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科学研究費助成事業　　研究成果報告書

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研究課題名（和文）High Performance Room-Temperature Thermoelectric Device using Colloidal Quantum Dot Superlattice

研究課題名（英文）High Performance Room-Temperature Thermoelectric Device using Colloidal Quantum Dot Superlattice

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研究成果の概要（和文）：本研究目的は高性能熱電材料用のコロイド量子ドット固体の開発。量子ドット固体では電気伝導率は熱伝導率から逸脱する可能性があることが予想されます。対処された課題は、秩序だった量子ドット組み立の形成制御であり、電気輸送を強化し、量子閉じ込め効果を維持し、電荷密度を調整します。量子ドット組み立の形態と電子特性の制御を行う新しい方法を確立しました。 高い電子移動度を達成するために、量子ドットを架橋する新しい機能性リガンド分子が発見されました。コア@シェル量子ドットを形成することにより、高い電荷担体ドーピング密度を実現しました。これらの調査結果は、量子ドットに基づく熱電材料を開発するための強力な基盤です。

研究成果の学術的意義や社会的意義
この研究の目的は、熱電用途の新しい材料を見つけることです。 量子ドットの使用に関する理論的予測は、15年以上にわたって提案されてきました。 ただし、材料の開発と処理の難しさ、および電荷キャリアの輸送についての理解の欠如により、実現するのは依然として困難です。 この研究の結果は、量子ドットを組み立て、熱電場に直接寄与する電子輸送特性を制御する方法の基礎を提供します。 新しいリガンド分子の発明、さまざまなタイプの量子ドットでの新しい動作、および材料処理の新しい方法は、さまざまな電子デバイス応用でのコロイド量子ドットのより大きな用途にも有益です。例えば太陽電池、光検出器、センサー、エネルギー貯蔵。

研究成果の概要（英文）：Nanostructuring was proposed to resolve the bottleneck in thermoelectric (TE) materials research. This project aims at developing colloidal quantum dot (CQD) solids for high-performance TE devices. In CQD solids, it is expected that the electrical conductivity can be decoupled from thermal conductivity, in addition to the prospect for enhanced Seebeck coefficient. The addressed challenges are the formation controls of well-ordered QD assemblies, enhance the corresponding electrical transport and to tune the charge density via various doping methods while preserving the quantum confinement effect. We established new processes to do on-demand controls of the QD assembly morphology and electronic properties. New functional ligands to crosslink QDs, to achieve high carrier mobility values, were discovered. High carrier doping density was realized by forming core@shell QDs that exclusively conduct electron. These findings are strong foundations to develop TE devices based on CQD solids.

研究分野：ナノ材料科学関連、応用物理

キーワード：colloidal quantum dots　thermoelectric　self-assembly　hybrid materials　carrier doping　charge transport　field-effect transistors　electrolyte gating

１．研究開始当初の背景

The challenges for developing thermoelectric materials are the complex relationship between the Seebeck coefficient, electrical conductivity and thermal conductivity that determine their *ZT* value. Improvement of one parameter usually negatively affects the other two. Nanostructuring is one of the most promising ways to develop superior thermoelectric materials [M. Dresselhaus, *Adv.Mater.*,2007]. It would enable us to decouple electrical conductivity from thermal conductivity. It was predicted that optimized nanostructured materials with a delta-like DOS might have a *ZT* value approaching 10 at room temperature [Humprey, *Phys.Rev.Lett.* 2005]. It has also been demonstrated that accessing the discrete energy level of QDs can modulate the thermopower and *ZT* value, even though it is still limited to single-QD devices at low temperature. The prospect of the utilization of CQDs for thermoelectric materials was first envisaged by theoretical studies. It was predicted that *ZT* values as high as 4 could be obtained in 5 nm diameter PbSe/PbS. So far, most efforts using QDs as thermoelectric materials have been limited to epitaxially grown materials, e.g. PbSeTe superlattice that demonstrated $ZT \sim 1.6$. As for the colloidal QDs, efforts have been made using Au QDs capped with $Sn_2S_6^{4-}$ Zintl ions and $Bi_{1-x}Sb_x$ alloy CQDs. Nevertheless, the *ZT* values were still low (<0.5) due to the low electronic conductivity, partly because of low carrier concentration. Furthermore, there is a severe limitation on the smallest feature that epitaxially-grown nanoparticle superlattices can achieve. The colloidal approach to form QDs is the only solution to fabricate features in the orders below 10 nm that are currently inaccessible, where the quantum confinement effect, beneficial for many applications including thermoelectric, will be significantly enhanced. The use of CQD for thermoelectric materials can be a breakthrough for the stagnation of research in this field with the condition that the charge conductivity in the QD assemblies is significantly improved.

