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METALLURGICAL EXAMINATION OF DEFECTIVE EXTRUDED SOLDER

by

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SUMMARY OF RESULTS

Samples of defective extruded solder and of the extrusion billets were submitted for examination.

The samples of extruded solder exhibited prominent blisters and laminations. Dark grease-like stains were found between some laminations. Analysis of the gas in a large blister indicated that it was primarily air.

Careful examination of samples of the multiple-pour billets showed laps and oxide inclusions between the different layers and also inclusions and shrinkage porosity within the billets. These could cause the types of defect observed, and were probably exaggerated by extrusion troubles such as entrapped air and organic dressing, extruding to too high a yield, insufficient cleaning of the container, etc.

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(11 pages, 7 illustrations)

INTRODUCTION

On July 30, 1959, a letter and two parcels containing defective extruded solder were submitted by Mr. D.K. Faurschou, B.C. Research Council, Vancouver 8, B.C.

Mr. Faurschou stated in his letter that the solder was provided by The Canada Metal Co. Ltd., 1428 Granville Street, Vancouver, and that the manufacturers had been troubled with "double skin" (laminations) and blister defects in the extruded sections. Mr. Faurschou said, further, that prior to extrusion, the solder was cast into billets 6 inches in diameter x 24 inches long and about six ladles of metal were used to pour each billet.

The samples included eight extruded pieces of 70-30 solder identified as exhibits No. 1 to No. 8, one extruded piece of 60-40 solder identified as exhibit No. 9, and eight pieces of ingot identified A1, A2, A3, B3, C3, ABC4, A5 and C5. Some of the extruded samples had been returned by a customer because of unsatisfactory performance.

Mr. Faurschou requested that the Non-Ferrous Metals Section examine the solder to determine why the double skin and blisters occur, and to suggest how the defects may be avoided.

VISUAL EXAMINATION

The samples of extruded solder exhibited prominent blisters and in some areas the defect was so deep seated that no blister formed but there was definite separation, causing the defect known to the manufacturer as "double skins", (see Figures 1 and 2). In some instances dark grease-like stains were seen between the two layers of metal and the interface was definitely oxidized. The dark stains dissolved readily in benzene.

The outside skins on the extruded solder varied in thickness from a very thin layer to a very heavy layer which constituted almost half of the extruded cross-section of the solder (see Figure 3).

On the samples from the billets, folds and voids in the metal were visible on sections from both the inside and the outside. In addition, large dross inclusions were often associated with these laps. It was apparent that the laps were generated by pouring the metal in successive batches rather than continuously, and that there was at least partial freezing of the surface of one batch before the next batch was poured. The dross inclusions appeared to be concentrated at the surface of the billet, and may be due to insufficient skimming of the metal surface in the mould, or may be generated by turbulent pouring of the next batch of metal, the dross thus generated being trapped at the interface. These defects were particularly evident at the top of the billet where apparently feed metal had formed a number of well defined layers, each with an interface. These defects were not apparent on the samples as supplied but could be clearly seen on bending the samples (see Figure 4). Some shrinkage porosity and dross was also found on the interior of the billet by this method.

ANALYSIS OF GAS IN BLISTERS

Unlike aluminum and copper, lead dissolves very little hydrogen in the liquid state and hence gas porosity from this source is almost unknown in lead alloys. The presence of large gas blisters in the solder therefore strongly suggested that air was entrapped, either during pouring the billet or in extrusion.

The gas from an unusually large blister was collected in a capillary tube under water. The length of the column of gas in the tube was used as a measure of the initial volume of gas and pyrogallol solution was injected into the capillary. Pyrogallol dissolves oxygen and turns red in the process so that a qualitative and semi-quantitative test for oxygen was obtained by measuring the length of the gas column after contact with pyrogallol.

