Fabrication of Thin-Film Cleaved Cavities Using a Bonding and Cleaving Fixture

D. W. McAlister, P. J. McCann, H. Z. Wu, and X. M. Fang

Abstract—**A novel method for the fabrication of cleaved-cavities has been developed that uses a copper plate assembly to support semiconductor layers after substrate removal. PbSe layers were grown through a combination of molecular beam epitaxy and liquid phase epitaxy on Si (100) substrates using CaF_2 and BaF_2 buffer layers. After growth the sample was bonded to the edges of a copper plate assembly epilayer down and the BaF_2 buffer layer was etched away allowing for growth substrate removal. This technique allows fabrication of cleaved Fabry–Perot resonant cavities by separating the copper plates after the substrate is removed.**

Index Terms—**AuIn, BaF_2, CaF_2, cleaved-cavity formation, epitaxial liftoff.**

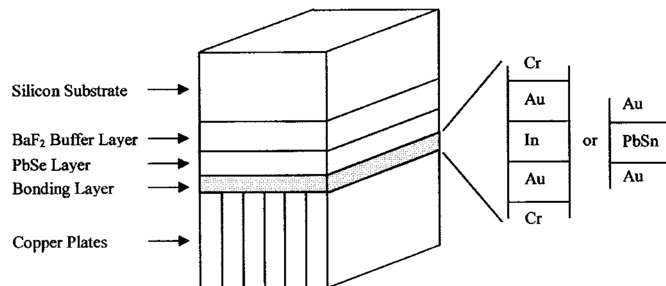

Fig. 1. PbSe/BaF_2/CaF_2/Si(100) structure shown bonded epilayer-down to a copper plate assembly. The copper plates were about 0.015 in (400 μm) thick.

I. Introduction

THE NEED for lasers working in the mid- to far-IR region of the electromagnetic spectrum has grown lately with the requirement to perform high-resolution gas spectroscopy in many areas of research and industry. The devices on the market today however, require cumbersome cryogenic cooling and can be quite costly to purchase and maintain. With advances in narrow bandgap semiconductor materials technology mid-IR lasers may reach higher working temperatures, and this should allow devices to be operated with compact thermoelectric (TE) coolers.

The compound semiconductor materials systems that are presently being used to fabricate mid-IR lasers are IV–VI semiconductors (lead salts), III–V semiconductors containing antimony, and III–V semiconductor quantum cascade (QC) structures. Lasers made from III–V (Sb) appear to be limited by nonradiative Auger recombination, which keeps emission under 250 K. QC lasers and IV–VI lasers both exhibit pulsed emission at or near room temperature, but the highest temperature for continuous wave (CW) emission from QC lasers is 160 K [1] whereas IV–VI lasers have demonstrated cw operation at 225 K [2]. For QC lasers, the QC superlattice structure itself may be the limiting factor in obtaining higher cw operating temperatures because folded acoustic phonon modes can arise which will prevent efficient active region heat dissipation. On the other hand, the limiting factor for IV–VI lasers is the thermally-resistive PbSe substrate (thermal conductivity of 4.2 W·m−1·K−1 @ 300 K). It has been predicted that a greater than 60° increase in operating temperature can be achieved by replacing the thermally-resistive lead salt growth substrate with a high thermal conductivity material such as copper [3]. This is possible by incorporating a selectively etchable release layer between the device layers and the substrate. The substrate can then be removed using an epitaxial lift-off technique much like those that have been developed for III–V semiconductor materials [4]. In the IV–VI system, BaF_2, a commonly used substrate material [5] and a buffer layer material for growth on silicon [6], can serve as an epilayer release layer. BaF_2 is water soluble, so it can be etched after epilayers have been bonded to the edges of a copper plate assembly without damaging the PbSe epilayer. After growth substrate removal, the IV–VI epilayer structure can then be cleaved to form Fabry–Perot resonant cavities by separating the copper plates. This letter describes this new cleaved-cavity fabrication technique [7] with particular emphasis on the IV–VI epilayer bonding procedure to copper.

II. Experimental Procedures

The samples used in the experiments were grown by a combination of molecular beam epitaxy (MBE) and liquid phase epitaxy (LPE). A typical epitaxial layer structure is shown upside down in Fig. 1. It consists of an LPE-grown PbSe layer, ~5 μm thick, that was grown on a (100)-oriented Si substrate with MBE-grown PbSe, BaF_2, and CaF_2 buffer layers. The thicknesses of these layers were 2000, 900, and 40 nm, respectively. LPE growth is needed because the MBE-grown PbSe layer has a high crack density due to thermal expansion mismatch with the Si substrate, but the subsequent LPE-grown layer is crack-free [6]. (It is believed that higher LPE growth temperatures allow plastic deformation of the PbSe layers via dislocation movement along higher order glide planes.) These

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LPE-grown PbSe layers were then bonded to the edges of a plurality of copper plates as shown in Fig. 1.

