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DEPARTMENT OF MINES AND TECHNICAL SURVEYS

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MINES BRANCH INVESTIGATION REPORT IR 58-193

# METALLURGICAL INVESTIGATION INTO THE FAILURE OF METAL PLATE FROM THE SIDE WALL OF AN AUTOCLAVE

by

**G. P. CONTRACTOR**  
PHYSICAL METALLURGY DIVISION

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METALLURGICAL INVESTIGATION INTO THE FAILURE OF  
METAL PLATE FROM THE SIDE WALL OF AN AUTOCLAVE

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SUMMARY OF RESULTS

Samples of badly damaged mild steel plate taken from the inside wall of a production autoclave were subjected to metallurgical examination to determine the cause of what was reported as crack formation. The samples did not show any evidence of the presence of cracks on the surface, or inside the metal. What appeared as cracks was the surface appearance produced by the slight undercutting of the plate metal by the rapidly agitating slurry in the autoclave.

The plate material was normal in regard to the chemical composition and microstructure, for a semi-killed steel.

The corrosion pattern on the plate surface and the presence of distorted grains of ferrite-pearlite in the pitted areas has led to the conclusion that the failure of the plate was caused by the combined action of pitting and erosion.

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\*Senior Scientific Officer, Physical Metallurgy Division, Mines Branch,  
Department of Mines and Technical Surveys, Ottawa, Canada.

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## INTRODUCTION

On September 24, 1958, three samples of steel plate were received at the Physical Metallurgy Division. The covering letter of the same date from Mr. A. Thunaes, Eldorado Mining and Refining Limited, Ottawa, Ontario, stated that the samples were from the walls of a leaching vessel (autoclave) which has been in operation for five years, and that fine cracks had developed in the area of the wall from which the samples were taken. It was requested that an investigation be undertaken to determine the cause of the crack formation.

An interim report in letter form was supplied to Mr. A. Thunaes on October 9, 1958.

## PROCEDURE

The following procedure was adopted for the examination of the samples.

- (a) Visual and magnaflux examination.
- (b) Chemical composition.
- (c) Hardness tests and metallographic examination.
- (d) Macro-etch.

(a) Visual and Magnaflux Examination

Figure 1 shows the condition of the samples as submitted by the client. The plate sample measured 5 in. square by about 7/10 in. thick. The other two samples were in the form of about 1 in. round lugs. The general surface condition of the plate was poor, being covered with tightly adhering product of corrosion and pitted areas with irregular and "curly" markings, indicating an advanced stage of corrosion. Figure 2 is a photograph of the plate after being sand-blasted and cleaned. It clearly shows the general pattern of corrosion.

Neither visual nor magnaflux inspection revealed the presence of cracks on the plate surface.

A close examination of the plate (see Figure 2) tends to indicate that the surface had undergone severe pitting erosion, a type of dynamic corrosion, in which the damage or the rate of corrosion is very rapid, resulting in unexpected deterioration of plant process equipment.

What appeared as fine cracks or crazing on the surface of the plate (see Figure 2) was in fact the surface appearance of the metal which had been slightly undercut by the turbulence of the slurry-charged liquid in the autoclave. This was confirmed subsequently by micro-examination of the metal and also by macro-etching of the section cut across the plate.

The corrosion pattern did not indicate any particular direction of the flow of liquid, but was indicative of a whirling agitation. There is little doubt that the corrosion pattern on the plate strongly suggests that abrasion conditions were present in the vessel, and that the rate and type of corrosion may have been affected by the quality of the plate material.

(b) Chemical Composition

The chemical analysis tabulated below shows that the plate was made of a semi-killed mild steel.

%		%	
C	0.17	Ti	<0.01
Mn	0.49	Ni	0.05
Si	0.02	Cr	0.15
S	0.031	Cb	Not detected
P	0.017	Sn	0.01 *

\* Spectrographic analysis

(c) Hardness Tests and Metallographic Examination

The average Brinell hardness of the plate was 115, the variation being in the range of 107 to 123. The average hardness is considered normal for the grade of steel under investigation.

