Synthesis of high-purity solid SiO_2 nanodumbbells via induced aggregation

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Abstract

Optically levitated nanodumbbells in vacuum are excellent candidates for thermodynamics, macroscopic quantum mechanics, precision measurements, and quantum sensing. Silica (SiO_2) material, with extremely low absorption of near-infrared light and rubust mechanical strength, has been the most potential material for this system. Here we synthesized high-purity solid SiO_2 nanodumbbells by introducing acetone for the induced aggregation of SiO_2 nanospheres. The nanodumbbells show high uniformity and the sizes are tunable. Previous experimental results demonstrated that the synthetic nanodumbbells can be applied in GHz nanomechanical rotors and can withstand the tensile strength of over 13 GPa. This work engineers a new material platform to advance levitated optomechanics.

Keywords: Silica (SiO_2), nanodumbbell, Stöber method, induced aggregation, levitated optomechanics

Introduction

Optically levitated nanodumbbells in vacuum are excellent candidates for torque sensing, rotational quantum mechanics, high-frequency gravitational wave detection, and multiple other applications [1-7]. However, large centrifugal force in high-speed rotation and the damage of photothermal effect require super tensile strength and extremely low absorption of light for the material. Silica (SiO_2), with low absorption of visible and near-infrared light and rubust mechanical strength, has been the most promising material in the field of optical levitation. Although the nanodumbbells can be synthesized by microemulsion method [8], wherein the surfactant molecules driving two nanospheres in a micelle and the nanodumbbell formed under depletion effects induced aggregation [9, 10]. Surfactants used to form microemulsions will introduce light-absorbing impurities into the SiO_2 and weaken the mechanical strength of the material [11-13], resulting in nanodumbbells being disassembled in our previous optical levitation experiments [4]. Stöber method, which does not use long-chain surfactants, is the most widely used wet chemistry synthetic approach to high-purity solid monodisperse SiO_2 nanospheres [14-16]. For the growth of nanodumbbells, induced aggregation of two nanospheres is the most important step. Ammonia was used to micro-sized SiO_2 particles, but it is ineffective on nanoparticles [8]. Therefore, it is of great significance to develop a method that can effectively aggregate SiO_2 nanospheres and achieve batch production of high-purity solid nanodumbbells.

Herein, by introducing acetone for the induced aggregation of SiO_2 nanospheres, we successfully synthesized the solid SiO_2 nanodumbbells via Stöber method. With a circularly polarized laser, the nanodumbbell can be drived to rotate beyond 1 GHz in high vacuum, which is the fastest nanomechanical rotor realized to date [4]. Remarkably, the ultimate tensile strength of the synthetic nanodumbbells exceeds 13 GPa, which is 2 orders larger than the bulk glass.

Experimental Section

Schematic Design. Fig. 1 shows the schematic illustration of the SiO_2 nanodumbbells synthetic processes. Solid SiO_2 nanospheres were synthesized via Stöber process (A), then acetone was added into the solution to induce aggregation of nanospheres (B). Next, a small amount of TEOS was added to grow silica shells under stirring (C). Finally, monodisperse nanodumbbells were obtained after the removal of nanospheres and other conglomerates by differential velocity centrifugation (D).

