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Mini Review

Shape-Selective Effect of Foreign Metal Ions on Growth of Noble Metal Nanocrystals with High-Index Facets

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Abstract

The introduction of foreign metal ions during the growth of noble metal nanocrystals has shown interesting shape-selective effect. This phenomenon has been attributed to the underpotential deposition of the foreign metal on the surface of the nanocrystals. As a result, the relative stability of crystal facets is changed, leading to the appearance of thermodynamically unfavorable ones. Based on this approach, a number of nanocrystals enclosed by high-index facets have been prepared. Due to their exotic shapes and highly active surfaces, these nanocrystals have found applications in catalysis and surface enhanced Raman scattering. Here we present some recent progress in using Ag, Cu or Pd ions to tailor the growth of noble metal nanocrystals bounded by high-index facets.

INTRODUCTION

Shape-controlled growth of noble metal nanocrystals (NCs) has attracted tremendous attention in recent years [1-3]. This ever-increasing interest arose mainly because of the unique shape-dependent properties of NCs made of metals such as gold, silver, platinum and palladium, as well as their great promise in applications as catalysts, chemical sensors, biomedical imaging contrast agents, and drug delivery vehicles [1-7]. To date, A large number of NCs with different shapes including rods [8-10], wires, [11-13], plates [14], polyhedrons such as cubes, cubooctahedrons, octahedrons, decahedrons, and icosahedrons, [15-18], and particles of complex shape profile such as hollow structures [19-21], core-shells [22,23], thorn- or star-shaped particles [24,25], and dentrites [26] have been prepared for noble metals. These structures are typically enclosed by surfaces with low-energy crystal planes including {100} and {111}. In the past few years, high-index faceted noble metal NCs started to emerge. Although fewer cases for NCs enclosed by high-index facets have been reported compared to low-index ones, researchers have made considerable progress in both controlled growth of such NCs and understanding of their growth mechanisms [27-42]. Typically, NCs with high-energy surfaces are more difficult to form. During the growth of metal NCs, high-index facets disappear quickly due to their high surface energy and low stability, leaving NCs enclosed by low energy surfaces. To grow NCs with high-index facets, one has to rely on strategies such as the use of adsorbates including surfactants and halides [43-48] to stabilize thermodynamically unfavorable crystal planes, or the manipulation of the reaction kinetics to promote the growth rate along different directions favoring high-energy planes [49,50]. Based on these strategies, a few high-index facets NCs with exotic shapes such as tetrahexahedron (THH) [51-55], trisoctahedron (TOH) [56-58], truncated ditetragonal prism (TDP) [59-61], bipyramid (BP) [62-64], trapezohedron (TZH) [65], hexoctahedron (HOH) [66,67] and concave polyhedrons [39,68-70] have been synthesized recently. The growth mechanisms of such crystals have been discussed in some latest reviews such as refs [71] and [72]. Here in this short review, we will focus on the use of foreign metal ions to assist the growth of high-index faceted noble metal NCs.

The introduction of foreign metal ions in solution-phase syntheses has shown a drastic morphology-selection effect on noble metal NCs [1,11,17,25,73]. During the NC growth process, the foreign metal ions may adsorb and deposit onto the surface of a metal NC and affect its growth behavior. This is because the strong bonding between the adsorbed foreign metal atoms (adatoms) and the metal substrate will change the mode of any further deposition of metal atoms. This deposition may also alter the adsorption of surface protection molecules in the growth solution. Therefore, the introduction of foreign metal ions represents a very attractive strategy for selective growth of NCs with tailored shapes. This strategy has been most successfully demonstrated for the growth of Au NCs [9,28,52,62,63,74-76]. To date, a wide variety of Au NCs with different shapes have been prepared with the assistance of Ag^{I} ions. For instance, Murphy and coworkers used Ag^{I} in a seed-mediated synthetic approach to grow Au nanorods [62]. Yang et al. also applied this strategy to a polyol synthesis and successfully obtained Au nanocubes [16]. In addition, many other shapes, such as Au octahedra [75], rhombic dodecahedra [28], bipyramids [9,63] have been demonstrated using Ag^{I}-assisted growth. Generally, the shape-selective effect of Ag^{I} ions has been attributed the underpotential deposition (UPD) of Ag on Au surfaces [1]. Liu and Guyot-Sionnest have proposed that the UPD of metallic Ag on the different facets of Au can cause symmetry-breaking of Au NCs [9]. In addition to Ag, UPD of other metal ions such as Cu^{II} has also shown shape-selective effect in the synthesis of Au NCs [73,77,78].

