Fig. 1.

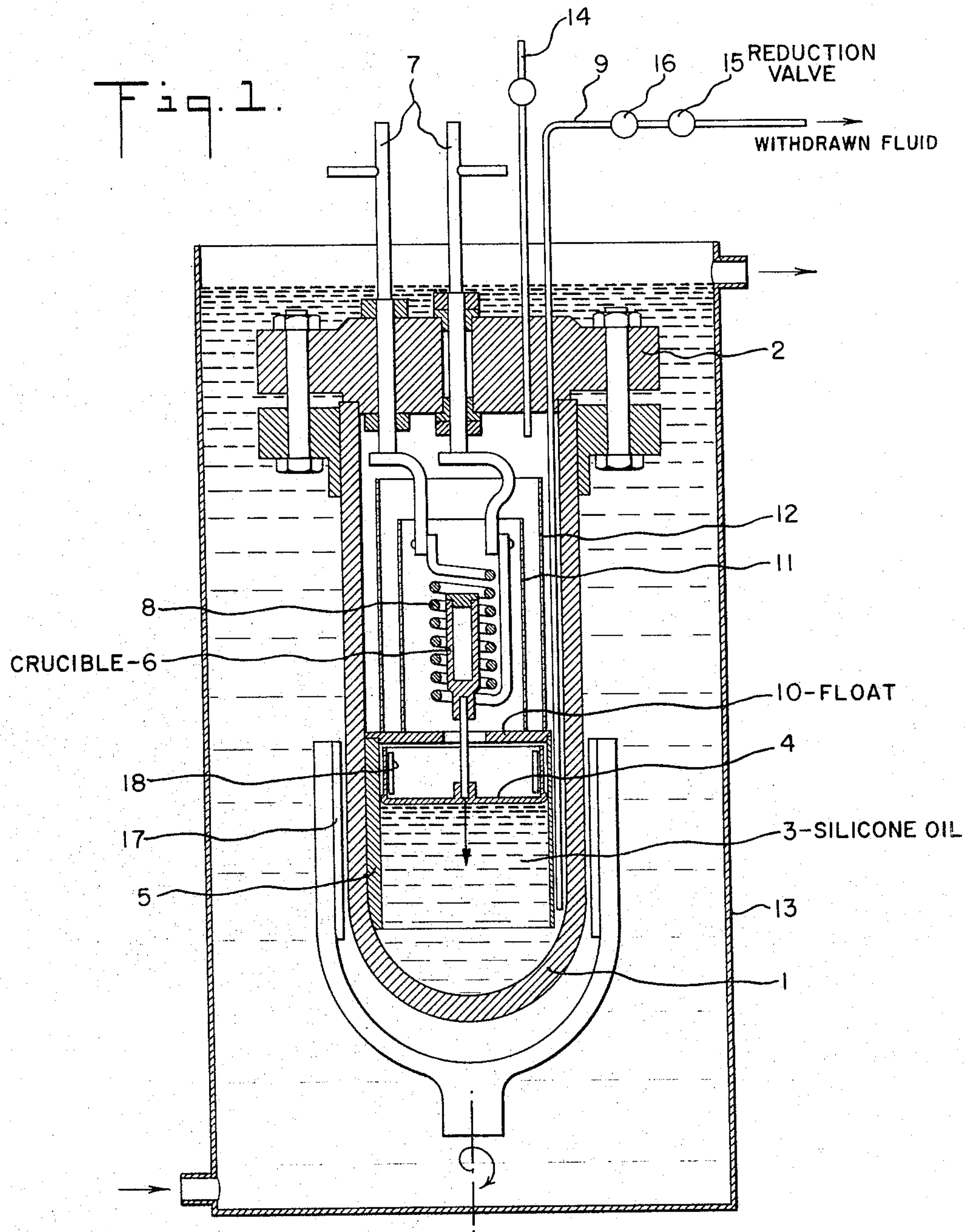

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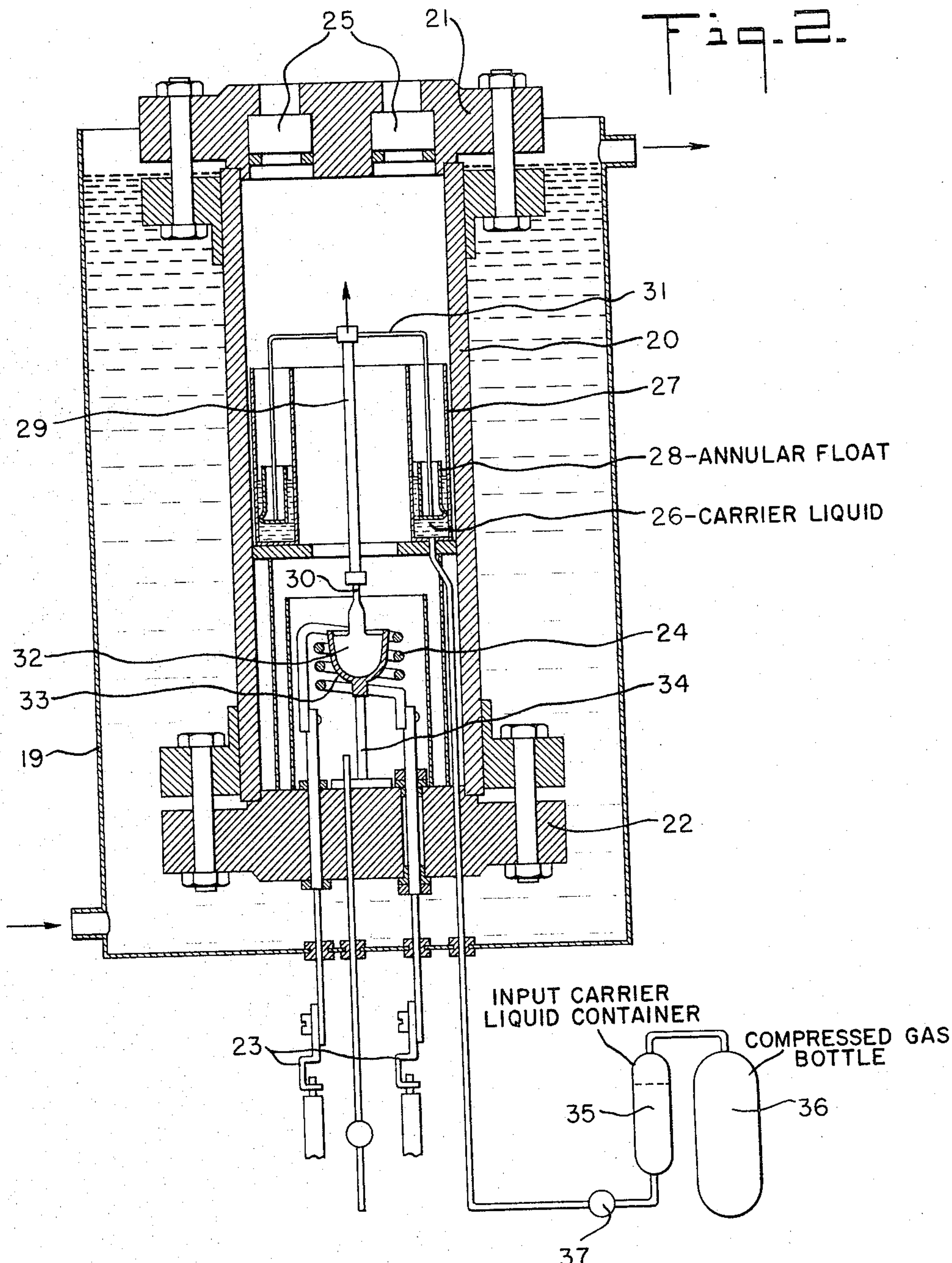
Fig. 2.



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PRODUCING AND REGULATING TRANSLATORY MOVEMENT IN THE MANUFACTURE OF SEMI-CONDUCTOR BODIES

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This invention relates to the manufacture of formed semi-conductor bodies and it has for its object to provide a novel and improved method and means for producing and regulating the translatory motion of such bodies during their formation.

Another object of the invention is to produce and control the translatory motion of the aforesaid bodies during their formation, with a high degree of accuracy and without the intervention of mechanical transmission means between the outside and the interior of the high pressure apparatus.

Still another object is to effect the foregoing result by lowering or raising the level of an auxiliary fluid which is located inside the pressurized apparatus, and to transmit such movement of said fluid in one way or another to the growing semi-conductor body.

Various other objects and advantages will be apparent as the nature of the invention is more fully disclosed.