The bonding medium between the PbSe and the copper needs to meet certain criteria for the proposed fabrication method to be successful. The main characteristics needed from the bonding medium are brittleness, high thermal conductivity, and a low bonding temperature. Brittleness (hardness) is needed so that when the copper plates are taken apart the bonding layer will break before it plastically deforms. Breaking will cause the epilayers to cleave whereas plastic deformation would not effectively transmit shear stress to the PbSe epilayer. High thermal conductivity will aid in delivering heat away from the active region, and a low bonding temperature is needed to limit interdiffusion within the epitaxially-grown laser structure and to reduce stress to the epilayers due to thermal expansion mismatch with the Si substrate. To fulfill these requirements, thin layers of metals were focused on to create the bonding medium.

Before metal deposition the copper plate assembly was boiled in solvent baths (acetone and then methanol) and then baked for 12 h at 80 °C. The PbSe epilayer sample was blown with dry N_2 to remove any particles. The first bonding medium tried consisted of electroplated Au on the PbSe sample and electroplated layers of Au, PbSn 60/40, and another layer of Au on the copper plate fixture. The bonding took place in an annealing furnace that had been pumped down to ~1 mtorr and backfilled with a 10% H_2/90% Ar gas mixture. The furnace was raised to 200 °C in 20 min and allowed to remain for 20 min. While in the furnace, pressure was applied between the PbSe epilayer and the copper plate fixture by the use of an approximately 13-g weight on the 1 cm^2 sample. The furnace was then turned off and allowed to cool down to room temperature before the sample was taken out.

The second bonding medium tried employed the Au–In system [8]. For this method the copper plate fixture was cleaned in the same fashion except for being baked out at 200 °C overnight in a vacuum chamber with a background pressure of ~10^{-6} torr. The sample and copper plate fixture were then coated with ~500 Å of Cr and ~2000 Å of Au by thermal evaporation. Indium shot was used to produce the alloy during bonding and the bonding was performed on a hot plate in air at 200 °C. First, the copper plate fixture was placed on the hot plate and pieces of indium weighing a total of about 0.0065 g were placed on the gold area to become molten. The sample was then placed coated side down on the molten indium and while pressure was applied a light scrubbing action was performed to break down any oxidation that had developed on the indium shot. After about 20 min at 200 °C the bonded assembly was left to cool slowly to room temperature.

Both alloys successfully bonded the sample to the copper fixture as tested by applying an in-plane force to the side of the silicon substrate. The silicon growth substrate was then removed by soaking the bonded assembly in deionized water for three to four days. The resulting structure is represented in Fig. 2(a). After the growth substrate was removed, the copper plates were taken apart as shown in Fig. 2(b). PbSe preferentially cleaves along the {100} planes so careful positioning must be used to align the preferred cleavage planes parallel with the copper plates. During bonding, the edges of the Si substrate, which had

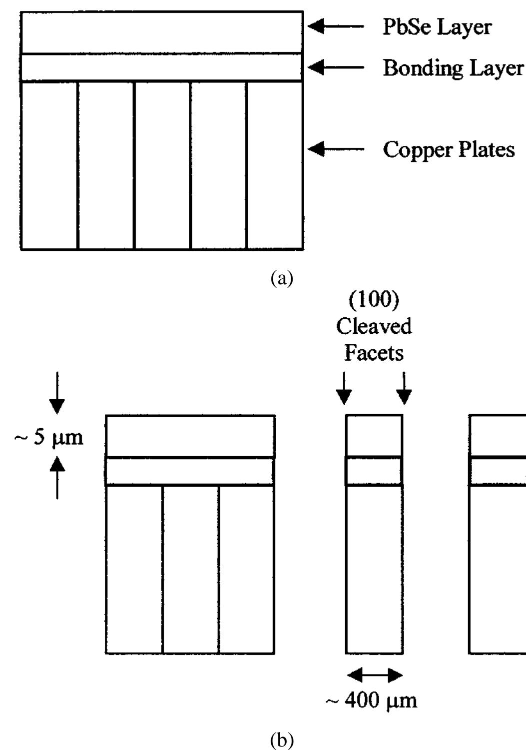

Fig. 2. Substrate removal and cleaving procedure after bonding. (a) PbSe bonded to edges of copper plates after dissolving the BaF_2 buffer layer in water. (b) Release of copper plates cleaves the epilayer into Fabry-Perot resonant cavities.

