

Electronic Supplementary Information

Formation of monolayer and few-layer hexagonal boron nitride nanosheets *via* surface
segregation

Mingsheng Xu ^{1,2,*}, Daisuke Fujita ^{3,4}, Hongzheng Chen ¹, Nobutaka Hanagata ^{5,6}

¹ State Key Laboratory of Silicon Materials, MOE Key Laboratory of Macromolecule Synthesis and Functionalization, and Department of Polymer Science and Engineering, Zhejiang University, Hangzhou 310027, P. R. China

² International Center for Young Scientists, National Institute for Materials Science, 1-2-1 Sengen, Tsukuba, Ibaraki 305-0047, Japan

³ Advanced Nano Characterization Center, National Institute for Materials Science, 1-2-1 Sengen, Tsukuba, Ibaraki 305-0047, Japan

⁴ International Center for Materials Nanoarchitectonics, National Institute for Materials Science, 1-2-1 Sengen, Tsukuba, Ibaraki 305-0047, Japan

⁵ Interdisciplinary Laboratory for Nanoscale Science and Technology, National Institute for Materials Science, 1-2-1 Sengen, Tsukuba, Ibaraki 305-0047, Japan

⁶ Graduate School of Life Science, Hokkaido University, Sapporo, Japan

*Corresponding author: msxu@zju.edu.cn (Mingsheng Xu)

Thickness estimation

Fe (LMM) transition	Substrate (sputtered for 0.4 min)	Buffer layer	Monolayer stack	Bilayer stack
Peak-to-peak magnitude	10695	7237	4871	3441
Thickness		0.31 nm	0.65 nm	0.91 nm

To estimate the thickness of h-BN films, we used Fe (LMM) Auger transition with the electron inelastic mean free path of ~ 1.2 nm for iron at ~ 703 eV based on inelastic attenuation model of the following expression. By considering the carbon contamination, we used the peak-to-peak magnitude of Fe (LMM) transition from the AES spectra after sputtered for 0.4 min as the $I_{\text{sub, pure}}$. However, we used the peak-to-peak magnitude of Fe (LMM) transition from the AES spectra without sputtering as I_{sub} because argon ion sputtering can damage the structure of h-BN and the buffer layer, and the carbon contamination at the buffer layer and h-BN regions is much less than at the bare substrate surface.

$$I_{\text{sub}} = I_{\text{sub, pure}} \exp[-(nd_0)/\lambda \sin(\theta)]$$

where I_{sub} is the peak-to-peak magnitude of the Fe (LMM) transition prior to sputtering by ion-gun sputtering, $I_{\text{sub, pure}}$ is the peak-to-peak magnitude of the Fe (LMM) peak after removing most of the carbon contamination (*i.e.*, 0.4 min sputtering time), n is the number of h-BN, d_0 is the theoretical thickness of single h-BN sheet (~ 3.4 Å), θ is the electron take-off angle (42° for the present Auger instrument), and λ is the inelastic mean free path (IMFP) of the Auger electrons for iron.

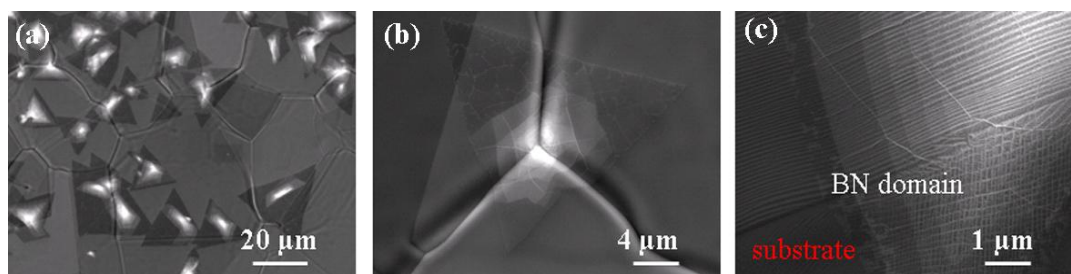


Figure S1. Typical SEM images of h-BN nanosheets synthesized by surface segregation from Fe-Cr-Ni alloy doped with boron and nitrogen.

