



Supplementary Materials

Formation of Nanospikes on AISI 420 Martensitic Stainless Steel under Gallium Ion Bombardment

Zoran Cenev ¹, Malte Bartenwerfer ^{2,*}, Waldemar Klauser ², Ville Jokinen ³, Sergej Fatikow ² and Quan Zhou ^{1,*}

¹ Department of Electrical Engineering and Automation, School of Electrical Engineering, Aalto University, Maarantie 8, 02150 Espoo, Finland; zoran.cenev@aalto.fi

² Department of Computing Science, University of Oldenburg, Ammerländer Heerstraße 114-118, 26129 Oldenburg, Germany; waldemar.klauser@uni-oldenburg.de (W.K.); sergej.fatikow@uni-oldenburg.de (S.F.)

³ Department of Chemistry and Materials Science, Aalto University, School of Chemical Technology, Tietotie 3, 02150 Espoo, Finland; ville.jokinen@aalto.fi

* Correspondence: m.bartenwerfer@uni-oldenburg.de (M.B.); quan.zhou@aalto.fi (Q.Z.); Tel.: +49-179-682-1971 (M.B.); +358-40-855-0311 (Q.Z.)

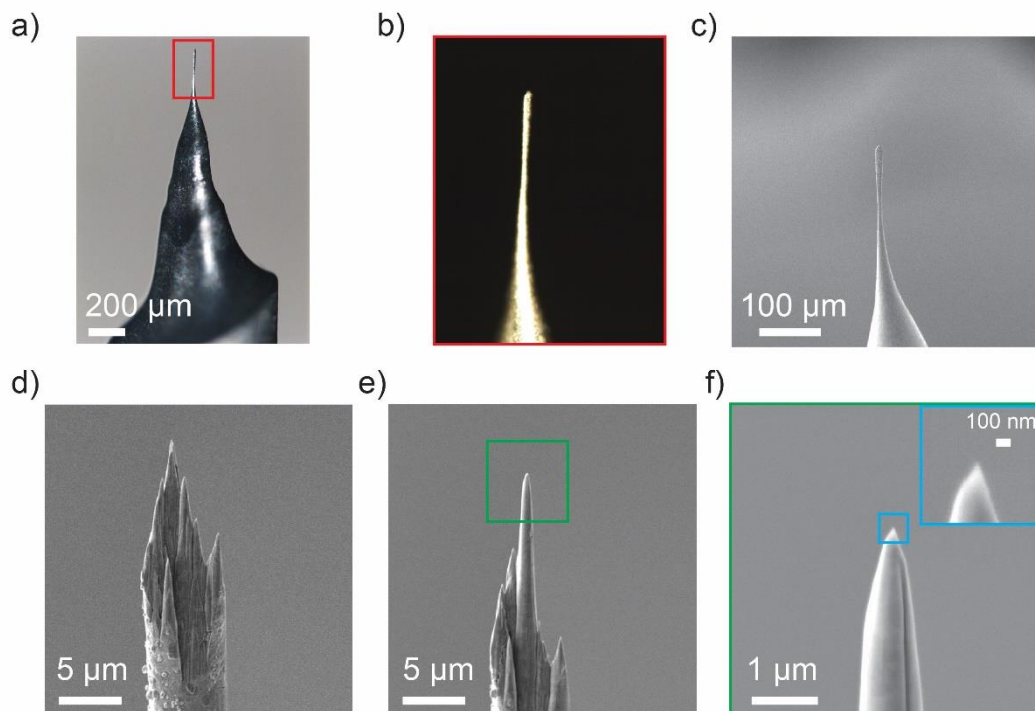


Figure S1. Intermediate steps of the fabrication process of martensitic stainless steel AISI420 needle with nanometer sharpness. a) Electrochemically thinned AISI 420 wire; b) close-up; c) SEM micrograph of the micrometer sharp tip before gallium irradiation. d) Nano-spikes formed after gallium irradiation in FIB/SEM dual system; e) FIB (with gallium ions) machining of surrounding spikes while isolating the middle one; f) The final nanospike with measured 15.15 nm diameter. Fitted circle indicating the measurement is given in Figure 1b, the inset is showing a close-in.

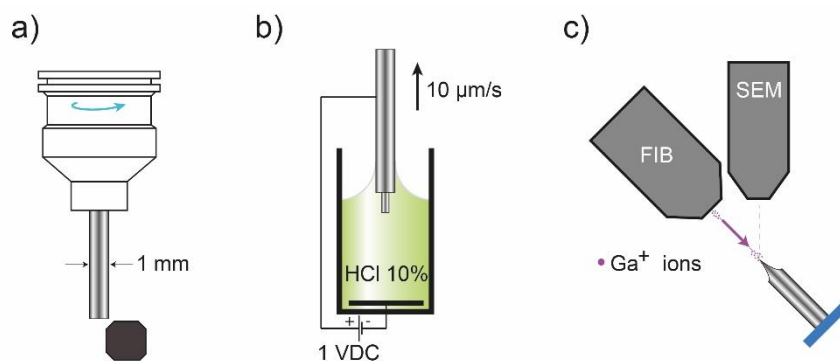


Figure S2. Illustration of the fabrication procedure of microneedle with nanopikes. a) Thinning by machining; b) Electrochemical etching in HCl bath with a constant speed of $10 \mu\text{m/s}$; c) Gallium irradiation within FIB/SEM dual system.

