# Thursday Morning, December 12, 2024

#### Thin Films and Surface Modification Room Naupaka Salon 4 - Session TF2-ThM

#### **Thin Films - Surface Modifications**

Moderator: Hyo-Chang Lee, Korea Aerospace University

# 10:20am TF2-ThM-8 Wafer-Level Glassblowing Process for Fabrication of 3d Micro-Resonators and Associated Imperfections Due to Surface Modifications and Change in Material Composition, Andrei Shkel, University of California Irvine

Glassblowing is an art that dates back over 2000 years. Today, glass blowing is used in a wide array of applications, including scientific glassware, optical components, consumer glass containers, and visual arts.

We introduced a fabrication process where glass micro-structures are blown on a wafer level, allowing thousands of 3D glass parts to be bult simultaneously. We reported, for the first time, a fabrication process for building atomically smooth, symmetric 3D wineglass and spherical shell structures, using low internal loss materials, fused quartz (FQ) and ultra-low expansion titania silicate glass (ULE TSG).

In order to minimize surface losses in resonant and optical applications highly smooth surfaces are required. Characterization of the glassblown samples before glassblowing and after revealed a two-fold improvement in surface roughness, from 0.4 nm Sa down to 0.23 nm Sa. Such improvements in surface roughness are attributed to viscous flow of the glass layer and the associated surface tension forces. The glassblowing creates an effect analogous to "stretching out" the angstrom-level wrinkles on the surface, lowering the surface roughness.

We found that glassblowing temperature and the rate of cooling are the most important parameters that affect the quality of the FQ and TSG layer after glassblowing. With slow cooling (~8 hours from 1700 C to room temperature), recrystallization and micro-cracks were observed on the surface. In contrast, when glassblowing followed rapid cooling by bringing temperature from 1700 C to 200 C within a minute, no recrystallization and micro-cracks were observed. The electron dispersive spectroscopy (EDS) was used to confirm that in the later case a homogeneous SiO2 and TiO2 distribution was the underlying reason. We concluded, the absence of recrystallization makes rapid cooling an essential step in glassblowing of micro-structures.

The technology for fabricating high-Q resonators includes mechanical grinding (utilized for removing the flaw-rich surface layer) and polishing, followed by (or alternated with) chemical cleaning and etching. Such treatment activates numerous physical-chemical processes on the glass surface. Our study included a comprehensive look into the surface effects and derived the technological approaches for chemical and thermal surface treatments.

A strong correlation between surface and thermal treatments and increase in the quality factor of 3D shell resonators have been established. We experimentally demonstrated that the method of fabrication leads to an unprecedented mechanical quality factor of such resonators, with the quality factor of over 3,000,000.

#### 10:40am TF2-ThM-9 Relationship between the Uniformity of the r- and Splane nanofaceted Substrate and the Nuclei Formation for Molecular Beam Epitaxial Layer of ZnTe on Sapphire, *Shumpei Tanaka*, *Masakazu Kobayashi*, Waseda university, Japan

Annealing of a sapphire m-plane (1-100) substrate produces a nano-meterscale faceted structure with alternating r- and S- planes [1]. It has been confirmed that (111)- oriented ZnTe layers with a single domain were grown by molecular beam epitaxy on sapphire S-plane (1-10-1) substrate, which was confirmed by the X-ray diffraction pole figure measurement. One domain ZnTe(111)/S-plane sapphire aligned layer structure was also confirmed from the layer grown on the r- and S- nanofaceted sapphire substrate (Fig.1(a)). With a 17.66° off of the r- and S-nanofacet substrates, (110)-oriented ZnTe thin films were grown on the substrate [2]. (111)oriented ZnTe layers were preferentially grown on the S-plane of the nanofacet structure, but the diffraction peaks from twinned (-1-1-1) ZnTe were also confirmed (Fig.1(b)). The off-angle of the substrate caused the reduction of the surface area ratio of the S-plane surface and increased the r-plane, resulting in an increase in the number of growth nuclei on the rplane.

The annealing duration of the substrate affects the height and size uniformity of the nanofaceted structure. The existence of large height

nanofacet structures would result in the preferential formation of nuclei on the large area r-plane and result in the twin formation. Extending the annealing time has shown to improve the uniformity of the nanofacet heights. Therefore, the uniform structure of the nanofacets could lead to reduce the number of nuclei on the r-nanofacet surface. The nuclei and rand S- plane nanofaceted structures on the substrate surface were analyzed by atomic force microscope (AFM). Figure 2 shows the AFM observation result of the surface right after the nuclei formation. The substrate was annealed at 1400°C for 20h and 1400°C for 40h, respectively. The nonuniform nano facet structure was confirmed only from the sample annealed for 20h. The sample annealed at 1400°C for 40h has nuclei formed on the Splane rather than r-plane, while the sample annealed at 1400°C for 20h has many nuclei formed on the r-plane with few formations on the S-plane. The sample with the high uniformity of nanofacet structure was considered to have preferential formation of growth nuclei on the S-plane.

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[1] S. Curiotto, and D. Chatain, Surf. Sci. 603, 2688 (2009).

[2] T. Nakasu, T. Aiba, S. Yamashita, S. Hattori, T. Kizu, W. Sun, K. Taguri, F. Kazami, Y. Hashimoto, S. Ozaki, M. Kobayashi, and T. Asahi, *J. Electron. Mater.* 45, 4742 (2016).

#### 11:00am TF2-ThM-10 Atomic Force Microscope-Based Surface Investigation of Low-Dimensional Materials and Fabrication of the Microscale Probes, Sangmin An, Jeonbuk National University, Republic of Korea INVITED

The atomic force microscope (AFM) is a crucial tool for investigating the surface properties of low-dimensional materials, such as zero-dimensional (0D) [1] and two-dimensional (2D) [2] nanomaterials. It offers exceptional high-resolution topographical imaging capabilities. This research aims to provide an overview of recent advancements in AFM techniques, focusing on the exploration of both 0D and 2D materials and their applications in 3D printing, guided by AFM. A novel approach is presented by integrating a nanopipette with a quartz tuning fork-based AFM [3], enhancing the utility and precision of AFM in these fields. Additionally, the research examines the current progress in microscale cantilever fabrication, highlighting significant developments in this area. This comprehensive review of advanced AFM techniques and their applications sheds light on the latest innovations in the field, setting the stage for future discoveries and technological advancements.

[1] S. Kim et al., Phys. Rev. X 8 (2018) 041046

[2] H. Kim et al., Nanoscale Adv. 5 (2023) 2271-2279

[3] C. Kim et al., Nano-Micro Lett. 14 (2022) 13

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