Solvo-thermal Preparation of Cadmium Telluride Nanoparticles from a Novel Single Source Molecular Precursor

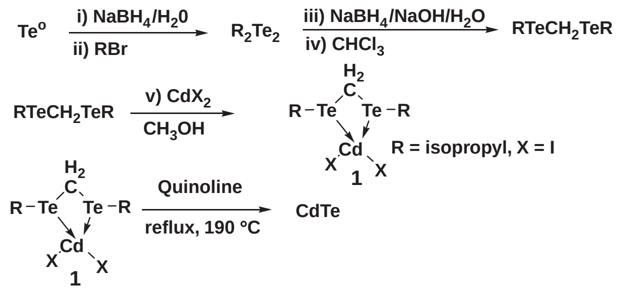
D. Selvakumar, Santosh Kumar Tripathi, Rajendra Singh, and M. Nasim*
MOCVD Division, Defence Materials & Stores Research & Development Establishment,
DMSRDE P.O., GT Road, Kanpur-208013, India

(Received September 5, 2007; CL-070960; E-mail: mocvd@rediffmail.com)

A convenient solvo-thermal route has been developed for the preparation of cadmium telluride nanoparticles using a novel single source molecular precursor. The X-ray diffraction pattern confirms the face-centered-cubic structure and average diameter of the nanocrystallite is calculated to be 29.5 nm. SEM image clearly shows the needle-like structure which is further supported by TEM image where agglomerated nanoparticles are clearly seen.

Nanoparticles of CdSe, CdTe, which are the compound semiconductors of elements in group II and VI of the periodic table, have been extensively investigated because of their exclusive features such as, size-dependent emission wavelength due to the quantum size-effect and strong luminescence and these particles are expected to be applied to various fields such as informational home electronics and biomarkers as new phosphores.[1–4] Nanosize semiconductor materials exhibit high emission efficiency when the surface state of the particles is controlled properly. A wide variety of capped nanoparticles have been prepared from metalorganic precursor through solvo-thermal route in presence of various capping agents.[5–8] However, most of these reactions require the conventional chemicals like dimethyl cadmium and diisopropyl telluride, which are extremely toxic, pyrophoric, and explosive.[9] The use of single-source molecular precursor for the preparation of nanoparticles avoids such potential problems. Although most of the work following the single-source molecular precursor route describes the preparation of sulfides and selenides, not much work has been done on tellurides.[10–18]

Here, we report a convenient method for the preparation of cadmium telluride (CdTe) nanoparticles under mild conditions from a novel single source molecular precursor, i.e. cadmium(II) complex of bis(isopropyltelluro)methane, **1**, reported earlier by us.[19] The precursor **1** is solid and neither extremely toxic, nor explosive. The CdTe nanoparticles have been prepared using single-source molecular precursor **1** according to Scheme 1. The lattice structure and the size of the prepared nanoparticles are calculated by X-ray powder diffraction (XRD) and the morphology has been studied by SEM and TEM techniques.



Scheme 1. Preparation of CdTe nanoparticles.

In a typical experiment for the preparation of nanoparticles (NPs), a solution of precursor **1** (0.5 mM, 0.367 g) dispersed in quinoline (10 mL) was injected dropwise into 25 mL of hot quinoline (190 °C) under inert atmosphere. The previously colorless solution turned dark brown and stirred at the same temperature for further 1 h. The reaction mixture was cooled to room temperature and further stirred for 2 h with the addition of an excess of methanol. The gray solid was separated from the flocculent solution by centrifugation and washed several times with methanol. The powdered particles were dried under vacuum and stored in argon atmosphere. The gray powder was not soluble in any of the organic solvents indicating the particles are not capped with quinoline.[20]