２．研究の目的

This project aims to develop, study the physical properties and fabricate high-performance room-temperature thermoelectric generators based on hierarchical assemblies of solution-processable colloidal quantum dots. Among the critical questions that this project would address are: (i) how the formations, order, and dimensionalities of the quantum dot solids give rise to different electronic transport properties?; and (ii) how charge carriers will interact collectively, determining the transport behavior in the CQD assemblies under high carrier density that will give rise to new functionalities of the materials, including the potential for high-performance thermoelectric materials. Therefore, the utmost priority of this project is to thoroughly investigate the formation of well-ordered and well-controlled QD assemblies that can enable high electrical conductivities.

３．研究の方法

(1) Synthesis of the colloidal quantum dots

The first step performed in this project is to transfer the capability to synthesize high-quality PbS colloidal quantum dots to our group. The PbS QDs are synthesized using the hot-injection method, with some modifications in the precursor choices and the post-synthesis cleaning procedures. We obtained high-quality PbS QDs with high monodispersity and purity as indicated by the precisions in tuning the desired excitonic peak, the width of this excitonic peak as well as the peak-to-valley ratio, where the value of >3 became the norm. The multiple cleaning steps to wash the excess oleic acid from the solutions and the QD surface become the key to obtaining device-grade nanocrystals that can be used for transistors or other devices that rely on charge carrier transport. Later, some of these techniques and their modification were also used to discover several new kinds of QD compounds (*J. Phys. Chem. C* 126, 5323 (2022))

(2) Deposition of colloidal quantum dot assemblies

We performed all steps of device fabrications inside N_2-filled gloveboxes. We performed several different deposition methods to deposit the colloidal QD assemblies. We examine the formation of QD assemblies by three different deposition methods: (i) conventional layer-by-layer spin-coating, (ii) a layer-by-layer slow dip-coating method, and (iii) assembly at a liquid/air interface, which results in different assembly morphologies. For most assemblies, we aimed at having all of the native insulating ligands replaced by shorter ligands that will assist the charge carrier transport. However, in later development, we modified one of the assembly methods to control the morphology through selective ligand stripping, by which oriented attachment of the QDs can be achieved. For spin coating methods, we performed layer-by-layer deposition, where the ligand exchange process was done for each layer. In the case of dip-coating, we performed layer-by-layer deposition using a special dip coater that can perform dips and withdraw with very slow motion and supports multiple-barrel operation, allowing subsequent ligand exchange process. As for the assembly at the liquid/air interface, we adapted a process to create Langmuir films with modifications where the QDs can interact with the supporting liquid substrates to allow either the ligand exchange process or ligand stripping process. Thus, the QD assemblies can be transferred onto a solid substrate using a Langmuir-Schaffer-like process.

(3) Characterizations

Morphological characterizations of the QD assemblies were performed using various techniques, including atomic force microscopy (AFM) and transmission electron microscopy (TEM). Low-resolution TEM was used to evaluate the assemblies of the QDs on a large scale. On the other hand, high-resolution TEM was utilized for different investigations: the orientation of the QD facets in the assemblies, the short-range orders, as well as to prove the existence of core@shell structure via Moire patterns of the atomic lattices inside the QDs. Furthermore, we also utilized scanning TEM and selective-area electron diffraction (SAED) spectroscopy for structure determination. Several X-ray techniques were also utilized, including Grazing Incident Small-Angle X-ray Spectroscopy (GISAXS) and Grazing Incident Wide-Angle X-ray Spectroscopy (GIWAXS). In order to determine the interactions between the crosslinking ligands and the QDs in the assemblies, various characterizations were performed, which include solution and thin-film Vis-NIR optical absorption spectroscopies, Raman spectroscopy, FTIR spectroscopy, as well as NMR spectroscopy.

