is not believed that this report contains any information that would warrant condemnation of the higher carbon retorts. The greater creep resistance of this material is valuable and the service conditions should be so adjusted as to permit its full development.

Recommendations:

1. All welds should be so located as to fall either well within a zone above 1600° F. or well outside of the zone below 800° F. The latter location would be preferable in view of the higher yield strength at low temperatures.

2. The above conditions should be maintained on those retorts used for magnesium distillation and calcium production. In these cases it might be necessary to locate the weld inside the hot zone to allow the use of a maximum amount of insulation.

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(Pages 15 to 23, following,)
(contain Figures 1 to 17.)

Figure I.

Retort Carbon Content Versus Service Life.

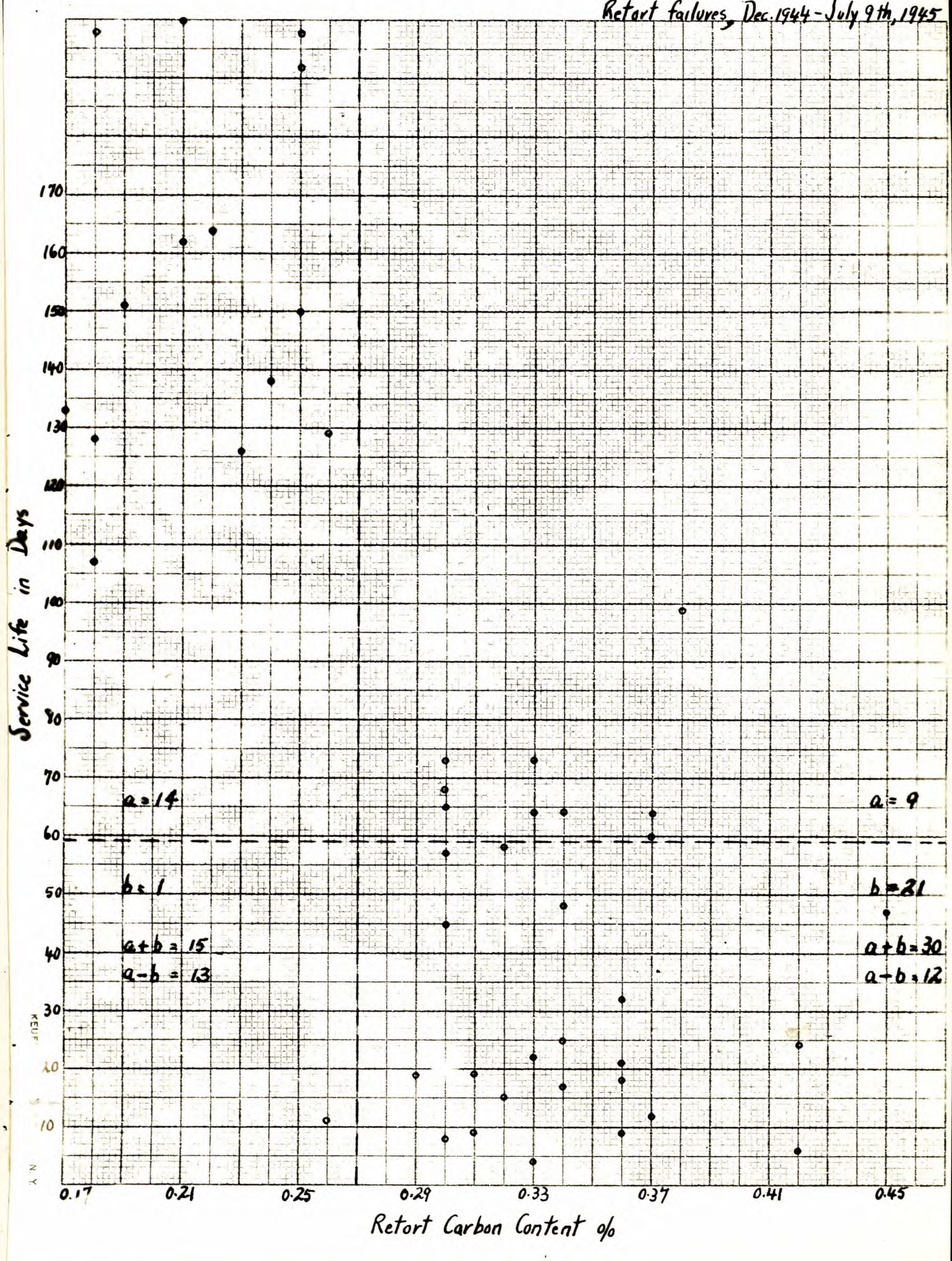
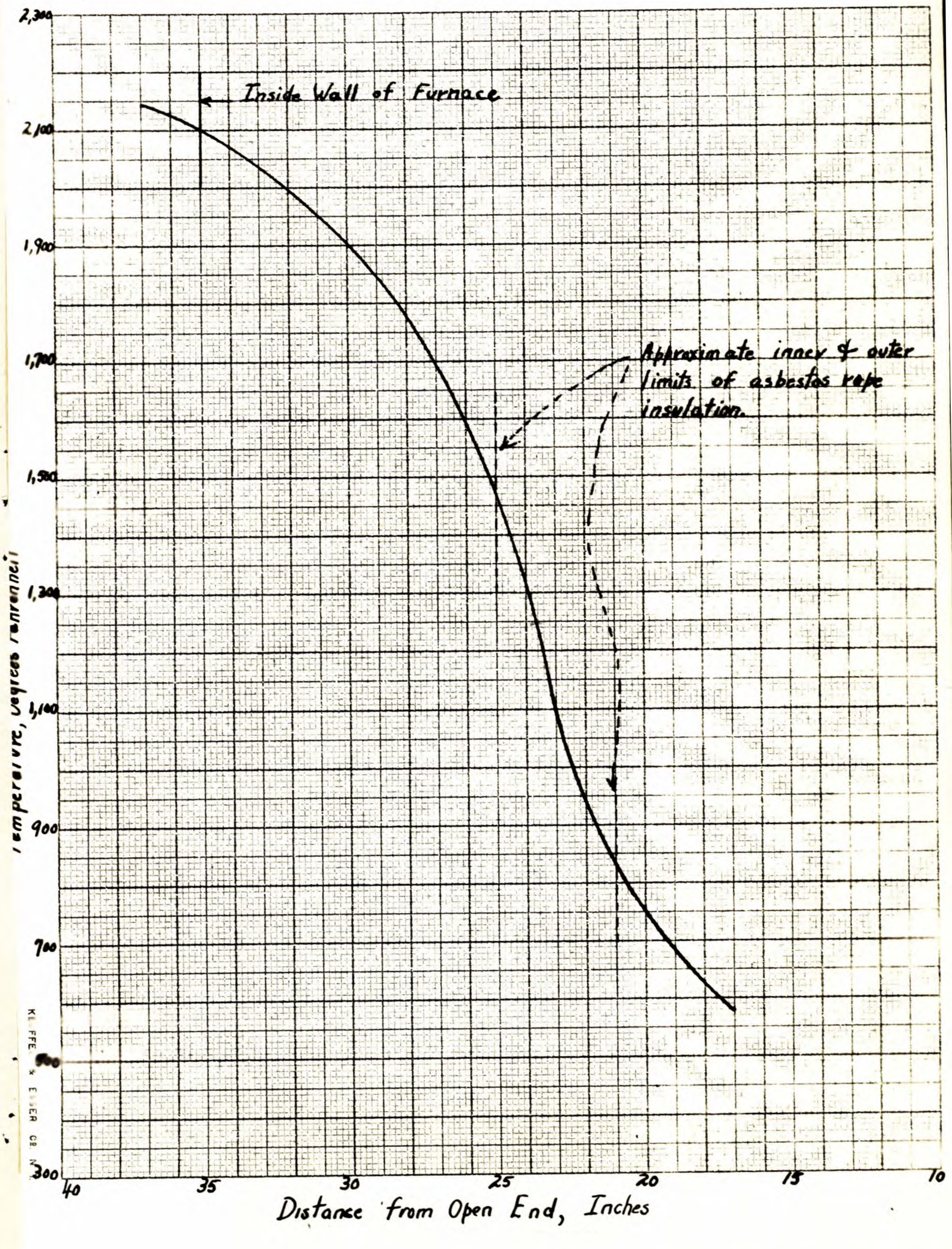


