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〈연구논문〉

Design of $Hg_{1-x}Cd_xTe$ OMVPE System and ARIIV Reactor Chamber

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$Hg_{1-x}Cd_xTe$ OMVPE System과 ARIIV Reactor Chamber의 설계 및 제작

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Abstract – The direct growth OMVPE system, designed specifically for direct growth of $Hg_{1-x}Cd_xTe$ using annular rectant inlet inverted vertical (ARIIV) reactor, was constructed. This paper presents the detailed technical approach on a newly designed ARIIV reactor that increases Hg incorporation, imposes uniformity, and avoids the needs for high temperature processing to create alloys by inter-diffusion approach.

요 약 – $Hg_{1-x}Cd_xTe$ 박막 성장을 위해 ARIIV(annular reactant inlet inverted vertical) reactor를 이용한 OMVPE(organometallic vapor phase epitaxy) 기구를 제작하였다. 본 논문에서는, 새로 고안한 ARIIV reactor 기술을 자세히 설명하였다. ARIIV reactor를 이용하면, 내부확산에 필요한 고온 처리 과정을 피할 수 있으며, Hg 혼합이 잘 된 균질한 $Hg_{1-x}Cd_xTe$ 박막을 얻을 수 있다.

1. Introduction

The convertional reactor configuration of $Hg_{1-x}Cd_xTe$ OMPVPE system has shown problems, with respect to reproducibly growing clean, low defect density, large area ($\geq 14\ cm^2$), $Hg_{1-x}Cd_xTe$ alloy epilayers having precisely controlled x ($0 \leq x \leq 1$), with compositional uniformities better than $\pm 1\%$ of the second decimal place in x and excellent electrical uniformities ($\pm 1\%$). Since these are necessary for the purpose of this study, we designed and constructed an all stainless steel reactor system and a modified inverted-vertical (IV) chamber (to be discussed in the next section) which can facilitate the delivery of Hg to the growth surface, without sacrificing the uniform growth characteristics obtained in IV reactor, because the thickness, composition and doping uniformities obtained in the IV reactor configuration exceed uniformitity requirments for staring arrays [1-3].

2. OMVPE System

The main components of an OMVPE reactor system designed for $Hg_{1-x}Cd_xTe$ epilayer growth are (1) a source of high-purity carrier gas, in most ins-

tances (but not necessarily) H_2; (2) temperature-controlled reactant source bubblers, usually in the form of specially designed stainless steel or commercially available stainless steel; (3) gas flow manifold and control system, including appropriate multiple gas flow paths with suitable valving and electronic mass-flow controllers, for acheving the desired reactant concentrations at the proper times in the substrate region; (4) a reaction chamber made of high-purity fused quartz for good visibility into the layer growth region, enclosing the substrate-supporting fused quartz pedestal and including a means of heating the substrate directly and controlling its temperature; and (5) a gas exhaust line, with appropriate filters for particle removal and provision for burn-off of H_2 and an evacuation pump for the gas mainfold system. In simplified form, the OMVPE reactor system designed for the growth of epitaxial $Hg_{1-x}Cd_xTe$ layers on suitable substrates is shown in Fig. 1. Ultra high-purity H_2 from a Nano-chem purifier and semiconductor grade Ar were directed, by suitable valving and parallel gas lines, toward a series of electronic mass-flow controllers (MFC). As shown at the center of Fig. 1, two of these carrier gas paths, after passing through MFC, join the input line of the gas manifold. This input line is connected to the main reactant bubbiers of DMCd (dimethylcadmium) and MATe (methylallyltelluride) alkyls. The bubblers were maintained by thermostatted refrigeration baths at the desired temperature. Temperature control of the elemental Hg bubbler and the Hg delivery line, extending from the bubbler to the annular chamber ("reactor" in the Fig. 1), was achieved by wrapping electrical heater tape around both components. This purpose was to prevent Hg condensation in the Hg delivery line, which was 10℃ warmer than the Hg bubbler, during the growth. The reactor in the Fig. 1 is an inverted-vertical configuration (to be discussed in detail in the next section). The filtered gas exhaust line and burn-off unit at the upper right of particle trap were located at the top of the hood which houses the OMVPE reactor system. The rotary-vane vacuum system was connected to the reactor for system leak checking.