The QD assemblies' charge carrier transport properties were characterized using field-effect transistor (FET) and electric-double-layer (EDL) transistor platforms. All fabrication processes and the measurements of the transistors and the other test devices were performed inside N_2 gloveboxes. In this project, we constructed a system where a vacuum cryostat is directly connected to an N_2 glovebox so that any measured samples will not be exposed to oxygen or moisture that can immensely alter the material properties. This system allows us to perform reliable electrical transport measurements and the thermoelectric measurement of a planar device. We also performed electrochemical-related measurements, including electrochemical impedance spectroscopy, cyclic voltammetry, and spectroelectrochemical measurements. These measurements are essential to determine the doping density of the QDs.

４．研究成果

Among the significant research results are:

(1) Discovery of a new crosslinking ligand that supports high hole mobility in QD assemblies

We demonstrate controllable p-type transport in PbS QD assemblies with record-high hole mobility values. We obtained hole mobility of 0.06 $cm^2 \cdot V^{-1} \cdot s^{-1}$ in transistors with a solid gate, which is at least 2~3 orders of magnitudes higher than any solid gate PbS FETs, and as high as 0.2 $cm^2 \cdot V^{-1} \cdot s^{-1}$ in transistors with ionic-liquid gate. The achieved hole mobility values are getting closer to the same order of magnitude of electron mobility value in any state-of-the-art PbS QD transistors. The keys to the demonstrations reported are the proper selection of molecular ligands to crosslink the QDs and the solvent for those ligand exchange processes. These two are found to be vital to achieve p-type doping in the QD assemblies. We screened molecular ligands with carboxylate groups that generally caused the p-type doping of PbS QD films and optimized their attachment conditions. Among these carboxylate ligands, thiophene-2,5-dicarboxylic acid affords PbS QD films with exceptionally high hole mobility. Traditional approaches and a particular benefit of our unique approach to screening from a library can never design such a ligand. Furthermore, using a solvent with high solvency power to the ligands significantly enhances the coupling between the QDs. The findings have strong general implications for developing p-type colloidal QD assemblies and, more general, including the efforts to enhance n-type transport. Indeed, the use of our ligand demonstrated higher mobility than the use of the shorter ligands, indicating that the length of the crosslinking ligand molecules cannot solely prejudge the electronic coupling between QDs. Furthermore, the solvent effect on the ligand exchange process is found to be a widespread occurrence for any ligand molecules, including those for enhancing n-type transport. (Published as *ACS App. Nano Mater.*, 1, 5217 (2018))

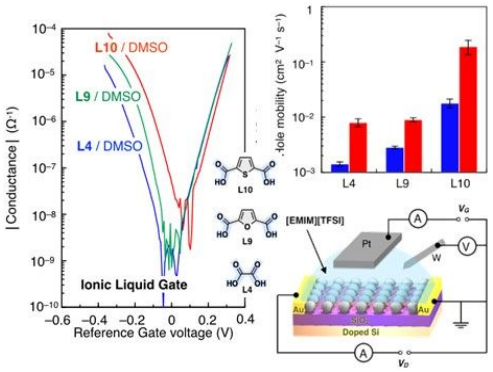

Figure 1. Comparison of I_D-V_G transfer curves of ionic-liquid-gated transistors of PbS QD with three crosslinking ligands, differ in size & molecular core, and the comparison of the corresponding hole mobility values obtained from both ionic-liquid-gated and solid oxide gate FETs.

