Pressure compression of CdSe nanoparticles into luminescent nanowires

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Oriented attachment (OA) of synthetic nanocrystals is emerging as an effective means of fabricating low-dimensional nanoscale materials. However, OA relies on energetically favorable nanocrystal facets to grow nanostructured materials. Consequently, nanostructures synthesized through OA are generally limited to a specific crystal facet in their final morphology. We report our discovery that high-pressure compression can induce consolidation of spherical CdSe nanocrystal arrays, leading to unexpected one-dimensional semiconductor nanowires that do not exhibit the typical crystal facet. In particular, in situ high-pressure synchrotron x-ray scattering, optical spectroscopy, and high-resolution transmission electron microscopy characterizations indicate that by manipulating the coupling between nanocrystals through external pressure, a reversible change in nanocrystal assemblies and properties can be achieved at modest pressure. When pressure is increased above a threshold, these nanocrystals begin to contact one another and consolidate, irreversibly forming one-dimensional luminescent nanowires. High-fidelity molecular dynamics (MD) methods were used to calculate surface energies and simulate compression and coalescence mechanisms of CdSe nanocrystals. The MD results provide new insight into nanowire assembly dynamics and phase stability of nanocrystalline structures.

INTRODUCTION

Oriented attachment (OA) of synthetic nanocrystals is emerging as an effective means of fabricating low-dimensional nanoscale materials (1–7). Through OA, nanostructures such as nanowires and nanosheets have been fabricated. Important applications in energy conversion have been successfully demonstrated (8–10). OA crystal growth for material synthesis occurs in solutions where active mobile clusters such as ions, clusters, or nanocrystals are self-assembled through direction-specific interparticle forces, such as dipole-dipole interactions found in several material systems (2, 11–14). Alternatively, capping agents or organic ligands (15, 16) have been used to modify or block certain crystal surface facets to inhibit their growth so that controlled growth of specific architectures, such as nanorods and nanowires, through unblocked crystal facets is achieved. More recently, block copolymer and surfactant self-assembly (13, 14, 17–21) have been used to confine oriented consolidation of necklace structures, including nanowires and bundles. Evolving toward the minimum free energy, OA leads to alignment of energetically favorable nanocrystal facets when the nanocrystals are coalesced to form various nanostructures (for example, nanowires) under ambient pressure (2, 11, 12, 22, 23). As a consequence, these nanostructures are generally limited to a specific crystal facet in their final morphology. Although OA is conducted at ambient conditions, our method, by application of external pressure, provides an additional processing parameter to change material structures via manipulating system energy. This enables the formation of new material configurations that affect new chemical and physical properties. In contrast to OA in solutions, pressure-induced synthesis is conducted by applying pressure to solid-state self-assembled arrays of spherical CdSe nanocrystals. When the external pressure is increased, the nanocrystal separation distance initially decreases. At pressures above a threshold, adjacent nanocrystals come into contact and then consolidate, enabling the formation of new classes of mechanically stable one-dimensional (1D) nanowires. To explore the high-pressure–induced synthesis, we prepared films of self-assembled spherical CdSe nanocrystal arrays by solvent evaporation and loaded them into a diamond anvil cell (DAC) to allow reversible compression under controlled and continuously monitored pressure conditions (see experimental details in Materials and Methods). Electron microscopy, in situ high-pressure synchrotron x-ray scattering, and optical spectroscopy were performed to investigate the formation mechanism of nanowires and the associated structure and structural evolution during increasing degrees of compression.