The building up of rod-like semi-conductor crystals from the melt is preferably done in accordance with the zone melting, Czochralski, or normal freezing process. In using these processes on sublimating substances and on substances with high vapor pressure or great tendency to decompose at the melting point, attempts have been made to melt said substances under appropriate excess pressure of the readily volatile component or of an inert foreign gas (see Patent 3,022,144 which describes the production of single crystals of zinc sulphide from the melt under a pressure of 3.5 to 10.5 kg./cm.^{2}).

The production of cadmium sulphide single crystals under an argon overpressure up to 105 atm. is explained by W. E. Medcalf and R. H. Fahrig (J. Electrochemical Soc. 105, 719; 1958).

The above crystal building processes require a very slow and even translatory motion of the melting crucible, of the heating device, or of the crystal itself. To create this movement, mechanical and hydraulic drive mechanisms are used, the movement being transmitted from outside the apparatus to the interior of the pressure vessel either by a shaft or by means of a magnetic clutch. The mechanical solution of transmitting such movement requires a slip packing between the axially movable and/or rotary shaft and the housing, with the attendant disadvantages of such packings. The magnetic power transmission, due to the comparatively high elasticity of such a clutch and the unavoidable bearing friction of the moving parts inside the apparatus, causes an unsymmetrical advancement.

The present invention provides a surprisingly simple method and means for producing and regulating the translatory motion when producing semi-conductor bodies in accordance with the lowering, deposition, zone melting or crucible drawing process under elevated pressure, during which no mechanical power transmission from outside to the interior of the high-pressure apparatus is required, and the above-mentioned disadvantages of the arrangements heretofore used are avoided. The method of the invention is characterized by the fact that the level of a liquid which we locate inside the pressurized apparatus

is lowered or raised as required. A float transmits the height of the liquid's level to the heating device, the crucible, the seed or the semi-condutor body.

The speed of the movement or the change of the liquid level can be regulated very exactly and evenly by the quantity of liquid piped in or withdrawn within a unit of time. It is useful to change the liquid's level by pumping in or withdrawing liquid through a pipeline leading inside the apparatus, the line being equipped with a regulating valve.

A particular advantage of our novel arrangement is the elimination of sliding packings and the movable drive elements with the required bearings inside the apparatus.

Our method can be used for producing element semi-conductor bodies, particularly when dosaging with easily volatile materials. Moreover it can be used to advantage for element and bonding semi-conductors which have a great vapor pressure at the melting point, e.g. tellurium and selenium, or which decompose easily, e.g. gallium arsenide. It can also be used for high-pressure separations and sublimations, as well as when using melting processes on sublimating substances. Furthermore it becomes possible, with slight changes, to use commercially available, relatively inexpensive laboratory autoclaves which are available for pressures of up to several 100 atmospheres, instead of expensive specially designed pressure devices.

The invention is described in connection with the accompanying drawings, in which:

FIG. 1 is a diagrammatic illustration of an apparatus embodying our method of producing and regulating the translatory motion of a semi-conductor body during its formation; and

FIG. 2 is a diagrammatic illustration of a modification of the invention.

In referring to the drawings we shall describe only so much of the well-known prior art formation of the semi-conductor bodies as is necessary to an understanding of our invention.

The apparatus of FIG. 1 is used for producing cadmium sulphide crystals in accordance with the normal freezing process under an inert gas pressure of up to 250 atmospheres excess pressure. As pressure container we use a conventional laboratory autoclave consisting of the lower vessel **1** and the lid **2**. The lower vessel **1** is filled with silicone oil **3** to a suitable level. The float **4** seated in the oil **3**, and guided without friction by a collar **5** inside the vessel **1** (somewhat like a piston in a cylinder) carries the melting crucible **6** filled with cadmium sulphide powder. Water-cooled power lines **7** are brought in through the lid **2** of the autoclave to the carbon spiral **8** which serves as a heating element. Also, a small-gauge pipe **9** is built pressure-tight into the lid **2**. This pipe **9** is immersed in the silicone oil **3** at least to the height of the oil reduction level so that it is always immersed in the oil.

The disc **10** on collar **5** and the sheet metal members **11** and **12** arranged concentrically around the heating element **8** serve as heat radiation protection. The entire autoclave stands in a vat **13** through which water circulates, as illustrated, for cooling purposes. The inert gases are piped in through supply line **14**.