The gas lines of the system were made exclusively of Type 316 stainless steel tubing, which was carefully cleaned properly pickled and passivated

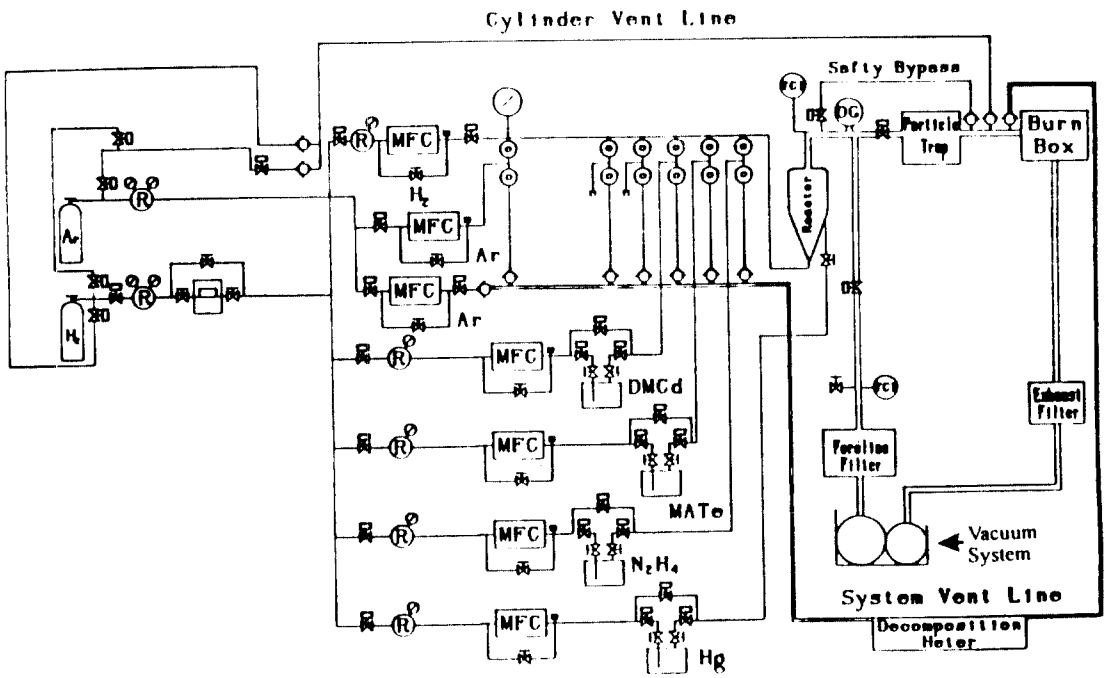

Fig. 1. Simplified schematic diagram of OMVPE reactor system for II-VI epitaxial growth.

on the interior surface prior to assembly. All permanent joints were formed by heliarc welding, or VCR fittings. Gas-activated solenoid-operated fast-respc-nse valves were used exclusively in the manifold system, with a low-dead-space construction to minimize the mass-flow inertia of the gas handling of the system and to maximize flow-rate control critical to various steps in the OMVPE growth sequence.

3. ARIIV Reactor Chamber Design

There are several fundamental generic problems that have to be overcome in the development of an ideal reactor chamber configuration. The first of these is the intrinsic difficulty of devising a laminar, mass transfer controlled system having a high degree of spatial uniformity. Bounary layer formation in the *entrance regions* would strongly mitigate against achieving such uniformity.

The second of these problems is that in order to achieve thermal decomposition a heated substrate has to be employed, the presence of such heated surfaces will give rise to thermal natural conviction, which again tends to produce spatial non-uniformities in the transport rates at the solid surfaces.

The Spire reactor chamber has provided an ingenious solution to this dilemma by seeking to estabish a balance between the forced and the buoyancy driven convective flows-achieving good results, but only a partial success. Furthemore, the production of heterostructures with a good degree of abruptness could be quite difficult in such a system.

Numerous recent effors have been aimed at the utilization of a stagnation flow concept, where a gas stream, possibly a distributed gas stream is made to impinge on a single wafer. These approaches represent promising avenues, but the extent to which the ultimate process objectives can be achieved (*i.e.* a very high degree of spatial uniformity, in the 1~2 % range and a high degree of abruptness) is still not clear.