RESULTS

Structure of CdSe nanowires

Figure 1A and its inset show a representative transmission electron microscopy (TEM) image of the 1D CdSe nanowires and bundles in the nanowire arrays formed after high-pressure compression of the spherical nanocrystal arrays. The CdSe nanowire array consists of individual CdSe nanowires with 2D hexagonal packing. These nanowire arrays were collected directly from the DAC and then dissolved in organic solvents, such as toluene, to form stable homogeneous colloidal solutions. The nanowires have an average diameter of 5 nm, with an SD of 10%, consistent with the diameter of the starting spherical nanoparticles. The nanowires display uniform lengths roughly on the scale of ~600 nm and an aspect ratio of ~120. High-resolution scanning TEM (HR-STEM) images shown in Fig. 1B reveal an unusual nanowire morphology, both polycrystalline and twisted. Along the nanowire c axis, each nanowire consists of connected individual CdSe nanocrystal domains. The nanocrystal domains retain the faceted nature of the starting spherical CdSe nanocrystals, which aids crystallographic indexing of the nanocrystal facets. This analysis indicates that nanowires are formed through direct coalescence of spherical nanocrystals along the c axis. From the lattice spacings seen in HR-STEM imaging, we found that the nanocrystal coalescence leads to several

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nanocrystal lattice orientations, which are summarized in Fig. 1 [B (inset) and C]. The CdSe nanocrystals primarily coalesce along \( <111>/<111> \) orientations, with some other types of interface. This solid-state coalescence contrasts with OA growth in solutions, where freely mobile moieties, such as ions, small clusters, and nanocrystals, are typically locked in through specific facets \((1, 2, 5, 12, 22, 23)\). During solution nucleation and growth, these free nanocrystals can rotate under the driving force of the favorable facet interactions. Structural relaxation is possible through solution-mediated mass redistribution or atomic rearrangement to eliminate defects, such as the neck structures between nucleated nanocrystals, leading to the formation of smooth and single crystalline nanowires \((12, 22)\). In situ synchrotron x-ray results indicate that the compressive stress induces neither nanocrystal rotation nor preferential nanoparticle alignment during compression and pressure release (fig. S2 and Fig. 2). We propose that this accounts for the observed random coalescence, the lack of neck structures between adjacent nanocrystals, and a twisted morphology instead of smooth single crystalline nanowires. This random coalescence occurred regardless of CdSe nanocrystal size, as shown in Fig. 1D.

**In situ high-pressure small- and wide-angle synchrotron x-ray scattering characterizations**

The consolidation of CdSe nanocrystals and the formation of CdSe nanowires were further explored using in situ high-pressure small-angle (HP-SAXS) and high-pressure wide-angle (HP-WAXS) synchrotron x-ray scattering characterization. Figure 2 shows the SAXS and WAXS data collected from the same specimens during compression and pressure release. The results reveal pressure-induced phase transitions at both the atomic scale and mesoscale. Figure 2A indicates that at the atomic scale, the initial wurtzite (WZ) CdSe nanocrystals remained as WZ with increasing pressure up to \( \approx 7 \) GPa. At about 7 to 8 GPa, phase transformation from WZ to rock salt (RS) occurred \((24)\). The nanocrystals then remained as RS with increasing pressure up to about 15 GPa. When pressure was released from 15 GPa, the nanocrystals remained in the RS structure until 0.7 GPa, only reverting to the zinc blende (ZB) structure when pressure was released to ambient pressure. The atomic phase transition of CdSe nanocrystals under pressure uniquely differs from the formation of metallic nanowires, such as gold nanowires, that do not exhibit atomic phase transition \((25, 26)\). The WAXS peak intensity as a function of azimuthal angle, measured during the entire compression and release cycle, exhibits only characteristics of powders (see fig. S2) \((27)\). No preferential alignment of the nanoparticles was observed in either the starting nanoparticle arrays or those formed during compression and pressure release. At the mesoscale, the SAXS patterns shown in Fig. 2B indicate that before compression, the nanocrystals were arranged in a standard face-centered cubic (fcc) lattice \((fmc3m)\) with a unit cell parameter \( a_{\text{fcc}} = 9.8 \) nm (also see fig. S3). Upon continuous
increase of pressure, the SAXS peaks continuously shifted to higher angles (lower $d$-spacing), indicative of shrinkage of interparticle distances. This shrinkage is more directly revealed in Fig. 2C, where the $d$-spacing of the first Bragg peak is plotted as a function of pressure. It can be seen from this compression curve that the $d$-spacing continuously decreased and the nanocrystals continuously approached each other with increasing pressure. The nanocrystals were in contact at a threshold pressure of ~9.5 GPa, where a minimal $d$-spacing was reached. When the pressure was released from 9.5 GPa to ambient condition, the SAXS peaks and the $d$-spacing returned to the original positions of starting materials, indicating that the change of the unit cell was reversible in the pressure range between 0 and 9.5 GPa. However, Fig. 2B indicates that when the pressure was increased above 9.5 GPa, the contacting nanocrystals began to consolidate, accompanied by a transformation of the mesophase from fcc packing to 2D hexagonal packing. This change in the phase and the corresponding unit cell dimension appears irreversible. This mesophase transition is illustrated in Fig. 2D, which shows the $d$-spacing ratio ($R$) at different pressures. In the fcc nanocrystal mesophase, $R = d_{311}/d_{111}$; in the 2D hexagonal mesophase, $R = d_{20}/d_{10}$.

