

# X-ray Photoemission Spectroscopy for Non-Destructive Analysis of Si Trench Bottoms

**S. Murase, T. Higashi, T. Kawashima, K. Inagaki, K. Arima**

*Department of Precision Engineering, Graduate School of Engineering,  
The University of Osaka, Japan*

Three-dimensional (3D) structures with high aspect ratios (ARs) have become standard in highly integrated semiconductor devices [1,2]. Ensuring high yield requires stringent cleanliness; however, non-destructive evaluation of cleaning in 3D structures remains limited, despite extensive studies on flat surfaces. To address this issue, we aim to develop a novel non-destructive method for evaluating cleaning performance at the bottoms of 3D nanostructures. Specifically, we apply angle-resolved X-ray photoemission spectroscopy (AR-XPS) to 3D structures such as deep trenches, embedding heterogeneous “landmark” elements selectively embedded at the bottoms as vertical markers.

In this talk, we examine the feasibility of the proposed method by obtaining XPS spectra of Si trenches with different ARs (1–7). To this end, Si trench structures were fabricated with gold (Au) selectively embedded at the bottoms using a wet etching process known as metal-assisted chemical etching (MACE) [3, 4]. AR-XPS measurements of these structures revealed a strong take-off angle (TOA) dependence of the Au 4f signal, particularly at higher ARs. This indicates that the embedded Au serves as an effective marker for aligning the sample and detector axes. AR-XPS was also conducted after removing Au from the trench bottoms. The resulting Si 2p spectrum exhibits a clear component corresponding to bulk Si (Fig. 1b), clearly distinguishable from that of Au-embedded samples (Fig. 1a), indicating that the signal originates from the trench bottoms. In other words, Fig. 1 demonstrates that MACE-fabricated Si trenches possess chemically distinct surface conditions at the top and bottom, enabling separation in Si 2p XPS spectra without additional surface treatments [5]. The proposed method is expected to be used in evaluating wet and dry cleaning processes at the bottoms of high-aspect-ratio structures.

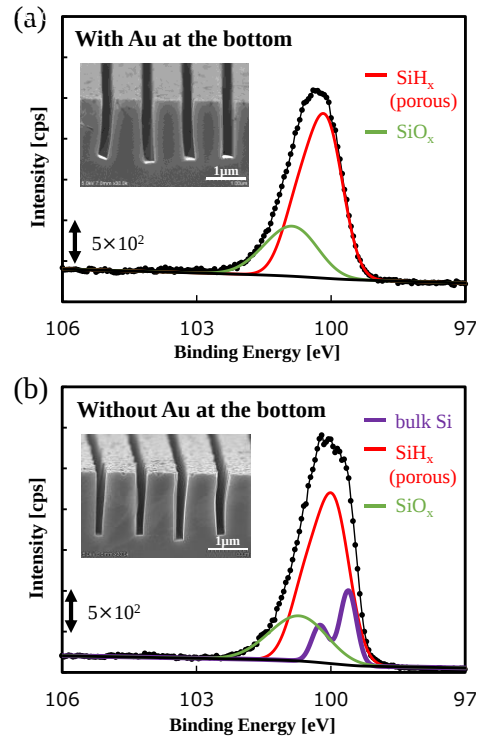


Fig.1. Si 2p spectra taken at the take-off angle of 90°. (a) With Au embedded at the bottoms of Si trenches. (b) After Au removal.

- [1] Y.-G. Liaw et al., *Solid-State Electron.* **126**, 46–50 (2016).
- [2] C.-J. Sun et al., *IEEE J. Electron Devices Soc.* **8**, 1016-1020 (2020).
- [3] Z. Huang et al., *Adv. Mater.* **23**, 285–308 (2011).
- [4] K. Arima, J. Li, S. Murase, K. Inagaki, *ECS Trans.* **114**, 17–26 (2024).
- [5] S. Murase et al. (in preparation).

## Supplementary information:

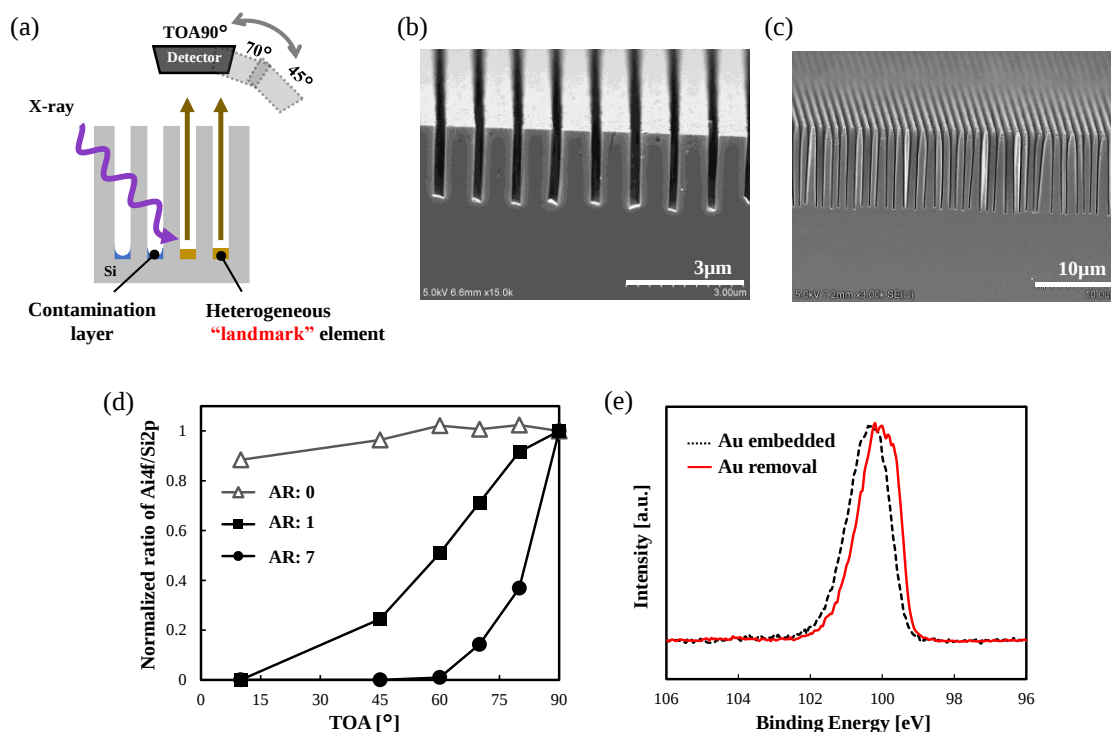


Fig. S1. (a) Proposed method for evaluating cleaning properties at the bottoms, (b, c) Cross-sectional scanning electron microscopy images of Si trenches after MACE, with aspect ratios (ARs) of (b) 8, (c) 37. (d) Peak-area ratios of Au 4f to Si 2p as a function of take-off angles (TOAs) for a Si trench structure containing embedded Au at the bottom. The vertical axis is normalized to the value at a TOA of 90°. (e) Overlaid Si 2p XPS spectra at a TOA of 90° from samples with and without embedded Au.