The discovery of UPD can be traced back to 1949 when Rogers and coworkers found that the deposition of Ag on platinum electrode shifted to a potential more positive than that predicted by Nernst equation [79]. To date, extensive studies have been conducted for the UPD of various metal ions on the crystal surface of another metal [80]. It has been found that with properly controlled growth, the UPD of the foreign metal atoms can promote the formation of high-index facets of NCs. Here we will discuss some recent development on the shape-selective growth of high-index faceted noble metal NCs based on the strategy of UPD of foreign metal species.

Shape-selective Effect of Ag^{I} Ions

$AgNO_3$ has been employed as a shape-selective agent in various approaches for the growth of Au NCs. High-index faceted shapes including THH, elongated THH (ETHH), TOH, TDP, BPs have been reported. A typical THH can be considered as a cube with each face being "pulled out" from the center to form a rectangular pyramid. Therefore, 24 faces can be found in a THH. Sun et al. firstly reported Pt NCs with THH shape grown on glassy carbon electrode via an electrochemical square-wave potential route in 2007 [51]. However, THH Au NCs were not obtained until two years later when Wang et al. produced elongated THH (ETHH) Au NCs (Figure 1a) [52]. Their synthetic route was based on seed-mediated growth, in which Au seeds were mixed with a growth solution containing both $HAuCl_4$ and $AgNO_3$ at 5:1 ratio in the presence of ascorbic acid and cetyl trimethylammonium bromide (CTAB). A yield of 95% for the ETHH Au NCs was achieved using this method. They also found that the reduction rate of Au with ascorbic acid at different pHs is important – while ETHHs were obtained with high yield at low pHs with slow reduction of $HAuCl_4$, at high pHs when the growth of Au NCs was too fast, irregular shapes of Au were produced.

Based on a similar seed-mediated growth route, Guo et al. prepared THH Au NCs in a growth solution with small quantity of $AgNO_3$ (Figure 1b) [81]. Instead of using CTAB as stabilizing agent which is believed to account for the tendency of forming quasi-onedimensional NCs, they used didodecyl dimethylammonium bromide (DDAB) at the beginning of the reaction to assist the growth of quasi- THH. When the growth process was switched to a binary surfactant system containing both CTAB and DDAB, well-shaped THH Au NCs were formed. They found that the use of DDAB is critical to form THH Au NCs instead of elongated rods. This was attributed to the change of reaction kinetics, as well as the double C_{12} hydrophobic tails that have strong liquid/solid interfacial absorption and thus may affect the shape of the resulting NCs.

Interestingly, when CTAB was replaced with cetyl trimethylammonium chloride (CTAC) in another similar growth condition, Mirkin et al. obtained concave Au nanocubes, again with the assistance of $AgNO_3$ [82].Similar to THH, the concave Au nanocube also has 24 high index facets that can be derived by "pushing in" each of the six faces of a cube from their centers (Figure2a,b) [82]. The facets of the concave Au nanocubes were indexed as {720}. The formation of such concave structure was attributed to the use of both Cl- (from CTAC) and Ag^{I}. In a later work of the same group, they studied the effect of Ag adsorption on the shape control of Au NCs (Figure 2c-f) [61]. By varying

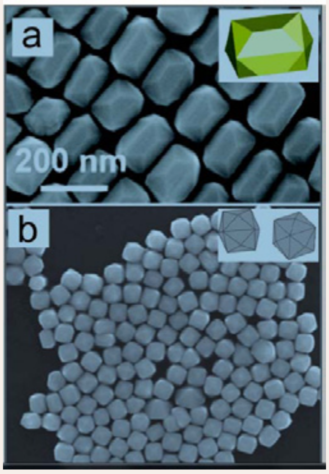

Figure 1 (a) Elongated THH Au NCs. Adapted with permission from ref 52. Copyright 2009 American Chemical Society.
(b) THH Au NCs. Adapted with permission from ref 81. Copyright 2010 The Royal Society of Chemistry.