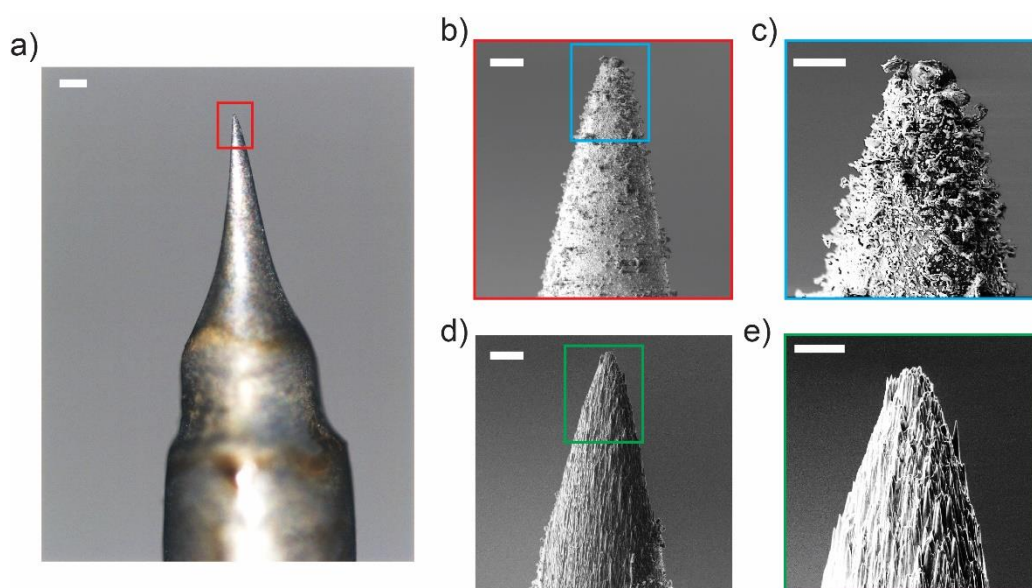


Figure S3. Intermediate steps of the fabrication process of martensitic stainless steel AISI420 microneedle with nanopikes. a) Thinned AISI 420 wire by machining and electrochemical etching; b) SEM micrograph of the micrometer sharp tip; c) close-up of b). d) SEM micrograph of the gallium irradiated tip. e) close-up of d). Scale bar is $100 \mu\text{m}$ in a), $20 \mu\text{m}$ in b) and d) and $10 \mu\text{m}$ in c) and e).