(2) Controls of morphology and charge transport of QD solids by variations of assembly methods

We demonstrate the control of charge carrier transport and charge carrier accumulation in assemblies of highly crosslinked colloidal QDs with various degrees of order. Modifying three different methods to assemble and crosslink the QDs (i.e. spin coating, dip coating, and assembly at liquid/air interface assembly), three different classes of QD assembly morphologies are attained with distinctive electronic characteristics. Most importantly, the dip-coating process achieved hierarchical porous assemblies, while the assembly formed compact crosslinked superlattices at the liquid/air interface. For the first time, the charge carrier transport in crosslinked PbS colloidal QDs is directly compared systematically with different

kinds of assembly morphology resulting from different deposition methods. It owes to the capability to successfully demonstrate operational FETs with active holes and electron enhancement for all deposition methods on either solid or liquid gates. In particular, for depositing QDs using liquid/air interface assembly, almost none have been able to report working solid-gate transistors, especially with active electron enhancement. We demonstrate the highest areal capacitance of an EDL capacitor electrode that consists of a very thin assembly of materials, which, in its further development, will be suitable for practical supercapacitor electrodes.

In contrast, even though it accumulates the least carrier density than the others, the liquid/air interface assembled PbS QD film demonstrates EDL transistor performance with the highest electron mobility and record-high current modulation ratio (>10^5). The high carrier mobility signifies the importance of the film assembly's compactness and the superlattice's orders in determining the charge carrier transport in the planar direction, which will be vital for their uses for thermoelectric devices. The findings reported in this manuscript will have substantial general implications for developing colloidal QD solids and their practical uses for both energy harvesting and energy storage devices. In particular, discoveries and techniques in this manuscript will establish possibilities to create materials on-demand for diverse applications based on QDs as the building blocks. (Published as *NPG Asia Materials*, 12, 33 (2020)).

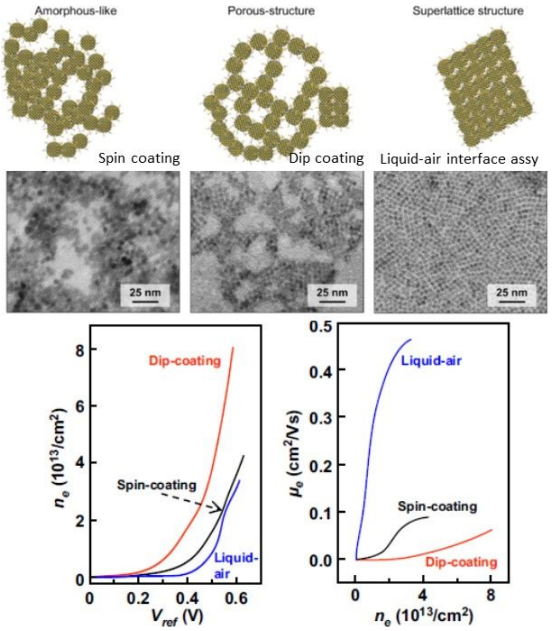

Figure 2. Comparison of the QD assembly morphologies that are deposited with three different methods. The difference of the morphologies lead to variations on the capability of accumulating charge carriers as well as the carrier-density dependent charge mobility values.

(3) Tuning the electronic transport properties of the QD solids by ligand coverage and oriented attachment controls

Inspired by the significant improvement of the charge carrier transport achieved by the QD assemblies fabricated using the liquid/air interface assembly technique, we demonstrate precise controls of ligand coverage by modifying this deposition technique, where ligand coverage degrees tune both the assembly structures and electronic properties. By controlling the ligand coverage degree of the QDs at the individual level, PbS QDs were assembled into different types of superstructures (random, square superlattice, and honeycomb superlattice) (Fig. 3). The formation of these superstructures was achieved via facet-selective ligand stripping via variations of supporting solvents (i.e., methanol, dimethyl sulfoxide, and acetonitrile) in a liquid/air interface assembly technique. The superstructures achieved were formed in a well-controllable manner and extended over μm^2 scale. Complete detailed structural analysis and formation mechanism of these controllable superlattices were thoroughly investigated by multiple approaches involving electron microscopy and diffraction, synchrotron-based grazing-incident small-angle x-ray scattering, and nuclear magnetic resonance spectroscopy. These three types of QD assemblies demonstrate different electronic transport properties depending on the degree of the ligand coverage. The QD assemblies with the medium coverage of ligand, which formed square superlattice of PbS QDs, displayed the best charge carrier transport properties with high electron mobility (> 0.2 cm^2/ V·s) and high transistor current modulation ratio (>10^5) that are among the record highest. It is because DMSO that has adequate solubility of oleic acid can mediate the formation of the atomically connected QD square lattice (oriented attachment). The different degrees of ligand coverage produced mainly insulating assemblies despite the extensive formation of the honeycomb-like superlattice since it is a double-layer structure of the hexagonal lattice. The oleic acid coverage control gives a new viewpoint to control self-assembly structures and electronic properties. The findings will