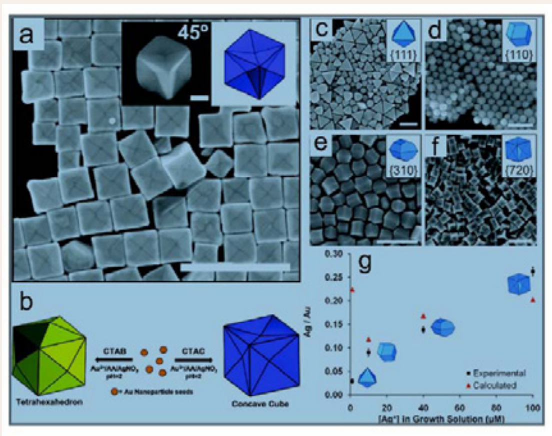

Figure 2 (a) Concave Au nanocubes prepared in the presence of $AgNO_3$(scale bar: 500 nm, inset scale bar: 200 nm); and (b) Shapechange caused by switching from CTAC to CTAB. Adapted with permission from ref 82. Copyright 2010 American ChemicalSociety. (c-f) Shape evolution of Au NCs with the change of AgNO3 concentration (scale bars: 200 nm); (g) Elemental analysisof Ag/Au ratio of each Au NC. Adapted with permission from ref 61. Copyright 2011 American Chemical Society.

the concentration of Ag^{I} added to the $HAuCl_4$ growth solution in the seed-mediated growth process, Au NCs enclosed by {110}, {310} and {720} were obtained, corresponding to rhombic dodecahedra, truncated ditetragonal prisms, and concave cubes, respectively. Detailed elemental analyses were performed on each shape to obtain the ratio of Ag to Au on the nanocrystal surface and compared with the model of each facet (Figure 2g). It was found that a monolayer or submonolayer of Ag was covered on these facets, indicating that the adsorption of Ag species, either in the form of UPD Ag or AgCl, plays a pivotal role in controlling the shape of the Au NCs.

The gold precursor used in all the above methods is $HAuCl_4$. When switching to a special Au precursor - - Au^{I}-tetra(ethylene glycol) (Au^{I}-TEG) complex which was generated *in situ* by reducing Au^{III} to Au^{I} in TEG, Xia et al. prepared penta-twinned Au nanorices enclosed by both {611} and {111} facets [64]. In this approach, $AgNO_3$ in trace amount had to be introduced for the formation of high-index facets. It was found that although the addition of Ag^{I} ions was not affecting the nucleation of Au NCs, it indeed altered considerably the growth process. The amount of Ag^{I} used in the reaction controlled the relative amount of typical penta-twinned decahedral Au NCs and nanorices. This is because the use of Ag^{I} promoted the growth rate along {111} direction and reduced the growth rate along {100} direction. As a result, a high-index {611} facet, which can be considered as the vector sum of five {100} and one {111}, started to appear in the resulting NCs.

Shape-selective Effect of Cu^{II} Ions

In addition to Ag^{I} ions, Cu^{II} ions have also shown shape-selective effect on noble metal NCs with high-index facets. Xia et al. successfully obtained Ag NCs with concave surfaces by reducing $AgNO_3$ with ascorbic acid (Figure 3) [83]. When the synthesis was done without the use of Cu^{II}, concave Ag octahedrons were formed. However, if the reaction was performed by introducing Cu^{II} ions, the shape of Ag NCs evolved from concave cube to octapod, and eventually to concave TOH -- all three shapes are enclosed by highindex facets. The shape-selective effect of Cu^{II} was examined by switching $Cu(NO_3)_2$ to $CuCl_2$ or $CuSO_4$. All forms of Cu^{II} ions showed the same result – the presence of Cu^{II} ions promoted the growth of Ag {111} facets with suppressed growth of {100} facets. The mechanism was related to the UPD of Cu on Au surface.