The pyrogallol became deep red in colour indicating the presence of oxygen while the gas column contracted about 40 percent indicating 40 percent by volume oxygen with or without carbon monoxide, carbon dioxide or hydrogen sulfide. The remaining 60 percent of the gas was inert to pyrogallol and very probably was nitrogen. The accuracy of the quantitative measurements was only fair, and this experiment suggests that the gas in the blister was essentially air, although the indicated oxygen:nitrogen ratio was somewhat greater than the 1:4 normally expected in air.

MICRO EXAMINATION

Specimens of the extruded and ingot solder were mounted and polished. Examination of the polished sections indicated that the outer skins adhered to the underlying metal in some areas and in other areas was quite non-adherent (see Figure 5).

A micro section of the metal just under the ingot surface showed that rough surface defects, indicative of turbulent pouring, and oxide skins extended into the ingot (see Figure 6). Similar defects were found in the extruded sections (see Figure 7).

DISCUSSION

From the general appearance of the samples of defective solder, it is apparent that a number of factors could be contributing to the problem. The defects could originate in the ingot or in the extrusion process.

Considering the ingot samples first, bonding of the successive metal pours was not complete and voids and oxide inclusions in the ingot were associated with folds on the ingot surface. These defects could be minimized by reducing the number of successive pours per ingot, by careful skimming, and by taking particular care to avoid turbulence when pouring. Shrinkage cavities, open to the atmosphere in the top of the ingot, could account for some of the bubbles in the extruded solder. With a low melting point material, such as solder, these could easily be eliminated by some form of "hot top" or heater to ensure directional solidification. In addition, it is apparent that the heavily lapped top of the billets should be cut off before extrusion, if hand feeding is continued.

In the process of extrusion there are several very likely sources of the defects shown by the samples. It is considered very probable that the blisters on the solder contain air. Excessive clearance between the ingot and the container walls could allow air to enter and become entrapped in the container and finally be forced into the extrusions to form blisters.

There is very strong evidence that the blisters and double skins are a result of butting a new ingot up to the end of the previous one which may have become concave in the final stages of extrusion, if too high an extrusion yield has been sought. When the new ingot is pushed into the hollow, air is trapped between the two layers of metal, which may only partially weld, and the result is a double skinned and blistered bar.

The dark grease-like stains between the metal layers may be mould dressing, dressing from the die, container, dummy block, etc., or grease accidentally dropped on the billet. Using semi-continuous extrusion without butt end discard, this defect would probably occur at the beginning of a new ingot and, hence, at the end of the previous ingot. The central core of solder, shown in Figure 3, was impregnated with the grease-like material. These, and similar inclusions would account for the poor workability of some of the solder.

In order to decrease the amount of air between the ingots and the container walls, it is suggested that the ingot be introduced with a temperature gradient so that the hot end of the ingot is against the die. When the ingot is subjected to pressure, the metal will upset from the die toward the dummy block and the air will be displaced in the same direction.

If successive ingots are extruded, care should be taken not to over-extrude any ingot, and excessive use of organic dressings should be avoided. Depending on the type of die used, extrusion ratio, etc., it should be possible to establish tables for good extrusion practice which the operator can follow. It is also advisable to clean out containers regularly to ensure that there is no excessive build-up of surface oxides on the container. This can readily be accomplished by the periodic use of a cleaning ring on the end of the ram.

Although it is difficult to make an accurate assessment and recommendations at long range, it would seem that small isolated blisters are probably associated with casting practice and are probably due to small inclusions, turbulent pouring, etc. Large isolated blisters which are relatively clean may be due to shrinkage pipes in the ingot (these alloys should not be particularly prone to inter-dendritic shrinkage because of their constitution). These may have bridged over, and so may not be obvious from a superficial examination of the billet.

Any coring of an extrusion, in which the central area is totally or partially separated from the surface, is almost always associated with poor extrusion practice although, in some instances, extensive surface laps in the ingot may be responsible. The repeated occurrence of chains of blisters, and seams with dirty oxidized interiors is, again, usually associated with poor extrusion practice, although billets with gross oxide and dross inclusions can give similar defects. In general, defects associated with extrusions are in a definite plane in the extrusion, and show continuity over a considerable length. Defects from poor billets tend to be distributed more uniformly through the cross-section and to be more sporadic in occurrence.

