Development of Large Diameter Semi-Insulating Gallium Oxide (Ga_2O_3) Substrates

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Abstract

Beta-phase gallium oxide (β-Ga_2O_3) is an emerging ultra-wide bandgap semiconductor targeting next generation high power switching and RF electronics. β-Ga_2O_3 possesses a bandgap (E_g) of approximately 4.9eV and an estimated electric field breakdown (E_{br}) of 8 MV/cm. This is 2 to 3 times larger than that of silicon carbide (SiC) or gallium nitride (GaN) with a Baliga Figure of Merit 4X that of GaN [1]. Early demonstrations of Schottky diodes have achieved 1.6 kV breakdown [2], while MOSFETs as high as 740 V [3] and lateral device electric fields of at least 3.8 MV/cm [4]. β-Ga_2O_3 single crystals can be produced by crystallization from a melt utilizing high growth rate processes such as Czochralski (CZ) or Edge-defined Film-fed Growth (EFG). This offers a pathway for low cost manufacturing of large diameter native substrates. SYNOPTICS has successfully scaled CZ growth of β-Ga_2O_3 single crystals from self-nucleated grains on iridium wire to (010) oriented 60-mm diameter boules. Currently, the Air Force Research Laboratory is supporting the development of bulk growth, fabrication and polishing processes. In this paper, we will review SYNOPTICS development of manufacturing processes for large diameter, semi-insulating β-Ga_2O_3 substrates.

INTRODUCTION

Wide bandgap semiconductors, namely SiC and GaN, have enabled revolutionary advances for both RF power amplification and power switching electronics. This despite no viable native substrate for GaN. Both semiconductor technologies are undergoing unprecedented growth in both military and commercial markets. In recent years, β-Ga_2O_3 has emerged as an attractive candidate for next generation high power electronics and high frequency transistors. This is primarily due to its ultra wide bandgap, very high electric field breakdown strength and availability of low cost native substrates. β-Ga_2O_3 possesses a bandgap of approximately 4.9eV and a theoretical critical field strength (E_c) of 8 MV/cm [Fig. 1]. This translates into a Baliga Figure of Merit (BFOM) estimated to be several times higher than that of SiC or GaN. The critical field strength is also a key factor in Johnson's Figure of Merit (saturation velocity-critical electric field product, $v_{sat} \cdot E_c$) used to describe RF operation. The commercial availability of bulk β-Ga_2O_3 substrates has enabled rapid device progress since the first β-Ga_2O_3 MESFET demonstration in 2012 [1]. Breakdown voltages for β-Ga_2O_3 Schottky Diodes have reached 1.6 kV, while breakdown voltages for MOSFETs are as high as 740 V [3]. Lateral device electric fields of at least 3.8 MV/cm^3 and vertical device electric fields of 5.1 MV/cm^3 have been demonstrated, along with on-currents of 1.5 A/mm [5].

	Si	GaAs	4H-SiC	GaN	β-Ga_2O_3
μ (cm^2/V·s)	1400	8000	1000	1200	250
E_{CRIT} (MV/cm)	0.3	0.4	2.5	3.3	8
ε_{REL}	11.8	12.9	9.7	9	10
v_{SAT} (10^7 cm/s)	1.0	1.2	2.0	2.5	1.1
λ (W/m·K)	150	50	370	130	10, 30
BFOM ($\mu\varepsilon E_C^3$)	1	15	340	870	2870
JFOM ($v_{SAT}E_C$)	1	2	17	28	29
HCAFOM ($\varepsilon\mu^{0.5}E_C^2$)	1	5	48	85	255

Fig. 1 Comparison of common semiconductor bulk properties.

CRYSTAL GROWTH

Ga_2O_3 is known to form five crystalline polymorphs. The β form is the only stable polymorphic phase at melt-based growth temperatures of approximately 1800C at atmospheric pressure. Growth of β-Ga_2O_3 was first reported by Tomm et al. in 2000. Unlike vapor sublimation processes required for SiC and GaN, β-Ga_2O_3 single crystals are grown using conventional melt-based techniques such as CZ or EFG enabling the possibility of manufacturing large diameter, low cost substrates. Tamura Corporation offers EFG β-Ga_2O_3 substrates of differing orientations and doping in sizes ranging from 10x15-mm for (010) and 50-mm for ($\bar{2}01$). Controllable n-type conductivity is possible using silicon, germanium or tin [6-8]. Both Fe and Mg dopants act as deep acceptors to obtain high resistivity. Growth of β-Ga_2O_3 is highly unstable at required growth temperatures. The high melting temperature and oxidizing environment of β-Ga_2O_3 requires the use of iridium crucibles. This is particularly challenging for CZ growth as iridium crucibles will easily oxidize in an atmosphere of only a few percent of oxygen [9, 10]. Galaska et al. [11] has shown both theoretically and experimentally that scale-up of β-Ga_2O_3 crystal size is strongly affected by the formation of metallic gallium in the melt. Further scale up to 100-mm diameter crystals will