The use of Cu^{II} for controlling the shape of noble metal NCs was also demonstrated by Kuang et al [66]. In their study, Cu UPD was successfully applied to grow Au-Pd alloy NCs with a special hexoctahedral (HOH) shape which is enclosed by 48 high-index facets (Figure 4). The HOH Au-Pd NCs were prepared by the coreduction of H_2PdCl_4 and $HAuCl_4$ with ascorbic acid in the presence of octadecyl trimethylammonium chloride (OTAC). Cu^{II} ions were introduced in the form of $Cu(CH_3COO)_2$. The resultant Au-Pd HOHs were bounded by {431} facets. Similar to the concave Ag NCs, the UPD of Cu, which takes place at a potential 0.15 V more positive than that of $Cu^{I}/Cu0$ (0.521 V), plays an important role in controlling the shape. This Cu adlayer can be oxidized by $PdCl_4^{\ 2-}$ or $AuCl_4^{-}$ *via* galvanic replacement reaction. As a result, Pd can be deposited on the Au surface along with the deposition of Au, leading to the formation of Au-Pd alloy.

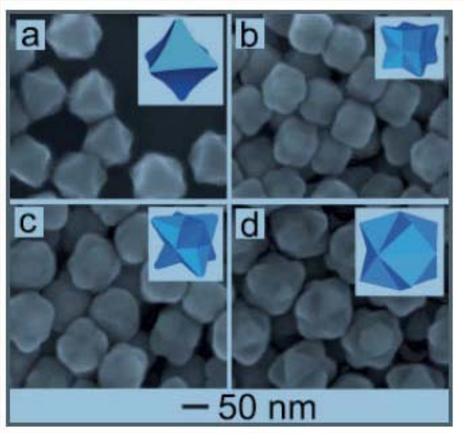

Figure 3 Concave Ag NCs prepare in the presence of CuII ions.Adapted with permission from ref [83]. Copyright 2011 Wiley.

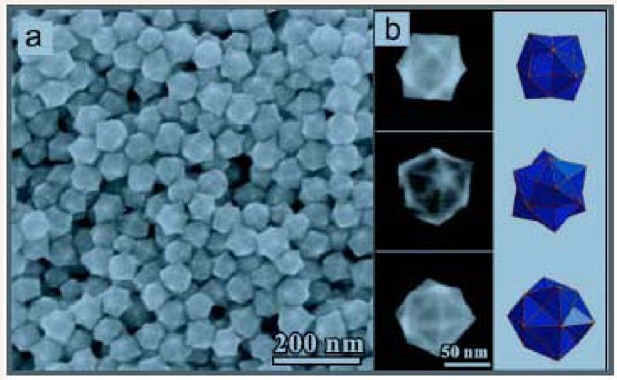

Figure 4 HOH Au-Pd alloy NCs. Adapted with permission from ref [66]. Copyright 2011 American Chemical Society.

Synergistic Effect of Two Foreign Metal Ions

For all the above-discussed cases where foreign metal ions were employed to tailor the growth of Au NCs enclosed by high-index facets, only one type of metal ions was introduced in the growth solution. If two foreign metal species are introduced simultaneously in an Au NC growth process, their deposition on Au surface may behave differently since the deposition mode of one metal on another metal surface can be significantly affected by their physicochemical properties such as atomic radii and bond dissociation energies [84]. Therefore, the interference of the deposition of two different foreign metals on Au surface may give rise to unusual shape-selective effect [85].