The third problem of conventional $Hg_{1-x}Cd_xTe$ reactor chamber, whether of the horizontal or the vertical configuration, is the introduction of hot Hg liquid into the upstream portion of the OMVPE reactor that leads to a lack of process control, because the hot Hg liquid reacts with Te and Cd alkyls. This configuration also should keep the entire reactor warm (about 240℃), to prevent the elemental Hg condensation in the reactor chamber wall. As result of the hot Hg vapor and the high temperature of hot reactor wall, the pre-crackings of Cd and Te alkyls is produced before the reactant gases reach to the growth surface and causes an unpredictable composition of the $Hg_{1-x}Cd_xTe$ alloy. epilayer.

Considerable attention, therefore, was given to the design of the reactor chamber, to facilitate elemental Hg transport to the growth surface in such a way that reactions between hot elemental Hg and organometallics in the system plumbing and upstream portions of the reactor chamber is avoided. Uniformities of thickness, composition and dopant incorporation superior to any other thin film growth process have been demonstrated, repeatably, in the IV reactor [1-3].

The IV reactor chamber assembly includes a reactor tube, a reactor cap, a fused quartz pedestal, a graphite susceptor, and a thermocouple sheath. The key difference between the IV reactor chamber [3] and the ARIIV reactor chamber is the addition of an annular reactant inlet(ARI), showen in Fig. 2. The Hg delivery approach used here is an important modification to the proven IV reactor design, in order to employ it for $Hg_{1-x}Cd_xTe$ growth. The unassisted pyrolytic OMVPE approach for $Hg_{1-x}Cd_x$-Te alloy growth consisted of the utilization of Hg vapor, and Cd and Te alkyls which completely decompose about 100 times faster than their total contact time with a growth surface (substracte) at temperatures of $\leq$300℃. Under these conditions the growth rate is independant of substrate temperature, and it is only necessary to control the flow rates of Hg vapor, and the Cd and Te alkyls in order to control the compositions.

Our unique approach delivers Hg to the growth surface, through an annular reactant inlet, shown in Fig. 2. This approach permits Cd and Te alkyls to be introduced in the normal way (from the bottom of the reactor chamber), while heated elemental Hg is introduced into the reactor at the substrate surface through the annular reactant inlet (ARI). This eliminates Hg liquid from the upstream gas

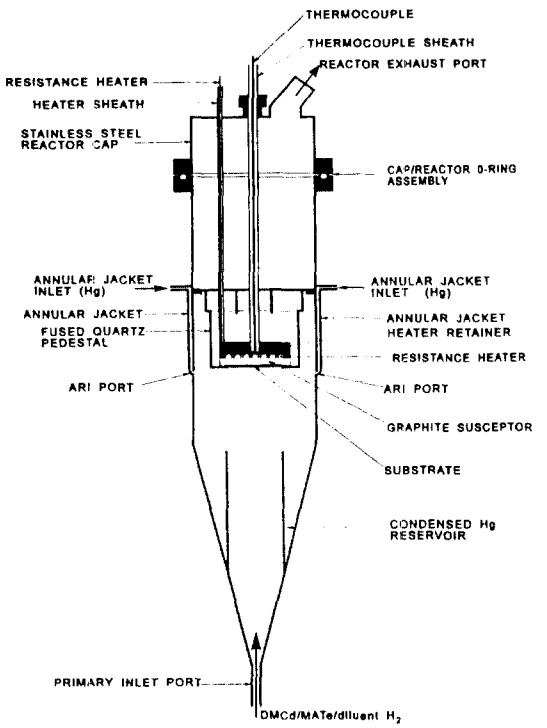

Fig. 2. Cross-sectional drawing of the ARIIV reactor chamber assembly.

flow and increases the impingment of Hg vapor onto the growth surface by a factor of than 100 for a given Hg vapor flow rate; it also increases the rate at which Hg vapor can be delivered to the growth surface. In addition, this approach facilities complete mixing of Hg with Cd and Te alkyls over the entire substrate surface.