Fig. 2. Pressure-induced structural evolution of CdSe nanocrystal mesophase during compression and release. (A) Representative integrated data from WAXS images of CdSe nanocrystal arrays at varied pressures. (B) Representative integrated data from SAXS images of CdSe nanocrystal arrays at varied pressures. $r$ is used to display the releasing pressure. Below 9.5 GPa, the HP-SAXS patterns are consistent with fcc mesophase. Mesophase transformation of the nanocrystal arrays starts at ~9.5 GPa, followed by an intermediate phase, where the overall distorted structures cannot be indexed by either fcc or 2D hexagonal mesophase. After releasing the applied pressure, the HP-SAXS patterns are consistent with 2D hexagonal mesophase. (C) The $d_{111}$-spacings change with the pressure during compression and release. (D) $d$-spacing ratios ($R$) at different pressures. In the fcc nanocrystal mesophase, $R = d_{311}/d_{111}$; in the 2D hexagonal mesophase, $R = d_{20}/d_{10}$.

Optical property and correlation to structure
The results of optical absorption experiments shown in Fig. 3 indicate that the optical properties of nanocrystals correlate with the
phase transition during compression and pressure release. In particular, the absorption peak continuously shifted to blue for pressures up to ~7 GPa. When the pressure was released from ~7 GPa, the absorption peak returned reversibly (see fig. S4). Considering that the absorption peak reflects the direct gap specific to a crystal structure, this result is consistent with the complete recovery of WZ observed from the HP-WAXS results shown in Fig. 2A. However, when the pressure was increased above ~7 GPa, the sharp absorption edge abruptly disappeared in Fig. 3. We believe that this is caused by the observed phase transition from the direct bandgap WZ to the indirect bandgap RS, indicated by the WAXS results in Fig. 2A. Pressure was then released after reaching a maximum value of ~14.8 GPa. Upon complete release of the pressure, a new absorption peak appeared at 594 nm. According to the TEM and HP-SAXS results, CdSe nanowires were formed when pressure was completely released, and this new peak can be attributed to the CdSe nanowires. The red shift and broadening of the excitonic peak of the nanowires were probably caused by the incomplete fusion (or growth) of the nanoparticles into nanowires, which is consistent with results on the nanowires synthesized from OA. Increased grain size through more complete fusion of nanoparticles would enable stronger delocalization, thereby narrowing of bandgaps (6, 28). The nanowires exhibited a quantum yield of about 1.6% immediately after the pressure release, with a slowly increasing quantum yield over time thereafter. This is attributed to relaxation of residual stresses within CdSe quantum dots after pressure release (29). To validate this, we relaxed residual stresses further by annealing samples at 200°C (fig. S5). As indicated in fig. S5, the quantum yield of annealed samples significantly increased from 1.6 to 17%. The crystal structure remains the same after annealing (fig. S5). Compared to a luminescence quantum yield of less than 1% in typical chemically synthesized CdSe nanowires, the high luminescence quantum yields obtained here are indicative of high crystalline quality nanowires (11), which also has been observed to increase nanowire mechanical strength (30, 31).