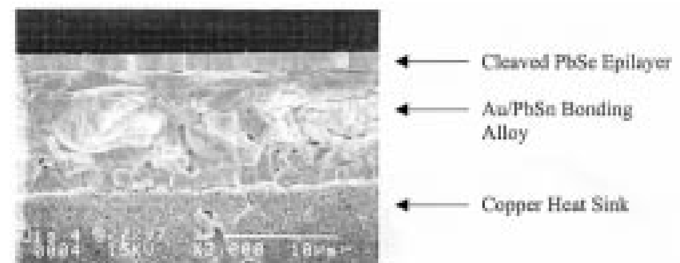

Fig. 3. A scanning electron micrograph showing the cross section of the copper plate-epilayer combination after bonding and cleaving using Au–PbSn bonding metallurgy.

been cleaved in the ⟨110⟩ directions, were aligned at a 45° angle to the copper plates to give the correct alignment. Copper plates taken from the center of the assembly thus contained (on their edges) layers of PbSe with {100} cleaved facets. The cleave quality and the morphology of the bonding medium were observed using scanning electron microscopy.

III. Results

Fig. 3 shows a cleaved facet of a PbSe epilayer bonded to a single copper plate using Au–PbSn bonding metallurgy. Although cleaves were formed from this procedure, the Au–PbSn alloy gave low reproducibility and typically resulted in less than 25% layer adhesion to the copper plates. The micrograph does not show a smooth cleaved facet. This is most likely attributed to poor alignment between the {100} cleavage planes of the epilayer and the copper plates. In contrast to the Au–PbSn alloy, the AuIn alloy gave high reproducibility with greater than 95%

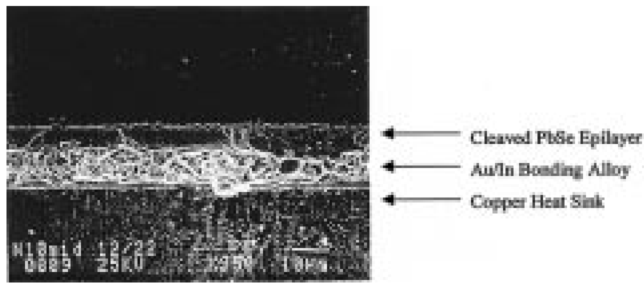

Fig. 4. A scanning electron micrograph showing the cross section of the copper plate-epilayer combination after bonding and cleaving using Au–In bonding metallurgy. The AuIn gives higher reproducibility with greater than 95% layer adhesion.

layer adhesion to the copper plate assembly. Fig. 4 is a scanning electron micrograph showing a smooth (100) cleaved facet of a PbSe layer bonded to copper with Au–In. The thickness of the bonding metal is about 10 μm, the same as for the Au–PbSn bonded sample shown in Fig. 3 and consistent with the total thickness of the deposited metals.

IV. Discussion

The success in using Au–In bonding metallurgy can be explained by considering the interaction of gold and indium during the bonding procedure. At 200 °C, the 0.0065 g of indium forms an approximately 9-μm-thick liquid layer over the 1×1 cm^2 sample area. This indium, sandwiched between two 0.2-μm-thick gold layers, will alloy with the gold through a solid-liquid diffusion process. A mixed phase of AuIn and $AuIn_2$ is created at the interface between the molten indium and the gold with the $AuIn_2$ layer bordered by the molten indium [9]. As indium continues to diffuse through the $AuIn_2$ layer the indium will react with the AuIn to create more $AuIn_2$. This process continues until the equilibrium condition between $AuIn_2$ and a 1.7 wt.% gold liquid phase is reached. It is believed that the formation of high melting temperature intermetallic compounds, AuIn (509.6 °C) and $AuIn_2$ (540.7 °C), as part of the bonding medium ensures sufficient adhesion and room temperature brittleness to facilitate cleaving the PbSe layer during copper plate separation.

The Au–PbSn–Au bonding medium should also produce intermetallics but the quality of the electroplated layers was not sufficient to ensure large area bonds. In this scheme, the scrubbing motion should not prove helpful since an Au layer covers the PbSn layer and thus oxidation is less of a concern.

V. Summary

Epitaxial layers of PbSe have been bonded to a copper plate assembly using both Au–PbSn and Au–In metallurgy. The layers were effectively transferred to the copper by dissolving a BaF_2 buffer layer that was grown between the PbSe layer and the silicon growth substrate. PbSe layers were then cleaved by releasing the copper plates. Samples bonded with Au–In metallurgy produced greater than 95% layer adhesion with high reproducibility and resulted in smooth facets when PbSe layer cleaving was performed. This new epitaxial layer manipulation method should enable the fabrication of cleaved-cavity IV–VI lasers that have excellent active region heat dissipation properties. Work is continuing on using this novel fabrication technique to obtain mid-IR lasers with CW operating temperatures in the TE cooler range.

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