require correspondingly higher oxygen concentration in the growth atmosphere, up to 100%.

EXPERIMENTAL

SYNOPTICS conducted 39 growth attempts for this development activity. A water-cooled, "bell-jar" type of CZ puller capable of growth temperatures in excess of 1800°C was used. This type of puller is commonly used for manufacturing a variety of laser crystals such yttrium aluminum garnet (YAG) and gadolinium gallium garnet (GGG) crystals. These pullers were not designed to employ an overpressure and as such all growths were conducted at one atmosphere of pressure. Crucibles ranged in size from 3.25 to 3.75 inches in diameter. Crystals were grown with an inductively heated iridium crucible in 91% CO_2 and 9% O_2 atmosphere. Galaska et al. has shown that a slight overpressure or increased oxygen partial pressure can suppress volatilization of gallium oxide into sub-oxide species. However, growth in an oxygen rich environment does expose the iridium crucible to increased oxidation. This amount of oxygen was adequate to decrease the evaporation of the molten melt as well as minimize the oxidation rate of the iridium crucible. Crystals were approximately 60-mm in diameter and 40-mm long [Fig. 2]. The growth rate varied from 2 - 3 mm/h at a crystal rotation of 2.0 rpm. In each case crystals were grown along the <010> crystallographic direction, parallel to both (100) and (001) cleavage planes. Crystals with (010) orientation are presently preferred orientation for epitaxial growth [11].

Fig. 2 Typical as-grown Mg-doped β-Ga_2O_3 boules.

β-Ga_2O_3 possesses two strong cleavage planes parallel to the (100) and (001) planes which have strong electrical, optical and thermal anisotropy due to its monoclinic crystal structure. Growth along the <010> axis is also subject to cleavage plane inducement of grain boundaries, twins and cracks. Figure 3 highlights typical defects observed following crystal growth. Twinning and cleaving are by far the most recurring experienced, but bubbles and iridium inclusions are also prevalent. Twins can form with domain boundaries parallel to the (100) cleavage plane during the cone portion of growth, and will propagate throughout the run. The majority of the crystals grown experienced some degree of twining requiring the boules to be cored to smaller, single grain diameters. Decomposition of the melt species directly onto the seed crystal prior to the seed being dipped into the melt can act as a nucleation site for defect formation. Maintaining the seed pristine prior to dipping is critical for high quality crystalline growth. One approach under investigation is to introduce gas flow near the growth interface to minimize sub oxide volatilization on the seed. Approximately 15% of all growth starts were abandoned due to the formation of crucible leaks. These crucibles required welding repairs prior to subsequent use. Overall, 82% of all growth starts were successful in yielding material suitable for subsequent processing. [Fig. 4].

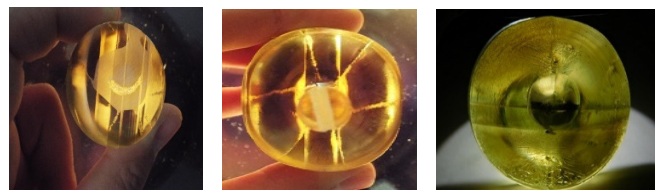
Fig. 3 Common defects in as-grown β-Ga_2O_3 crystals; a) twins, b) cleaves and c) bubbles/inclusions.

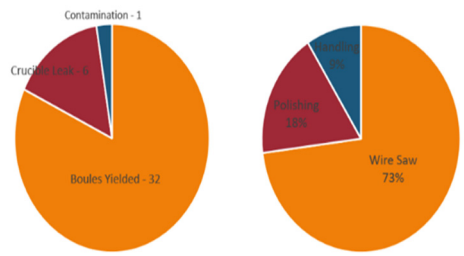

Fig. 4 Yield failures for a) crystal growth, b) fabrication and polishing.