Molecular dynamics simulation of CdSe coalescence
To understand the energetics of phase transformations described above, molecular dynamics (MD) simulations were performed to compress a synthetic bulk crystal composed of half ZB and half WZ structures. The initial configuration, projected along a <111> ZB or <0001> WZ direction, is shown in Fig. 4A, where the hexagonal {111} ZB or {0001} WZ plane is indicated by a hexagon. The system was then compressed for 1.2 ns at a temperature of 1500 K and a pressure of about 5 GPa. The same projected configuration is shown in Fig. 4B, where a square indicates that the initial hexagonal configuration transforms to a square plane. Examination of the unit cell of the square confirms that the system corresponds to an RS crystal structure. The MD simulation was continued for another 1.3 ns to release the stress, and the final projected image is shown in Fig. 4C, in which the system recovered back to a hexagonal symmetry characteristic of {111} ZB or {0001} WZ. More detailed analysis indicated that the system after pressure release also contains a mixture of ZB and WZ. However, different ZB and WZ domains are stacked along the direction normal to the image, as opposed to the left and the right bicrystals shown in Fig. 4A. Hence, Fig. 4 (A and C) appears different. The modeling results in Fig. 4 are consistent with the experimental findings that, during compression, the initial WZ nanocrystals were driven to RS structure and were recovered to a stable ZB structure upon pressure release. Further, the simulation illustrates how the RS structure acts as an intermediate phase without a change in the initial and final crystallographic orientations. For example, the initial WZ {0001} and the final {111} ZB planes shown in Fig. 4 (A and C) are essentially the same hexagonal plane.

Fig. 3. In situ UV-vis absorption spectra of CdSe nanocrystal arrays during compression and pressure release. r is used to show the releasing pressure.

Fig. 4. MD simulation of pressure-induced nanocrystal phase transition and consolidation. (A) Initial configuration. (B) Intermediate configuration at pressure ~5 GPa. (C) Final configuration after pressure release. Two MD simulation cases of sintering showing (D) the formation of the <111>/<111> interface and (E) the formation of the <111>/<210> interface.
Direct MD simulations were also performed to study sintering of two CdSe ZB particles of 5.5 nm diameter (25, 26). Figure 4 (D and E) shows two representative cases, where the two particles were misaligned arbitrarily. After MD simulations at 800 K for 0.2 ns with the cell dimension along the axis of the two particles shrinking at a strain rate of \(3.75 \times 10^{-5} \text{ ps}^{-1}\), the two particles in the first case coalesced on a \(\{111\}\)/\(\{111\}\) interface, as shown in Fig. 4D, and the particles in the second case coalesced on a \(\{111\}\)/\(\{210\}\) interface, as shown in Fig. 4E. Time-resolved observation indicates that during sintering, the interface orientations are mostly random due to the lack of rotation before the contact.

MD simulations were further used to calculate energies of a variety of CdSe ZB surfaces (see fig. S6) (32, 33), which indicated that \{111\} and \{110\} surfaces of CdSe nanocrystals have the lowest energies. As a result, the nanocrystal facets were mostly composed of these two surfaces before the compression. If sintering proceeds with little adjustment of orientation, interfaces are likely to contain one \{111\} plane or one \{110\} plane because of the high probability of encountering such a surface when nanocrystals come into contact. Once such a \{111\} or \{110\} plane is present, we hypothesize that the other crystals find a matching plane when they come into contact. This process appears consistent with the atomistic coalescence process shown in Fig. 4 (D and E) and contrasts with the solution-processed OA, where interfaces tend to contain high-energy surface planes because systems can remove these unfavorable planes by forming interfaces through crystal rotation. Hence, the calculated surface energies appear consistent with the conclusion drawn from the experimental observations that coalescence occurred nearly randomly with little crystal rotation. Finally, we point out that although the coalesced particles were RS crystals, the RS structure only acted as an intermediate phase because it does not affect the initial and the final orientations. For example, Fig. 4 (A to C) indicates that the initial \{111\} ZB or \{0001\} WZ planes before compression were still \{111\} ZB or \{0001\} WZ planes after pressure release, despite intervening transformation to \{100\} RS under pressure.