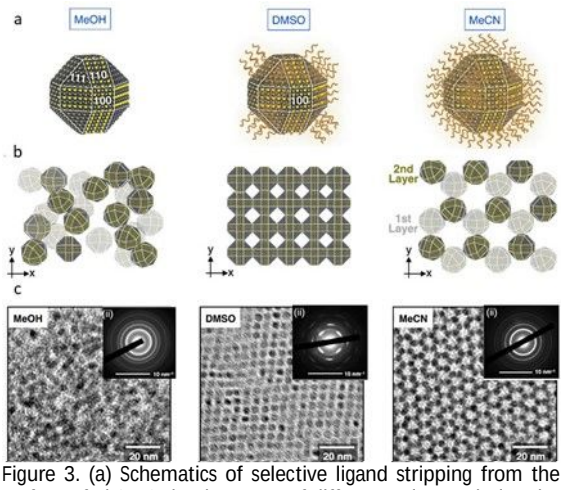

Figure 3. (a) Schematics of selective ligand stripping from the surface of PbS NCs by the usage of different solvents during the assembly process. (b) Schematics of the formed assemblies, and (c) the corresponding transmission electron microscopy images. (insets) the selected area electron diffraction (SAED) images.

have strong general implications for developing QD superlattices and understanding their electronic properties. In particular, it will establish possibilities to discover many exotic emerging electronic and optical properties, as predicted by many theoretical studies. Furthermore, the strategy for ligand coverage controlled assemblies can be used as a strategy template to control the coverage of any other types of ligands. In the end, we demonstrated the possibility of achieving band-like transport in colloidal QD superlattices and achieving band-filling by field-induced doping. It is in our greater interest to control the assembly that supports high electrical conductivity. At the same time, keep suppressing the corresponding thermal conductivity low and preserving the quantum confinement nature for their applications as thermoelectric materials. (Published as *Nanoscale*, 11, 20467, (2019); Nanoscale 13, 14001 (2021); and Nature Communications 14, 2670 (2023)).

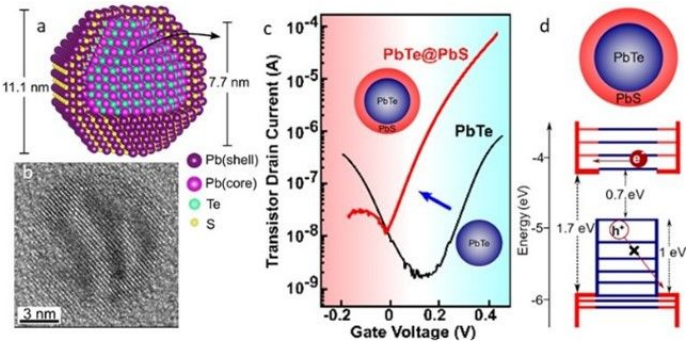

Figure 4. (a) A schematic of a PbTe@PbS core@shell nanocrystals, and (b) the corresponding high-resolution TEM image. The moiré stripe pattern arises from the interference by the PbS and PbTe lattice at the NC core. (c) The comparison of I_D–V_{ref} transfer characteristics of EDLTs using PbTe@PbS NCs (red) and PbTe core-only NCs (black), indicating that shelling significantly modifies the carrier transport. (d) Energy diagram of the core@shell NC.

