

## CENTRIFUGE FOR SOLUTION GROWTH OF SEMICONDUCTOR LAYERS: AN APPLICATION OF MAGNETIC BEARINGS

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### ABSTRACT

Three generations of centrifuges equipped with active magnetic bearings (AMBs) have been successfully used for research work in liquid phase epitaxy. The centrifuges have served to prepare semiconductor layers of excellent quality and purity. The strong interest in using AMBs for the preparation of semiconductor material lies in the fact that they can induce the rapid rotation of a large and heavy crucible if it is placed in an extremely clean vacuum or hydrogen environment of a crystal growth apparatus. The AMBs do not need lubrication nor are they subject to wear and particle generation by abrasion.

### 1. INTRODUCTION

Semiconductor layer growth from liquid solutions - *Liquid Phase Epitaxy* (LPE) - ensures a minimum of defect density of the material, i.e. material of excellent quality.

At the Max Planck Institute Stuttgart, active magnetic bearings in centrifuges have been used for LPE since 1979. For bringing together a crystalline plate, which is called substrate, and a liquid solution, growth of the crystalline layers requires a transport system. The liquid solution constitutes the feeding phase for the growth of the crystalline layers. It has been shown that centrifugal forces in a rotating crucible allow solutions to circulate, completely and adequately speedily, from a container to a substrate and - after layer growth - back from the surface of the grown layer into a container[2,3].

Rotation of the crucible with the help of magnetic bearings provides the opportunity to transport solutions inside the crucible, avoiding contamination from lubricants which are used in mechanical bearings. It also prevents contamination through wear and abrasion from mechanical bearings.

Only AMBs are capable of operating under UHV (Ultra High Vacuum) conditions at temperatures of between 300 and 950 °C of a crucible fastened to one end of the rotor shaft. The centrifugal growth technique has been successfully applied in pilot experiments for growing high quality layers of Si, SiGe, and GaAs on planar and profiled 4" substrates[4-7]. The capability of the centrifugal technique for multi-layer growth on smaller substrates, whose growth faces are planar or profiled, has been demonstrated[2,3].

The advantages of LPE in the production process consist in low energy consumption as well as in ecological benefits.

The need for high quality semiconductor layers which during the past years has already been urgent for micro- and opto-electronics, has recently increased remarkably due to the successful application of such layers for photovoltaic devices.

We have now developed novel applications of centrifugal forces for growing crystalline semiconductor layers[8]. With these new techniques centrifugal forces are caused not only to transport the solution but in addition to separate the solute from the solution. The new technique offers advantages for growing crystals, for it makes possible high growth rates at low temperatures as well as iso-thermal growth.

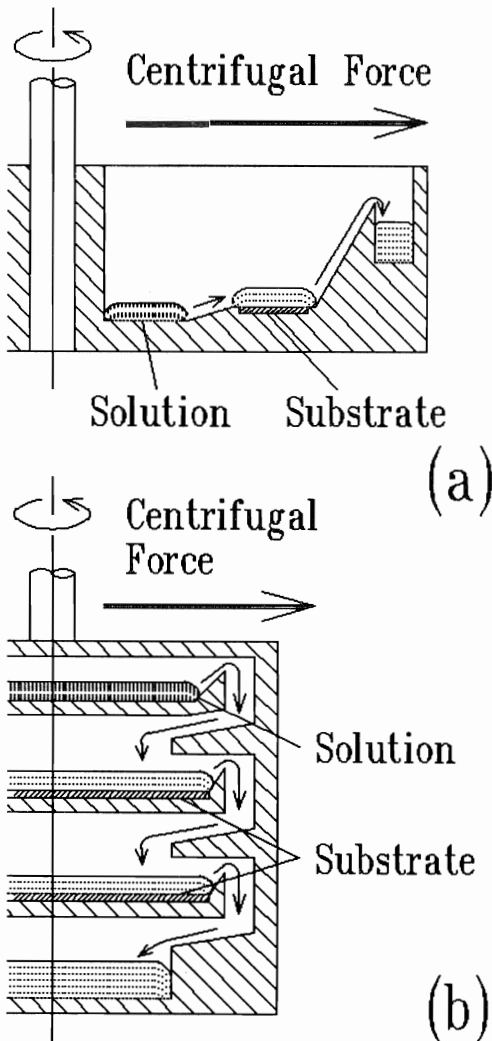
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### 2. OPERATIONAL PRINCIPLE OF A LIQUID PHASE EPITAXY CENTRIFUGE

Centrifugal force can be applied for several purposes in semiconductor liquid phase epitaxy.