**DISCUSSION**

The combined experimental characterization and simulation indicate that mechanical compression can drive the formation of new microstructures of CdSe nanowires. The CdSe nanoparticles are surface-stabilized by organic monolayers, which initially prevent nanoparticles from aggregation or contact. At ambient pressure, ordered fcc nanoparticle arrays are formed through balanced nanocrystal interactions, such as van der Waals forces, hydrophobic attraction, and charge-charge interaction, leading to alkane chain interdigitation that locks in the final fcc nanocrystal mesophase (34). The ligand-coated nanoparticle assembly exhibits exponent force against external compression. An external pressure is necessary to overcome the force to induce nanoparticle coalescence (34). During compression at low pressure, pressure is not sufficient to break the force balance between adjacent nanoparticles. Hence, the spherical nanoparticle arrays remain in the fcc mesophase while shrinking and reversibly springing back when pressure is released, as verified by the SAXS results (Fig. 2, B to D). However, when the pressure is higher, continuous compression increasingly breaks the force balance between adjacent nanoparticles and drives the nanoparticles to contact and ultimately coalesce, forming nanowires. In the fcc mesophase, the closest interparticle spacings are between \{220\} planes. Consequently, particles touch and coalesce along the close packing \(<110>\) direction first, forming 1D nanowires. During this process, partial dissociation of ligands at the contact points of the nanocrystal surfaces likely occurs to allow particle connection (12). Because the starting CdSe nanocrystal arrays are in a solid-state phase that does not allow facile rearrangement of nanocrystals, this coalescence occurs in a random pattern within adjacent nanocrystals, leading to unusually twisted polycrystalline nanowires. The pressure-induced nanocrystal assembly mechanism discovered here may provide a flexible material synthesis route because morphology and architecture can be readily tuned to produce desired properties independent of crystal facet orientation.

In summary, we have developed a facile mechanical compression method to fabricate luminescent semiconductor nanowires from ordered spherical CdSe nanoparticle arrays. The fabrication process relies on pressure-induced phase transitions and coalescence from both atomic scale and mesoscale. The long-range ordering of starting nanoparticle assembly and its mesophase orientation are important for the formation of the nanowires. The fabrication method is simple and convenient to be implemented for large-scale synthesis of new nanostructures. The ease of the compressive process facilitates fundamental understanding of nanomaterial coupling through energy transfer. We envision that this method could apply to a variety of other semiconductor and magnetic materials (35, 36).

**MATERIALS AND METHODS**

**Synthesis of CdSe nanocrystals and ordered arrays**

Se powder (14.7 mmol, 1.16 g) was dissolved in 7.22 g of trioctylphosphine (TOP) to make a 1.70 M stock solution. CdO (1.88 mmol, 0.24 g), octadecylphosphonic acid (3.76 mmol, 1.26 g), and trioctylphosphonic oxide (31 mmol, 12 g) were loaded into a three-neck flask, degassed, and purged with Ar. The mixture was heated to 150°C and pumped for 1 hour before it was heated to 300°C under Ar. TOP (6 g) was injected into the flask, and the mixture was heated to 355°C. Se/TOP (1.72 ml) stock solution was quickly injected into the flask. The heating mantle was removed after 20 s to 3 min. In the case of 5-nm CdSe, 1-min reaction time was used. The reaction mixture was cooled to room temperature. The CdSe nanoparticles were precipitated using ethanol and centrifuged at 4000 rpm. The crude product was further purified by dissolving in a small amount of toluene and precipitated again. The CdSe nanoparticle solution was filtered through a 0.45-μm polycrystalline filter before use. CdSe (100 mg/ml) nanoparticle (with or without 20 weight % polystyrene) solution in toluene was drop-casted on Si wafers to form thin films of CdSe arrays. A small piece of film was peeled off and transferred to the DAC for high-pressure measurement.