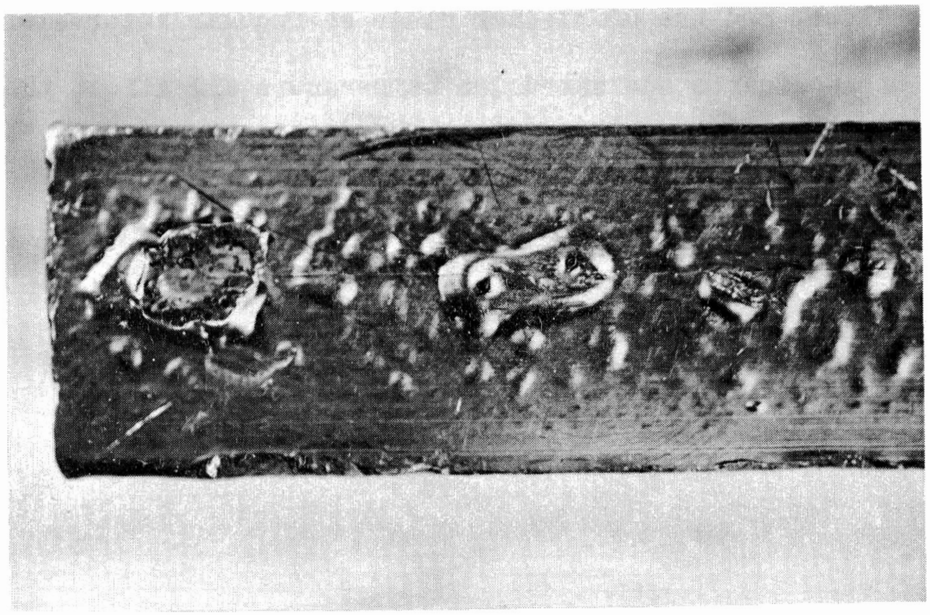
CONCLUSIONS

Of the defects examined in the solder bars, some came from the billets, whereas others were definitely associated with extrusion.

It is recommended that billet quality be improved by the adaption of melting or holding equipment so that a complete billet can be poured in one operation. Also, care should be taken that the metal is cleaned and skimmed before pouring and that pouring should be without turbulence. Some form of hot top to ensure the absence of shrinkage porosity is also recommended, and it may be necessary to cut off and discard the feed head of each billet.

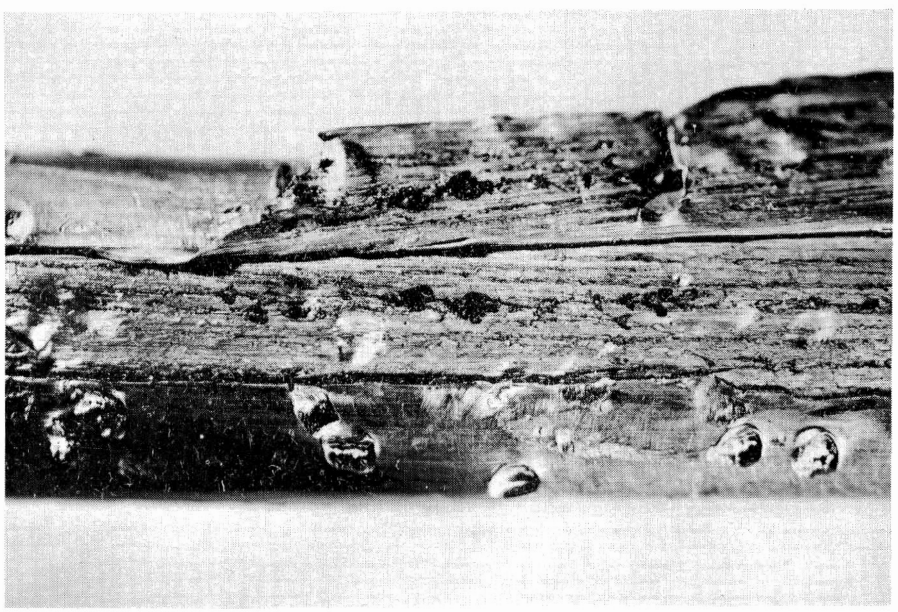
Improvement in extrusion practice can probably be effected by attention to such details as container and billet clearance, extrusion yield of billets relative to various dies, gradient heating of billets, and use of cleaner rings at regular intervals. Die design, use of lubricants and extrusion temperature all affect the flow of metal in the container and hence the structure of the resulting extrusion.