### 2.1 Solution Transport

One of the functions of centrifugal forces in the LPE procedure is the transport of a solution. Figure 1 shows schematically two examples of our crucibles. The solutions are transported in the crucible by sequential action of centrifugal and gravitational forces. During the LPE growth procedure motion of the crucible components with respect to each other is not necessary. Attrition of the crucible components which could cause particle contamination of the system and of the epitaxial layers can thus be avoided. The solvent, which is usually a molten metal (In, Ga, or Bi), is first saturated with a semiconductor material such as Si or GaAs at high temperatures, i.e. 350 - 950 °C. The saturated solution is then transported on to the



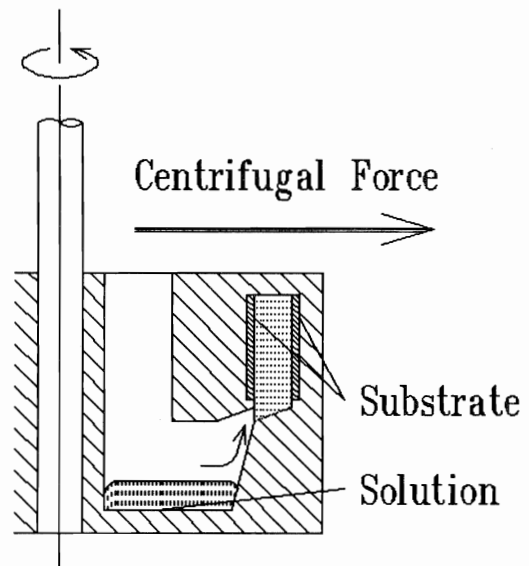
**FIGURE 1:** Schematics of Crucibles used for Centrifuge for Solution Transport. Substrates can be arranged either Out of Axis (a) or On Axis (b)

substrate. The substrate may be a crystalline plate of Si or GaAs which we call *wafer*. Next the crucible is cooled at a constant cooling rate of between 5 and 100 K/h. During the cooling the semiconductor solute material grows epitaxially on the substrate because the solubility of semiconductor materials in the solvent decreases with decreasing temperature. By removing the solution from the wafer growth can be terminated. During growth the crucible can be rotated at relatively low rotational speed in order to homogenize the solution.

Figure 1 (a) shows a LPE centrifuge crucible in which substrates are arranged outside the rotational axis. In the crucible shown in Fig. 1 (b), two 4"-diameter wafers are mounted in such a manner that the wafer centers coincide with those of the rotational axis.

### 2.2 Material Transport in the Solution

Another method for applying centrifugal forces consists in separating solute and solvent. One of the crucibles which has been used, for example, for growing Si from a solution of Ga saturated with Si is shown schematically in Fig. 2. First the solvent Ga is saturated at high temperature. Then the crucible set in rotation. As a result the solution moves into the upper part of the crucible. Provided crucible rotation is maintained, the concentration of Si increases at the surface of the substrate which is attached to the inside wall of the growth chamber because the molar volume of Si is larger than that of Ga. A Si substrate attached to the outside wall of the chamber serves as Si source. In short, the dissolved Si can be driven by centrifugal force towards the inner side of the growth chamber



**FIGURE 2:** Schematic Drawing of a Crucible used in a Centrifuge for Material Transport in the Solution

where we can place the substrate for epitaxial growth. Relatively high rotational speed of the crucible is required in order to create sufficiently large centrifugal forces. At a point which is, for example, 22 mm away from the rotational axis,  $2.0 \times 10^3$  rpm (revolutions per minute) are required to create  $100 g_n$ . Here,  $g_n$  means the standard acceleration of free fall ( $9.807 \text{ ms}^{-2}$ ).