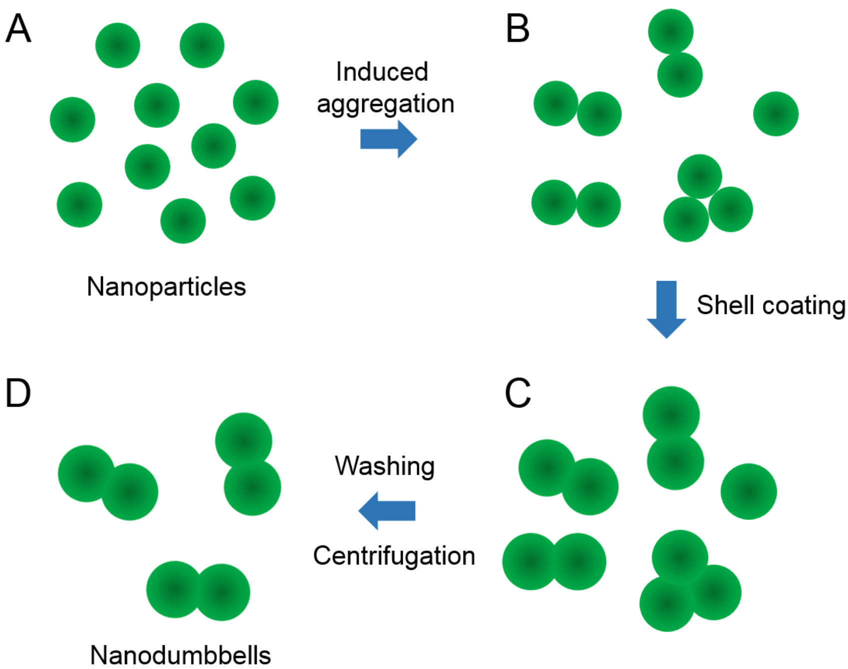

Fig. 1 Schematic illustration of SiO_2 nanodumbbells synthetic processes. (A) Solid SiO_2 nanospheres were synthesized via Stöber method. (B) Induced aggregation of the nanospheres by acetone. (C) Growing silica shells to enhance the mechanical strength of nanodumbbells. (D) Monodisperse nanodumbbells were obtained after the removal of nanospheres and other conglomerates.

Chemicals. All reagents are of the highest purity possible to avoid the introduction of impurities resulting in optical absorption. All lab glasswares are soaked in aqua regia for 24 hours and flushed with deionized water for 3 times. Tetraethyl orthosilicate (TEOS, 99.999%) was purchased from Aladdin reagent. Ammonia (26.5% w/w of NH_3 in H_2O), acetone (>99.9%) and ethanol (>99.9%) were all chromatographic grade and purchased from Sinopharm Chemical Reagent Ltd., China. Deionized water with a resistivity of 18.2 MΩ·cm (Milli-Q) was used for all rinsing processes. All reagents were used as received without further purification.

Nanodumbbell Synthesis. SiO_2 nanospheres with a diameter of 80 nm were synthesized by adding 2 mL of TEOS to a well-stirred mixture of ammonia (9.72 mL), deionized water (5.96 mL) and ethanol (200 mL). After stirring for 48 h, a colloidal solution of SiO_2 nanospheres with a diameter of about 80 nm was obtained. Then 20 mL of acetone was added into the colloidal solution and stirred for 24 h to induce aggregation, and 40 μL of TEOS was added under stirring for another 24 h to grow the silica shells. The silica shell can enable the nanodumbbells to have larger ultimate tensile strength [17, 18]. The precipitate of silica nanodumbbells with a diameter of 90 nm was obtained after washing with ethanol and centrifugation at 9000 r.p.m. for 10 minutes.

Purification of Nanodumbbells. For the requirement of optical levitation in high vacuum, the SiO_2 nanodumbbells must be high-purity to avoid the damage of photothermal effect. So the product was washed by anhydrous ethanol for seven times, and then transferred into a Teflon-lined stainless-steel autoclave (50 mL capacity) and heated at 90 °C for 12 h. Subsequently, the removal of nanospheres and other conglomerates will be carried out by differential velocity centrifugation [19-21]. After centrifuge the product at 1000 r.p.m. for 10 minutes, the supernatant was collected and centrifuged at 9000 r.p.m. for another 10 minutes. Furthermore, the product was centrifuged at 3000 r.p.m. for 5 minutes, the supernatant was collected and centrifuged at 6000 r.p.m. for another 5 minutes. Finally, the precipitate was dried under vacuum at room temperature overnight and the purified monodisperse SiO_2 nanodumbbells were produced.

Results and discussion

Based on above processes, batch production of high-purity solid SiO_2 nanodumbbells were synthesized successfully and Fig. 2 shows the SEM images of various growth stages. Fig. 2A shows the monodisperse solid SiO_2 nanospheres synthesized by Stöber method. For the requirement of super tensile strength of silica in optical levitation experiment, the SiO_2 nanospheres must be solid rather than mesoporous. Fig. 2B shows the aggregated nanoparticles induced by acetone, by now the nanospheres, nanodumbbells and other conglomerates are coexisting and the surfaces were not covered by shells. In order to further enhance the mechanical strength of nanodumbbells so that they will not be dismembered by large centrifugal force in high-speed rotation, an extra silica shell was coated with the previous aggregated nanoparticles (Fig. 2C). The diameter of silica cores will also be a little bit larger. Finally, after purification by washing and differential velocity centrifugation, monodisperse solid SiO_2 nanodumbbells were obtained (Fig. 2D).

