Technical University of Denmark



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

Gondo, Takashi; Kasama, Takeshi; Kaneko, Kenji

Published in: Microscopy

Link to article, DOI: 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, 63(suppl 1), i27. DOI: 10.1093/jmicro/dfu081

DTU Library Technical Information Center of Denmark

General rights

Copyright and moral rights for the publications made accessible in the public portal are retained by the authors and/or other copyright owners and it is a condition of accessing publications that users recognise and abide by the legal requirements associated with these rights.

- Users may download and print one copy of any publication from the public portal for the purpose of private study or research.
- You may not further distribute the material or use it for any profit-making activity or commercial gain
- You may freely distribute the URL identifying the publication in the public portal

If you believe that this document breaches copyright please contact us providing details, and we will remove access to the work immediately and investigate your claim.

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

- 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
- 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 $\rm TiO_2$ nanocrystals in mesoporous nanocomposite using energy filtered transmission electron microscopy tomography

Takashi Gondo^{1,*}, Takeshi Kasama², and Kenji Kaneko¹

¹Department of Materials Science and Engineering, Kyushu University, 744 Motooka Nishi-ku Fukuoka, 819-0395, Japan, and ²Center for Electron Nanoscopy, Technical University of Denmark, Building 307, DK-2800 Kongens Lyngby, Denmark

*To whom correspondence should be addressed. E-mail:

3te11019m@s.kyushu-u.ac.jp

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 (\sim 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 TiO_2 for AuNPs.

Materials and method

 Au/TiO_2 -SBA-15 was embedded into epoxy resin for electron microscopy and microtomed to about 30 nm thickness. EFTEM-tomography was operated at 120 kV and using Ti-L ionization edge via threewindow 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

Au TiO2

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

References

- 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.
- 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.
- 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 $GdBa_2Cu_3O_{7-\delta}$ superconductor films with $BaHfO_3$ artificial pinning centers by scanning transmission electron microscopy

Kazuhiro Yamada¹, Takeshi Nishiyama¹, Kenji Kaneko¹, Yukio Sato¹, Ryo Teranishi¹, Takeharu Kato², Akira Ibi³, Masateru Yoshizumi³, Teruo Izumi³, and Yuh Shiohara³

¹Department of Materials Science and Engineering, Kyushu University, 744 Motooka, Nishi-ku, Fukuoka 819-0395, Japan, ²Nanostructures Research Laboratory, Japan Fine Ceramics Center, 2-4-1 Mutsuno, Atsuta-ku, Nagoya 456-8587, Japan, and ³HTS Conductor Processing & Power Application Division, International Superconductivity Technology Center, 3-2-1 Sakado, Takatsu-ku, Kawasaki 213-0012, Japan

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).