metals

# Supplementary Materials: Synthesis of Metastable Au-Fe Alloy Using Ordered Nanoporous Silica as a Hard Template

**Paskalis Sahaya Murphin Kumar, Thiripuranthagan Sivakumar, Takeshi Fujita, Ramasamy Jayavel, and Hideki Abe**

Content



## Synthetic procedures

Reagents used for the synthesis were used as purchased without further purification: anhydrous Au(III) chloride ($AuCl_3$, Aldrich), iron acetylacetonate (Fe(II)acac, Aldrich), diethylene glycol dimethyl ether (anhydrous, 99.5%, Aldrich), $LiBH(C_2H_5)_3$ (super-hydride, 1M in THF, Aldrich), hexane (anhydrous, 95%, Aldrich), poly (ethylene glycol)-block-poly(propylene glycol)-blockpoly(ethylene glycol) ($EO_{20}PO_{70}EO_{20}$, average molecular weight 5800, Aldrich).

In a typical synthesis of SBA-15 (reported by Vinu et al [17]) as illustrated below: 4 g of the amphiphilic triblock copolymer was dispersed in water (30 g) and HCl solution (120 mL, 2 M) and stirred for 5 h. Thereafter, tetraethylorthosilicate (TEOS, 9 g) was added to the homogeneous solution under stirring. The collected gel was aged at 40 °C for 24 h and finally heated to 100 °C for 24 h. After that, the synthesized solid was calcined in flowing air at 540 °C to decompose the triblock copolymer.

## Characterization

**Powder X-ray diffractometry (*p*XRD):** The pXRD measurements were performed using Cu K$\alpha$ radiation ($\lambda$ = 0.15418 nm; X'Pert Powder Diffractometer, Panalytical) with an increment of 0.02 degrees in a range of diffraction angles from 10 to 90 degrees. An obliquely finished Si crystal (non-reflection Si plate) was used as a sample holder to minimize the background.

**Hard X-ray photoemission spectroscopy (HAXPES):** HAXPES measurements were performed using X-rays with a photon energy of 5.95 keV, at the undulator beamline BL15XU of SPring-8, Japan. The core-level states of the samples were examined at room temperature in UHV using a hemispherical electron energy analyzer (VG SCIENTA R4000). The total energy resolution was set to 220 meV. The binding energy was referenced to the Fermi edge of an Au thin film.

**Transmission electron Microscopy:** We used a 200kV transmission electron microscope (TEM and/or STEM, JEM-2100F, JEOL) equipped with two aberration correctors (CEOS GmbH) for the image- and probe-forming lens systems and an X-ray energy-dispersive spectrometer (JED-2300T, JEOL) for elemental mapping. The samples for TEM were prepared by dropping an ethanol suspension of the sample powder onto a commercial copper TEM grid coated with a collodion film. The sample was thoroughly dried in vacuum prior to observation.

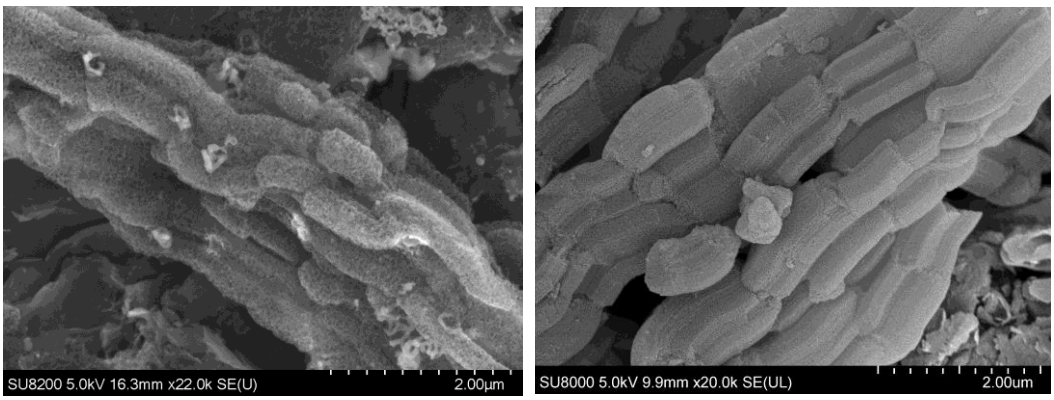


**Figure S1.** SEM images of the prepared nanoporous AuFe (left) and silica template (right).

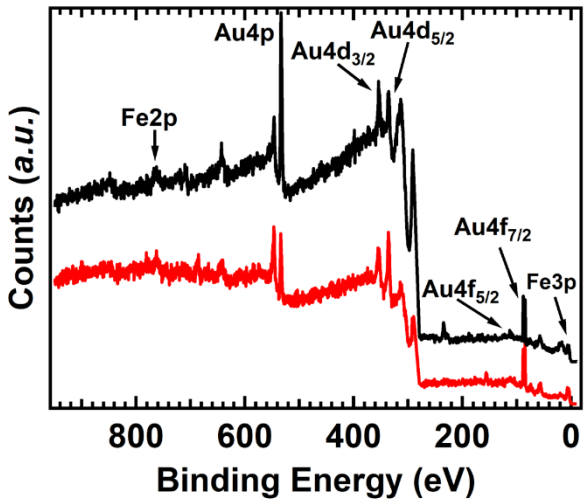


**Figure S2.** Hard X-ray photoemission spectroscopy (HAPES) profiles of the nanoporous Au-Fe (red) and AuFe nanoparticles (black) in a wide range of binding energy.