### 3. REQUIREMENT FOR MAGNETIC BEARINGS IN LIQUID PHASE EPITAXY CENTRIFUGE

#### 3.1 UHV Compatibility

In order to avoid contamination and oxidation of semiconductor materials liquid phase epitaxy is often carried out in a pure hydrogen gas chamber at atmospheric pressure, and the system must be evacuated before hydrogen flush. The magnetic bearing system has to be UHV tight. Ultimate pressure should be below  $10^{-8}$  mbar. In order to fulfill all requirements, the rotor should be enclosed in a vacuum tank. Bearing magnets, sensors, and motor-stator stay outside the tank. All of the materials used in the vacuum tank require very low out-gassing as well as very low vapor pressure at the operation temperature. Lamination of rotor parts is not appropriate. The vacuum tank including the rotor should be bakeable at  $300 \text{ }^\circ\text{C}$ .

#### 3.2 High Temperature Operation

The semiconductor crystals are grown at temperatures below  $950 \text{ }^\circ\text{C}$ . Crystal growth takes place in the crucible fastened at one end of the shaft. Heat flows from the crucible through the shaft towards the rotor and in addition hydrogen gas at atmospheric pressure conducts heat from the crucible towards the bearing. When the temperature of the crucible is as high as  $950 \text{ }^\circ\text{C}$ , the rotor near the bearing is, as has been estimated, warmer than  $150 \text{ }^\circ\text{C}$ . It should be mentioned that the shaft material must be chemically inert against hydrogen at high temperatures.

#### 3.3 Rapid Acceleration and Deceleration

Crucible acceleration up to the desired rotational speed, and its deceleration should be as rapid as possible in order to minimize the time for applying the solution to the substrate and for removing it. For example, with a Ga solvent and a starting growth temperature of  $930 \text{ }^\circ\text{C}$  and a cooling rate of  $60 \text{ K/h}$ , the layer grows to a thickness of about  $0.9 \text{ } \mu\text{m}$  during the first minute. This means that, if it takes 1 min to remove the solution totally from the substrate, the layer thickness at the place where the solution begins to withdraw would be  $0.9 \text{ } \mu\text{m}$  thinner than that at the opposite end. A similar situation exists when the solution is supplied onto the

substrate. In order to grow a layer of uniform thickness, rapid acceleration and deceleration of a crucible is, therefore, required.

#### 3.4 Flexibility of Magnetic Bearings

The total amount of solution in the crucible may differ according to each particular growth experiment. Moreover, the solution moves inside the crucible during rotation. Although the weight of the load, may be several ten to several hundred grams, it is relatively small compared to the total weight of the rotating part. The magnetic bearings have to cope with changing imbalance and a changing solution mass.

#### 3.5 Safety of the System

Our crucible rotates in a quartz-tube reactor filled at atmospheric pressure with hydrogen and is heated externally by a cylindrical electric furnace. The space between crucible and quartz tube is small in order to obtain satisfactory temperature uniformity.

In case of electric power failure, an uninterruptable power supply keeps the AMB system operational, and in case of a hardware failure, retainer bearings guarantee the safe rotation of the rotor until it stops. Precaution against hydrogen-oxygen explosion is indicated.