After the cadmium sulphide powder contained in the crucible **6** has been melted down completely at a temperature of about 1500° C., we start the lowering of the crucible. For this purpose the reduction valve **15** built into the pipeline **9** is opened, so that, due to the overpressure existing in the autoclave, the silicone oil **3** is pressed out from the latter. This pressure, which in any case is technically required for the process itself, thus serves at the same time as a power source for the lowering mechanism. To make possible a fine setting of the lowering speed by means of valve **15**, it is useful to include an automatic pressure reduction valve **16** in pipeline **9** as illustrated. The measurement of the lowering speed is done by determining the quantity of oil running out through pipeline **9** during a unit of time. As the level of the oil **3** is lowered, the float **4** and crucible **6** descend, as the semi-conductor continues to build up in the crucible.

In order to achieve a phase border surface directed as level as possible perpendicular to the direction of growth of the crystal, it is usually necessary to superimpose a rotary motion on the vertical translatory motion of the seed, crucible or rod. The speed of rotation is of a magnitude between 20 and 200 revolutions per minute. As indicated in FIG. 1, this rotary motion can be created without difficulty in our arrangement of the lowering mechanism by means of a magnetic clutch mounted inside the apparatus. This consists of an external magnet **17** which is disposed in vat **13** outside the autoclave and rotated by any suitable means (not shown) and internal magnet elements **18** mounted inside the float **4**. In the case of this apparatus an uneven movement is not troublesome, and, moreover, is not anticipated at the rotational speeds mentioned here. Of course, it is a prerequisite that the structural elements located between the external and internal magnet elements be made of non-magnetic materials, e.g. Remanit 1880 S. Since only the hydrodynamic friction of the float must be overcome, a comparatively small torque is sufficient.

The method of drawing crystals from the melt may be carried out as illustrated in FIG. 2. The autoclave vessel **20** which is again suspended in a water vat **19** for cooling purposes is in this case closed on top and bottom by lids **21** and **22**, respectively. The power leads **23** for the usual heater **24** are brought in through the lid **22**, while the upper lid **21** contains two windows **25** of thick quartz plates. One of such windows serves for observation, the other for artificial illumination of the inside of the autoclave. The vessel **27** mounted in the autoclave contains the carrier liquid **26**, in which is the float **28** which is open on top and is in the shape of an annulus or collar. The rod **29** at the lower end of which is attached, for instance, a mono-crystalline seed crystal **30**, is connected with the float **28** by means of the bow or stirrup **31**. The crucible **33** containing the melt **32** is fixed to the lower lid **22** by pin **34**.

Since an upward movement is required for drawing crystals, in this case the carrier liquid **26** is pressed into the float vessel **27** from a reservoir **35**, to raise the level of the liquid **26** in said vessel **27**. The required pressure, which must be somewhat greater than the overpressure in the autoclave, is obtained, for instance, from a commercially available compressed gas bottle **36**. The adjustment of the desired liquid input is set by means of regulator valve **37**. For zone melting which requires alternating movement possibilities, using the arrangement shown in FIGURE 2, the liquid is pressed in or alternately let out through the decompression valve.

The invention claimed is:

1. The combination with a closed pressurized vessel for the manufacture of semi-conductor crystalline bodies by a seed pulling operation, and means including a vertically movable element inside said vessel for inserting a seed crystal into a melt and pulling said seed crystal from said melt in a crucible to form a semi-conductor body and to build the body in a vertical direction with respect to the vertical movement of said element, of a body of liquid in said vessel, a float seated on said liquid and supporting said element, and means for varying the vertical level of said liquid to move said float and said element in a similar vertical direction.

2. The combination of claim **1**, in which said last-mentioned means comprises a pipe extending from outside said vessel into the interior thereof and having its lower end immersed permanently in said liquid, and means for opening said pipe to discharge liquid therethrough in response to pressure in said vessel.

3. The combination of claim **1**, characterized by the provision of means for increasing the volume of said body of liquid to raise the vertical level of said liquid and correspondingly raise said float and said element.

4. The combination of claim **1**, having means for imparting a rotary motion to said element.

5. In the process of vertically building a crystalline semi-conductor within a pressured vessel, the steps of inserting a seed crystal supported on and affixed to a float, into a melt contained in a crucible and withdrawing said seed crystal from the melt in a vertical direction by varying the level of the liquid on which said float is seated such that the float and seed crystal move away from the crucible to form said semiconductor body and to build said body in a vertical direction to a dimension dependent upon the vertical movement of the float within said vessel.

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