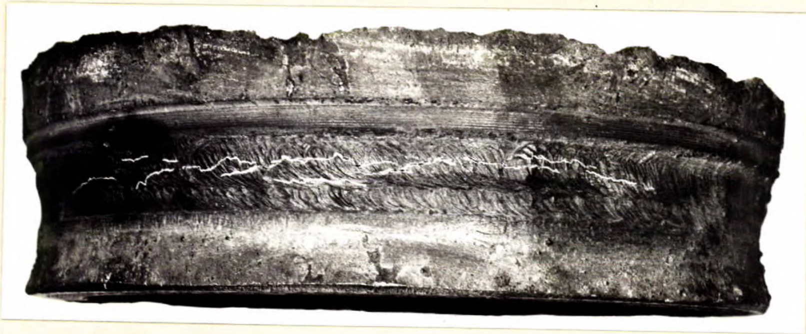
Figure 2.

Temperature of Outside Surface of Retort
Versus
Distance from Open End of Retort.



1924

Figure 3.



BOTTOM OF WELD OF SAMPLE NO. 1.

Note cracks and sharp machined change of section above weld.

(Approximately 1/3 size).

Figure 4.

TOP OF WELD OF SAMPLE NO. 1.

Note crack and sharp machined change of section above weld.

Figure 5.

SAMPLE NO. 1 AS RECEIVED.

Note cracks on inside, originating in casting defects.

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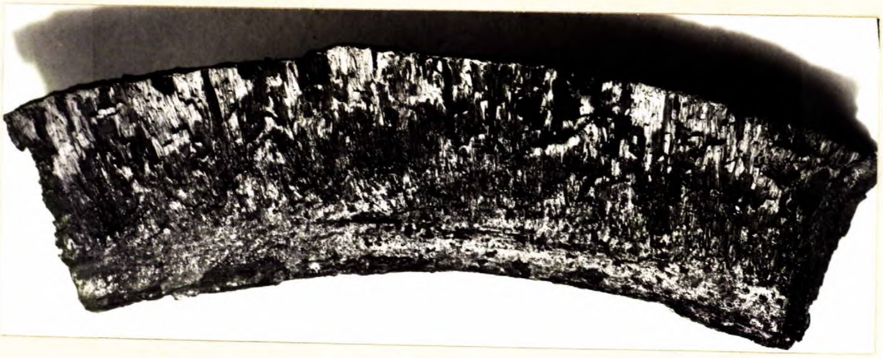
Figure 6.



SAMPLE NO. 2 AS RECEIVED.

Note "woody" type of fracture.

Figure 7.

SECTION OF TOP OF WELD OF SAMPLE NO. 1.

Note cracks in weld metal, extending down into barrel material. Dotted line is outline of weld. Note unfused root of weld.

Figure 8.

SECTION OF BOTTOM OF WELD OF SAMPLE NO. 1.

Note profusion of cracks in weld metal, crack in barrel material adjacent to weld, and unfused root at which a heavy crack originates. Dotted line is outline of weld.

(Approximately 1-1/2 times actual size).

Figure 9.

SECTION OF WELD OF SAMPLE NO. 2.

Note cracks in weld metal and adjacent metal. Dotted line is outline of weld.

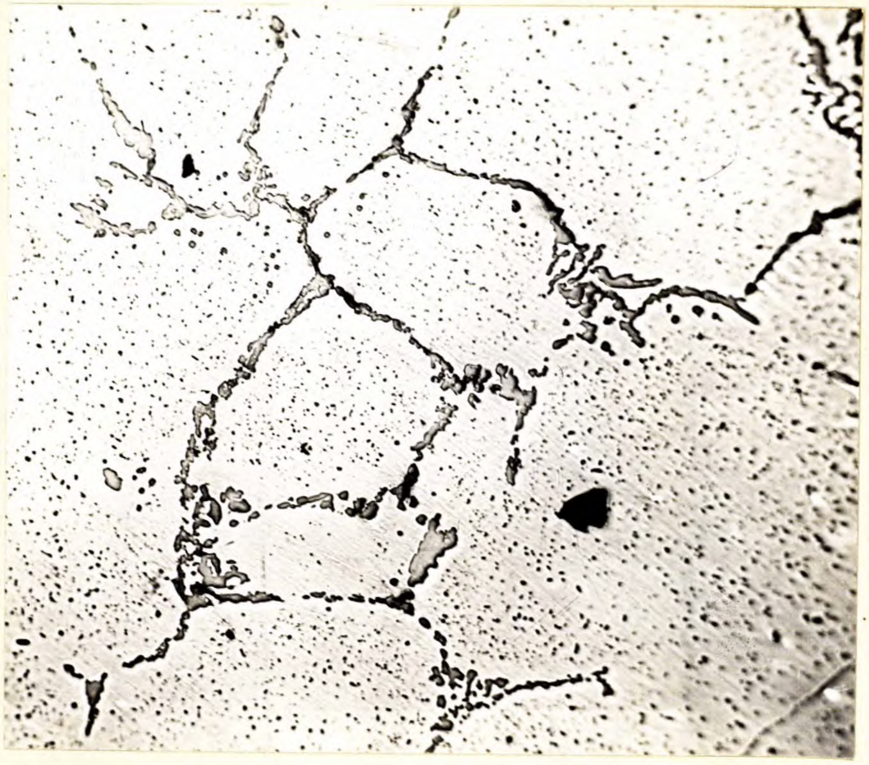
Figure 10.