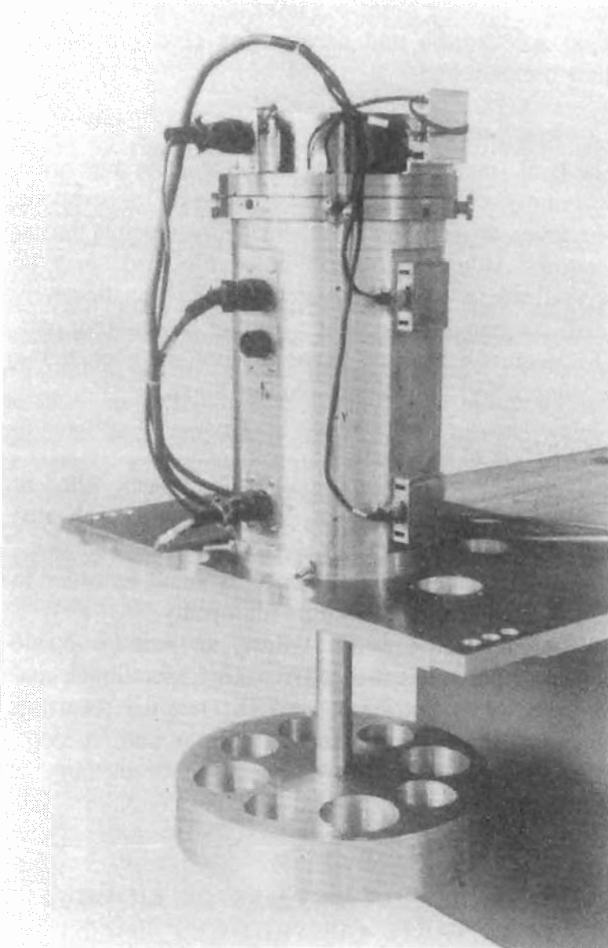
### 4. CONSTRUCTION DETAILS OF LIQUID PHASE EPITAXY CENTRIFUGES

#### 4.1 Overview

Since temperatures are between  $350$  and  $950^\circ\text{C}$  at the one rotor end, a great distance is needed between the magnetic bearings and the hot end of the rotor which is carrying the crucible. Therefore, the AMB controllers have to deal with long, elastic rotors whose center of mass lies outside the two radial magnetic bearings (Figs. 3 and 4).

Centrifuges having magnetic bearings on both sides of the crucible would provide better rotor-dynamics, but an "overhanging crucible" is easier to be loaded and unloaded.

In order to fulfill UHV requirements, the rotor can not have laminations. Magnetic losses should, therefore, be kept low by homopolar bearing magnets. The vacuum tank is to be made from non magnetic stainless steel and standard UHV flanges must be used to join the different components of the centrifuge. Therefore, the AMB dimensions have to be fitted to the available sizes and diameters of the UHV flanges, and the measurements of the rotor position have to be made through the vacuum tank wall. For this purpose, inductive sensors as well as magnetic sensors have been used.



**FIGURE 3:** Centrifuge III with a Crucible-dummy from Aluminum at the Lower End of the Rotor

#### 4.2 History

Ever since 1979, we have been applying three different types of vertically arranged magnetic bearings.

*Centrifuge I*, shown in Fig. 4 (a), was built in 1979 by H. Ulbrich at the Technische Universität München[1]. There, the crucible, which has a mass of about 400 g and a diameter of 70 mm was mounted on a long shaft whose diameter was 40 mm and the total rotor length was about one meter. A rotational speed of 4000 rpm was achieved in the epitaxy centrifuge I. This meant that almost the first bending critical speed was reached. Owing to the upright position which had its center of mass above the bearings and owing to the relatively low bearing force, the system required precise alignment in a vertical direction. During the years following 1979, the AMB control was provided with an analog state space controller. The rotor position was measured by magnetic sensors combined with Hall sensors. Analog power amplifiers from Hewlett Packard were used.

*Centrifuge II*, whose primary advantage lies in a much higher load capacity is shown in Fig. 4 (b). It was built in 1981 at the Eidgenössische Technische Hochschule in Zürich[2]. A crucible of about 4.3 kg was hung at the lower end of the rotor. Centrifuge II was equipped with an analog state space controller. The rotor position is measured by industrial encapsulated inductive sensors with a modulation frequency of 5 kHz. The rotor can reach a maximum rotational speed of 1000 rpm; it takes the rotor 18 seconds to reach 500 rpm.

Power amplifiers of the same kind are used in centrifuges I and II, and in both, the axial bearings are placed between rotor and crucible.

*Centrifuge III*, shown in Fig. 4 (c) was built by Mecos Traxler AG. The intention was to increase the crucible dimensions in order to be able to grow in each epitaxial run layers on several 4" wafers. A total rotor mass of almost 60 kg, with a crucible of 420 mm diameter and a mass of 27 kg is suspended by an axial bearing located at the top of the system.