In the ARIIV reactor, transport of Hg vapor to the ARI port is accomplished by introducing it into an annular reactor tube wall jacket near the top of the reactor. The annular jacket inlet was modified from a single inlet, which was designed at the first stage of system construction, to a double inlet at diametrically locations, in order to provide uniform pressure around the annular jacket. The annular jacket is enclosed by electrical band heater tape to prevent Hg condensation from the jacket wall. The purpose of the vertical offset between the entry port into the ARI port is to obtain uniform dispersal of Hg in the jacket before it enters the reactor at the ARI port. The Hg enters the reactor just below the substrate from a 360° arc which lies in a plane perpendicular to the reactor axis. The distance between the outside wall of the reactor tube and the inside wall of the jacket is 2 mm. This cross section is reduced to 1 mm at the ARI port to increase the Hg velocity as it enters the reactor, and to prevent other gases from back-streaming into the ARI port. The ARI port protrudes approxmately 1 mm into the reactor and injects Hg into the reactor in a plane perpendicular to the reactor axis.

In the ARIIV reactor chamber arrangement, the inside dilameter (ID) of reactor increases from 4 mm at the primary inlet port to a maximum of 89 mm at the top of the funnel shaped portion of the reactor. The angle between the reactor axis and the reactor tube walls in the funnel shaped portion of the reactor is 11.5°. Above this portion, the reactor the walls are parallel to the reactor axis. An 83 mm ID pedestal support ring is fused to the inner wall of the reactor tube, 70 mm below the mating surface of the reactor tube mating flange. A 45 mm diameter by 105 mm long glass tube is welded to the inside of the reactor, to form a reservoir condensed Hg.

The reactor chamber cap is fitted to the top of the reactor tube by means of mating flanges which are compressionally loaded against a viton gasket to form a vacuum tight seal. A 21 mm ID reactor exhaust port and a 6.3 mm ID thermocouple sheath inlet port are located at the top of the reactor cap. The thermocouple sheath inlet port opening is centered with respect to the reactor tube and it is parallel to the reactor axis. The exhaust port is connected to the system exhaust by a 24 mm ID stainless steel Ultra-Torr fitting. The reactor chamber cap system consists of 316 stainless steel.

The pedestal is a cup-shaped fused quartz cylinder, as shown in Fig. 3. The pedestal is coaxially supported within the reactor tube by an 86 mm diameter support collar which rests on the top surface of the pedestal support ring. The length and diameter of the pedestal are 59 mm and 74 mm, respectively. The side wall and the bottom surface are 2 mm thick.

The openings in the side wall, at the top of the pedestall, provide a flow path to the reactor exhaust

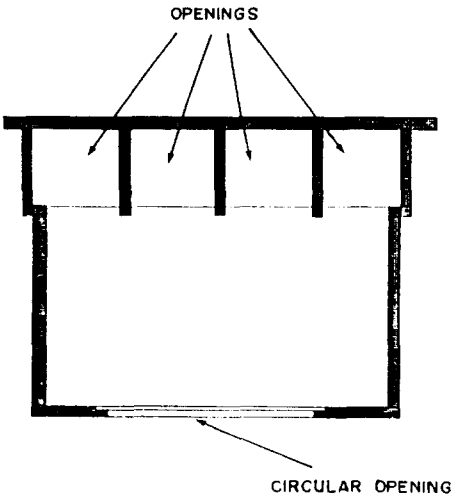

Fig. 3. Cross-sectional view of fused quartz pedestal.

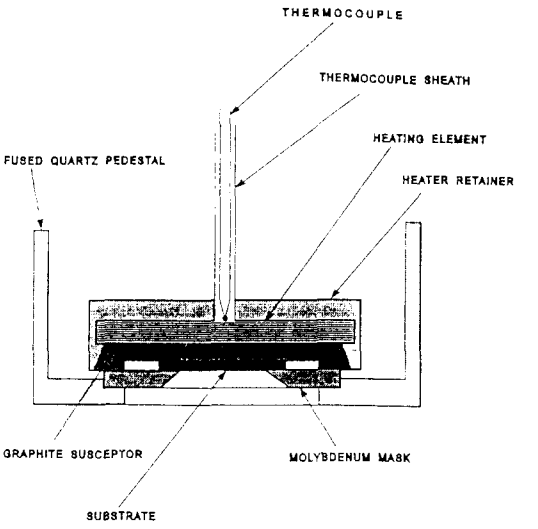

Fig. 4. Englarged view of the fused quartz pedestal, substrate, mask, and heater assembly.

for product gases. This is only one of several possible methods which can be emplyed to provide a flow path to the reactor exhaust.