X200, etched in 5 per cent HCl and
1 per cent picric acid in alcohol.

CONDENSER MATERIAL - SAMPLE 1.

Note fine carbides in grains and heavier carbides in grain boundaries.

Figure 11.



X200, etched in 5 per cent HCl and
1 per cent picric acid in alcohol.

RETORT MATERIAL - SAMPLE 1.

Note fine carbides in grains and heavier carbides in grain boundaries.

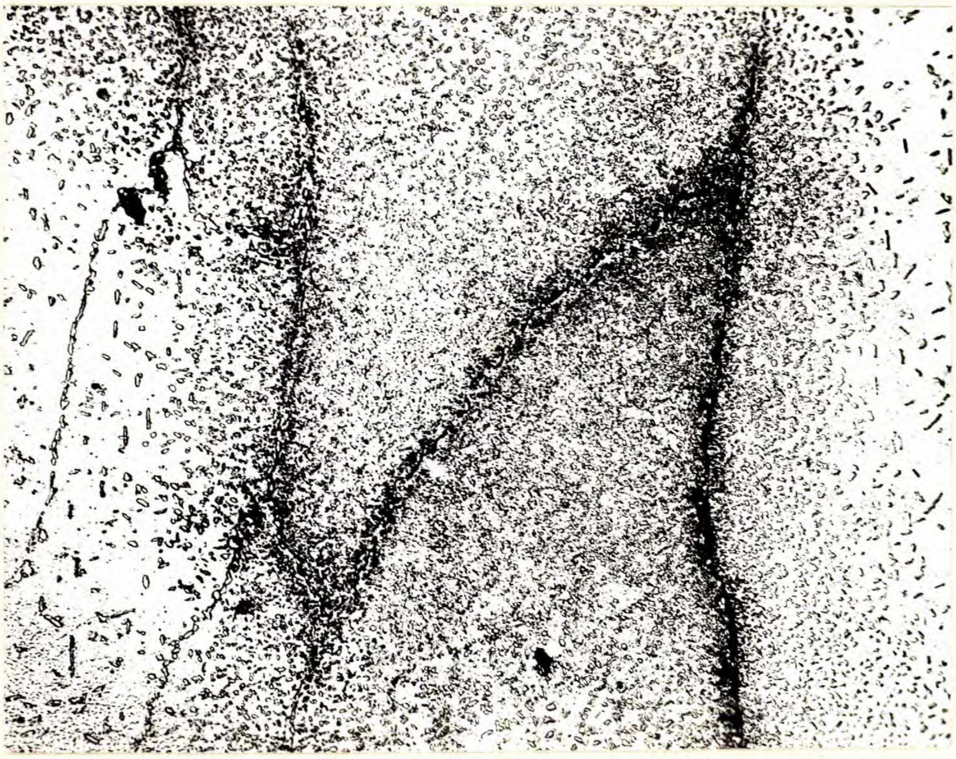
Figure 12.

X200, etched in 5 per cent HCl and 1 per cent picric acid in alcohol.

BARREL MATERIAL ADJACENT TO WELD - SAMPLE 1.

Note heavy carbides in grains and grain boundaries. Note cracks following grain boundaries.

Figure 13.



X200, etched in 5 per cent HCl and 1 per cent picric acid in alcohol.

ONE TYPE OF CARBIDE IN HEAVILY CRACKED AREAS OF WELDS.

Note fine carbides in grains and cracks following grain boundaries.

Figure 14.

X200, etched in 5 per cent HCl and
1 per cent picric acid in alcohol.

A SECOND TYPE OF CARBIDE FOUND IN HEAVILY CRACKED AREAS OF WELDS.

Note 90° orientation of carbides and cracks following grain boundaries.