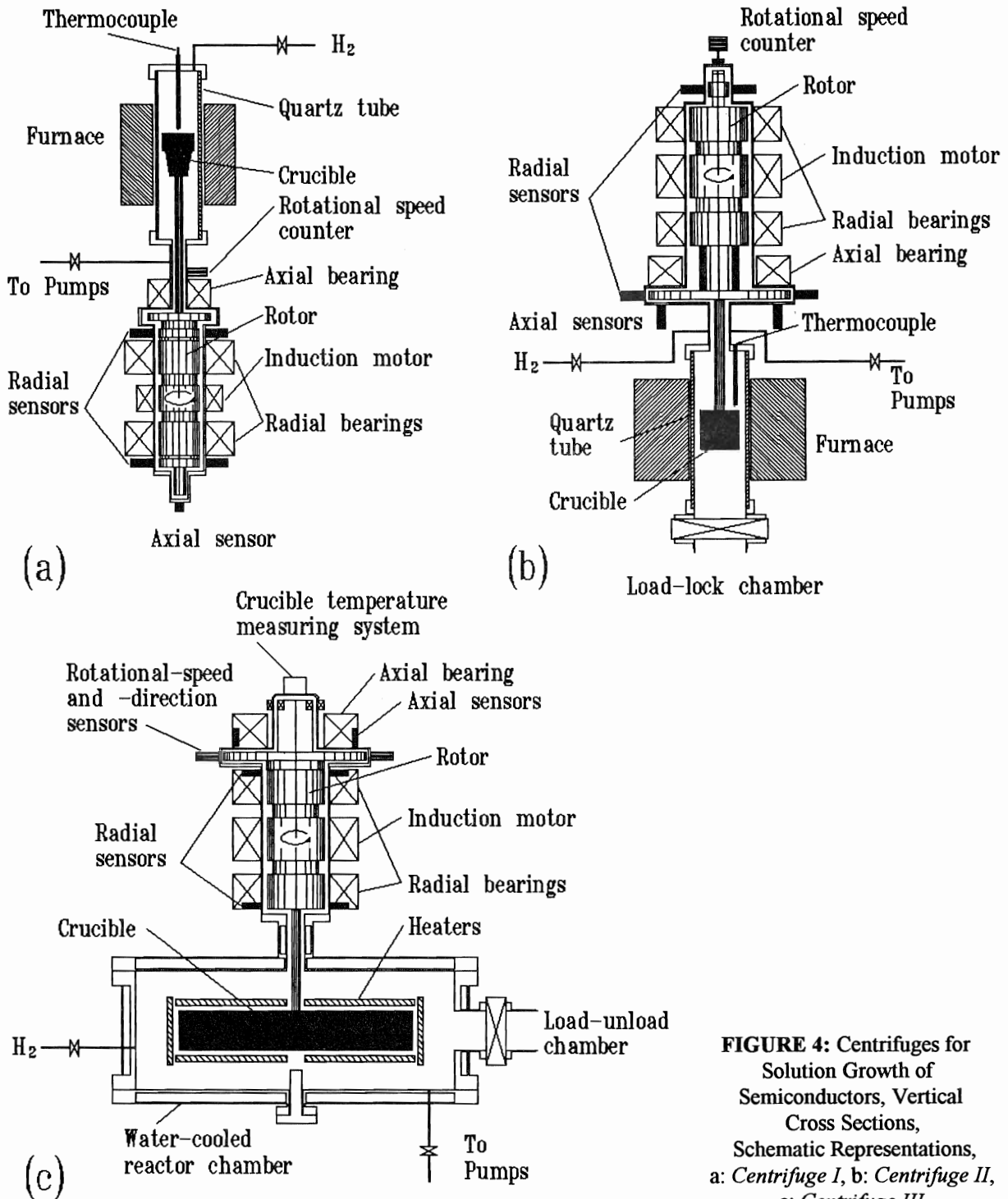
The AMB system is digitally controlled by a DSP. Switched power amplifiers are used to reduce loss at higher available power. To drive the inductive sensors, the configurable standard sensor-electronics allow modulation frequencies from 1 MHz down to 3 kHz. The frequency is set at 7.5 kHz in order to facilitate measurements through the vacuum tank. The rotor drive consists of a frequency inverter and an asynchronous motor. Laminations of the rotor are not permitted inside the vacuum tank. The squirrel-cage rotor, therefore, is made from massive iron.

