doi:10.1017/S143192761005600X
Microsc. Microanal. 16 (Suppl 2), 2010

Structural Characterization of Gold Icosahedron Crystals

S.V. Prikhodko[1], M. Pozuelo[1], L. Chen[2], S.D. Sitzman[3], R. Richards[2], S.Kodambaka[1]

[1] Department of Materials Science and Engineering, UCLA, Los Angeles, CA 90095
[2] Department of Chemistry and Geochemistry, Colorado School of Mines, Golden, CO 80401
[3]Oxford Instruments Nanoanalysis, 300 Baker Ave., Concord, MA 01742

Nanoparticles of noble metals, such as gold, have attracted attention owing to their size- and shape-dependent catalytic particles [1]. To-date, crystalline gold nanoparticles of a variety of shapes, cubic, octahedral, decahedral, dodecahedral and icosahedral, have been synthesized using several wet chemical methods [2]. Here, we report the room-temperature synthesis and characterization of gold icosahedrons of desired sizes between 50 nm and 400 nm. Our method involves the use of water as a solvent, glucose as a reducing reagent, and sodium dodecyl sulphate as the directing agent [3]. We determined their crystallinity and morphology using transmission electron microscopy (TEM) and scanning electron microscopy (SEM) coupled with electron backscattered diffraction (EBSD).

A high-resolution TEM image of gold nanoparticles with the shape of icosahedron is shown in Fig. 1A. The crystal structure was identified as face centered cubic (fcc) gold S.G.:Fm3m (225). Fig. 1B shows SAED from the particle and 1C a schematic of the diffraction spots. From the analyses of the diffraction pattern, we suggest the particle is composed of rotational fcc twins, which can be described as a result of rotation of the parent orientation (origin) of fcc crystal on 60° around {111}. In terms of their orientation relationship (OR) such twinning can be described as {111}o ǁ {111}t; <011>o ǁ <011>t (OR1), where the subscripts o and t represent orientations of the original and twin crystals. On top of it there is yet another type of OR, which can be described as: {111}o ǁ {112}t; <011>o ǁ <111>t, (OR2). Dark-field images (lower panel in Fig.1) reveal that the icosahedron is represented as an arrangement of smaller tetrahedral units. The possibility for the formation of a twinned icosahedron via the arrangement of tetrahedral units with {111} facets were reported earlier [4,5]. Results of EBSD study presented here is consistent with our TEM findings. EBSD patterns were taken on different faces of several icosahedron particles (example is shown in Fig.2). The obtained pole figures show the presence of the common rotational fcc twins (OR1) as well as crystals oriented toward the origin according to OR2. Moreover, EBSD significantly compliments the TEM results. It is difficult to orient individual icosahedron nanoparticles in TEM to measure the orientation of its facets. Whereas in SEM, when precautions are taken in selecting the icosahedron particles laying with their facet on the flat substrate (Si wafer substrate was used in our experiment), that would guarantee the facet to be parallel to the substrate. In all cases we have measured icosahedron faces oriented parallel to {111} planes.

[1] Y.W.Cao, R.Jin, C.A.Mirkin, *Science* **2002**
[2] D.Seo, J.C.Park, H.Song, *J. Am. Chem. Soc.*, **2006**, *128*, 14863.
[3] L.Chen, J.Hu, S.Prikhodko, M.Pozuelo, S.Kodambaka, H.Yang, R.Richards, *Nanotechnology* **2008**, (submitted).
[4] L.D.Marks, *Rep.Prog.Phys*. **1994**, 57,603.
[5] S. Ino, *J.Phys. Soc.* **1966**, 21, 346.