Indeed, Lu et al. employed a one-pot poly (diallyldimethylammonium chloride)-mediated polyol process to grow Au NCs with the assistance of Ag^{I} and Pd^{II} ions and found that various shapes including Au truncated ditetragonal prisms (TDPs) enclosed by 12 {310} facets, truncated THHs with both {111} and {310} facets, and multiple-twinned bipyramids can be obtained in high yields [59]. In this study, they found that when introduced separately, Ag^{I} and Pd^{II} ions caused {110} and {100} truncations of Au octahedra, respectively. The presence of both Ag^{I} and Pd^{II}, however, led to the growth of high-index facets. At low concentrations of Ag^{I}, simultaneous deposition of Ag and Pd on Au crystal surface led to the formation of truncated THHs, a shape partially enclosed by {310} facets. With the increase of Ag^{I} to Pd^{II} ratio, truncated ditetragonal prisms (TDPs) bounded by {310} facets were produced (Figure 5). This is the first report of Au NCs with TDP shape. Particles with this shape are enclosed with 12 faces: eight side faces parallel to the principal axis and two terminating faces located at each of the two ends. Elemental analyses indicate that these NCs are mainly composed of Au with a surface layer rich in Ag and Pd. These results indicate that Ag and Pd promote the formation of different facets -- while Ag^{I} causes the appearance of {110} facets, Pd^{II} may facilitate the development of {100} facets. Study of Pd adsorption on Au surfaces showed that $PdCl_4^{2-}$ forms ordered adlayers on all Au low-index facets [86]. UPD of Pd on Au(100) and Au(110) may also lead to alloy formation by adatom exchange diffusion. Although the relative bonding strength of UPD Pd on Au (110) and (100) is unclear, it is likely that the deposited Pd species on Au surface will cause change in the deposition mode of Ag.

In addition, $PdCl_4^{2-}$ may also replace Ag deposited on the Au surface *via* galvanic replacement reaction. Together, these factors lead to the formation of highindex {310} facets. When the AgI concentration is relatively high (>1/2 of $AuCl_4^-$ concentration), fivefold- twinned bipyramids were formed (Figure 6). The Au penta-twinned BPs have faceted sides and truncated tips.

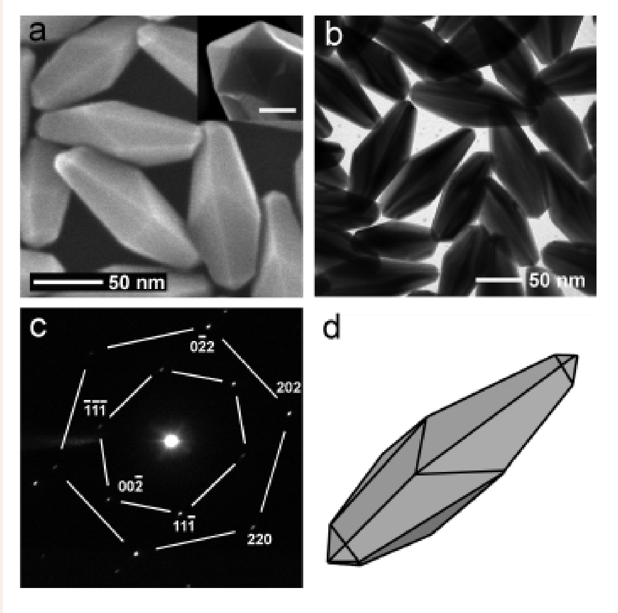

Figure 6 (a,b) Au bipyramids (BPs) synthesized at a ratio of Au:Pd:Ag=15:1:8. TEM image of the Au BPs shows twinplanes within the NCs. (c) SAED pattern of a BP shows the superposition of both [110] and [111] zones of fcc gold. (d) Schematic drawing of a Au BP. Adapted with permissionfrom ref [59]. Copyright 2011 American Chemical Society.

