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DEPARTMENT OF MINES AND RESOURCES

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Ottawa, October 10, 1946.

R E P O R T

of the

ORE DRESSING AND METALLURGICAL LABORATORIES.

Investigation No. 2126.

(Subsequent to Investigation Report)
(No. 2119, dated October 10, 1946.)

Metallurgical Examination of a No. 2 Catalyst Steel Column used for Ammonia Synthesis.

PART II. - Microscopic Examination.

(Copy No. 13.)

Bureau of Mines
Division of Metallic
Minerals

Physical Metallurgy
Research Laboratories

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Origin of Material and Object of Investigation:

In March of 1946, in the course of a visit, Mr. A. H. W. Busby, Superintendent of Physical Research, The Consolidated Mining and Smelting Company of Canada Limited, Trail, British Columbia, requested investigative work on a No. 2 Catalyst steel column which had failed in service while being used in the synthesis of ammonia. During the period from April to July, further information was supplied by the company on the service conditions encountered by the column.

Part I of this investigation (Investigation No. 2119, October 5, 1946) covered the mechanical and metallurgical examination of samples cut from the column.

Microscopic Examination:

A microscopic examination was made on two small samples which had been cut from the diffused copper and steel area of the lead-in port. The smaller of the two samples had been left at the Physical Metallurgical Research Laboratories by Mr. Busby, and the larger had been submitted by mail.

Samples were also cut from one of the pieces of the column forwarded by freight, and these were examined under the microscope.

Figure 1, taken at X100 magnification, shows the microstructure of the steel in the column. Microscopic examination of samples cut from various localities in the section failed to disclose any variation in the microstructure, and Figure 1 may be considered representative of the steel used in the column.

Figures 2 to 7, inclusive, are taken from the small sample of the diffused copper and steel area of the lead-in port. Figure 2, taken at X100 magnification, shows the copper (light) and the copper-steel area (dark). The copper which has formed a solid solution with the steel, causes it to etch up very dark.

Figure 3 shows large cracks in the copper-steel zone.

Figures 4 and 5 show cracks in the copper-steel zone extending into the heat-affected steel areas.

Figure 6, taken at X500 magnification, illustrates the structure of the copper-steel zone, consisting of undissolved copper in a background of martensite.

Figure 7 shows the heat-affected zone immediately adjacent to the copper-steel area.

(Continued on next page)

(Microscopic Examination, cont'd) -

Figure 8 illustrates typical manganese sulphide inclusions found in the steel. These inclusions, although large in size, are to be expected in a casting of this size.

(Figures 1 to 8 are)
(at end of report.)

Hardness Tests:

Hardness readings were made on the samples submitted, using the Tukon microhardness tester. Readings were taken in the copper-steel area as well as in the heat-affected zone. The results are as follows:

	Hardness Knoop	Hardness Rockwell "C" (Converted)
Steel-copper zone -	407 to 640	40 to 56
Heat-affected steel zone -	407 to 554	40 to 51

Discussion:

The coarse-grained structure of the steel in the column (see Figure 1) is to be expected for a casting of this size, since the deformation ratio resulting from the forging operation is comparatively small and very little breaking up of the original cast austenite grain had actually taken place. Because of this, and also because of the very long soaking time in the annealing operation, the refinement of the grains would not be very complete.

The microscopic examination proves very conclusively the existence of numerous cracks in the diffused copper-steel areas as well as in the adjacent heat-affected zone which extends to a depth of approximately 5/8 inch around the lead-in port. The penetration of the copper into the steel and the formation of a hard, brittle, crack-filled alloy leaves very little reason to doubt this as being the source of the

(Discussion, cont'd) -

failure of the column in the hydraulic test. (According to the A.S.M. Handbook, 1939, the maximum solubility of copper in alpha iron at 1560° F. is 1.4 per cent. The solubility drops down to 0.35 per cent at 1200° F., where it remains constant. Hence, the copper-rich epsilon phase is nearly always present at room temperature.)