(4) Shelling as a doping method for exclusive electron transport in lead-chalcogenide quantum dots

We demonstrate exclusive electron transport in the assemblies of core@shell PbTe@PbS colloidal NCs. Shelling the narrower bandgap PbTe with PbS shell is shown to be an effective strategy to dope narrow-bandgap CQDs to become exclusive electron-transporting with complete suppression of hole transport due to the formation of type-II heterojunction core@shell NCs. The demonstration of exclusive electron transport in this core@shell NCs is beneficial for their applications in devices or device components where ambipolar characteristics of the materials should be actively suppressed, such as in thermoelectric and electron-transporting layers in light-emitting diodes or solar cells. Typically, colloidal semiconductor NC assemblies demonstrate ambipolar characteristics where holes and electrons can be transferred from one NC to another. The current state-of-the-art ligand modifications, surface treatments, and doping can shift the position of the valence, conduction, and Fermi levels of the NCs relative to the others to make them have roles as either p-type materials or n-type materials in particular devices. However, those modifications do not alter the NC properties as ambipolar material. In FET measurements, the influence of those modifications will be manifested only by threshold voltage shifts of holes and electron accumulations. This research demonstrates exclusive electron transport in NC assemblies for the first time. The exclusive electron transport is defined by the complete suppression, due to localization, of hole transport, in addition to the enhancement of electron conductivity. Thus, PbTe@PbS core@shell NC is a purely n-type material with no possibility of hole transport. Electron conductivity as high as 9 S/cm and high electron mobility (> 1 cm^2/V.s) presented in this PbTe@PbS core@shell NC system is among the highest in the colloidal semiconductor NC field. Transistor measurements (solid-gate FETs and EDLTs) demonstrate the exclusive electron transport in large-scale assemblies. From the transistor characteristics, we can understand that the doping density by the shelling of the PbTe NC can reach 10^{13}/cm^2, corresponding to about two additional electrons/NC. It is the highest electron doping density ever reported in a colloidal NC system by chemical means on the surface of the NC. The use of electric double-layer transistors reveals that hole transport cannot be achieved in this PbTe@PbS core@shell system, despite the enormous amount of hole accumulation by the field-induced doping of the EDLT due to its capability to scan Fermi level much wider than solid-state gating. These proofs show that the wider-gap PbS shelling on the narrower-gap PbTe NC electron dopes the NC and simultaneously localizes the hole strongly by tighter confinement. The findings will have general implications for developing various colloidal NCs, particularly in designing other new core@shell NC systems beneficial for stabilizing or doping the core NCs. Furthermore, the findings will also be impactful for their potential practical uses for both energy harvesting devices where electron transport with a complete absence of hole transport is necessary, such as in solar cells and thermoelectrics. (Published as *ACS Nano*, 14, 3242 (2020) and *Applied Physics Letters*, 117, 173101 (2020))

To achieve the conceptual thermoelectric materials proposed by this project, we will still need to combine several other chemical doping techniques and shelling. So far, chemical doping is still unable to match the potential that field-induced doping might have promised in achieving conditions where the discrete electronic states of the QDs can be half-filled. All the findings obtained by this project will become solid foundations for further explorations to achieve high-performance thermoelectric materials.