**Characterizations**

TEM was performed on a JEOL 2010 TEM and an FEI Titan G2 80-200 STEM with a Cs probe corrector, operated at a 200-kV acceleration voltage. STEM images were recorded with a high-angle annular dark-field detector with the angular range of 60 to 150 mrad. SEM images were taken using a Hitachi S-5200 FEG microscope at 5 kV. Ultraviolet-visible (UV-vis) spectra were obtained on a Lambda 950 spectrophotometer (PerkinElmer). Photoluminescence (PL) spectra were measured using a Horiba FluoroMax-4 fluorometer with an excitation wavelength of 450 nm. In situ HP-SAXS and HP-WAXS measurements were performed on B1 station at Cornell High Energy Synchrotron Source (CHESS) with monochromatic x-ray radiation.
wavelength $\lambda$ of 0.485946 Å (37). The distance between the sample and a large-area MAR345 detector was 727.19 mm, as determined using both CoO$_2$ and silver behenate powder standard. Several small ruby chips were loaded into the DAC chamber as internal pressure probes to monitor the sample pressure by a standard pressure-dependent ruby fluorescence technique (38). Scattering images were calibrated and integrated using the Fit2D software.

**Simulation method**

The Cd-Se-Te analytical bond-order potential (BOP) (39) was used in the MD code LAMMPS (Large-scale Atomic/Molecular Massively Parallel Simulator) (40, 41) to perform the simulations. The BOP was tested with other simpler potentials (42), an additional advantage of the BOP is that it captures property trends of many metastable phases and defects. Time-averaged energies obtained from MD simulations were used to calculate surface energies. In addition to incorporating the finite temperature effects, time-averaged energies obtained from MD simulations were found, somewhat surprisingly, to be much more accurate than the static energies calculated from energy minimization simulations (43).

**SUPPLEMENTARY MATERIALS**

Supplementary material for this article is available at http://advances.sciencemag.org/cgi/content/full/3/5/e1602916/DC1

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Acknowledgments

Funding: This work is supported by the U.S. Department of Energy, Office of Basic Energy Sciences, Division of Materials Sciences and Engineering. CHESS is supported by the NSF and NIH/National Institute of General Medical Sciences via NSF award DMR-0225180. Research was carried out, in part, at the Center for Integrated Nanotechnologies, a US Department of Energy, Office of Basic Energy Sciences user facility. Sandia is a multimission laboratory operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy’s National Nuclear Security Administration under contract DE-AC04-94AL85000.

Author contributions: H.F. conceived the idea and designed the experiments. X.Z. performed the simulation and modeling. P.L. performed STEM characterizations. K.B. and B.L. performed statistical analysis of TEM images and optical characterizations. B.L., K.B., and Z.W. performed synchrotron experiments and analysis. M.S. performed PL imaging. All authors commented on the manuscript and contributed to the writing of the manuscript.

Competing interests: H.F. and B.L. are inventors on U.S. patents 9,180,420 B1 ("Tuning and synthesis of metallic nanostructures by mechanical compression," 10 November 2015) and 9,187,646 B1 ("Tuning and synthesis of semiconductor nanostructures by mechanical compression," 17 November 2015). All other authors declare that they have no competing interests.

Data and materials availability: All data needed to evaluate the conclusions in the paper are present in the paper and/or Supplementary Materials. Additional data available related to this paper may be requested from the authors.

Submitted 21 November 2016
Accepted 8 March 2017
Published 5 May 2017
10.1126/sciadv.1602916

Citation: B. Li, K. Bian, X. Zhou, P. Lu, S. Liu, I. Brener, M. Sinclair, T. Luk, H. Schunk, L. Alarid, P. G. Clem, Z. Wang, H. Fan, Pressure compression of CdSe nanoparticles into luminescent nanowires. Sci. Adv. 3, e1602916 (2017).