Metallographic examination of the unetched microsections cut across the plate showed the presence of a fairly large amount of non-metallic inclusions (Figure 3) in the form of stringers of sulphides and oxides. The amount present, however, is the usual distribution found in commercial semi-killed steels. Segregation impurities or their solid solutions in steel as well as stress, either applied or residual, are notable for their ability to form anodic areas. In the present case, it is likely that the presence of the inclusions may be responsible, to some extent, for accelerating the rate of corrosion, and for giving rise to crack-like formation (crazing) on the badly pitted surface of the plate.

The microstructure of the plate was normal (Figure 4) and consisted of uniformly distributed grains of ferrite and areas of dark lamellar pearlite. Figures 5, 6, 7 and 8 show the cross-sectioned microstructure of the pitted surface and the metal underneath the

surface. It was observed that in general the pits (Figures 5 and 6) were of various sizes and shapes, and were randomly scattered.

One of the most significant aspects of microexamination was the evidence that displacement and distortion of ferrite grains (see Figures 5, 7 and 8) had taken place in the metal adjacent to the corroded surface. This is strongly suggestive of the mechanical nature of damage. In other words, it was obvious that the plate was subjected to the combined action of pitting and erosion corrosion.

(d) Macro-etch

A transverse section of the plate along AA-BB, Figure 2, was etched in 1:1 HCl at 165°F for about 30 minutes. The results (Figure 9) showed a clean, uniform structure without any evidence of piping, macro-segregation or the presence of cracks extending from the damaged surface to the interior.

#### DISCUSSION

Figure 8 clearly illustrates the mechanism of corrosion in the present case. It tends to indicate that the impact of rapidly agitating slurry<sup>\*</sup> in the autoclave has torn out a part of the plate, and has caused plastic flow or deformation of the metal grains. This cycle repeats many thousands of times. In case of relatively low strength metal, like the mild steel plate under investigation, erosion would be resisted for a period of time, but breakdown of large areas that acquire the appearance of pitting takes place followed by erosion if abrasive conditions are present, such as liquids moving at

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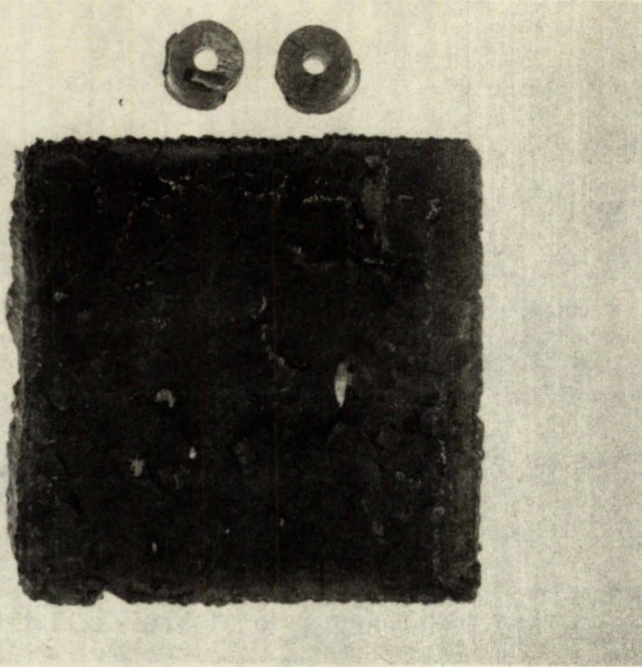
\*The slurry contained about 50 per cent solids by weight, and the solids consisted of uranium ore containing quartz, feldspar, hematite and lesser amounts of sulphides, pitchblende and chlorite.

substantial velocities, solid in suspension (i.e. slurries), marked turbulence, and impingement. Some of these conditions were certainly present in the autoclave. Furthermore, it is likely, as stated before, that the presence of non-metallic stringers (Figure 3) in the plate metal may have accelerated the rate of corrosion, and may have contributed to the crack-like (crazing) formation or pattern on the corroded surface.