５．主な発表論文等

〔雑誌論文〕　計9件（うち査読付論文　9件／うち国際共著　9件／うちオープンアクセス　2件）

１．著者名 Liu Liming、Bisri Satria Zulkarnaen、Ishida Yasuhiro、Hashizume Daisuke、Aida Takuzo、Iwasa Yoshihiro	４．巻 1
２．論文標題 Ligand and Solvent Effects on Hole Transport in Colloidal Quantum Dot Assemblies for Electronic Devices	５．発行年 2018年
３．雑誌名 ACS Applied Nano Materials	６．最初と最後の頁 5217～5225
掲載論文のDOI（デジタルオブジェクト識別子） 10.1021/acsanm.8b01205	査読の有無 有
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１．著者名 Liu Liming、Bisri Satria Zulkarnaen、Ishida Yasuhiro、Aida Takuzo、Iwasa Yoshihiro	４．巻 11
２．論文標題 Tunable electronic properties by ligand coverage control in PbS nanocrystal assemblies	５．発行年 2019年
３．雑誌名 Nanoscale	６．最初と最後の頁 20467～20474
掲載論文のDOI（デジタルオブジェクト識別子） 10.1039/C9NR07101F	査読の有無 有
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１．著者名 Miranti Retno、Shin Daiki、Septianto Ricky Dwi、Ibanez Maria、Kovalenko Maksym V.、Matsushita Nobuhiro、Iwasa Yoshihiro、Bisri Satria Zulkarnaen	４．巻 14
２．論文標題 Exclusive Electron Transport in Core@Shell PbTe@PbS Colloidal Semiconductor Nanocrystal Assemblies	５．発行年 2020年
３．雑誌名 ACS Nano	６．最初と最後の頁 3242～3250
掲載論文のDOI（デジタルオブジェクト識別子） 10.1021/acsnano.9b08687	査読の有無 有
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１．著者名 Septianto Ricky Dwi、Liu Liming、Iskandar Ferry、Matsushita Nobuhiro、Iwasa Yoshihiro、Bisri Satria Zulkarnaen	４．巻 12
２．論文標題 On-demand tuning of charge accumulation and carrier mobility in quantum dot solids for electron transport and energy storage devices	５．発行年 2020年
３．雑誌名 NPG Asia Materials	６．最初と最後の頁 33
掲載論文のDOI（デジタルオブジェクト識別子） 10.1038/s41427-020-0215-x	査読の有無 有
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1．著者名	4．巻
Miranti Retno、Septianto Ricky Dwi、Ib??ez Maria、Kovalenko Maksym V.、Matsushita Nobuhiro、Iwasa Yoshihiro、Bisri Satria Zulkarnaen	117
2．論文標題	5．発行年
Electron transport in iodide-capped core@shell PbTe@PbS colloidal nanocrystal solids	2020年
3．雑誌名	6．最初と最後の頁
Applied Physics Letters	173101
掲載論文のDOI（デジタルオブジェクト識別子）	査読の有無
10.1063/5.0025965	有
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2．論文標題	5．発行年
Carbon-Based Quantum Dots for Supercapacitors: Recent Advances and Future Challenges	2021年
3．雑誌名	6．最初と最後の頁
Nanomaterials	91～91
掲載論文のDOI（デジタルオブジェクト識別子）	査読の有無
10.3390/nano11010091	有
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1．著者名	4．巻
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2．論文標題	5．発行年
Evidence of band filling in PbS colloidal quantum dot square superstructures	2021年
3．雑誌名	6．最初と最後の頁
Nanoscale	14001～14007
掲載論文のDOI（デジタルオブジェクト識別子）	査読の有無
10.1039/d0nr09189h	有
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Miranti Retno、Septianto Ricky Dwi、Kikitsu Tomoka、Hashizume Daisuke、Matsushita Nobuhiro、Iwasa Yoshihiro、Bisri Satria Zulkarnaen	126
2．論文標題	5．発行年
π-SnS Colloidal Nanocrystals with Size-Dependent Band Gaps	2022年
3．雑誌名	6．最初と最後の頁
The Journal of Physical Chemistry C	5323～5332
掲載論文のDOI（デジタルオブジェクト識別子）	査読の有無
10.1021/acs.jpcc.2c00266	有
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1．著者名 Septianto Ricky Dwi、Miranti Retno、Kikitsu Tomoka、Hikima Takaaki、Hashizume Daisuke、Matsushita Nobuhiro、Iwasa Yoshihiro、Bisri Satria Zulkarnaen	4．巻 14
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〔産業財産権〕

〔その他〕

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６．研究組織

	氏名 （ローマ字氏名） （研究者番号）	所属研究機関・部局・職 （機関番号）	備考

７．科研費を使用して開催した国際研究集会

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８．本研究に関連して実施した国際共同研究の実施状況

共同研究相手国	相手方研究機関			
スイス	ETH Zurich	EMPA		
オーストリア	IST Austria			
インドネシア	Institut Teknologi Bandung			