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E.I.du Pont de Nemours & Co.
Explosives Department
Atomic Energy Division
Wilmington, Delaware

October 10, 1955

MEMORANDUM TO FILE

From: Edgar E. Hayes

Trip Report
FMPC - September 20, 1955
(Meeting of Thorium Working Committee)

INTRODUCTION

A meeting of the Thorium Working Committee was held at FMPC on September 20, 1955. Present were T. C. Runion and Lou Levy of NLO, W. T. Warner, of FAO, C. Showalter of OROO and G. L. Tuer and E. E. Hayes of du Pont. This Committee was appointed at the final meeting of the NLO-SR Thorium Scheduling Committee, held at FMPC on June 9, with the following objectives which were outlined in the minutes of this meeting.

1. Evaluation of recycle metal.

2. Evaluation of the results of pile loadings in process.

3. Evaluation of the results of exposure of future small test lots for development work.

4. Improvement of metal quality (process and final product).

5. Evaluation of NLO development work on cost reductions as influenced by process efficiency.

6. Establishment of standard analytical methods and procedures hardness, density, etc.

7. Improvement of thorium metal specifications (physical and chemical).

8. Members of the Committee will keep each other informed of progress in off-site development work.

9. Keeping of NLO and du Pont management, AEC, and MDAC informed of the progress made, particularly the maintenance of liaison between du Pont and NLO.

Since the appointment of this Committee, FMPC has received a production order for the manufacture of 300 tons of thorium ingot metal. This includes the 100 tons that du Pont has requested (50 tons of ingot for a possible future full load of 1" diameter slugs and 50 tons of ingot for (1) Nuclear Metals development work on thorium co-extruded tubes and (2) sufficient metal for fabrication of tubes and/or Mark IV slugs for in-pile test irradiations). The balance of 200 tons is for "stockpile". To attain this production, FMPC will operate Plant 9 on a 3-shift—5-day-a-week basis.

SUMMARY

The current status of the thorium production program was discussed and a limited development and evaluation program was outlined which it is felt will (1) clear up some of the more important question marks in thorium metal quality and (2) develop a more satisfactory method for processing solid metal scrap.

The basic parts of this immediate program are - (1) SRL would evaluate ingot sections of as-cast double arc-melted virgin metal in order to classify typical ingot defects and determine their frequency of occurrence. (2) Du Pont would request for evaluation purposes ingot sections from typical recycle ingots which incorporated vacuum-induction melting for the consolidation of recycle metal in the form of consummable electrodes for arc-melting. (3) After completion of item (2), du Pont would then request approximately 200 slugs, made from suitably processed recycle metal, which would be used for canning and steam autoclave tests.

This program should lead to definite specifications (and/or classifications) for ingots to be stockpiled for future AEC use.

DISCUSSION

Most of the discussions centered around (1) ingot quality and (2) processing and evaluation of recycle metal. In addition to these major subjects, other items covered were methods to reduce the total amount of recycle metal, evaluation of thorium made with "commercial" calcium, reasons for hardness requirements of metal for Nuclear Metals, review of some process changes at Horizons in their development of electrolytic metal, review of available irradiation data on thorium to date, need for a new cross-lab check on hardness data, etc. Throughout the day's discussions it was emphasized by the du Pont representatives that our main concern on metal quality was (1) soundness of as-cast metal (since this has been one of the primary causes of slug rejections), and (2) oxide content whether it be in virgin or recycle metal, primarily since we consider product loss after irradiation to be directly proportional to the oxide content.

Ingot Quality

Although over 300 virgin double arc-melted ingots have been made at FMPC to date, there is insufficient data available as to the severity and frequency of basic ingot defects. For the recently completed production orders for du Pont, most of the rejected material was for defects which probably originated as basic ingot defects, such as shrinkage cracks. Rejections for "porosity" may also have been related to ingot quality, such as low density or segregation of impurities. It was pointed out that sound metal for our tubular program was of utmost importance. Also, for the stockpiling of cast ingots the extent and nature of ingot defects should be characterized. Because of the high pressures of production requirements, there has been no complete evaluation of ingot quality. Furthermore, there has been some differences in opinion between NLO and du Pont personnel on the actual quality of ingot metal and these differences may have resulted from either or both insufficient sampling and evaluation methods.

It was suggested by Tuer and Hayes that an ingot evaluation program be carried out and that this be done at SRL, if NLO could prepare cut ingot sections. SRL would examine these sections by etching, after first machining the surfaces smooth, for determination of shrinkage cavities, shrinkage cracks, and porosity. Variations in hardness throughout the cross-section would be determined. (Reported hardness values on the most recent cast metal have been higher for the metal produced for the last production order. This difference may be caused merely by the difference in method of taking the measurements. Hardness values are now taken on the as-cast surface of the ingots, rather than on a cut surface somewhat below the top, which may be harder because of higher impurity concentration.)

If possible, SRL would make some determinations on chemical segregation and density variations within the ingot as well as a limited micro-examination.

Mr. Schowalter of OROO stated that sufficient metal for this evaluation (~10 ingots) could easily be made available for this phase. It was agreed that du Pont would request metal for this program through the normal channels.

