Centrifuge for Epitaxial Growth of Semiconductor Multilayers

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Abstract

We describe a vacuum centrifuge which consists of a long vertical rotor that is at its upper end electromagnetically suspended inside a vacuum tank. The design is arranged such that it fulfills the requirement for a thoroughly clean vacuum recipient. Experiments are discussed which illustrate the application of the centrifuge to liquid phase epitaxy of silicon multilayers.

Introduction

Preparation for the production of high quality epitaxial semiconductor layers requires conditions of extreme purity. Impurities and foreign atoms, even when present in minute traces, can impede the uniform growth of new crystalline material on the substrate and thereby impair the required structural perfection of the multilayers. In addition, impurities may alter the electrical and optical properties of the epitaxial layers.

Liquid phase epitaxy (LPE) requires crystal growth systems that provide for brief dwell times for the liquid applied to the substrates. Transporting the solution by centrifugal force in a rotating crucible is, therefore, advantageous. **Short** dwell times of the solutions ensure growth of the desired **thin** crystalline layers on the substrates. In growth systems equipped with conventional bearings for rotary motions, however, the quality and uniformity of the opto-electronic material properties and the reproducibility of the growth- results suffer severely if contaminated by lubricants, or because of wear and abrasion.

Magnetic bearings provide a unique opportunity for avoiding such difficulties [1,2]. The vacuum centrifuge described in the following text uses magnetic bearings and meets all requirements of liquid phase epitaxy. The description will be followed by a brief outline giving basic principles of LPE and explaining the features of operation of the rotating crucible within the centrifuge. Finally, a few examples of high quality silicon multilayers grown in the centrifugal system will serve to demonstrate the advantages which the centrifugal technique utilizing magnetic bearings possesses.

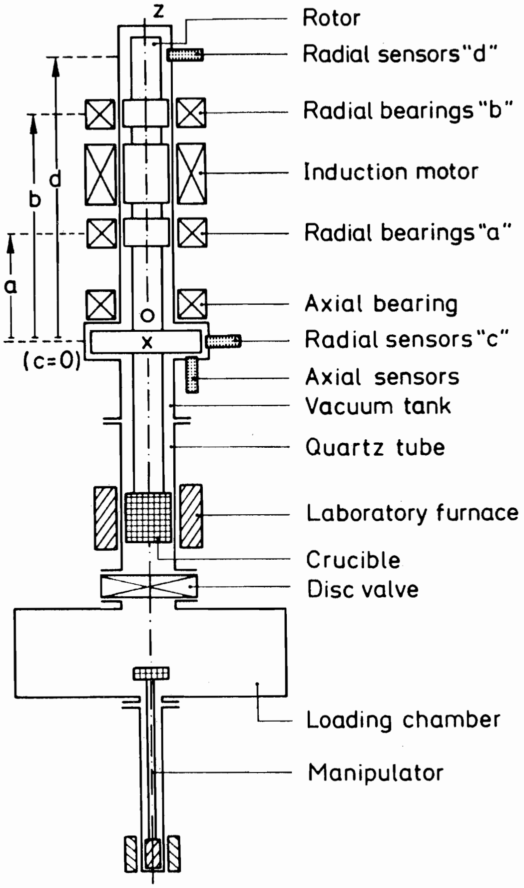

Fig.1. LPE centrifuge with contactless electromagnetic suspension of the rotor, schematic longitudinal section after [1-4]. The bearing and sensor plane positions a, b, c and d are measured from the centre of gravity at x(c=0). The planes are perpendicular to the z-axis (see also Table 1).