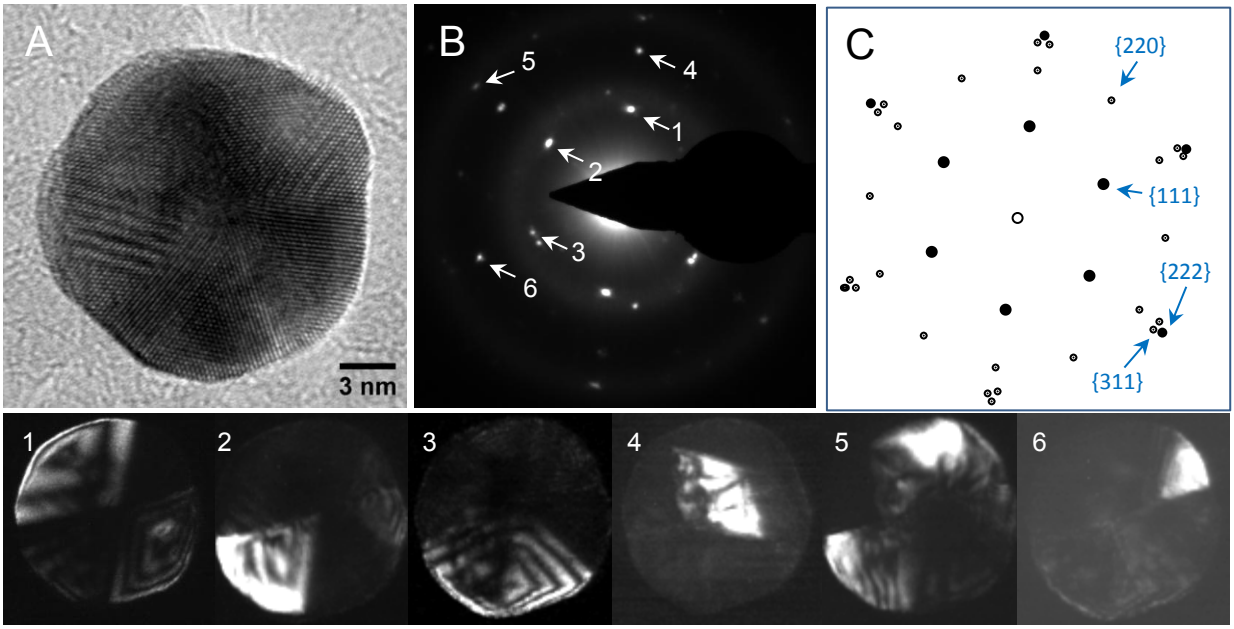
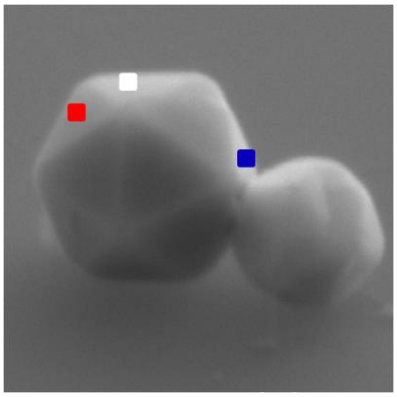


Fig.1. (A) HRTEM image of the gold icosahedron particle. (B) SAED pattern of the particle. (C) Schematics of indexing. 1-6) Dark-field images of the particle using different diffraction reflections shown in (B): 1-3 - {111}, 4,6 – {220} and 5 – {311} and {222} reflections.



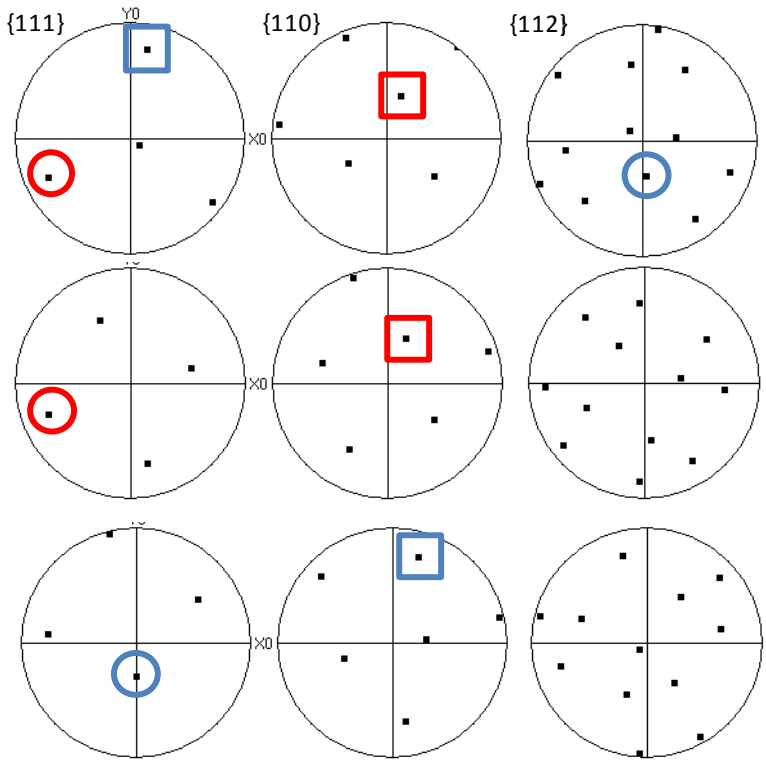


Fig.2. SEM image of gold icosahedron particles and pole figures {111}, {110} and {112} (left, center and right col. correspondingly) obtained using EBSD. Orientations of three different facets of an icosahedrons, marked in the SEM image with white, red and blue dots (size of the probing area was about 20 nm). Top panel of the pole figures represents orientation of the icosahedron facet marked with the white dot (origin). Central panel shows pole figures of the face highlighted with the red mark. This face oriented toward the origin with OR1. Parallel planes and directions of OR1 are highlighted with red circles and squares respectively. Lower panel represents pole figures of the blue facet. It is oriented toward the origin according to OR2. Corresponding parallel planes and directions are highlighted with blue circles and squares.

https://doi.org/10.1017/S143192761005600X Published online by Cambridge University Press