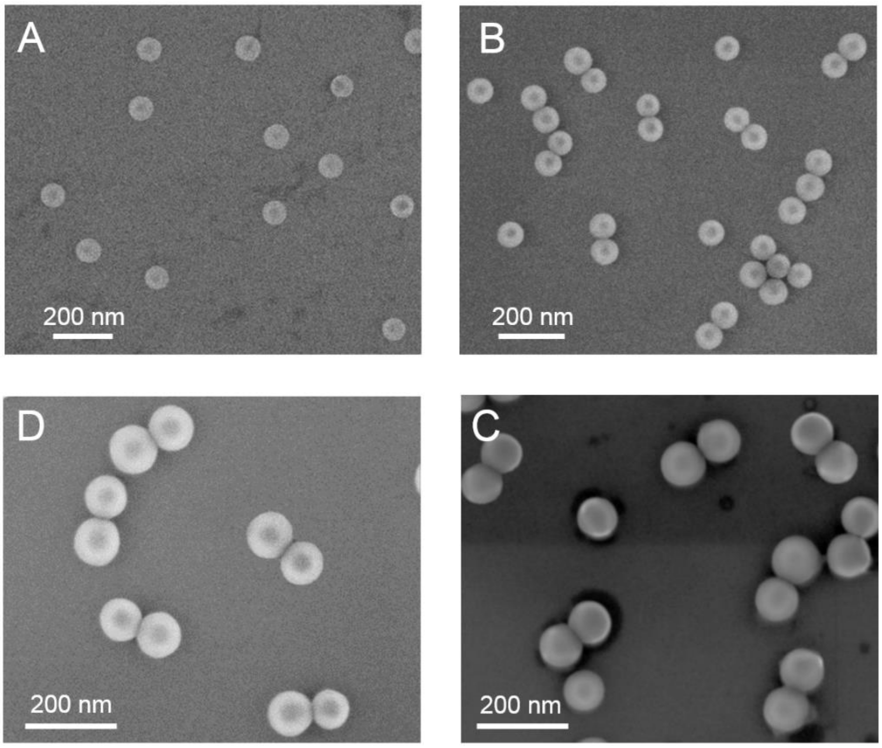

Fig. 2 SEM images of various growth stages. (A) monodisperse solid SiO_2 nanospheres. (B) The aggregated nanoparticles induced by acetone. (C) Extra silica shells were coated with the previous aggregated nanoparticles. (D) High-purity solid SiO_2 nanodumbbells.

The diameter of nanodumbbells is tunable via our method. As shown in Fig. 2, we synthesized various nanodumbbells with a diameter of 90 nm (Fig. 3A), 160nm (Fig. 3B) and 200nm (Fig. 3C) by aggregating nanospheres with different nanoparticle sizes. Remarkably, the calculated result prosed that the ultimate rotational speed of nanodumbbells under optical levitation would increase as the size of the nanodumbbells decrease [4]. The faster rotation of the nanorotor will be more conducive to quantum sensing and precision measurements.

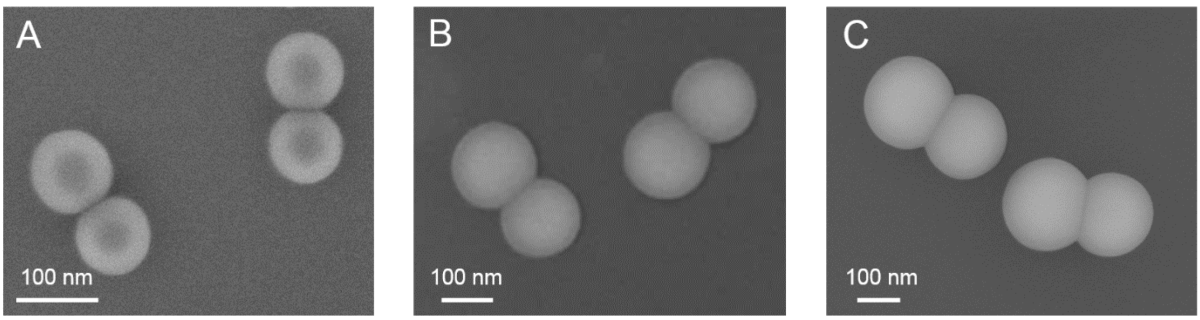

Fig. 3 SEM images of size-tunable nanodumbbells. Various nanodumbbells with a diameter of 90 nm (A), 160nm (B) and 200nm (C). These nanodumbbells were synthesized by similar process and aggregating nanospheres with different nanoparticle sizes.

Conclusions

Herein, batch production of high-purity solid SiO_2 nanodumbbells were synthesized via Stöber method by introducing acetone for the induced aggregation of SiO_2 nanospheres. By avoiding the long-chain surfactant, the synthetic nanodumbbells remain solid structure and extremely low absorption of light. Experimental results showed that our SiO_2 nanodumbbells can be drived to rotate beyond 1 GHz with a circularly polarized laser in high vacuum, the ultimate tensile strength of the synthetic nanodumbbells exceeds 13 GPa. This approach provides an excellent candidate material in optically levitated field for quantum sensing, precision measurements and other applications.

Data availability

All data are available from the corresponding author(s) upon reasonable request.

Acknowledgements: None.

Conflict of interest: The authors declare no competing financial interest.

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