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DEPARTMENT OF MINES AND TECHNICAL SURVEYS

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MINES BRANCH INVESTIGATION REPORT IR 58-1

PREPARATION OF BISMUTH ALLOYS
CONTAINING VARIOUS AMOUNTS OF SODIUM AND RUBIDIUM

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Physical Metallurgy Division

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Introduction:

In a letter (reference M2-15-63) to Dr. John Convey, Director, Mines Branch, dated 28 March 1957, a request was made by Dr. D. C. MacPhail, Director, Mechanical Engineering Division, National Research Council of Canada, Ottawa, for samples of (a) bismuth--8-9% sodium alloy, and (b) various bismuth-rubidium alloys (Bi--5%, 1%, 0.1% and 0.01% Rb).

The present report describes the preparation and analysis of these bismuth alloys.

Preparation of Bismuth-Sodium Alloy:

From previous experience (see P.M. Test Report No. 10,406, Nov. 1, 1956), the charge for the 8-9% Na alloy was calculated on the basis of 10% Na to allow for melting losses.

An electric resistance furnace was used to melt the alloy, the bismuth (approximately 18 lb) being held in a 5 in. diameter graphite crucible. When the temperature of the metal reached about 400°C, the sodium was added in the form of lumps (about 1 1/2" cubes). The sodium had been stored under kerosene, most of which was wiped off just before alloying. The crucible was flushed with argon throughout the alloying operation and this prevented excessive burning of the sodium.

During alloying, which took a considerable period of time (owing to the large volume of sodium to be added), the temperature of the melt rose to about 530°C. This heating effect was presumably due to heat of reaction or solution, as the furnace was turned off during the alloying period.

The melt temperature was then raised to about 600°C and the alloy poured into two preheated, iron ingot moulds. Considerable oxidation occurred at the surface of the ingots during solidification.

Samples were taken from the centre and end of each ingot for analysis by the Flame Photometer, and the following results were reported by the Mines Branch Chemical Laboratory:

| | | | |
|-------------|---|---------------|----------|
| Small ingot | - | centre sample | 9.67% Na |
| " | " | - end sample | 9.12% Na |
| Large ingot | - | centre sample | 9.88% Na |
| " | " | - end sample | 9.78% Na |

It will be seen that a higher recovery of sodium was obtained than had been anticipated. This may have been a result of the size of the melt, which was considerably larger than in the earlier melt (see P. M. Test Report No. 10, 406).

Preparation of Bismuth-Rubidium Alloy:

1. Materials

The rubidium metal was supplied by the National Research Council and was contained in four evacuated pyrex vials (containing respectively, 5 g, 5 g, 10 g and 4 g).

The melting point of rubidium is given as 39°C (ASM Metals Handbook, 1948), but it was noted that part of the contents of the vials was liquid at normal room temperature (approximately 20°C). This suggested that the rubidium was contaminated with elements which lowered its melting point. The presence of potassium was later confirmed during analysis (see below).

2. Alloying Method

The Bi-1% Rb alloy was prepared first, as it could be made with one vial of rubidium and could subsequently be used as a master alloy for the preparation of the Bi-0.1 and 0.01% Rb alloys.

500 g of bismuth was melted in a graphite crucible

(1 1/4" inside diameter) and the temperature raised to about 400°C. At this point and throughout the subsequent operations the furnace was flushed with argon. One end of a vial containing 5 g of rubidium was immersed in the molten bismuth and broken off under the liquid. The fracture of the tube was localized by means of a scratch which had previously been filed on the neck of the vial.

In this way, burning of the rubidium was kept to a minimum.

The tube was held under the bismuth for about 1 1/4 hours, during which time the temperature of the melt was raised from about 400°C to 600°C. At the end of this period a considerable volume of solid alloy was still present in the end of the tube and this persisted even up to 620°C. However, after a further 15 minutes the tube appeared to be substantially empty and alloying complete.

The explanation of this behaviour was assumed to be that a high-melting-point intermetallic compound was formed between the bismuth and the rubidium and that this dissolved comparatively slowly in the bulk of the alloy. No reference to a bismuth-rubidium equilibrium diagram could be found in the literature but the behaviour of the alloy would be consistent with a system similar to that for bismuth-sodium.

The alloy was cast, at about 300°C, into a steel mould. Some exudations of a low-melting-point constituent were noticed on the surface of the solidifying slab and the lowest temperature at

which liquid was present was estimated to be approximately 220°C.

The weight of the slab was 470 g. Two samples were taken and submitted for analysis.

The problems connected with analysing the alloys are discussed further below, but the result obtained for this alloy (nominally 1.0% Rb) by X-ray spectroscopy was: 0.8% Rb \pm 0.1%.

Part of the slab was used as a master alloy to make the 0.1% and 0.01% Rb alloys, straightforward melting techniques being used in an argon atmosphere.

The bismuth-5% Rb alloy was made in a similar way to the 1.0% Rb melt, the remaining rubidium (19 g) being added by breaking the ends of the vials under the liquid bismuth (380 g). This was rather more alloy than had been called for (about 200 g), as it was necessary to have an adequate volume of liquid in which to immerse the vials.

3. Analysis

Considerable difficulty was experienced by the Mineral Dressing and Process Metallurgy Division in analysing for rubidium in the alloys. The flame-photometric method, which had been used for the sodium and potassium in previous alloys made in this program (see P.M. Test Report No. 10, 406), could not be used in the present case as other alkali metals (principally potassium) were present and interfered.

X-ray spectroscopy, which was used for the analysis of the 1.0% alloy mentioned above, was used but some of the results

seemed unreliable.

The colourimetric method was used to determine the bismuth (and the rubidium by difference), but this was not satisfactory for the lower rubidium contents.

The most satisfactory method for the 5.0%, 1.0% and 0.1% Rb alloys appeared to be to determine the total alkali chlorides present chemically. The results were expressed as percent rubidium but actually included unknown amounts of other alkali metals. The results obtained by this method are given below:

| <u>Nominal Composition</u> | <u>Actual Composition</u> |
|----------------------------|---------------------------|
| (% Rb) | (% Rb) |
| 5.0 | 3.4 |
| 1.0 | 0.65 |
| 0.1 | 0.13 |

(No satisfactory result for the 0.01% Rb alloy was obtained.)

Approximately 28% of ordinary rubidium is beta-emitting Rb⁸⁷ and this suggested the possibility of a radiation method of analysis. It was found, however, that the energy of the radiation was too low for practical measurements.

In this connection, if further rubidium alloys are to be made, it is suggested that a known proportion of an artificial radioactive isotope of Rb be added with the main addition and the resulting alloy analysed by a radiation method.

WAP: (PES)PG.

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