The sudden formation of a great quantity of localized heat (at least 1060° C. is necessary to melt the copper), resulting in the plastic deformation of the adjacent steel area, followed by a cooling period, must have resulted in the formation of thermal stresses of very great magnitude, in a range from 150,000 to 210,000 p.s.i. in tension. This hypothesis can be easily verified by simple calculation.

Thermal stresses built up around the electrical lead-in port may be calculated with an approximation by several different ways. The simplest method uses Lame equations, with the assumption that the decrease (d^1) of the inner radius (b'') of the outer cylinder (i.e. material not affected by heat) plus the increase (d'') in the outer radius (b'') of the inner cylinder (i.e. material affected by heat) produced by a negative pressure, assumed to be acting on the radius (b'') marking the boundary of zones affected and not affected by heat should be equal to d the thermal expansion of b'' by heating up to t°C.

On the above basis the following equations may be used:

$$d^1 = \frac{b''p}{E}\left(\frac{b''^2+C^2}{C^2-b''^2}+\mu\right)$$

C = for simplification, assumed very large as compared to b''

$$d^1 = \frac{b''p}{E}(1+\mu)$$

$$d'' = \frac{b''p}{E}\left(\frac{a''^2+b''^2}{b''^2-a''^2}-\mu\right)$$

$$d^1+d'' = d = tnb''$$

$$p = -\frac{tnE(b''^2-a''^2)}{2b''^2} = -75{,}300 \text{ p.s.i.}$$

t assumed 500°C.

The thermal stress on surface a'' internal side of lead-in port:

$S_{tangential(r=a'')}$ = 210,300 p.s.i.

(Discussion, cont'd) -

The assumption that the above-named factors are to be blamed for failure is also supported by the fact that the crack occurred through the strongest section of the flange where the inside diameter is 750 mm. and outside diameter is 1,670 mm. as compared to the central portion of the column where the outside diameter is 1,170 mm.

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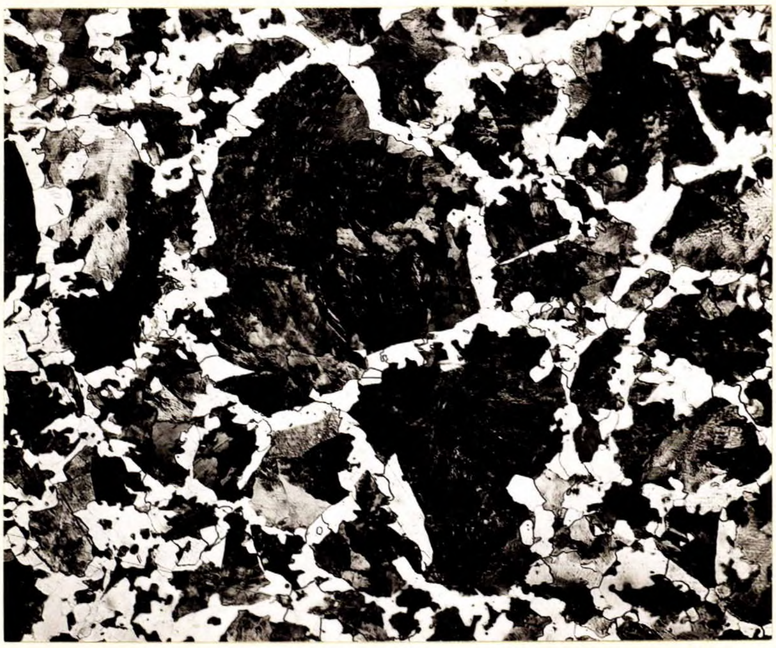
(Figures 1 to 8 follow,)
(on Pages 6 to 9.)

Figure 1.



X100, nital etch.

MICROSTRUCTURE OF THE FAILED AMMONIA STEEL COLUMN (ANNEALED).