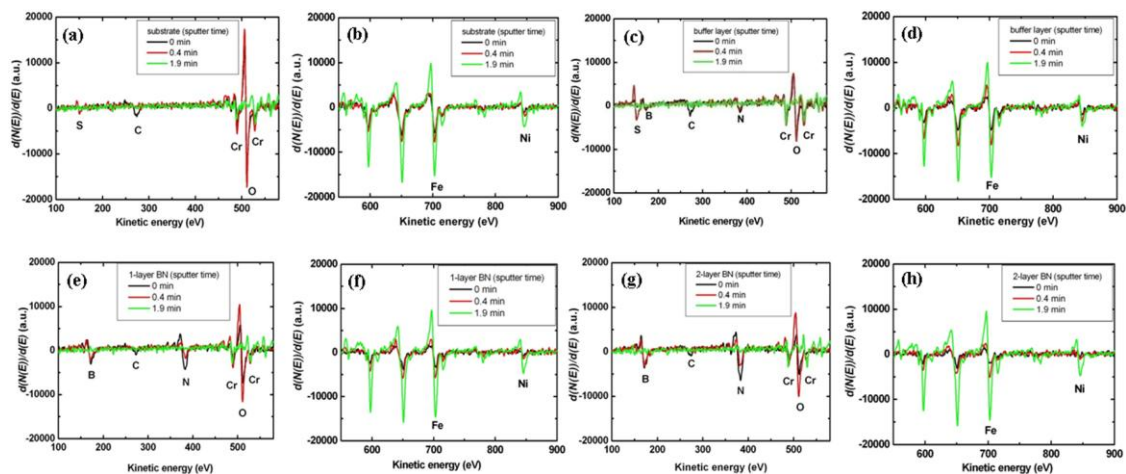


Figure S2. AES spectra show elemental evolution as a function of sputtering time, acquired at (a)-(b) substrate region, (c)-(d) B-N buffer layer region, (e)-(f) monolayer h-BN region, and (g)-(h) bilayer h-BN region of the sample in Figure S3.

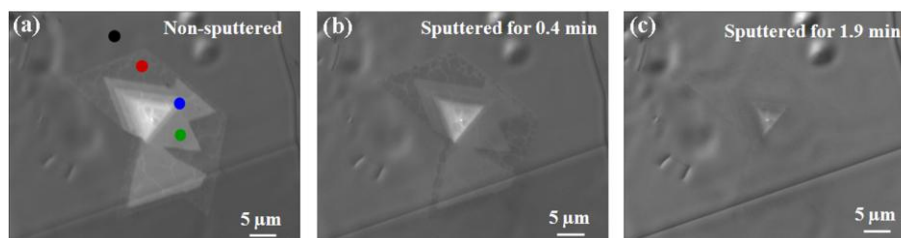


Figure S3. SEM images of h-BN flakes acquired before and after sputtered for different times. The black, red, blue, and green spots represent the substrate, B-N buffer layer, monolayer h-BN, and bilayer h-BN, respectively.

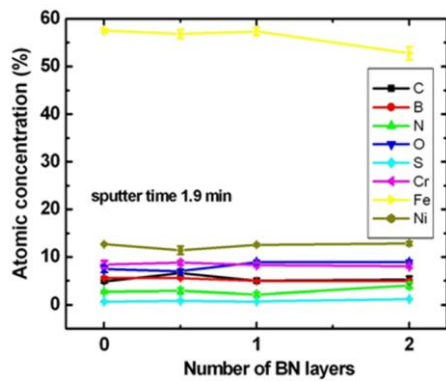


Figure S4. Atomic concentration of h-BN sample after sputtered for 1.9 min.