Conclusive formation of cadmium telluride is evident from the X-ray powder diffraction data. The XRD[21] pattern of the decomposed product is shown in Figure 1 and the data can be indexed to face-centered-cubic (fcc). The lattice parameter is calculated to be $a = 6.43 \pm 0.003$ Å, which is in agreement with that of cadmium tellurium reported in the JCPDS diffraction file.[22] From the width of XRD peaks, the average crystallites diameter of the nanoparticles is estimated to be 29.5 nm from the Scherrer equation.[23]

SEM[21] image (Figure 2a) reveals the entangled needle-like highly crystalline morphological features at the nanometer scale. TEM[21] micrograph (Figure 2b) clearly shows that these needles are self-assembled aggregates of several spherical cadmium telluride nanoparticles. In this image, pearl-neck-lace agglomerate of the nanoparticles is seen in great abundance. The average diameter of the nanocrystallites is calculated in the range of 29–30 nm. It is obvious due to strong and long-ranged dipole–dipole interaction in the semiconductor nanocrystals, which leads to the formation of pearl-neck-lace-like nanostructure. Tang, N. A. Kotov and Giersig[24] reported the spontaneous reorganization of CdTe nanoparticles into crystalline nanowires upon controlled removal of the protective shell of organic stabilizer. The intermediate step in the nanowire formation was found to be pearl-neck-lace aggregates. Strong dipole–dipole interaction is believed to be the driving forces of nanoparticle self-

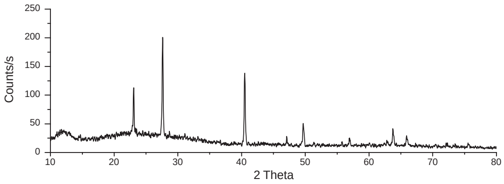


Figure 1. XRD pattern of cadmium telluride nanoparticles.

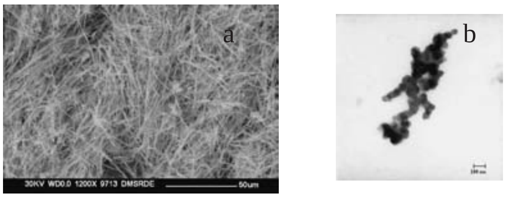


Figure 2. a) SEM and b) TEM images of cadmium telluride nanoparticles.

organization. We have prepared CdTe nanoparticles without using any surfactant or capping agents, which produce pearl-neck-lace-like nanostructures. These findings demonstrate the collective behavior of nanoparticles as well as a convenient, simple technique for production of one-dimensional colloids suitable for subsequent processing into quantum-confined superstructures, materials, and devices.

In conclusion, a convenient and cost effective solvo-thermal route has been developed for the preparation of nanoscale cadmium telluride needle-shaped particles in quinoline/methanol medium without any further size selection procedure. The use of other capping agents for the preparation of different size nanoparticles from single-source molecular precursors are under progress.

The authors are thankful to Director of Defence Materials & Stores Research & Development Establishment for encouragement, support and permitting this work for publication. The authors are also thankful to Dr. R. Ramani of Defence Materials & Stores Research & Development Establishment for TEM and D. Bharathimohan of Hyderabad University for XRD analysis.

