

Apparatus for crystal growth

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The design of an apparatus based on Bridgman's method, enabling visualization of the growth process and regulation of the crystallization rate, for obtaining single crystals from a melt in a school laboratory is presented. Conditions for obtaining single crystals of several substances are given.

1. Introduction

Large single crystals of good quality are rare in nature. Therefore, most large single crystals for technical applications and scientific investigations are produced in laboratories under controlled conditions. In principle, preparation of single crystals can be achieved in three ways: by crystallization from the solid, liquid or gas phase. A number of methods have been developed for growing single crystals (Buckley, 1958; Tarjan & Matrai, 1972; Wilke, 1977). The choice of the most suitable method for the preparation of single crystals of a particular substance depends not only on their nature and physico-chemical properties, but also on the intended application of the single crystal, the quality desired and the available laboratory equipment.

In many cases single crystals are obtained by growing from the melt, which is in essence controlled cooling growth. In comparison with other methods for single-crystal preparation this method is easily controlled, and it achieves a higher crystallization rate than other methods. With the exception of crystal growth from water solutions, this process has been the most thoroughly investigated from the technical standpoint. Crystal growth from the melt is widely applicable but has limitations regarding non-melt-stable materials.

2. The apparatus

Cabric & Pavlovic (2000) have previously presented a design of an apparatus based on Stöber's method (p. 89 of Buckley, 1958; p. 271 of Wilke, 1977), with the aim of regulating crystallization rates in Tamman test tubes, for school laboratory work. In the present paper, two set-ups (Figs. 1 and 2) based on Bridgman's method (p. 76 of Buckley, 1958; pp. 172–179 of Tarjan & Matrai, 1972; p. 286 of Wilke, 1977), enabling exact and easy regulation of the crystallization rate (starting from zero) along Tamman test tubes with visualization of the growth process, are constructed. The substance to be crystallized is placed in a Tamman test tube (p. 77 of Buckley, 1958; p. 178 of Tarjan & Matrai, 1972; p. 260 of Wilke, 1977). The tube is then lowered slowly through a furnace by raising the water level in the vessel, as shown in Fig. 1. When the temperature of the melt at the bottom of the Tamman test tube decreases below the crystallization temperature, only a few crystal seeds will form and one will overgrow the others (p. 263 of Buckley, 1958; Fig. 2 in Cabric & Pavlovic, 2000) producing a single crystal.

The crystallization rate along the test tube is regulated by the rate at which water is dripped into the vessel (Fig. 1), *i.e.*

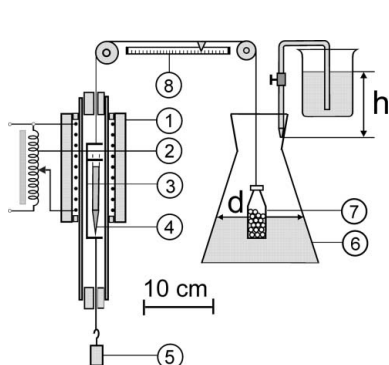


Figure 1

Apparatus for crystal growth. (1) Electroresistant tube furnace, (2) continuously changeable transformer, (3) hanger ('fishhook' in the shape of the letter 'E' made of rigid wire), (4) Tamman test tube, (5) weight, (6) vessel (*e.g.* flowerpot or pitcher), (7) buoy (bottle with marbles, pearls or sand) and (8) scale (ruler).

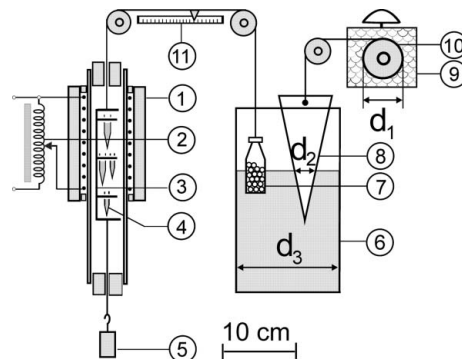


Figure 2

Alternative apparatus for crystal growth. (1) Electroresistant tube furnace, (2) continuously changeable transformer, (3) hanger ('comb' made of rigid wire), (4) Tamman test tubes, (5) weight, (6) vessel, (7) buoy (bottle with marbles, pearls or sand), (8) wedge (*e.g.* made of wood), (9) lowering assembly (*e.g.* watch movement), (10) pulley (*e.g.* spool of thread) and (11) scale (ruler).

Table 1

Crystallization conditions for some substances (Gilman, 1963; Tarjan & Matrai, 1972; Wilke, 1977).

Substance	Melting temperature (K)	Test tube material	Test tube diameter (cm)	Test tube lowering rate (cm h ⁻¹)
Naphthalene	353.4	Pyrex	1.3–2	0.1–0.4
Anthracene	490.2	Pyrex	2.5	0.6–1.2
Sn	505.1	Pyrex	0.5	2.5–13
Bi	544.6	Graphite, Pyrex	0.5	2.5–16
Pb	600.6	Graphite	2.5	0.5–2.5
Zn	692.7	Pyrex	0.5–1.5	2.6–12

with the height (h) and the cross section of the vessel (d). Knowledge of the crystallization rate and perfection of the crystal along the test tube enables determination of the most suitable crystallization rate of the substance (Cabric *et al.*, 1994; Cabric & Pavlovic, 1997). The design of an alternative apparatus for regulation of several crystallization rate intervals (starting from zero) along a set of Tamman test tubes is shown in Fig. 2. The crystallization rate along the test tubes is regulated by the rate at which the cross sectioned body (wedge) is lowered into a vessel containing water (proportional to d_1), and the ratio of the cross sections of the cross

sectioned body (proportional to d_2) and the vessel containing the water (proportional to d_3) (Cabric *et al.*, 1997). Using different shapes and dimensions of Tamman test tubes (pp. 175–179 of Tarjan & Matrai, 1972; p. 260 of Wilke, 1977) and the vessel (Fig. 1) or wedge and hanger (Fig. 2) (*i.e.* different crystallization rate intervals), students can grow larger and better quality single crystals (*e.g.* from substances with low melting temperatures, as shown in Table 1).

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