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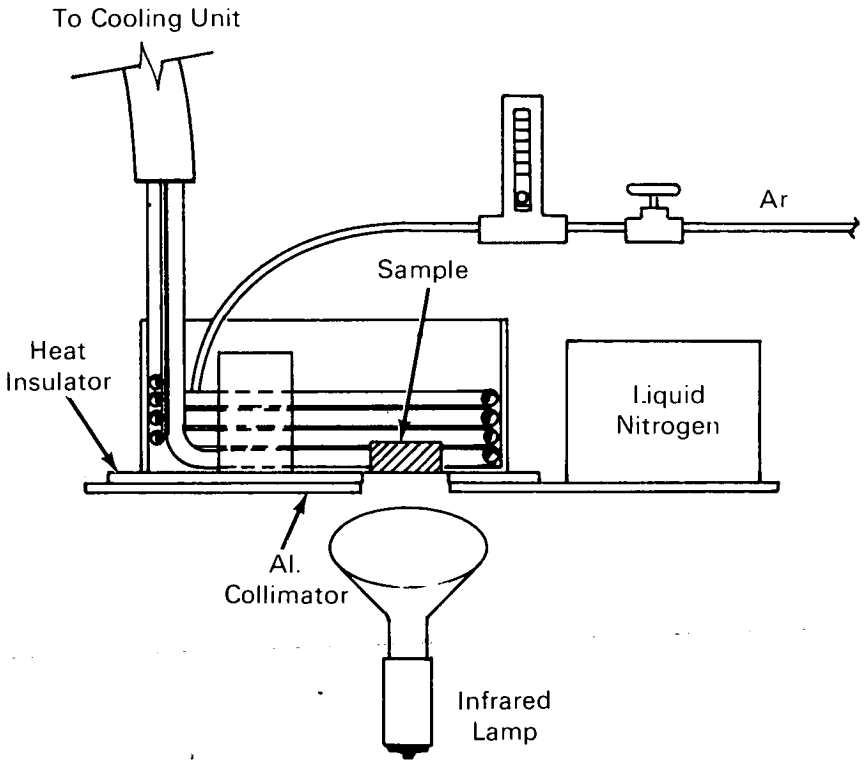
Method for Copper Staining of Germanium Crystals



The problem:

To develop a method for copper staining of germanium crystals. Copper staining, by white-light illumination of a sample immersed in a Cu^{++} solution, has been used for decoration of Si crystals. Germanium devices, however, could be decorated only by various adaptations of techniques based upon preferential chemical etching and electrolytic plating.

The solution:

Excellent decoration of Ge crystals by copper staining. The method can be used to show nonuniformities in Ge crystals prior to Li drifting, and as a tool for study of the diffusion and drifting of Li in germanium.

How it's done:

The key conditions for proper decoration of Ge crystals by copper staining are a low solution temperature of 3°C, illumination of the sample by infrared light, and careful positioning of the light source relative to the germanium sample so as to minimize absorption of the infrared light. The staining solution contains 20 g of $CuSO_4 \cdot 5H_2O$ and 1 cm^3 of 48% HF per liter; it must be kept near the freezing point for development of clear stains.

(continued overleaf)

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The use of light is essential in copper staining, since the technique is based on a photovoltaic effect causing more electrons to accumulate on n-type material, and therefore preferential plating of the Cu^{++} ions. Since infrared radiation is absorbed by the Cu^{++} solution, the sample is irradiated through the glass bottom of the dish, and therefore through the sample rather than through the top of the solution.

The surface to be stained is carefully lapped, and dried with a jet of dry nitrogen. The sample is then immersed in the solution kept at 3°C by an ethylene glycol cooling coil. After a 30-second delay, the infrared light is turned on for from 2 to 4 minutes, depending on the size of the sample. The liquid-nitrogen-cooled collimator allows longer exposure if desired, without causing a serious rise in temperature of the staining bath.

During the staining exposure, Ar is bubbled into the bath at 3 liter/min to provide stirring without which the heat currents rising from the faces of the device cause severe distortion of the stain. A piece of copper pipe is located in the bath to promote circular motion of the solution.

Reference:

For details see E. J. Rivet, *Rept. UCRL-18430* (Univ. of California, Berkeley, Aug. 1968); *Nuclear Instr. Methods* **37**(2), 349 (1969).

Notes:

1. This information may interest crystallographers and manufacturers of radiation detectors.
2. Inquiries may be directed to:

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Patent status:

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

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