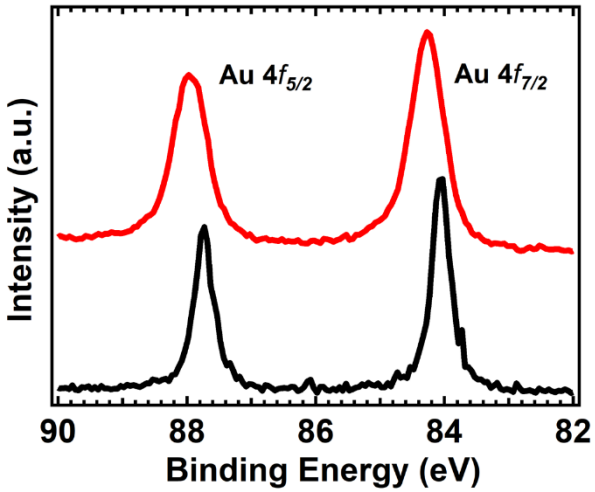


**Figure S3.** Hard X-ray photoemission spectroscopy (HAPES) profiles of the nanoporous Au-Fe (red) and Au nanoparticles (black) in the Au 4*f* region.

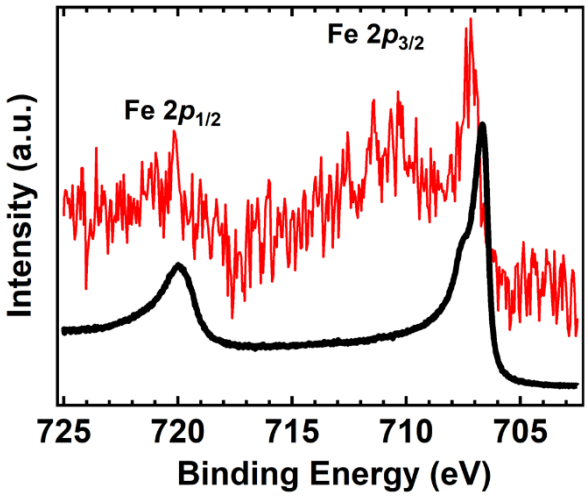


**Figure S4.** Hard X-ray photoemission spectroscopy (HAPES) profiles of the nanoporous Au-Fe (red) and Fe powder (black) in the Fe 2*p* region.

The Au to metallic Fe (Fe0) ratio was determined from Figures 2S-24:
The XPS peak area for the Au $4f_{7/2}$ emission = 11090 ± 1100.
The cross section for the Au $4f_{7/2}$ emission = 1.614.
The Au $4f_{7/2}$ peak area/cross section = 11090/1.614 = 6870 ± 690.
The XPS peak area for the $Fe^0$ $2p_{1/2}$ emission = 11435 ± 1100.
The cross section for the $Fe^0$ $2p_{1/2}$ emission = 4.026 [1].
The $Fe^0$ $2p_{1/2}$ peak area/cross section = 11435/4.026 = 2840±280.
The Au/$Fe^0$ ratio = 6870/2840 = 2.42±0.24.

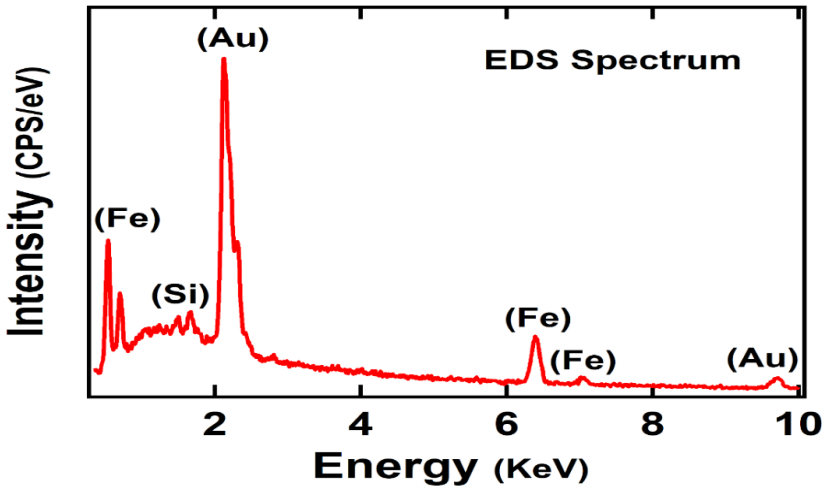


**Figure S5.** Energy dispersve spectroscopy (EDS) profile for the nanoporous Au-Fe.

**Table S1.** Chemical composition of the nanoporous Au-Fe determined by EDS.

| Element | Line type | Wt% | Wt% Sigma | Atomic % |
|---|---|---|---|---|
| Si | K Series | 2.12 | 0.23 | 0.49 |
| Fe | K Series | 27.13 | 0.81 | 57.75 |
| Au | M Series | 70.75 | 0.82 | 41.76 |
| Total | - | 100.00 | - | - |