Vacuum epitaxy centrifuge

Figure 1 shows a schematical longitudinal section of the epitaxy centrifuge. The epitaxy crucible is mounted at the lower end of a vertical rotor. The rotor's upper end constitutes a part of a contactless electromagnetic suspension arrangement inside a thin-walled vacuum tank [2]. The bearing magnets and the rotor drive are outside the tank. The upper part of the tank surrounds the rotor and consists of a stainless steel tube. A flange connects the stainless steel tube with a quartz recipient. The lower end of the rotor and the crucible are surrounded by a quartz recipient. The crucible is heated from outside the quartz tube by a laboratory furnace. To reduce contamination of the system, the crucible is loaded and unloaded through a loading chamber. The loading chamber is flushed with pure hydrogen and is equipped with a magnetic feedthrough and a manipulator. Since temperatures of about 1000°C may be required in the crucible, the magnetic bearing and the rotor drive are water-cooled. The cooling jacket provides also for the mechanical rigidity of the system, Fig. 2.

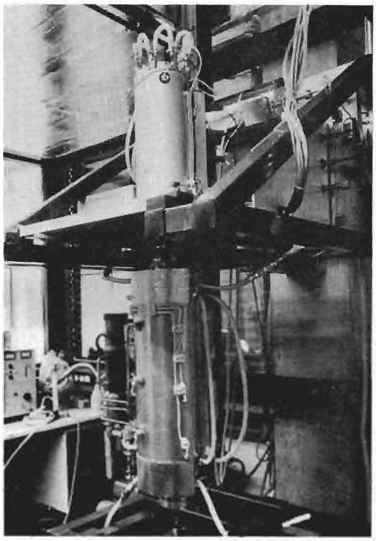

Fig.2. Upper part of LPE growth system with centrifuge. Loading chamber and manipulator are removed.

Magnetic bearing surrounded by cooling jacket

Rotor and magnetic bearing

Figure 1 illustrates the arrangement of bearings and sensors. Figure 3 shows the rotor of the vacuum centrifuge. The various physical system parameters are indicated in Table 1. The electromagnet which generates the bearing force is part of an automatic control loop. The radial gap between rotor and bearing is 1.8 mm wide. More than half of the distance is filled by

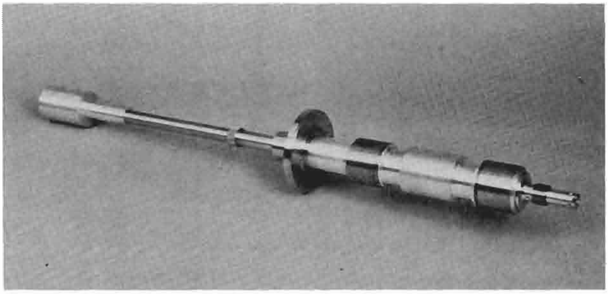

Fig.3. Rotor with two bearing rings for the radial bearing (right), support disk for the axial bearing (center) and payload crucible (left).

the 1 mm thick wall of the vacuum tank. As a consequence, rotor movements in the tank are restricted to 0.4 mm. In order to prevent tank damage owing to "overload" or sudden stoppage of the rotor, two graphite rings are fastened in the tank and serve as emergency bearings. The radial bearing has an 80 mm inner diameter and the comparatively large gap width mentioned above. The ratio of bearing force to control current is approximately 60 N/A. The gain factors are selected so as to achieve a maximum radial force of 60 N. The bearing's stiffness obviously depends on the dimensioning of the controller and is frequency-dependent. Bearing forces are fully effective up to a frequency of about 30 Hz. As shown in Fig. 4 they decrease with a further increase of the frequency. The system must be sufficiently vibration-free [5].

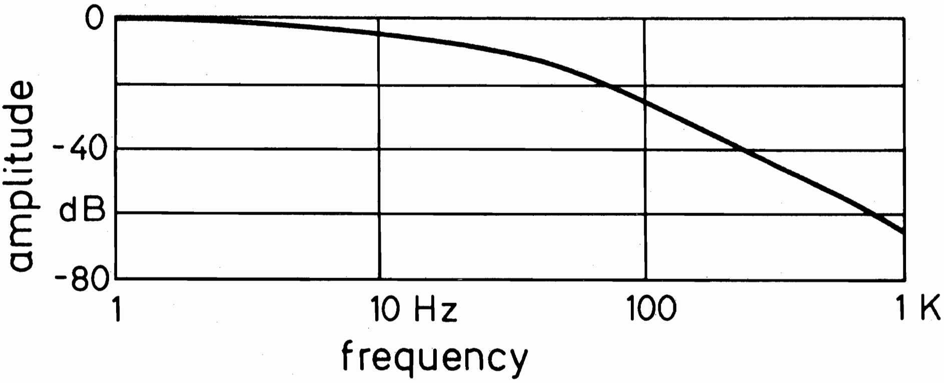

Fig.4. Amplitude-frequency relation of radial rotor motion.

