⌫ Retour au sommaire

3. Bridgman and Stockbarger methods

The powdered materials are inserted in a conical crucible and melted after dehydration and/or outgassing. A temperature gradient is established in the furnace and allows for the crystallisation to start from the cone shaped bottom of the crucible. In the Bridgman technique, the crucible is slowly moved in the temperature gradient (figure 19), while the temperature of the furnace is slowly decreased in the Stockbarger method.

Both techniques are very convenient for the growth of moisture sensitive materials : fluoride or mixed anionic compounds (oxide fluorides, chlorofluorides...). Several examples are listed in table 3.

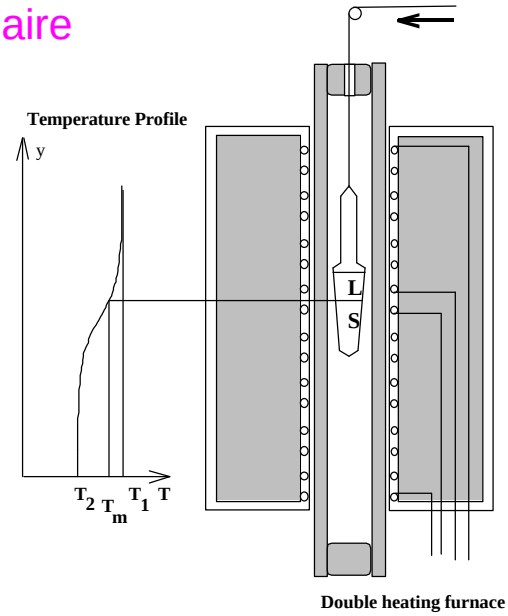

Figure 19. - Principle of Bridgman growth technique.

Table 3: Conditions of crystal growth of selected materials by Bridgman technique.

	Material	Structure type	Displacement rate (mm/h)	T (°C)	ΔT (°C/cm)	Starting Materials
Fluorides	Rb_2KYF_6	Elpasolite	1.5	1090-750	25	Rb_2KYF_6
	$K_{1-x}Rb_xAlF_4$		0.6	600-400	15	$K_{1-x}Rb_xAlF_4$
	$KCaF_3$	Perovskite	0.6		10	$KCaF_3$
	$BaThF_6:Ce^{3+}$	Tysonite	0.5	1160	20	BaF_2,ThF_4,CeF_3
Mixed anions	$K_3MoO_3F_3$		1.0	600-400		$K_3MoO_3F_3$

4. Verneuil and skull melt methods

Compounds with very high melting temperatures were primarily grown by one of the precursor methods of crystal growth : the Verneuil or flame fusion method (figure 20). The starting powder is fused in a flame and slowly drops onto the growing "boule".

Corundum Al_2O_3 crystals are mainly produced by this technique in a H_2/O_2 flame at 2000°C. Colored varieties are used as lasers (ruby $Al_2O_3:Cr^{4+}$) or gem imitations (ruby, sapphire $Al_2O_3:(Fe^{2+},Ti^{4+})$. TiO_2 rutile and $SrTiO_3$ (named fabulite !) were also grown for diamond imitation.

The skull melt process is dedicated to phases which present electronic or ionic conduction at high temperature. The sintered powder is heated by high frequency induction (4 MHz, 100 kW) in a cooled crucible. The inner part of the sample melts and is protected from the atmosphere and from crucible pollution by the solid outer part (the skull, name given by the inventor Osiko). Crystals are grown by a slow decrease of the HF heating.

Pure zirconia ZrO_2 (T_m=2750°C) and stabilised cubic zirconia (CaO or Y_2O_3 doped ZrO_2) are synthesised by this technique. This last phase is a convincing diamond simulant.

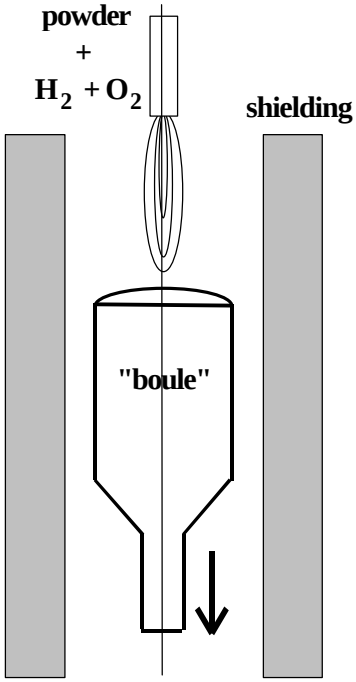

Figure 20.
Principle of Verneuil growth method.