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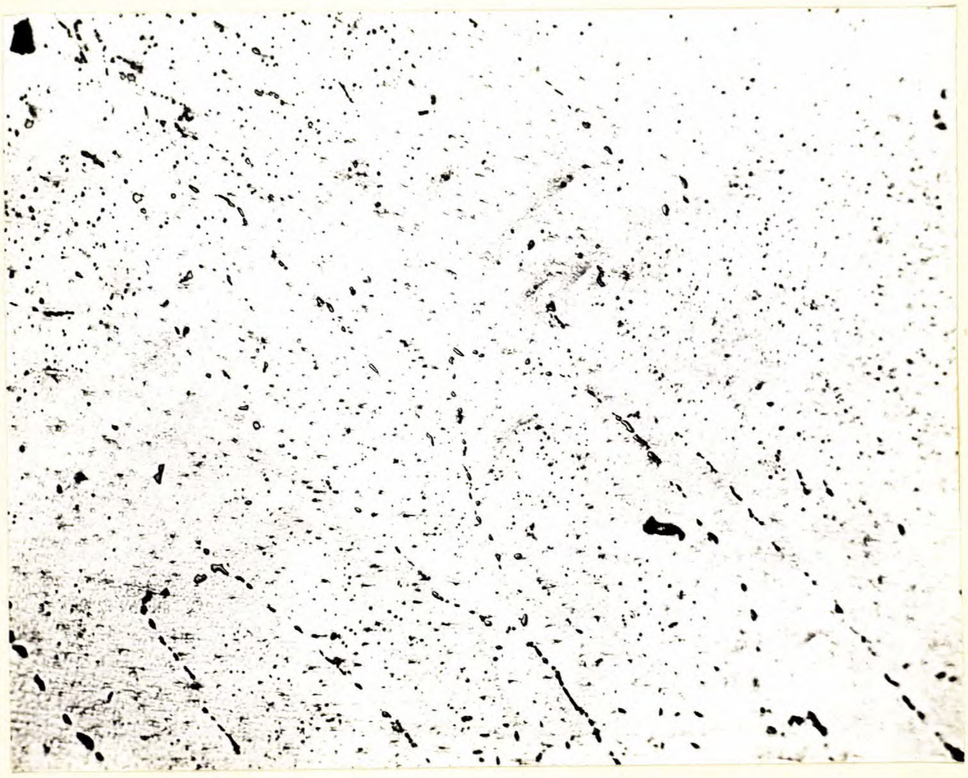
Figure 15.



X200, etched in 5 per cent HCl and
1 per cent picric acid in alcohol.

TYPICAL STRUCTURE OF UNCRACKED WELDS.

Note paucity of carbides.

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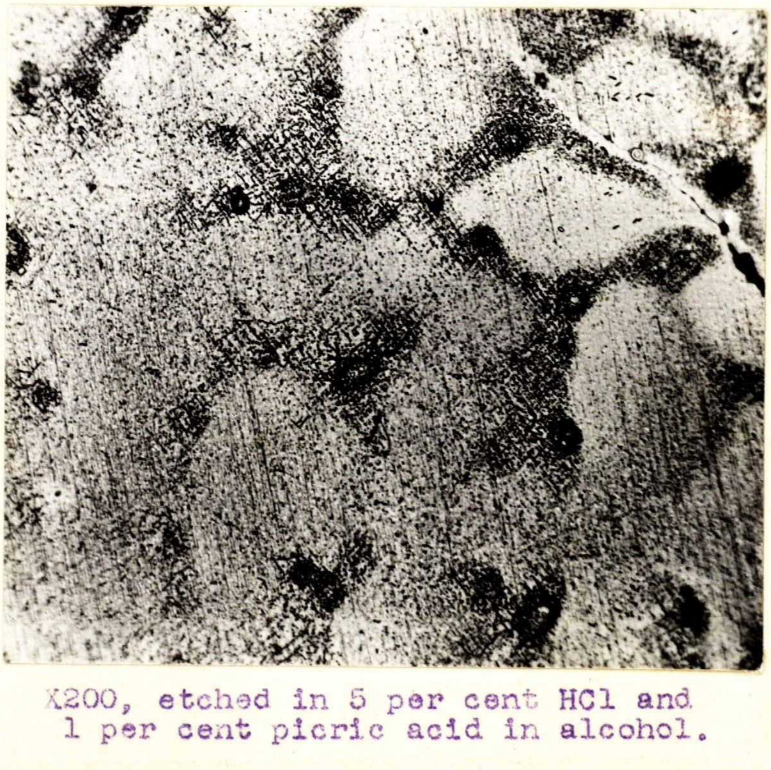
Figure 16.

X1000, etched in 5 per cent HCl and
1 per cent picric acid in alcohol.

STRUCTURE OF DEFORMED 35/15 MATERIAL AFTER
HEATING AT 1400° F. FOR 18 DAYS.

Note 90° orientation of carbide particles.

Figure 17.



X200, etched in 5 per cent HCl and
1 per cent picric acid in alcohol.

STRUCTURE OF UNDEFORMED 35/15 MATERIAL AFTER
HEATING AT 1400° F. FOR 18 DAYS.

Note that there is no preferred orientation
of carbide particles and that carbides
are migrating to grain boundaries.

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