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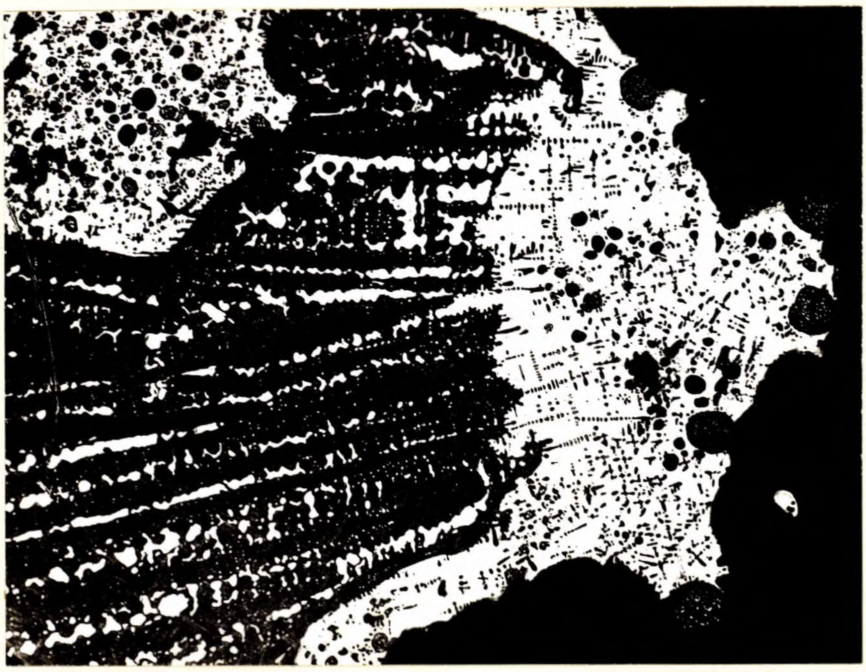
Figure 2.



X100, nital etch.

GENERAL VIEW OF DIFFUSED COPPER (LIGHT) AND COPPER STEEL (DARK) AREA OF THE ELECTRICAL LEAD-IN PORT.

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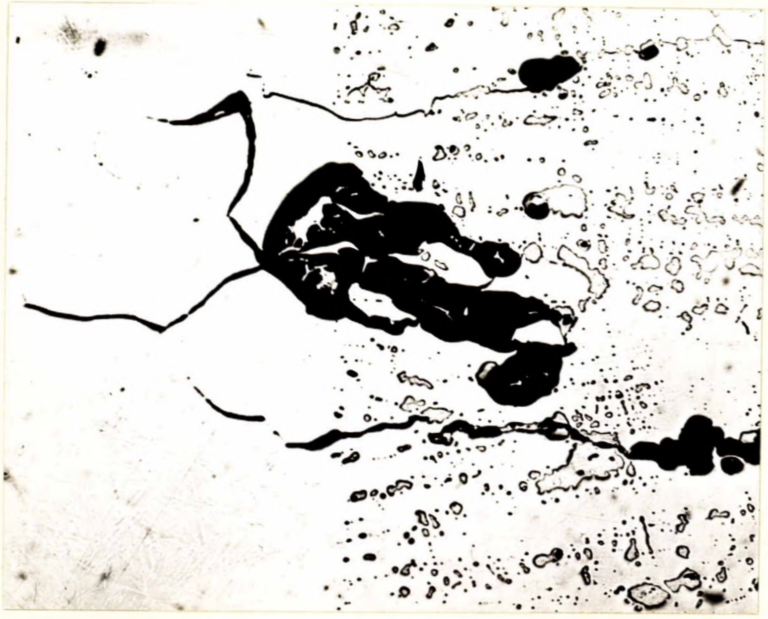
Figure 3.

X100, unetched.

LARGE CRACKS IN THE COPPER-STEEL ZONE.