GWT/JOE/RJB



(X3)

<u>Figure 1</u>. - View of exhibit No. 1, 70-30 extrusion, showing large blisters.



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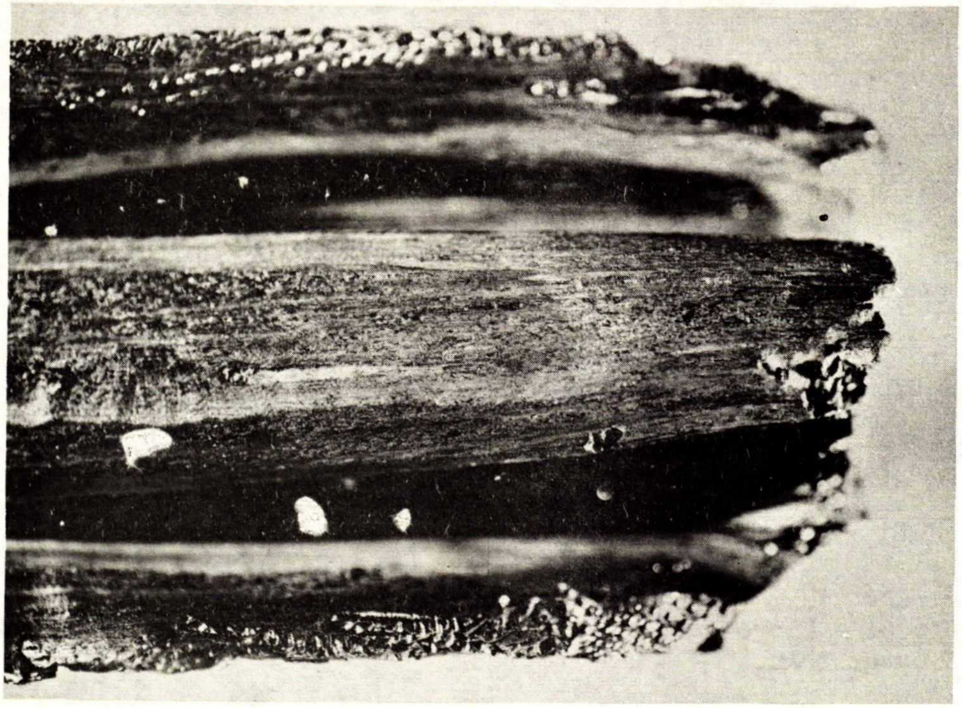
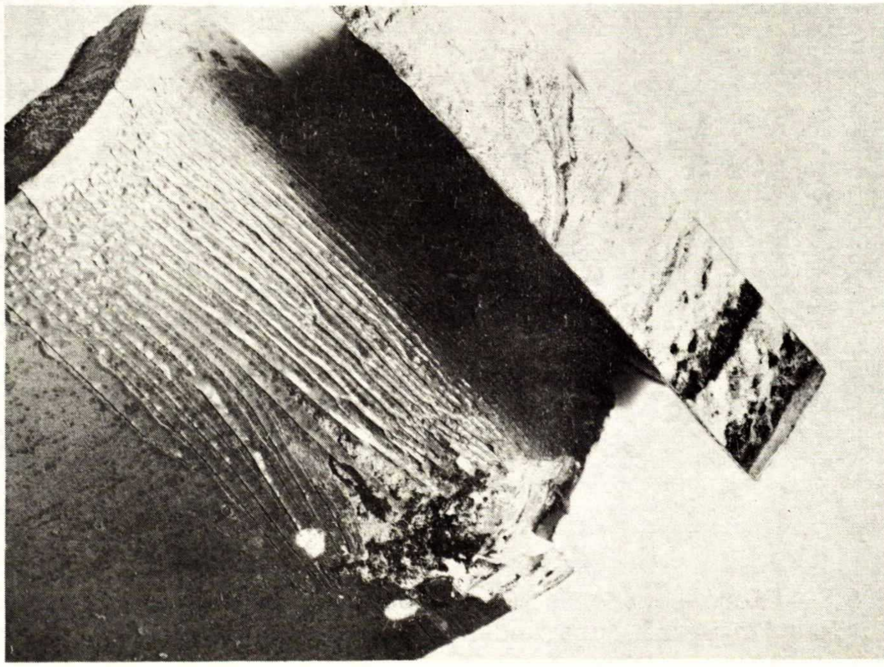
<u>Figure 2</u>. - View of exhibit No. 9, 60-40 extrusion. The outer skin has been bent away from the central core and dark stains are shown at the interface.