The presence of two strong cleavage planes parallel to the (100) and (001) planes also complicate β-Ga_2O_3 fabrication and polishing. These crystal planes are highly susceptible to mechanical stress and can easily cleave during processing. Crystals up to 60-mm in diameter were cored, sliced and polished using a combination of mechanical and chemical mechanical polishing processes. Boule area free of twins and other macro defects were cored into smaller diameter boules or seed material to maximize useable yield [Fig. 5]. The coring process was shown to induce some cleaves to form along the active planes. A Takatori multi-wire saw with a 150µm diameter wire and boron carbide slurry was used to slice the boules. Wire saw rollers with slicing pitches ranged from 750µm to 900µm. Use of both roller pitches experienced excessive substrate breakage. Approximately 73% of all substrate yield losses were attributed to the wire sawing process [Fig. 4]. This was predominately due to uncontrollable wire jumping and cleave formation. As such, a variety of potting techniques and epoxies were explored to improve wire stability and reduce thermal stress [Fig. 5]. This approach greatly improved wire stability and improved substrate yield.

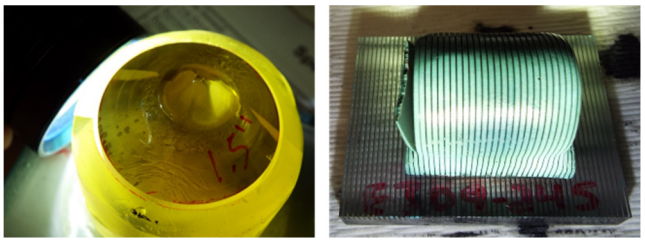

Fig. 5 a) Cored 50-mm boule, b) epoxy encapsulated sliced 25-mm boule.

As-cut substrates underwent a three step polishing process consisting of lapping, pre-polish and chemical mechanical polish (CMP). Due to the thinness of the substrates and the susceptibility to cleaving, special care was required at all times when handling substrates. Lapping required the development of a high rate removal process that doesn't induce additional subsurface damage. An Al_2O_3 based slurry with both soft and hard platens were used. The softer spindle facsimile pad with a 40µm Al_2O_3 slurry resulted in poor throughput and excessive breakage. A much harder 48" cast iron platen with a 20µm Al_2O_3 slurry greatly improved throughput but further development of a gentler, higher removal rate process is necessary. Development of a pre-polishing process investigated use of diamond and Al_2O_3 based slurries combined with a lead plate spindle and a hard pad polisher. A metallic spindle with a 1µm Al_2O_3 based slurry provided the better results. However, removal rate was slow and the pH of the slurry chemistry requires adjustment. The final CMP process only removed a few microns using a felt pad and colloidal silica slurry with a pH up to 12. AFM measurements [Fig. 6] confirmed sub-nm surface roughness is readily reproducible but a new cleaning protocol to remove residual slurry residue is required to provide an epi ready surface.

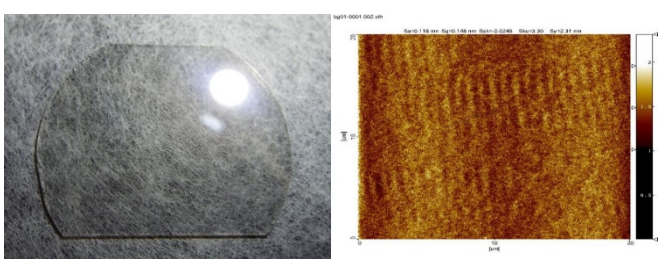

Fig. 6 a) 25-mm double sided polished substrate with unintentional flats, b) AFM image of 20X20µm scan.

A 3-step process for polishing as-cut β-Ga_2O_3 substrates up to 50-mm has been developed and demonstrated. The lapping, pre-polish and CMP processes scaled from a single substrate to 7x25-mm [Fig.7]. Both single side and double side polishing was demonstrated. Optimization of platens and slurry chemistry is necessary to further mitigate cleaving and substrate breakage.

Fig. 7 Scale up of β-Ga_2O_3 polishing process from single substrate to 7X25-mm substrates.

CONCLUSIONS

β-Ga_2O_3 is a novel ultra wide bandgap semiconductor with attractive intrinsic properties and ability to be grown with melt-based processes. Initial manufacturing processes have been established for the crystal growth, fabrication and polishing of up to 50-mm diameter Mg-doped and Fe-doped (010) β-Ga_2O_3 substrates.

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