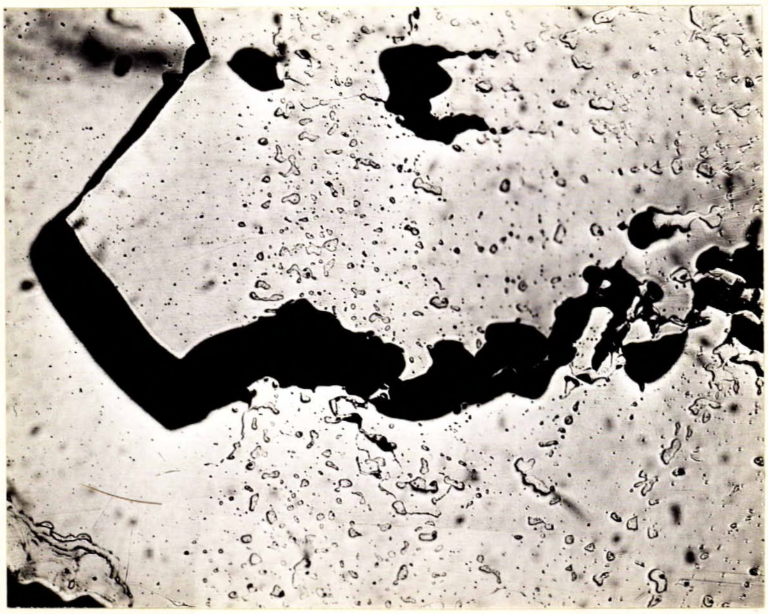
Figure 4.



X100, unetched.

Note continuation of cracks from copper-steel zone (right) into steel area (left).

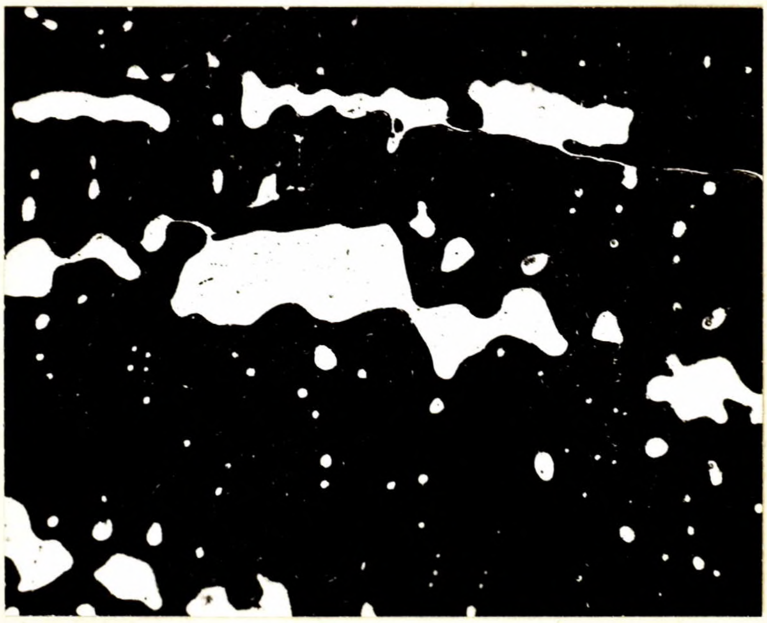
Figure 5.



X100, nital etch.

Note continuation of cracks from copper-steel zone (right) into steel area (left).

Figure 6.



X500, nital etch.

COPPER-STEEL ZONE SHOWING UNDISSOLVED COPPER (WHITE) IN A BACKGROUND OF MARTENSITE (DARK).

Hardness, 40-56 Rockwell "C".

Figure 7.

X100, nital etch.

HEAT-AFFECTED STEEL PORTION IMMEDIATELY ADJACENT TO THE COPPER-STEEL ZONE AND PROBABLY CONTAINING SOME DISSOLVED COPPER.

Note large grain size.
Hardness, 40-50 Rockwell "C".

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Figure 8.

X100, unetched.

SHOWING TYPICAL LARGE INCLUSIONS, PROBABLY MANGANESE SULPHIDE.

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