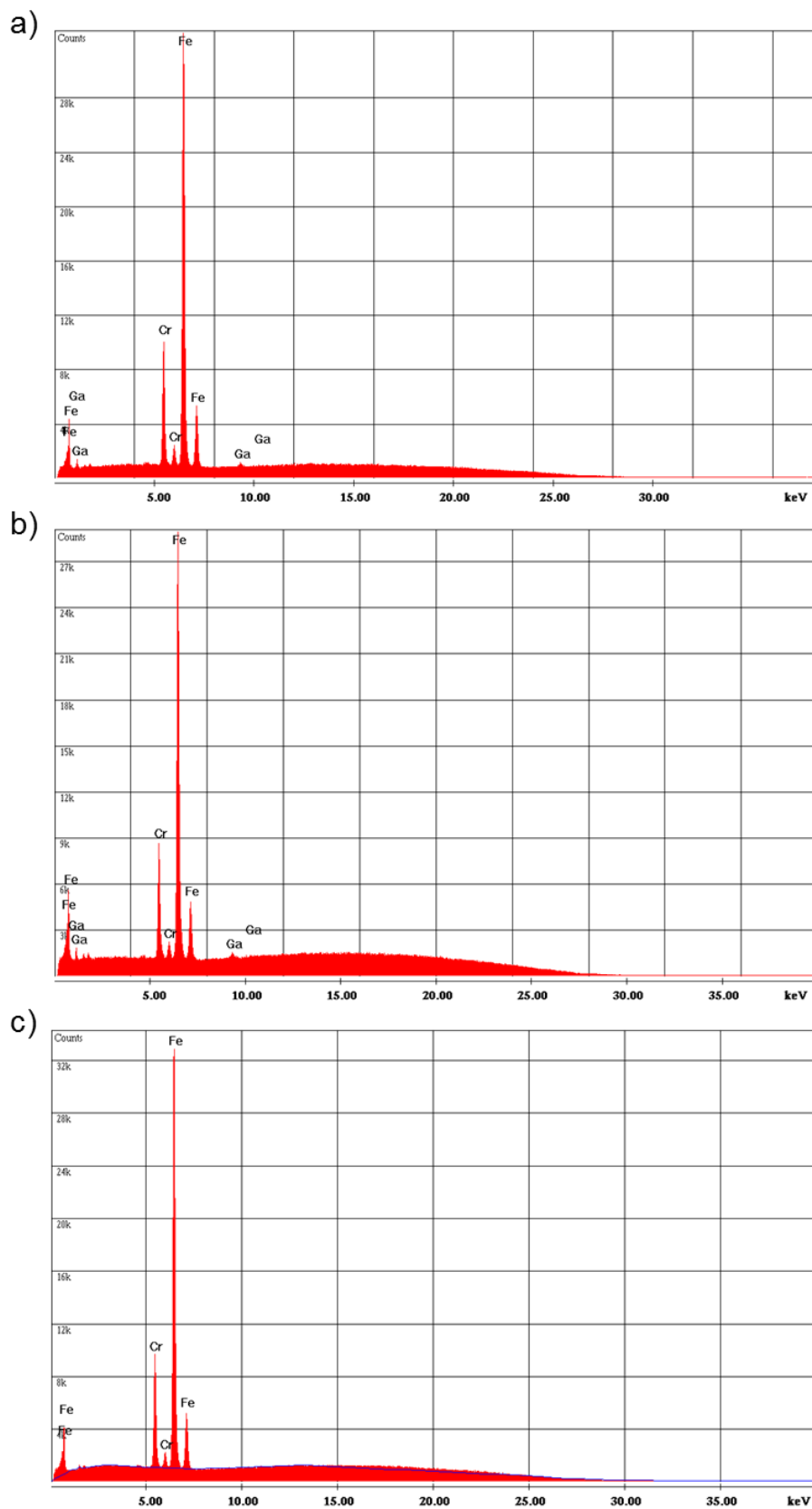


Figure S4. Energy-dispersive X-ray spectroscopy (EDX) results of a) the completely irradiated trench; b) a spot on a single nanopike; c) non-irradiated area.

