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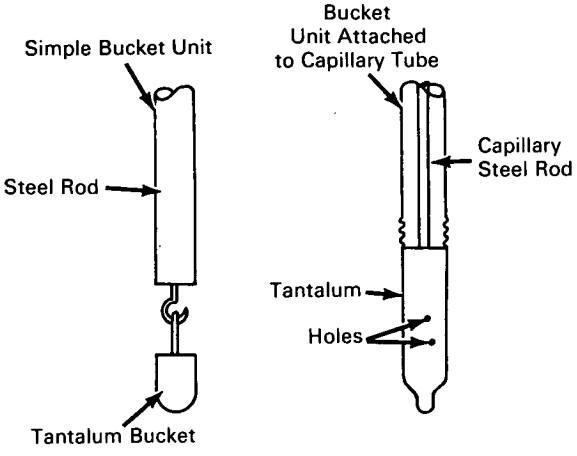
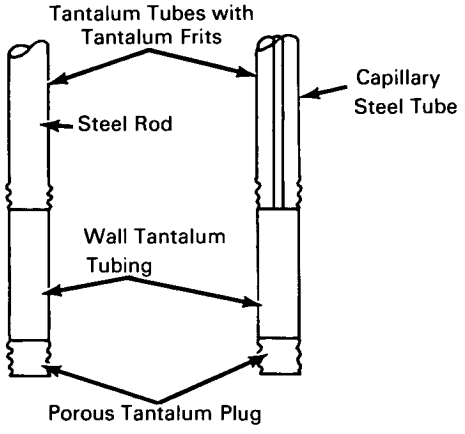
AEC-NASA TECH BRIEF

Two Techniques Enable Sampling of Filtered and Unfiltered Molten Metals



SAMPLERS FOR OBTAINING UNFILTERED SAMPLES OF METAL SOLUTIONS



SAMPLERS FOR OBTAINING FILTERED SAMPLES OF METAL SOLUTIONS

The problem:

To obtain samples of unsaturated liquid-metal solutions and two-phase metal–metal and metal–salt solutions. In two-phase systems, a provision must be made for sampling the lower phase.

If a filtered sample is desired, the solute element is filtered off the solid phases in suspension. The sample tube and the filter material must be inert to the sample solution and structurally strong. To avoid sampling errors, drainage of any sample liquid out of the sample tube must be prevented. Drainage reduces the concentration of the liquid phase and increases the weight percent of the solid phase (by forming crystals on the solid phase). After sampling, the entire sample must be removed for analysis because the solute in the sample may separate during cooling.

For an unfiltered sample, the solute element is obtained by drawing off part of the supernatant liquid, and there must be no loss of supernatant liquid or mother liquor.

The solution:

Filtered samples are obtained by filtering the solutions under the action of a pressure differential through a plug of a porous filter material fitted into the end of a sample tube. Unfiltered samples are obtained by using a perforated or open bucket with an attached steel extension rod; however, a capillary-tube extension rod used with a perforated bucket is a more reliable setup.

How it's done:

For obtaining filtered samples, a fritted filter material is selected that is inert to the sample solution. The fritted plug is fitted into an inert and structurally strong sample tube and is held in place by roller-sealing or force-fitting. The end of the tube not containing

(continued overleaf)

the plug is attached to an extension rod. During sampling, the rod extends above the melt.

The sample solution is forced into the sample tube by external pressure on the solution surface or by application of a vacuum through a capillary tube inserted into the sample tube. The lower phase of a two-phase solution is sampled by blowing inert gas through the capillary tube and the frit while the tube is lowered through the upper phase. The pressure differential depends on solution properties, porosity of the frit, and wetting of the frit by the solution. To prevent sample loss: (1) drainage of the sample liquid out of the sample tube is prevented by the frit and (2) the pressure outside the tube or the vacuum within the tube is maintained until the sample solidifies. The entire sample is removed either by peeling the tube away from the sample or by dissolving the sample out of the tube.

For obtaining unfiltered samples, a perforated or open bucket is attached to the steel rod and lowered into the solution. Bucket samplers ensure against loss of supernatant liquid or of motor liquor. A more reliable assembly is a capillary tube steel rod attached to a bucket containing holes. With this setup, a sample can be drawn through the holes regardless of whether the sample liquid wets the sampler. The lower phase of a two-phase system is sampled by blowing inert gas through the capillary while the bucket is lowered through the upper phase.

Notes:

1. Obtaining filtered samples usually takes 2 minutes (including preliminary heat-up of the sample tube to prevent solidification of the sample on it) with a pressure differential of 10 to 20 psia.
2. Obtaining unfiltered samples takes about 5 seconds (excluding a 1-minute heat-up).
3. Because the solutions used were zinc-rich, only graphite, tantalum, tungsten and certain of its alloys, and various ceramic materials could be used in the sampling system. A tantalum frit and a tantalum sample tube were chosen because a graphite frit in Vycor tubing caused interactions of the sample with the filter, occasional fracture of the plug, and occasional breakage of the tube. Tantalum frits are available in coarse, medium, and fine porosities.
4. Stainless steel filters in thicknesses of 1/16 to 1/4 inch are available in a number of porosities. These filters are used for sampling alkali, alkaline earth, and cadmium metal solutions. Fritted filters of nickel, Monel, platinum, gold, silver, and tungsten are also available.
5. Additional details are contained in: "Sampling of Liquid Metal," by I. O. Winsch, et al, ANL-7088, September 1965. This report is available from the Clearinghouse for Federal Scientific and Technical Information, Springfield, Virginia 22151; price $3.00 (microfiche copies, $.65).
6. Inquiries concerning this innovation may be directed to:

   Office of Industrial Cooperation
   Argonne National Laboratory
   9700 South Cass Avenue
   Argonne, Illinois 60439
   Reference: B67-10034

Source: I. O. Winsch, K. R. Tobias,
R. D. Pierce, and L. Burris, Jr.
Chemical Engineering Division
(ARG-150)

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 Commision
Chicago Operations Office
9800 South Cass Avenue
Argonne, Illinois 60439