#### CONCLUSIONS

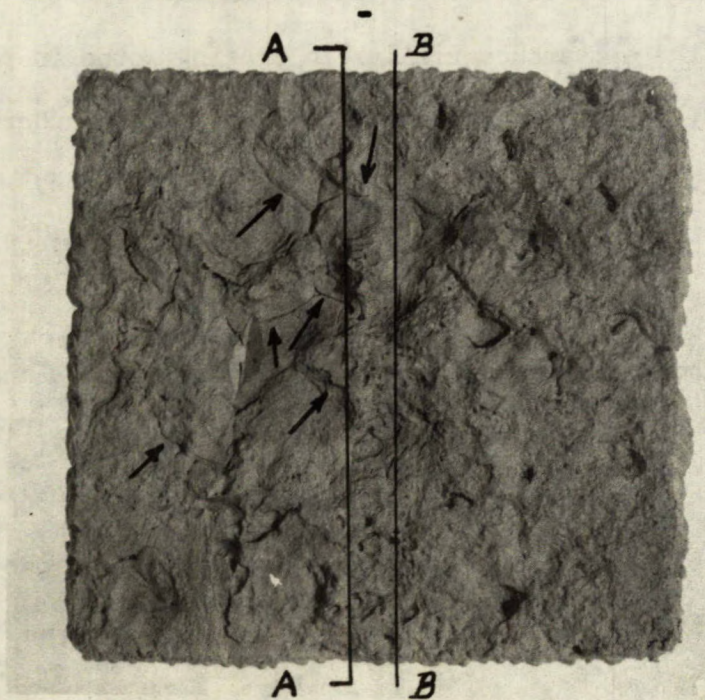
1. The damaged plate was made of semi-killed mild steel.
2. Neither visual nor magnaflux inspection revealed the presence of cracks on the plate surface.
3. The damaged surface of the plate was badly pitted and showed a fine crack-like appearance similar to crazing found in pottery. The crack-like formation is attributed to the slight undercutting of the metal by the fast-moving slurry in the autoclave.
4. The microstructure of the plate was normal for a semi-killed mild steel.
5. Both the corrosion pattern on the plate surface and the presence of distorted grains of ferrite-pearlite in the pitted areas strongly suggest that abrasion conditions were present in the autoclave, and that the failure of the plate was caused by the combined action of pitting and erosion.





(Approx.  $\frac{1}{2}$  actual size)

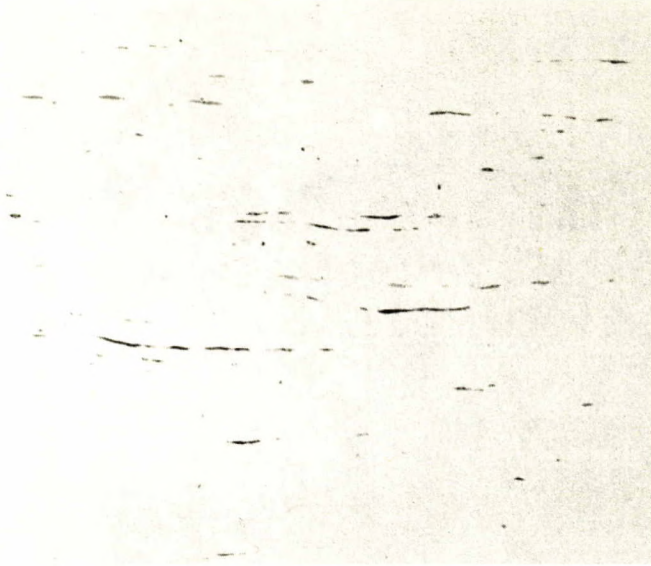
Figure 1. - Photograph of the samples of plate and lugs in the as-received condition.



