

Letter to the Editor

A simple device for growing single crystals of reactive materials

In a recent paper McEwen and Touborg (1973) discuss the possibilities of growing single crystals of the rare earth metals. The serious problem of their high chemical reactivity results in the impossibility of finding a suitable crucible material. As is well known this problem also holds for many other substances. A method overcoming these difficulties is the use of a water cooled crucible, first described in a German patent (Siemens 1926). This method was later expanded to the purification of semiconductors employing a horizontal boat (Warren 1957) and to the *in situ* melting and zone refining of metals (Sterling 1957, Sterling and Warren 1961). The purpose of this paper is to describe an extremely simple device employing a water cooled boat used for growing single crystals of Dy.

The assembly is shown in figure 1. The horizontal boat is simply pressed into a copper tube being cooled by water.

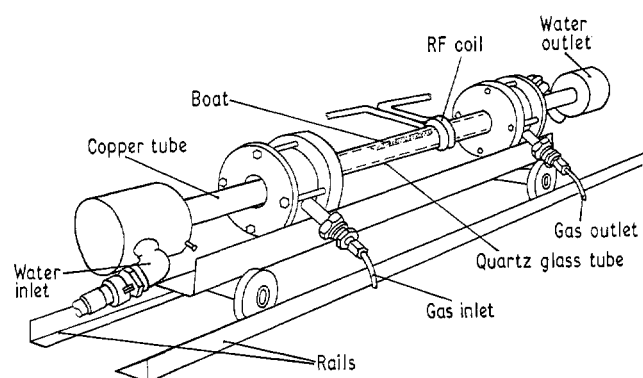


Figure 1 Sketch of the device

The boat is surrounded by a quartz tube making it possible to use an atmosphere of any desired gas. The copper tube and the quartz glass tube are held by two flanges lying on a little wagon. By means of this wagon the whole assembly can be pulled through a fixed RF coil.

Using this assembly we succeeded in growing crystals of Dy. Commercially available distilled Dy metal was put into the boat and melted forming a rod $7\text{ mm} \times 7\text{ mm} \times 100\text{ mm}$. This was achieved by repeatedly sweeping the molten zone over the whole length of the boat. The subsequent crystal growth was achieved with a pulling speed of 15 cm h^{-1} .

Afterwards the material was annealed at a temperature of 1300°C , and the wagon pulled at last three times through the RF coil with a speed of 1.5 cm h^{-1} . Before the onset of melting the assembly was scavenged with gettered argon. Then the gas outlet valve was closed to avoid any contamination of the reactive material with traces of oxygen from the argon gas. The oxygen content of the metal is critical for the production of large single crystals (Whittaker 1968).

During the melting and annealing an overpressure of 100 Torr was maintained. The result of this growing procedure were centimetre crystal grains. The largest crystal obtained was $7\text{ mm} \times 7\text{ mm} \times 28\text{ mm}$. X ray back reflection photographs of this crystal did not show lineage substructure. An indication of the quality of this crystal is the sharpness of the transition from the ferromagnetic to the antiferromagnetic state at 85 K as measured by a neutron depolarization experiment (Rauch *et al.* 1971). Internal stresses would lead to a marked broadening of the transition curve because of the high magnetostriction effect in Dy. Measurements on commercially available Dy crystals showed a broadening of the transition up to 2°C in contrast to less than 0.1°C of our crystal.

Finally it is to be noted that the advantages of this device are: (i) the processes of melting, zone refining, crystal growth, and recrystallization can be done subsequently without any handling of the material between the different steps; (ii) using a cold crucible avoids any difficulties due to reaction with the crucible material; (iii) even at high evaporation rates the melt may be inspected simply by turning the quartz glass tube; (iv) a very low gas volume is used thus reducing the contamination of the melt by oxygen etc; (v) the extremely simple construction.

More details of the construction can be obtained upon request to the authors.

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References

- McEwen K A and Touborg P 1973 *J. Phys. F: Metal Physics* **3** 1903-9
- Rauch H, Seidl E and Zeilinger A 1971 *Z. Angew. Phys.* **32** 109-113
- Siemens and Halske A G 1926 *German Patent No 518499*
- Sterling H F 1957 *British Patent No 889615*
- Sterling H F and Warren R W 1961 *Nature, Lond.* **142** 745
- Warren R W 1957 *British Patent No 827676*
- Whittaker M 1968 *J. Crystal Growth* **4** 317

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