References and Notes

1 V. L. Colvin, M. C. Schlamp, A. P. Alivisatos, *Nature* **1994**, *370*, 354.
2 D. L. Klein, R. Roth, A. K. L. Lim, A. P. Alivisatos, P. L. McEuen, *Nature* **1997**, *389*, 699.
3 B. A. Ridley, B. Nivi, J. M. Jacobson, *Science* **1999**, *286*, 746.
4 S. H. Kim, R. G. Mediros, D. A. A. Ohlbergf, W. R. Stanley, J. R. Health, *J. Phys. Chem. B* **1999**, *103*, 10341.
5 C. B. Murray, D. J. Norris, M. G. Bawendi, *J. Am. Chem. Soc.* **1993**, *115*, 8706.
6 M. Kuno, J. K. Lee, B. O. Dabbouri, F. V. Miculec, M. G. Bawendi, *J. Chem. Phys.* **1997**, *106*, 9869.
7 J. E. B. Katari, V. L. Colvin, A. P. Alivisatos, *J. Phys. Chem.* **1994**, *98*, 4109.
8 X. Peng, L. Manna, W. Yang, J. Wickham, E. Schere, A. Kadacanich, A. P. Alivisatos, *Nature* **2000**, *404*, 59.
9 a) J. J. Eiscj, *Organometallic Synthesis*, Academic Press, New York, **1981**, Vol. 2. b) A. L. Wayda, M. Y. Davensbourg, *Experimental Organometallic Chemistry, A Practicum in Synthesis and Characterization*, ACS Symposium Series 357, American Chemical Society, Washington D.C., **1988**. c) D. F. Shriver, M. M. Drezdon, *The Manipulation of Air Sensitive Compounds*, Wiley, New York, **1986**.
10 T. Trindade, P. O'Brien, *Adv. Mater.* **1996**, *8*, 161.
11 T. Trindade, P. O'Brien, X. Zhang, *Chem. Mater.* **1997**, *9*, 523.
12 B. Ludolph, M. A. Malik, *Chem. Commun.* **1998**, 1849.
13 M. Chunggaze, J. McAleese, *Chem. Commun.* **1998**, 833.
14 M. A. Hines, P. Guyot-Sionnest, *J. Phys. Chem. B* **1998**, *102*, 3655.
15 M. A. Malik, M. Zulu, P. O'Brien, G. Wakefield, *J. Mater. Chem.* **1998**, *8*, 1885.
16 T. Trindade, P. O'Brien, X. Zhang, M. Motevalli, *J. Mater. Chem.* **1997**, *7*, 1011.
17 a) N. Revaprasadu, M. A. Malik, P. O'Brien, G. Wakefield, *Chem. Commun.* **1999**, 1573. b) P. Sreekumari Nair, N. Revaprasadu, T. Radhakrishnan, G. A. Kolawole, *J. Mater. Chem.* **2001**, *11*, 1555. c) T. Trindade, P. O'Brien, *Adv. Mater.* **1996**, *8*, 161. d) M. Green, P. O'Brien, *Chem. Commun.* **1999**, 2235. e) B. Ludolph, M. A. Malik, *Chem. Commun.* **1998**, 1849.
18 Y. Wada, D. Niinobe, M. Kaneko, Y. Tsukahara, *Chem. Lett.* **2006**, *35*, 62.
19 D. Selvakumar, R. Singh, M. Nasim, G. N. Mathur, *Phosphorus, Sulfur Silicon Relat. Elem.* **2001**, *172*, 247.
20 M. A. Olshavsky, A. N. Goldstein, A. P. Alivisatos, *J. Am. Chem. Soc.* **1990**, *112*, 9438.
21 X-ray diffraction (XRD) pattern were measured by using a Philip PW 1700 series automated powder deffractometer using Cu Kα radiation at 40 kV/40 mA with a secondary graphite-crystal monochromator. Samples were supported on glass slides (5 cm). The concentrated toluene solution was slowly evaporated at room temperature on to a glass slide to obtain a sample for analysis. The SEM images were recorded on JEOL JSM-35 CF scanning electron microscope at recording voltage of 10 kV. The samples were gold coated prior to examination under microscope and the TEM images were recorded on JEOL JEM CX II transmission electron microscope at an operating voltage of 80 kV. At first, a colloidal suspension of CdTe was formed in methanol. A fine 400 mesh copper grid coated with colloidal film was taken and this was coated again with a layer of carbon using a vacuum evaporator to enhance the conductivity. Two drops of the colloidal suspension was taken on the grid and was air dried completely before microscope viewing.
22 JCPDS file no. JSPS#75-2083.
23 H. P. Klig, L. E. Alexander, *X-Ray Diffraction Procedures*, John Wiley and Sons, New York, **1959**.
24 Z. Tang, N. A. Kotov, M. Giersig, *Science* **2002**, *297*, 237.

Published on the web (Advance View) December 1, 2007; doi:10.1246/cl.2008.34