Table 1. Physical System Parameters for the Centrifuge

Rotor length	L	=	1	m
Rotor mass	m	=	9	kg
Radial moment of Inertia	I_x	=	.616	kg m^2
Axial moment of Inertia	I_z	=	.0092	kg m^2
Rotational speed	Ω	=	1000	rpm
Force-current factor	k_i	=	66	N/amp
Force-displacement factor	k_s	=	80	N/mm
Bearing and sensor positions (Fig.1):				
Bearings "a"	a	=	118	mm
Bearings "b"	b	=	346	mm
Sensors "c"	c	=	0	mm
Sensors "d"	d	=	411	mm

Description of the crucible and epitaxial growth of multilayers

In LPE, single crystalline layers are deposited from saturated solutions on usually single crystalline substrates. The solvents applied for this purpose consist of molten metals saturated with the substance which is to be deposited epitaxially. The saturated solutions are brought into contact with the substrate, Fig. 5. Cooling of the system causes an epitaxial layer to grow on the substrate. When the solution is removed from the surface of the grown layer the epitaxial growth is terminated.

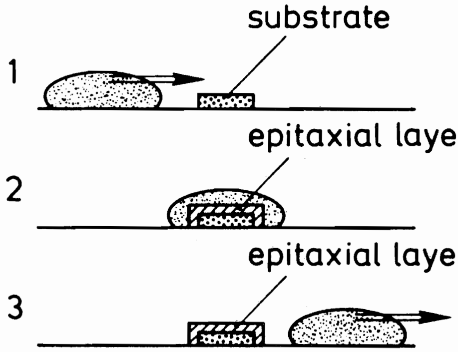

Fig.5. Liquid Phase Epitaxy (LPE). The three basic stages in layer growth procedure:

1 solution and substrate are brought into contact

2 cooling of the system: growth of the epitaxial layer

3 solution is removed from substrate.

One of our graphite crucibles of simple design which allows for the basic processes which are illustrated in Fig. 5 is schematically shown in Fig. 6. This crucible comprises four containers for solutions as indicated by the number "1" in Figs. 6a and b.

One way in which the growth procedure of the npn-multilayer can occur is the following [1]: Two of the containers opposite to each other, as for example those close to A and C, are filled with As- and Ga-doped, saturated solutions, respectively. Then two substrate wafers are placed in positions A and C, and two undoped wafers serving resaturation of the solutions are placed at positions B and D. When the crucible starts to rotate, the solutions flow from container position 1 to the growth positions by which they cover the substrates 2, whereby filling the gap-like channels above the substrates A and C. In this particular example, the crucible then rotates with 230 rpm.

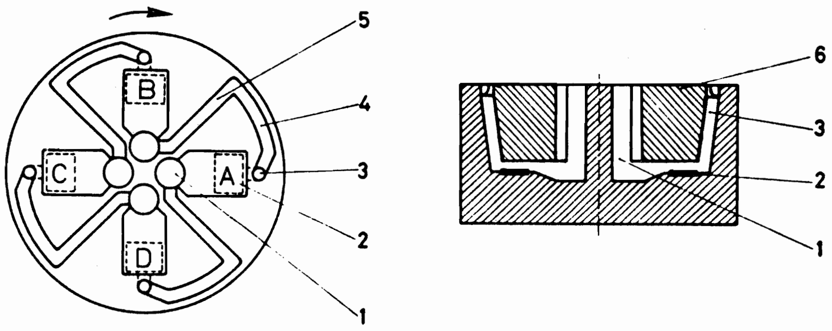

Fig.6. Crucible of the LPE centrifuge (a) cross section, (b) longitudinal section. The crucible itself contains no moving parts. The solutions are transported by centrifugal force. (1) containers for solution, (2) substrates, (3), (4) and (5) solution-flow channels, (6) substrate holder blocks.

