



AEC-NASA TECH BRIEF



AEC-NASA Tech Briefs describe innovations resulting from the research and development program of the U.S. AEC or from AEC-NASA interagency efforts. They are issued to encourage commercial application. Tech Briefs are published by NASA and may be purchased, at 15 cents each, from the Clearinghouse for Federal Scientific and Technical Information, Springfield, Virginia 22151.

Spectrographic Analysis of Bismuth-Tin Eutectic Alloys by Spark-Ignited, Low-Voltage AC-Arc Excitation

The problem:

To develop an analytical method for determining individual stainless-steel components in a molten bismuth-42 w/o tin eutectic to help determine the solubility of Type 304 stainless steel in such alloys. An accuracy of better than $\pm 10\%$ was required. The large number of samples and low element concentrations in some alloys made standard wet chemical techniques impractical. Analysis by emission spectroscopy appeared to be advantageous from the standpoint of detection sensitivity and speed. However, direct dc-arc excitation of the solid material was not satisfactory because of inadequate standards. Also, a preliminary examination of pins cast showed that segregation of impurities occurred during sample solidification.

The solution:

A spectrographic determination, utilizing the high sensitivity and precision of the spark-ignited, low-voltage ac-arc excitation of samples rendered homogeneous by dissolution. Concentrations of chromium, iron, manganese and nickel were determined in bismuth-tin eutectic alloys. These impurities could be determined over a concentration range of a few parts per million to 1% with accuracies of $\pm 10\%$.

The high sensitivity of this mode of discharge compares favorably with conventional dc-arc methods. Good accuracy is attained, and the gain in precision can present distinct advantages over dc-arc techniques.

How it's done:

To obtain the standards, several alloy melts were prepared from high-purity bismuth and tin matrices and were analyzed spectrographically by the dc-arc

method. The material that was free of stainless-steel constituents was used as a matrix in the preparation of solution standards, and as a blank in the dilution of samples of high-impurity content. To approximate the composition of samples and standards as closely as possible, a portion of a Type 304 stainless steel disc for use in the dissolution studies was analyzed chemically.

Solution standards were prepared by dissolving 100-mg portions of the pure alloy matrix in a hydrochloric-nitric acid mixture. The nitric acid was subsequently removed. Appropriate volumes of a standard stainless-steel solution and a cobalt internal standard were added to produce a series of alloy matrices.

Analytical samples were received as 1/4-in.-diameter pins. A series of drillings was taken along the length of the pins. A 100-mg sample portion was weighed and dissolved in a hydrochloric-nitric acid mixture. Again the nitric acid was removed. Then 0.1 ml of a 100- $\mu\text{g}/\text{ml}$ cobalt internal standard solution was added, and the sample was brought to 10 ml with 6 N hydrochloric acid.

Both copper and silver rods were used as supporting electrodes for the spectrographic analysis. The 1/4-in. electrodes were cleaned and machined to a smooth surface. A 0.1-ml sample aliquot equivalent to 1 mg of alloy, was divided equally between two electrodes, and these were dried under infra-red heat lamps.

Samples and standards were exposed in triplicate. Spectra were recorded on Eastman Kodak 103-0 plates, which were developed under standard conditions. Spectral line transmissions were measured with a nonrecording microphotometer. The line transmission readings were converted to line intensity

(continued overleaf)

ratios by means of a Respectra calculator. The element concentrations were evaluated using the experimental analytical working curves.

Notes:

1. Additional information is available in "Spectrographic Analysis of Bismuth-Tin Eutectic Alloys by Spark-Ignited, Low-Voltage AC-Arc Excitation," ANL-7331, May 1967. This report is available from the Clearinghouse for Federal Scientific and Technical Information, Springfield, Va. 22151; price: \$3.00 (microfiche copies, \$0.65).
2. This information may be of interest to metal research laboratories.

3. Inquiries concerning this innovation may be directed to:

Office of Industrial Cooperation
Argonne National Laboratory
9700 South Cass Avenue
Argonne, Illinois 60439
Reference: B69-10081

Source: E. A. Huff and S. J. Kulpa
Chemistry Division
(ARG-10288)

Patent status:

Inquiries about obtaining rights for commercial use of this innovation may be made to:

Mr. George H. Lee, Chief
Chicago Patent Group
U.S. Atomic Energy Commission
Chicago Operations Office
9800 South Cass Avenue
Argonne, Illinois 60439