The bottom surface (base) of the pedestal has a 46 mm centered circular opening, as indicated in Fig. 3, 5. The inside surface of the pedestal base has a 0.1 mm deep recess which extends, concentrically, 2.5 mm beyond the 46 mm diameter opening. Substractes (50 to 51 mm diameter) are supported on the 2.5 mm annulus, growth face downward, on the inside bottom of the pedestal. Smaller circular or irregularly shaped substrates are supported on a 51 mm diameter, 1 mm thick molybdenum mask which rests on the 2.5 mm annulus of the pedestal base. In this situation, substrate growth surfaces are exposed to the reactant gases through one or more openings in the mask, as shown Fig. 4.

Heating is accomplished by a direct resistance pancake heater, which is made of flexible high watt-density electric sheath heater wire coiled in the stainless steel heater retainer. The heater retainer rests on a graphite susceptor which in turn sits directly on the back surface of the substrate and inside the pedestal. The graphite susceptor is 63.5 mm and 57.5 mm in bottom and top diameter, respectively, and 5 mm thick. The bottom of the graphite susceptor also contains a 51 mm diameter concentric recess. A susceptor with a 0.1 mm deep recess is used for mounting 2 inch diameter substrates. The pedestal and susceptor recesses, in conjunction with the substrate (or substrate and mask), form an interlocking arrangement which centers the substrate and susceptor with respect to the reactor tube axis.

In the absence of exhaust pumping, the total flow rate that minimizes back streaming near the reactor tube walls (optimum total flow rate) is determined by the exhaust pressure and by the smallest cross-sectional area through which reactant products must pass, downstream from (above) the primary reactor inlet port. The reactor chamber exhaust pressure for all experiments reported in this paper was 760 torr. The minimum cross-sectional area for gas flow through the reactor was 3.46 cm^2, located at the exhaust port. The optimum total flow rate through the reactor is 2.9 standard liters per minute (SLPM). This flow rate minimizes the size of eddy currents, and localizes their position to the reactor tube wall just below the bottom surface of the pedestal.

4. Inverted-Vertical Reactor Design Purpose and Function

The inverted-vertical configuration, where in reactant gases are introduced below a downward facing

substrate and the reactant products are exhausted above the substrate, utilizes the buoyancy effect to remove hot reactant products from the reactor and to suppress the formation of backstreaming eddy currents [3]. The purpose of the 11.5° angle between the tube wall and the reactor axis is to minimize the dead volume between the inlet port and the substrate. The 11.50 angle was selected because it is the smallest angle that can be reproducibly made by conventional glass shop techniques.

Stagnation point flow and symmetry are the goals of most vertical reactor designs. The IV reactor achieves these goals by virtue of the inverted configuration and the orientation of the substrate surface perpendicular to the gas flow direction. Back-streaming eddy currents do not form at the growth surface because the buoyancy of the hot gases keeps them in contact with a downward facing surface until they are pushed beyond the radius of that surface and permitted to flow in an upward direction.

The reactor exhaust port, in the cap, is not centered with respect to the reactor axis. This asymmetry in the IV reactor design does not effect the growth uniformity when the reactor is used at atmospheric pressure because the pressure gradient in the reactor is created at the inlet port. However, growth uniformity under low pressure operating conditions, where the pressure gradient in the reactor is created at the exhaust port, may require that the exhaust port be concentrically positioned above the pedestal. Concentric positioning of the exhaust port would provide the best possible uniformity in pumping speed about the pedestal radius.

The arrangement of the pedestal, substrate, and susceptor within the reactor performs two functions, aside from the primary function of attaining the IV configuration, which are describe below.

Particulates cannot fall onto the growth surface because the substrate faces downward. Flaking of reactor tube wall deposits rarely occurs, except during very long growth runs, because this reactor tube is designed for cold wall pyrolytic OMVPE growth. When flakes do fall from the reactor tube wall, they just above the primary inlet port; where they swirl about in the inlet gas stream. The flakes do not enter the 4 mm diameter inlet tube until it is disconnected from the 6.3 mm OD stainless steel inlet tube.

Shielding the growth surface from impurities emanating from hot reactor parts is the other function of the pedestal design. The reactant and product gases do not come into contact with any hot surface, except the substrate, until they are downstream from (above) the growth surface. The hot reactor components are located in a region of high velocity (pedesal side walls), or a region of minimal flow (inside the pedestal) which is well isolated from the input gas stream and the growth surface.

The characterization performance of II-VI OMVPE system using IV and ARIIV reactor chambers has been demonstrated in other papers [3-7].

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