If the temperature of the revolving crucible is reduced, growth of epitaxial layers on the substrates A and C results. Increasing the rotational frequency of the crucible causes the solutions to be withdrawn from the substrates. Growth is then terminated and the solutions move upwards through the flow-channels 3 into the storage-channels 4. The solutions remain there as long as the crucible rotates at higher speed, e.g. in this example 600 rpm. When the rotational frequency is reduced again, the solutions fall through the channels 5 (Fig. 6a) into the inner containers close to B and D. If the frequency of the boat is thereupon once more increased up to 230 rpm, the solutions creep into the gaps above the wafers B and D. These wafers being undoped, the solutions become, with an increase of the temperature of the crucible resaturated at the expense of the wafers B and D. During further increases, decreases and again increases of the rotational frequency of the boat, the solutions move on to substrates C and A. There, during times of temperature reduction, the next epitaxial layers grow. This growth cycle for pn- and np-structures can be repeated many times during one run. Both the operation of the epitaxy centrifuge and the epitaxial growth process are computer controlled. After charging the crucible the automated epitaxy apparatus produces single- or multilayers according to a chosen program. It needs no supervision.

Examples of grown multilayers

The repetitive and clean operation of the epitaxy centrifuge serves to grow high quality multilayers, such as pnpn-structures [4,5] or other sequences of layers. Fig. 7 shows a silicon pnpn-multilayer grown from two different saturated solutions of silicon in Indium, doped with Ga and As, respectively. The scanning electron micrograph of a photo-etched cleavage face of this sample shows that the thickness of the individual layers can be precisely controlled because of the automation of the system. An example which shows the defect-free growth of multilayers with even much thinner individual layers, as observed with the help of transmission electron microscopy, is given in Fig. 8, and the sequence of As- and Ga-doped layers is clearly visible. No crystalline defects have been formed during

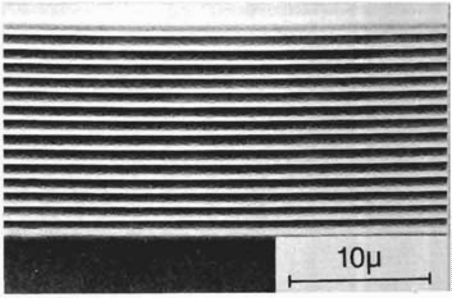

Fig.7. Silicon pnpn-multilayer grown in centrifuge. Photoetched cleavage face, SEM-micrograph. p-layer $10^{18}cm^{-3}$ Ga, n-layer $10^{18}cm^{-3}$As [7].

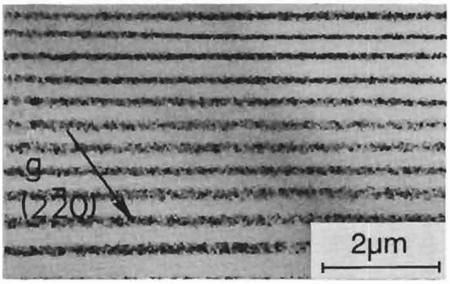

Fig.8. Transmission electron micrograph of pnpn-multilayer. Cross-section specimen, g diffraction vector. The multilayer is free of defects.

growth. This result is mainly due to the cleanliness which has to be observed during the whole growth process. It has to be noted that in the two cases illustrated in Figs. 7 and 8 the interfaces are remarkably planar which is a prerequisite for the technological application of multilayers.

Other examples of epitaxial growth, in particular the growth of multilayers on structured substrates, and of selective epitaxial growth yielding silicon on insulators, are given in the literature [1,4-6].

Conclusions

The vacuum centrifuge equipped with magnetic bearings has been successfully used in many experiments for the growth of semiconductor multilayers by liquid phase epitaxy. The operation of the automated system has proven to be reliable, economical and productive of multilayers having highest crystalline and electronic quality.

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