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PROPOSAL: DIRECT INGOTING OF UNPICKLED BUTTON
FOR ECONOMIC AND PRODUCT QUALITY GAINS

AUTHOR

R. H. Bond

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PROPOSAL: DIRECT INGOTING OF UNPICKLED BUTTONS
FOR ECONOMIC AND PRODUCT QUALITY GAINS

Classification Cancelled and Changed To

By

R. H. Bond

Plutonium Chemistry Laboratory
Research and Engineering Operation
Chemical Processing Department

HANFORD ATOMIC PRODUCTS OPERATION
RICHLAND, WASHINGTON

September 21, 1964

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PROPOSAL: DIRECT INGOTING OF UNPICKLED BUTTONS
FOR ECONOMIC AND PRODUCT QUALITY GAINS

I. INTRODUCTION

The final product from the Reduction Operation at the present time is an unalloyed button of reasonably high purity and density. Considerable effort is being directed toward improvement of button purity and surface finish, or appearance. A direct ingoting step is proposed in place of button pickling as a practical means of improving metal quality and appearance. A second ingoting step is proposed to further improve process economics through improved radiographic integrity.

II. PROCESS PROPOSAL--DIRECT INGOTING

Ingoting of buttons immediately following reduction appears to be a more direct and practical approach to definition and improvement of metal purity and improvement of product appearance.

The proposed process change places button ingoting within the scope of the Reduction Operation. The final product from the Reduction Operation will then be an ingot of uniform composition (no segregation), reliable density and plutonium content (no slag inclusions or voids).

The ingot could be subdivided in this operation, ready for Foundry use; or it could be cast as an ingot of assured quality for off-site shipment.

III. DETAILS OF PROPOSED PROCESS

1. No change of button-breakout procedure is anticipated. The presence of a calcium cap or adhering slag and crucible on the button should cause no difficulties in subsequent processing.
2. Pickling will be eliminated, if possible. (Pickling is believed to be unnecessary in the proposed process, but verification will be necessary.)
3. The crude button will be heated in a tantalum crucible in a simple ingoting stack to separate the pure metal from button surface materials. Two variations are possible for this step:
 - a. The pure metal can be dribble-poured, for maximum purity, at a crucible temperature not exceeding the metal's melting point by more than 10 C. The ingot so obtained would show some bottom-to-top impurity variation.
 - b. The pure metal can be plug-poured (chill-cast), for uniform composition of the ingot and optimum ingot appearance, at a crucible temperature not exceeding 700 C.

The melting operation, as described, should leave an unmelted, button-surface shell of impure metal, more or less equivalent to the surface removed by pickling in the present process. The calcium cap should remain with the button residue. (Calcium melts at 810 C) The bell jar atmosphere for this operation should be inert gas (argon or helium) at a low vacuum (ca. 5" Hg) to avoid vaporizing calcium.

The ingot from this step is free of the calcium cap, surface or occluded tramp materials (slag, crucible), and much segregated impurity (nitride, oxide). It still contains much volatile metal.

The ingot may be loaded out in this form, as the final Reduction Operation product, having better purity and appearance than a pickled button. Alternatively, this ingot may be processed through a second, higher-temperature step for removal of volatiles and further improvement of metal quality.

IV. ANTICIPATED GAINS OF PROPOSED PROCESS

The direct ingoting process offers the following potential gains:

1. Elimination of button pickling.
2. Improvement of metal quality, because of the initial low-temperature separation of the metal from button-surface impurities and occluded impurities. Low-temperature pour assures maximum retention of suspended material in the skull. Initial high-temperature ingoting (present procedure) dissolves and blends many of the trace impurities that may be present.
3. Improvement of product appearance. Present efforts to improve button-surface appearance by polishing would become unnecessary. (The extra cost of polishing would be avoided.) The product would be an ingot having a smooth surface and uniform shape.
4. Subdivision for blending. The metal from this first ingoting operation could be poured as segments for blending in FAB.
5. Improvement of analytical reliability. Button sampling at present is based on sampling alternately from the top of one button and bottom of the next one. This should give a statistically high impurity value, because a bottom sample should be nearly representative of the whole button, whereas a top sample should be appreciably higher in some impurities than the whole button. A single sample from the uniform ingot should show a statistically lower impurity level, even if a true improvement of purity had not been obtained through ingoting. A true improvement of purity should be obtained, in addition to the statistically apparent improvement.