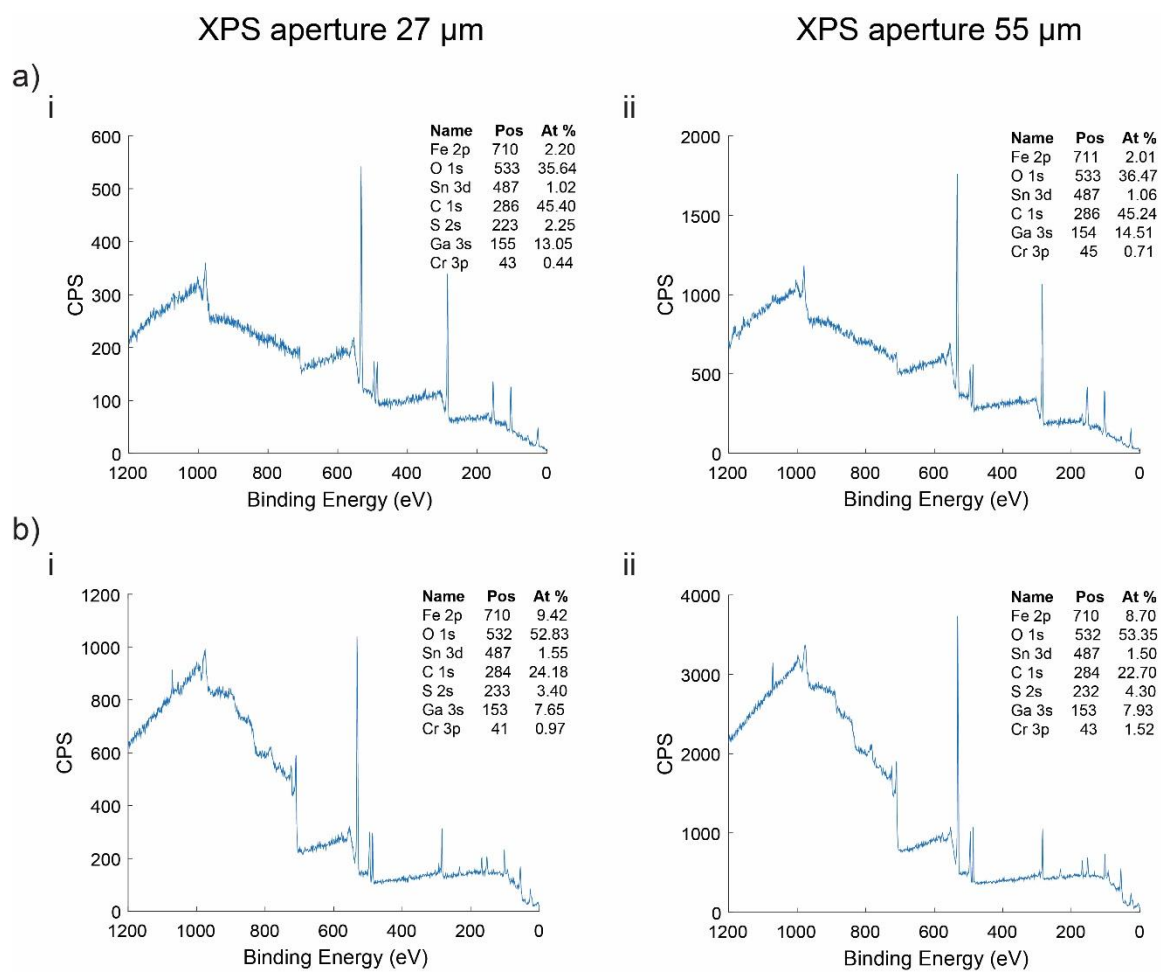


Figure S5. X-ray Photoelectron Spectroscopy (XPS) results of a) a ~35 μm in diameter gallium irradiated trench; b) non-irradiated area. i and ii denote measurement with XPS aperture of 27 and 55 μm , respectively.

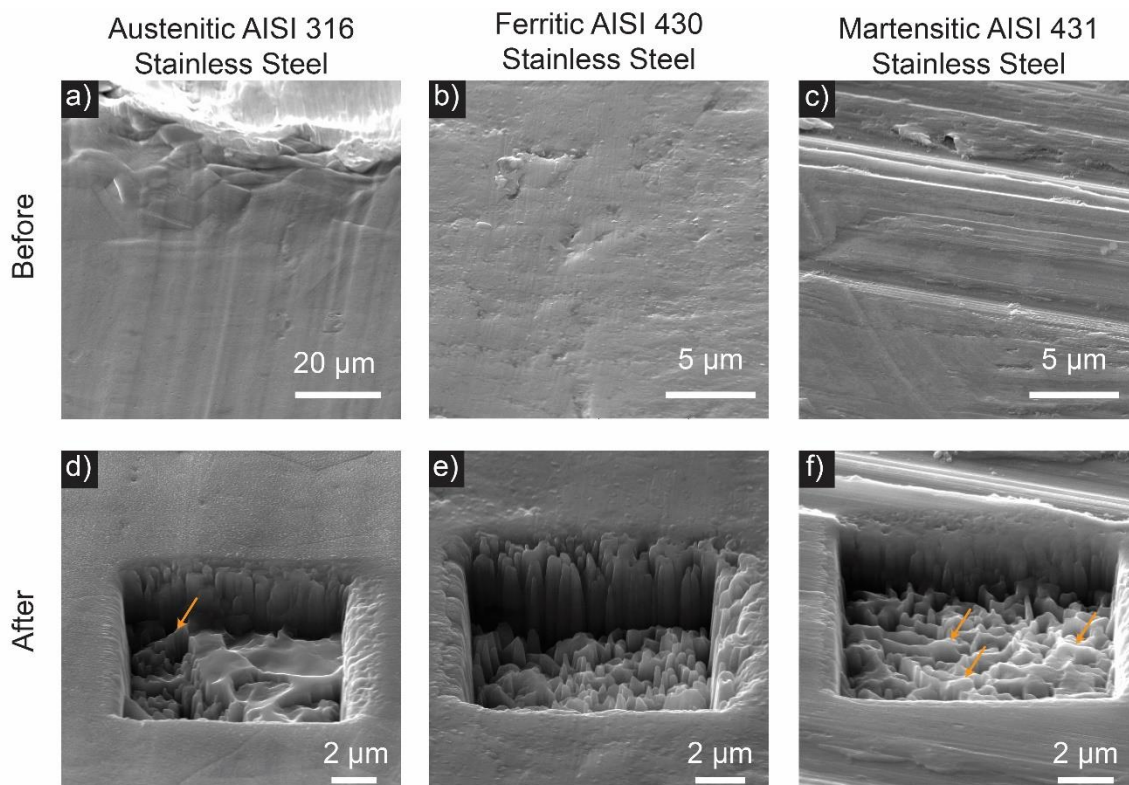


Figure S6. Gallium irradiation of austenitic AISI 316 (a, d), ferritic AISI 430 (b, e), and martensitic AISI 431 (c, f) stainless steel plates with a dose of $19.4 \text{ C}/\mu\text{m}^2$. a-c) Before and d-f) after gallium irradiation. Orange arrows denote the so-called nano-cliffs.