(Approx.  $\frac{3}{5}$  actual size)

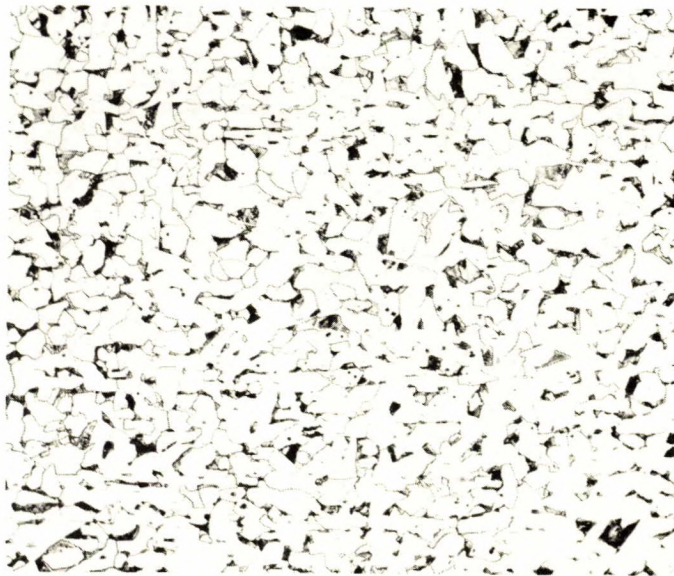
Figure 2. - Photograph of the plate after being sand-blasted and cleaned. Note the presence of what appeared to be cracks (arrows), and the areas of large and small corrosion pits.





(Unetched; magnification X100)

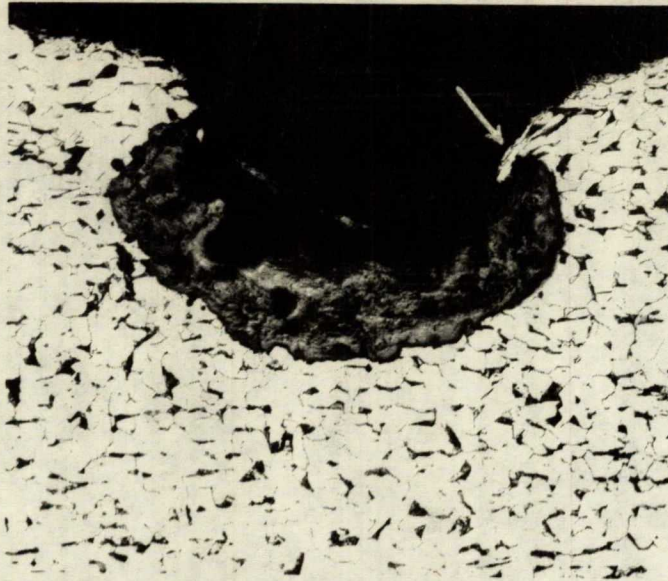
Figure 3. - Photomicrograph of the plate metal showing stringers of non-metallic inclusions.



(Etched 2% nital; magnification X100)

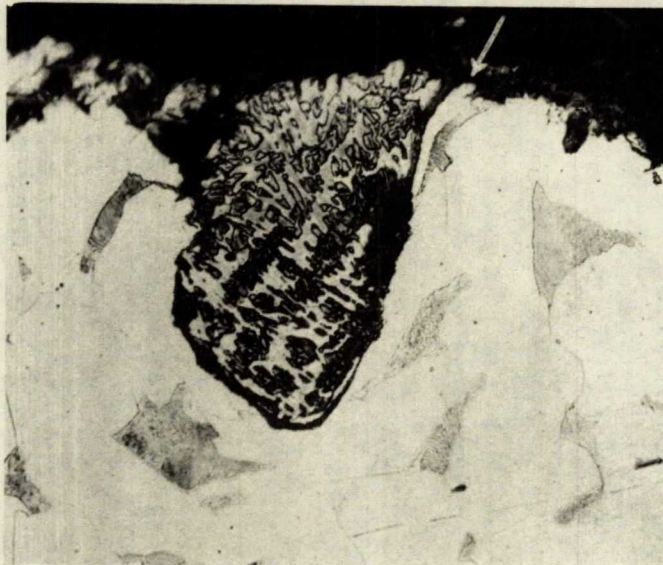
Figure 4. - Microstructure of the plate metal showing grains of ferrite (white) and pearlite (dark).