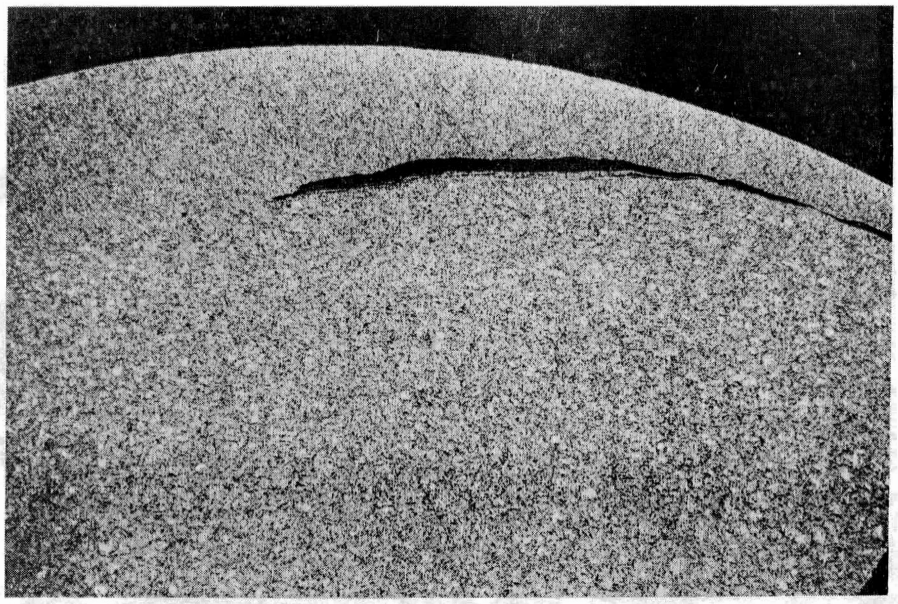
(X3)

Figure 3. - View of exhibit No. 9, 60-40 extrusion. Note heavy outer skin. The inner core of this sample was impregnated with a black grease-like material.