Applications of High-index Faceted NCs

It is not surprising that due to their exotic shape profiles, high-index faceted NCs are expected to exhibit interesting catalytic and optical properties and may find a wide variety of applications. Indeed, high chemical activity has been demonstrated from a number of Au NCs enclosed by high index facets. The high chemical activity of high-index facets NCs is mainly because of the high density of atomic steps and kinks on their high index facets. It has been found that the oxidation peak of elongate THH Au NCs with {730} facets started to show at 1.18 V vs. Ag/AgCl in 0.1 M H_2SO_4 solution compared to a higher potential of 1.35 V for octahedral Au NCs enclosed by low index {111} facets [52]. When used for formic acid oxidation, THH Au NCs enclosed by {520} facets also exhibited a low peak current potential of 0.56 V vs. Ag/AgCl, in comparison with 1.27 V for traditional polycrystalline Au electrode with {111} surface [81]. Enhanced catalytic activity was also observed for other shapes of Au NCs. For instance, HOH Au-Pd NCs have shown excellent performance towards electrooxidation of formic acid with high methanol tolerance [66]. An oxidation current 5 times higher than Pd black was attained from HOH Au-Pd alloy NCs, while their electrochemical active surface area was not affected by the presence of methanol. Xia et al. also showed that Au nanorices enclosed by {611} facets started to catalyze CO oxidation at a relative low temperature of 140 °C, while for Au spheres with similar size, no activity was observed at temperatures below 200 °C [64]. More importantly, the nanorices did not show any noticeable change in shape after CO oxidation tests at 270 °C, indicating that high stability can be attained from high-index faceted NCs. In another work, Gang et al. deposited a monlayer (ML) of Pt via galvanic replacement reaction with a Cu UPD monolayer on Au TDPs [60]. They examined the hydrogen evolution and oxidation reactions on the resultant Au(TDP)-Pt(ML) NCs and found much higher activity per Pt surface area than spherical Pt NCs. In this case, the Au TDP served as a template to translate the high-index facet to its supported materials.

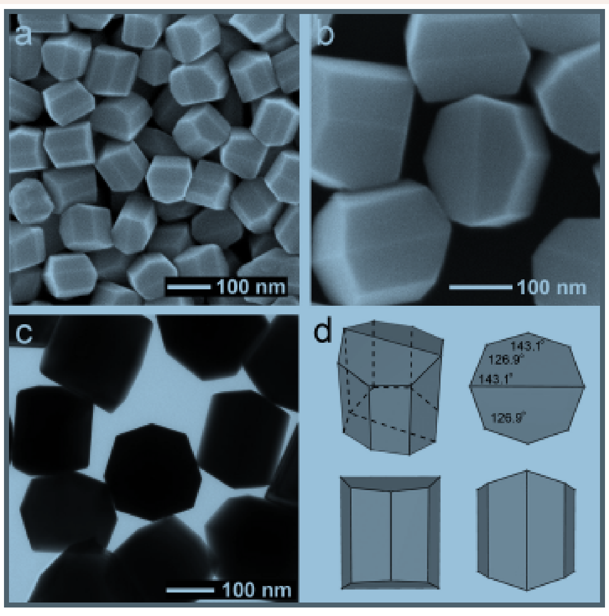

Figure 5 (a,b) TDP Au NCs synthesized with a ratio of Au:Pd:Ag= 15:1:1.6. (c) TEM image of the Au NCs. (d) Schematic drawing of a TDP enclosed with {310} facets and itsprojections along the three indicated viewing angles. Adaptedwith permission from ref [59]. Copyright 2011 American Chemical Society.

For high-index faceted Ag and Au NCs, interesting surface plasmon resonance (SPR) and surface enhanced Raman scattering (SERS) properties have been demonstrated. Xia et al. found that the SPR peaks of Ag NCs continuously shift from 425 to 530 nm from concave octahedron to concave TOH [83]. They also employed the NCs absorbed with 1,4- benzenedithiol molecules for SERS tests and obtained an SERS enhancement factor of 5.7×10^5 for concave TOHs, 10 times higher than that of octahedrons. This improved SERS enhancement factor was attributed to the larger density of intra-particle gaps, tips, and edges existing among concave TOHs than octahedrons. Strong SERS was also observed from HOH Au NCs enclosed by {321} facets [67].

SUMMARY AND OUTLOOK

The use of foreign metal ions to achieve controlled growth of noble metal NCs with high-index facets is still in its infancy. Although some mechanistic studies have been attempted to understand the UPD of foreign metal species on NC surface to gain better control over the resulting shapes, in most cases, this strategy still remains as a trial-and-error approach. Due to the complexity of the NC growth processes involving metal precursors, foreign metal ions, anions, surfactants, and surface functional molecules, it is difficult to isolate the effect of a single species. Often times, it is the combination of multiple factors that leads to the formation of different NC shapes. Therefore, further systematic study is necessary to elucidate the underlying mechanisms. Strategies to isolate the effects of different species present in NC growth process may offer some clues.

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