#### 4.2 Retro-fit

Many years after it was set up centrifuge I needed complete renovation. Centrifuge II needed a larger crucible. Both centrifuges were, after 14 and 11 years, respectively, of operation, retro-fitted by Mecos with a digital controller of the most advanced technology. This guaranteed future operation.

In *centrifuge I*, the former magnetic sensors were replaced by inductive sensors which had identical dimensions. They are now driven by a sensor electronics identical with the one in centrifuge III. The power amplifiers have remained unchanged. New technology is represented by digital controllers which allow easy adaption and tuning to new requirements.

In *centrifuge II*, only the control rack with the analog controller was replaced. The digital controller rack was built with compatible connectors. The exchange of the old analog with the new digital control, and the necessary identification and tuning was accomplished within one day.



**FIGURE 4:** Centrifuges for Solution Growth of Semiconductors, Vertical Cross Sections, Schematic Representations, a: Centrifuge I, b: Centrifuge II, c: Centrifuge III

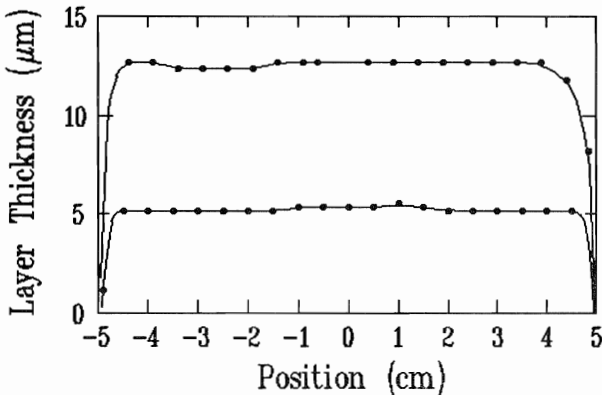
**5. EPITAXIAL LAYERS GROWN BY LPE CENTRIFUGES**

The epitaxy *centrifuge I* has been applied for growing semiconductor crystals based on the function of material transport in the solution as described above.

Using *centrifuge II*, we have prepared intentionally and unintentionally doped n- and p-type Si layers on 4"-diameter mono- and poly-crystalline Si substrates, and GaAs layers on 4"- diameter GaAs wafers. The thickness uniformity of Si layers grown on a 4"-

diameter Si wafer is shown in Fig. 5 and, in both examples, is better than  $\pm 4.9\%$ , excluding a peripheral 5 mm region.

We have also grown on small substrates multilayers with more than 10 thin p- and n-type Si and GaAs layers, using the same centrifuge but another crucible.



**FIGURE 5:** Thickness Uniformity of Si Epitaxial Layers grown on 4"-diameter Monocrystalline Si Wafers

Various surface topologies of epitaxial layers, such as pyramid, roof, or wall shapes have been obtained by employing patterned or partially masked substrates. These layers and layer structures have been and will be applied for semiconductor devices and solar cells.

## 6. CONCLUSION

Active magnetic bearings, successfully applied for liquid phase epitaxy centrifuges of different design for more than ten years, are the essential components of epitaxy centrifuges. They open new areas for solution growth of semiconductor layers. By applying AMBs, crucible rotation in clean vacuum or hydrogen environment has become possible.

By retro-fit of the used centrifuges by replacement of the older controller electronics with modern digitally switched power amplifiers, new data for future equipment architecture could be gained - which shows the wide modularity of the active magnetic bearing technology.

Using this technology has made possible upscaling of centrifuges so that liquid phase epitaxy technique can be applied in industry and provide us with the urgently required capability for large scale production of high quality materials for solar cells, photo- and particle-

detectors, and SOI (Semiconductor On Insulator) structures.

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