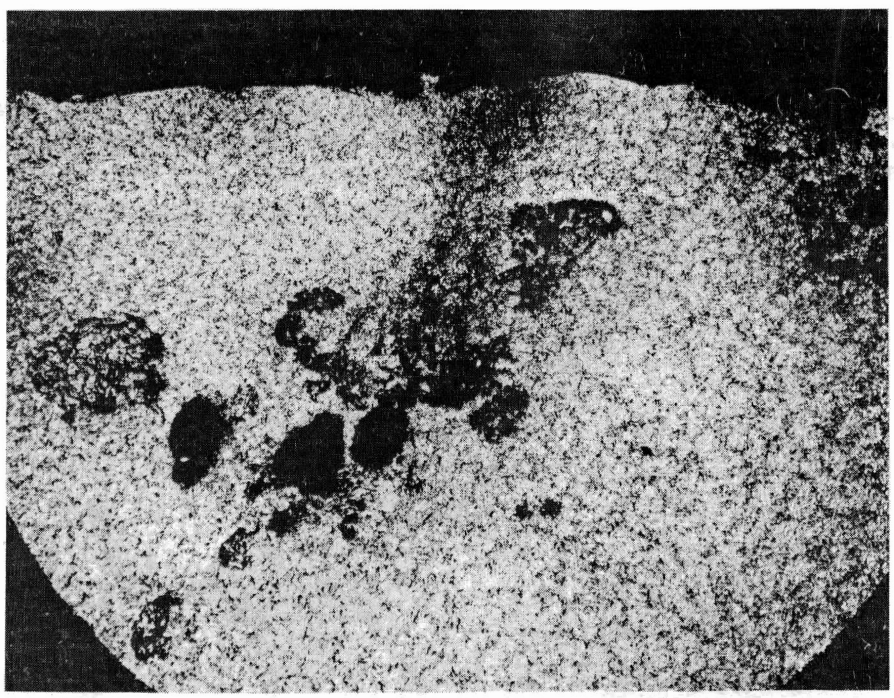
(X2)

Figure 4. - Billet sample bent at surface lap showing depth of unbonded area and presence of inclusions. Note clean unbonded surface, from between successive pours, shown on upper billet sample.



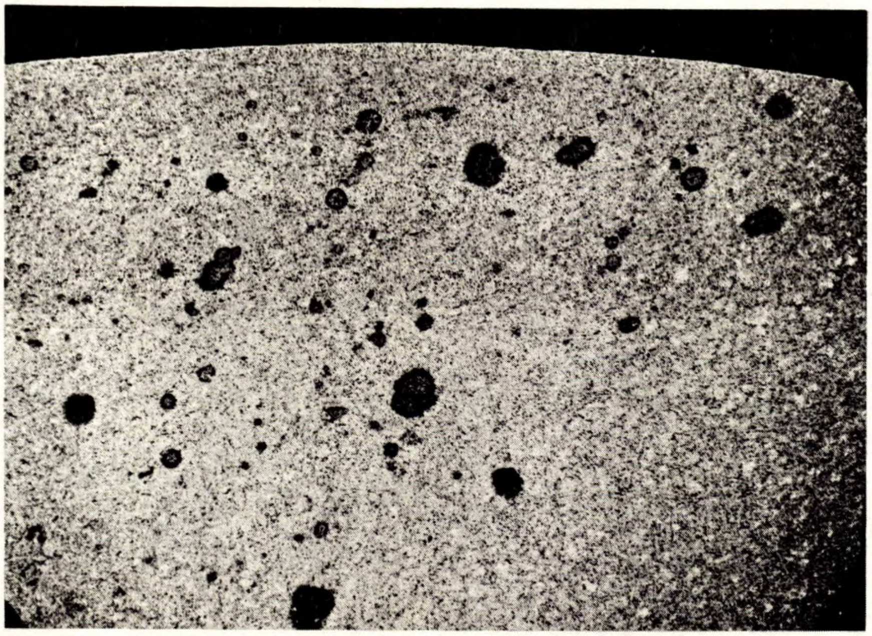
(X36)

Figure 5. - Micro-section of exhibit No. 9. Note only partial welding of outer skin.



(X36)

Figure 6. - Micro-section of ingot sample A5, showing voids and inclusions in metal just under the folded billet surface.



(X36)

Figure 7. - Micro-section of exhibit No. 2, showing voids and inclusions in the extruded section.