Processing of Recycle Metal

Fernald is not satisfied with the current method of processing recycle metal. It has proven very time-consuming and costly to fabricate consummable electrodes from recycle metal and the quality of the final ingot is considerably poorer than virgin metal with respect to oxide; - average of ~3-1/2% vs average of 1-1/2% for virgin metal. Fernald believes that most of this

pickup is occurring in the electrode preparation step. They believe that induction melting of recycle would be much better from both quality (with respect to oxide pickup) and cost standpoints but have understood that there has been a top-level decision against any induction melting of thorium for reactor use. We pointed out that this was true for the metal now under irradiation, but that since the objective of the Committee was a development program every consideration should be given to alternate methods of processing when there are good technical and economic reasons for doing so. We agreed that induction melting of recycle for purposes of consolidation into electrodes for consumable arc-melting appears very promising and would give them whatever support we could to have such development carried out. The most feasible way of doing this was considered to be a request from du Pont for ingot slices from two typical recycle ingots processed in this manner for evaluation purposes. Du Pont will request these ingot slices through the normal channels.

Evaluation of Recycle Metal

There was considerable discussion concerning what tests recycle metal should be subjected to in order to determine its suitability for reactor use. Du Pont representatives pointed out that steam autoclave tests on pinholed canned slugs would be the quickest way to obtain reliable information on how metal quality might affect pile performance of recycle metal. In the course of canning recycle slugs, data would also be obtained on the effect of secondary phases (particularly oxide) on bond quality. In the autoclave tests itself, data on the relative corrosion behavior of recycle metal would be obtained. Approximately 200 recycle metal slugs would be required for this phase of the program. A request for these will be made after the evaluation of the recycle ingot metal slices at SRL is completed.

With respect to a reactor test of recycle metal, it was pointed out that under the present Savannah River reactor conditions of low thorium burn-up (with accompanying low heat generation) a limited scale reactor test might not be too meaningful since (1) thorium has already demonstrated its ability not to change dimensions significantly under irradiation and (2) failures of canned slugs, under these reactor conditions, would probably be the result of sheath failures.

Irradiation Data

G. L. Tuer presented all the currently available data on thorium irradiations at SRP. These included the L-2, L-3, and R-5 loads. These data (length, diameter and warp changes) show quite satisfactory behavior from a dimensional standpoint at an exposure of 1200 g/t.

Use of Commercial Calcium

NLO has obtained some commercial calcium from New England Lime for trial reductions. Analysis of this material is aluminum 1/2%, magnesium 2-1/2 to 5%, and nitrogen 0.1 to 0.3%. It is hoped that this test will prove whether or not this type of less expensive calcium will give a satisfactory product. (Early experience at Ames indicated that metal might be of poor quality because of excessive Al and N contents in the final metal; 1/2# of Ca is required for each pound of thorium reduced.)

Methods to Reduce Amount of Recycle Metal

Two possible methods for reducing the amount of recycle metal (fewer machine turnings) were discussed. These were (1) swaging of extruded to final diameter and (2) fusion welding of as-cast ingot surface to improve surface quality. It is not expected that any work will be done on either process since (1) the NLO development budget for fabrication has been drastically reduced and (2) there is no available equipment, either NLO or SRL, for fusion welding.

Hardness Requirements for Extended Surface Shapes

The basic reason for a hardness requirement on as-cast metal was reviewed by E. Hayes. It was stated that the softest possible metal is desired for the Nuclear Metals co-extrusion process with an upper limit of Brinnell 60. It was emphasized that this is an arbitrary value based on present data. We stated that we would be willing to determine the suitability of harder metal once a satisfactory co-extruded element had been attained with metal in the $\leq$ 60 BHN range. Data was presented showing the increase in extrusion constant of thorium (at co-extrusion temperature) as a function of room temperature hardness.

Mr. Runion requested that NLO representatives be authorized to visit Nuclear Metals to observe the thorium co-extrusion process so that they could obtain first-hand information on the quality requirements for this process.

Cross-Lab Check on Hardness

It was agreed that a new thorium sample would be prepared for a cross-lab check on hardness values. This is particularly important since new hardness equipment is now being used at NLO for all ingot hardness determinations.

Horizons Process

The basic changes in the Horizons process during the past year were briefly reviewed. The process for chloride preparation now employs a precipitation of oxycarbonate from a nitrate solution. This is followed by chlorination with chlorine. The larger electrolytic cells are now internally heated. Prior to the end of this fiscal year, at which time development work will be terminated, a large-type cell should be completed and tested out. This cell will have a capacity of 16 pounds of metal per hour at a current capacity of approximately 7000 amperes.

Kroll Process

Very little discussion was held on this activity, except that NLO may cut back sharply its support of this program at the Bureau of Mines, Albany, Oregon.

Arc-Melting Work at SEP

Mr. Runion expressed interest in the small-scale arc-melting of calcium-reduced powder at SEP. Specific details of this work were not available, but these were promised prior to the next meeting.

The next meeting of this Committee was tentatively scheduled for December at Savannah River.

In Summary, the major items on which it was agreed that definite action should be taken were as follows:

By SRL:
1. Evaluate typical ingot sections from ten virgin metal ingots for ingot defects.
2. Evaluate typical ingot sections from at least two recycle metal ingots (employing vacuum induction-melting for consolidation of consummable electrodes.)
3. Test canned recycle metal slugs by steam autoclaving after satisfactory completion of items 1 and 2.

By FMPC:
1. Prepare ingot slices for shipment to SRL.
2. Investigate induction-melting as an alternate method of consolidating recycle metal.
3. Prepare virgin ingots from metal reduced by "commercial" calcium.

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