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Published in:
Microscopy (Oxford, England)

Link to article, DOI:
[10.1093/jmicro/dfu081](https://doi.org/10.1093/jmicro/dfu081)

Publication date:
2014

Document Version
Publisher's PDF, also known as Version of record

[Link back to DTU Orbit](#)

Citation (APA):
Gondo, T., Kasama, T., & Kaneko, K. (2014). 3D visualization of TiO₂ nanocrystals in mesoporous nanocomposite using energy filtered transmission electron microscopy tomography. *Microscopy (Oxford, England)*, 63(suppl 1), i27. <https://doi.org/10.1093/jmicro/dfu081>

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rod- and plate-like morphologies with homogeneous dispersion in GdBCO. In addition, growth directions of these precipitates were found with wide angular distributions from growth direction of GdBCO. Anisotropy of J_c in the magnetic fields was probably enhanced by various growth directions and homogeneous dispersion of nanosized BHO within GdBCO.

References

1. Takahashi K, Yamada Y, Konishi M, Watanabe T, Ibi A, Muroga T, Miyata S, Shiohara Y, Kato T, Hirayama T (2005) Magnetic field dependence of J_c for Gd-123 coated conductor on PLD-CeO₂ capped IBAD-GZO substrate tapes. *Supercond. Sci. Technol* 18: 1118–1122
2. Tobita H, Notoh K, Higashikawa K, Inoue M, Kiss T, Kato T, Hirayama T, Yoshizumi M, Izumi T, Shiohara Y (2012) Fabrication of BaHfO₃ doped Gd₁Ba₂Cu₃O_{7-y} coated conductors with the high I_c of 85 A=cm-w under 3 T at liquid nitrogen temperature (77 K). *Supercond. Sci. Technol.* 25: 062002

doi: 10.1093/jmicro/dfu080

3D visualization of TiO₂ nanocrystals in mesoporous nanocomposite using energy filtered transmission electron microscopy tomography

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Introduction

Mesoporous silica, SBA-15, is one of the best candidate for the supporting material of catalytic nanoparticles because of its relative large and controllable pore size and large specific surface area [1]. So far, various nanoparticles, such as Au, Pt and Pd, have been introduced into the pore for catalytic application [2]. The size of nanoparticles supported inside SBA-15 is restricted by that of the pore, and they are usually ranging from 2 nm and 50 nm in space.

It is necessary to anchor the nanoparticles within pores to avoid segregation / sintering of them. However, it is difficult to anchor them within pores in the case of use of deposition-precipitation method due to extreme low iso-electric point (IEP) of silica (~2). Therefore, TiO₂ nanocrystals (IEP 6-8) were then introduced to anchor AuNPs [3].

In this study, EFTEM tomography was applied to examine the effectiveness of TiO₂ for AuNPs.

Materials and method

Au/TiO₂-SBA-15 was embedded into epoxy resin for electron microscopy and micromotted to about 30 nm thickness. EFTEM-tomography was operated at 120 kV and using Ti-L ionization edge via three-window method. Prior to EFTEM, STEM-HAADF tomography was also carried out for visualizing AuNPs and for comparison.

Result and discussion

Figure 1 shows 3D-volume of AuNPs and TiO₂ nanocrystals from EFTEM-tomography. TiO₂ nanocrystals in the porous material were successfully visualized using EFTEM -tomography, and local relationship between AuNPs and TiO₂ nanocrystals were revealed. A large number of TiO₂ nanocrystals were randomly distributed in the SBA-15. It was found that most AuNPs were directly on the exposed TiO₂ nanocrystals. It implies that TiO₂ nanocrystals were exposed on the surface of the pore and anchored AuNPs inside the pores.

Key words: EFTEM, 3D-Electron tomography, Mesoporous silica, TiO₂, Au, nanoparticle

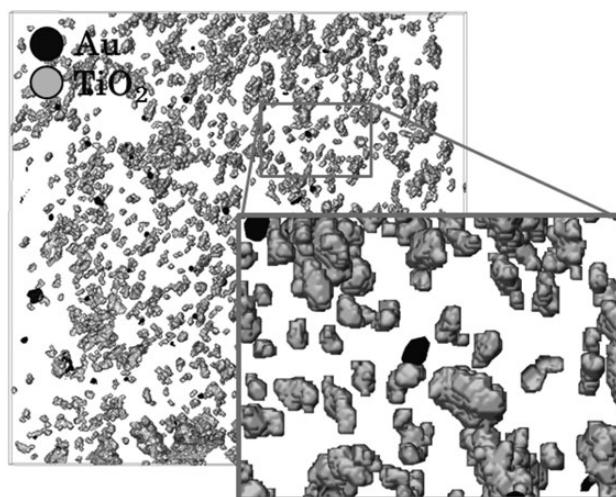


Fig. 1. 3D volume of AuNPs and TiO₂ nanocrystals

References

1. Zhao D., Feng J., Huo Q., Melosh N., Fredrickson G.H., Chmelka B.F., Stucky G.D. (1998). Triblock copolymer syntheses of mesoporous silica with periodic 50 to 300 Angstrom pores. *Science* 279, 548–552.
2. Zhu J., Konya Z., Puntes V.F., Kiricsi I., Miao C.X., Ager J.W., Alivisatos A.P., Somorjai G.A. (2003). Encapsulation of metal (Au, Ag, Pt) nanoparticles into the mesoporous SBA-15 structure. *Langmuir* 19, 4396–4401.
3. Takashi G., Kenji K., Takeshi N., Kazuhiro Y., Zineb S., Jonathan S.B., Paul A.M., (2014). Microstructural Analysis of Au/TiO₂-SBA-15 Nanocomposite. *Microscopy and Microanalysis* 20, 1001–1007.

doi: 10.1093/jmicro/dfu081

Microstructural characterization of GdBa₂Cu₃O_{7-δ} superconductor films with BaHfO₃ artificial pinning centers by scanning transmission electron microscopy

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Critical current (I_c) of superconductor films under magnetic field is strongly influenced by dispersions and morphologies of artificial pinning centers (APCs) in general [1]. BaHfO₃ (BHO) is acknowledged as the best candidates of APCs for REBCO films, which shows utmost thickness dependence and isotropic angular dependence of I_c values for REBCO films [2]. Moreover, several researchers have focused on the nanostrains caused by the lattice mismatch at the interface between APCs and REBCO matrix, which are also the source for enhanced vortex pinning of the REBCO films [3]. In this study, we investigated to examine the nanostrain at the interface using spherical aberration (C_s) corrected scanning transmission electron microscopy (STEM).