(Etched 2% nital; magnification X100)

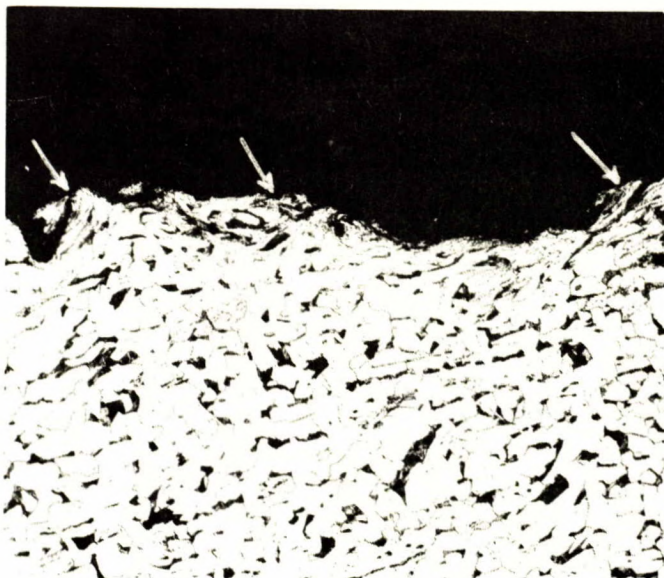
Figure 5. - Photomicrograph showing a typical corrosion pit. Note the presence of distorted grains of ferrite (arrow).



(Etched 2% nital; magnification X500)

Figure 6. - Photomicrograph showing a very fine pit filled with either slurry or corrosion product. Note the distorted ferrite grain (arrow).





(Etched 2% nital; magnification X100)

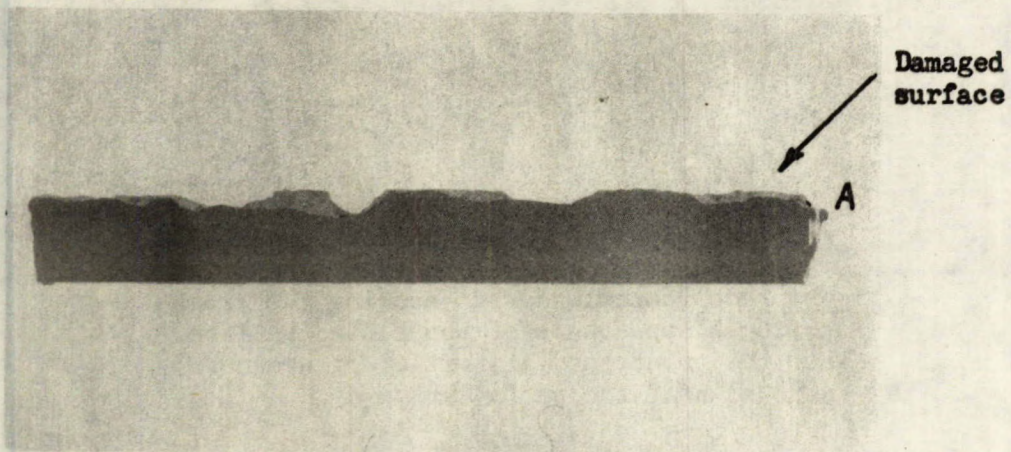
Figure 7. - Photomicrograph showing the cross-sectional appearance of the pitted surface. Note the pronounced distortion of grains (arrows) near the surface.



(Etched 2% nital; magnification X500)

Figure 8. - Photomicrograph showing the tearing of surface metal and also how it is deformed by the impact of agitating slurry. Arrows indicate distorted metal.





(Approx. 4/5 actual size)

Figure 9. - Transverse section along AA, Figure 2, deep-etched in 1:1 HCl. Note the absence of piping, macro-segregation or crack formation from the surface to the interior.

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