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6. Improvement of density reliability. A button is produced occasionally which is poorly segregated or consolidated. The button density is appreciably lower than the true metal density because of the presence of slag inclusions, or voids. Routine ingoting prior to density determination would eliminate doubt as to correctness of metal density.
7. Accountability improvement. The first-step ingot would be the final product from the Reduction Operation. Its weight, impurity analysis, and assay would be reliable because of elimination of occluded slag and suspended non-metallics. As such, its plutonium content would be well defined. These conditions should permit a more meaningful accountability across the Reduction Operation. Plutonium value for transfer purposes would be defined reliably. The button residue from the ingoting operation should not constitute a more serious recovery problem than it poses in FAB. The uncertainties of plutonium content of the residue would then be coupled with the uncertainties of powder and slag. Accountability of pickle liquor would be eliminated. The accountability within the Fabrication Operation would be simplified.
8. Economic gains. The man-power requirements and costs for single-step, direct-ingoting should be no greater than for pickling. The proposed process is expected to improve overall acceptance rate for shapes, hence an overall economic gain is expected.
9. Decrease of radiation exposure. Ingots produce slightly less gamma and neutron radiation than buttons. Since almost all handling of metal will be done as ingots (sampling, density check, load-out, storage), exposure will be decreased. If the expected improvement of acceptance is achieved, a further reduction of radiation exposure should materialize through decreased recycle, inspection, and recovery operations.

V. REQUIRED EQUIPMENT AND PROCESS CHANGES

The hood presently used for pickling should be convertible to ingoting, in terms of space and geometry. Modification of this hood to include a casting furnace base and vacuum source should not be an insurmountable problem. Installation of a high-frequency work coil and temperature instrumentation will be required.

The availability of the power and vacuum facilities, and space for their installation, will require evaluation.

The time required for ingoting, from stack to breakout, is not expected to exceed 45 minutes to an hour (button heated to not more than 700 C, poured into a cold ingot mold). Hence, one casting stack should be adequate to process the output from the three reduction furnaces.

VI. PROPOSED SECOND INGOTING STEP

Casting charges for Fabrication use are prepared at present by blending

metal from the several feed sources (buttons, briquet ingots, miscellaneous metal) in proportion to feed source and in amount very close to required charge size. Optimum radiographic acceptance has been achieved partly through this blending and partly through multiple ingoting (in addition to other factors). Multiple ingoting permits segregation and elimination of suspended solids. It also permits effective outgassing of the melt. Removal of both solids and volatiles appears essential to high radiographic integrity of castings.

Outgassing of volatiles contained in button metal can be accomplished much more easily before blending than after blending. For this reason a high-temperature outgassing step is proposed for all button metal to be used by Fabrication. This outgassing step is desirable, whether coupled with the direct ingoting step proposed above, or for buttons routed directly to Fabrication.

The outgassing step could be applied to all metal from the Reduction Operation. In this case, metal for off-site shipment would be of better quality than the buttons shipped at present.

VII. DETAILS OF SECOND INGOTING STEP

Several alternatives are available for this step, and will require evaluation. Further removal of suspended impurities (nitride, oxide) will be accomplished by the second ingoting pour, regardless of time, temperature, or pour conditions. The effectiveness of outgassing will depend very much on actual process conditions.

Effective outgassing can be achieved quite rapidly by heating to high temperature (ca. 1100 C) with stirring (see HW-83736) or by a more prolonged heating, without stirring, at lower temperature. Recent FPT evaluations of the DAB process appear to indicate that effective stirring can be obtained by electromagnetic means, when the metal is in a silicon nitride crucible, without excessively high temperature (based on observed effective DAB blending at 900 C or less). The possibility of effective outgassing under these conditions warrants evaluation.

VIII. ANTICIPATED GAINS FROM THE SECOND STEP

The major gain anticipated from this second ingoting step is improvement of radiographic acceptance rate. Evidence from PCL investigations of the DAB process and briquet recovery demonstrated the need for multiple ingoting to assure radiographic integrity.

The absolute requirement of effective outgassing has not been proved. The elimination of volatiles from the metal is believed to be a contributing factor to radiographic integrity of casting batches prepared in the PCL. Elimination of volatiles should decrease the possibility of voids in castings, as well as formation of segregatable intermetallic compounds of volatile metals.

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This second step purification applies, at present, only to metal routed to the Fabrication Operation. The outgassing would be done in FAB, using existing facilities.

At some future time, economics may justify the double ingoting of all metal produced. Both steps would then be incorporated into the Reduction Operation. The gain from this change would be an improvement of product quality available to off-site customers. This, in turn, could improve customer relations and competitive position.

R. H. Bond

R. H. Bond

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