Nanomaterials

Room Mauka - Session NM-WeP

Nanomaterials Poster Session

NM-WeP1 Quantum Confinement Effect and Photocatalytic Properties of Cu₄SnS₄ and Cu₄SnS₄-Au Quantum Dots, Wenchao Liu, Nanjing Tech University, China

We developed an improved hydrothermal method with water-oil twophase reaction system to synthesize size-controllable and oil-soluble Cu₄SnS₄ (CTS) quantum dots (QDs). The formation and growth mechanism of CTS QDs was proved to proceed via three steps: nucleation of $C_{31}S_{16}$ nanoparticles, diffusion of $Sn^{4\ast}$ into $Cu_{31}S_{16}$ nanoparticles and formation of regular hexagonal nanoprisms via Ostwald ripening. The water-oil interface plays an important role in controlling nuclei process and growth speed of CTS QDs. The average size and size distribution of CTS QDs can be controlled efficiently through adjusting the solvent ratio of toluene/water. Nearly monodisperse CTS QDs can be synthesized when the toluene volume is larger than 30 mL. More importantly, this improved hydrothermal method is a general and low cost method to synthesize not only chalcogenide based QDs but also other complex compound QDs. The blue-shift of absorption edge and broadening of Raman bands were observed due to the quantum confinement effect. The exciton Bohr radius of CTS QDs was calculated to be 3.3-5.8 nm by using the first principle calculation. We further use the CTS quantum dots as seeds to produce CTS-Au heterostructured nanoparticles. Such heterostructured nanoparticles are shown to have excellent photocatalytic properties.

NM-WeP2 Magnetic Strain Imaging of a Main Pole Tip and an Upper Shield in a Hard Disk Drive, Kenta Butsugan, Kansai University, Japan

Hard disk drives (HDDs) play an important role as large-scale information storages. Currently, in perpendicular recording, the main pole is keenly confined to apply magnetic fields to a finite portion. Therefore, remanent magnetization tends to appear at the pole tip. The magnetic fields produced by the remanent magnetization erase data recorded on magnetic media. The head-induced data erasure, especially erase-after-write, is a serious problem for the single-pole writer. We observed a head to reveal the erase-after-write phenomenon using magnetic strain imaging.

Scanning probe microscopes (SPMs) can sensitively detect changes in the gap between the tip and sample, with high in-plane resolutions. Strain imaging, based upon the excellent

capabilities of SPMs, detects changes in the gap induced by the modulation of a physical property to be imaged, and images the modulated property of the sample together with its topography. Strain imaging was applied to observe the piezoelectric/ferroelectric properties of samples, and then magnetic observation was presented.

When a magnetic sample is subjected to a magnetic field, strains are generated in the sample. Because the strains involve factors that depend on the magnetization of each domain, we can observe the magnetic domain structures by imaging the strains via an SPM. We pointed out magnetostriction, magnetic forces, and electromagnetic interaction between the magnetization and eddy currents as the causes of the strains. Any magnetic probe is not required with this method, because domain structures are imaged by the detection of surface displacements. The applied magnetic fields are well-estimated and precisely controllable, while the stray fields from the magnetic probes used in magnetic force microscopy are not controllable and not easy to estimate.

Our observed samples were heads having the main pole tips of 60-nm and 30-nm width. One of them was multilayer pole tip. We observed magnetic imaging of the tips with respect to the write coil currents. We present these results.

NM-WeP3 Electrical and Optical Properties of Barium Titanate Nanoparticles Contained in Mesoporous Silicate Thin Film, Atsushi Kohno, T. Tajiri, Fukuoka University, Japan

Integration of size-controlled nanoparticles into thin films is attractive for applications to functional materials and devices owing to their unique properties related to the reduced size. We have synthesized the nanoparticles of dielectric materials in the one-dimensional pores of mesoporous silicate SBA-15 thin films formed on silicon and quartz substrates and have investigated the characteristics of nanoparticles. Ferroelectric nanoparticles are attractive for memory application especially as ultra-small scale devices, and the ferroelectricity of nanoparticle is

interesting from the viewpoint of materials sciences. In this research we focus on the nanoparticles of perovskite-type ferroelectric materials. The synthesis of barium titanate (BaTiO₃) nanoparticles in the pores of SBA-15 thin film were developed, and the electrical and optical properties of the nanoparticles have been investigated.

Mesoporous silicate SBA-15 has been synthesized using Ethylene oxide/propylene oxide/ethylene oxide triblock copolymer and tetraethylorthosilicate. The mixture solution was coated on p-Si(100) and quartz substrates by spin-coating, and then the coated samples were dried and sintered. The SBA-15 thin film samples were dipped in BaTiO₃ precursor solution. The SBA-15 films which contained BaTiO₃ precursor solution in the pores were sintered in a furnace at 700 °C for crystallization. In the case of electrical measurement Au electrodes were made on the films.

X-ray diffraction showed that the one-dimensional pores in SBA-15 thin film were aligned parallel to the surface and stacked keeping well-ordered hexagonal symmetry. The pore diameter was estimated to be about 5 nm from the analyses of X-ray diffraction and reflectivity, as detail of the evaluation method was reported elsewhere. It was confirmed that the stacked pore structure was never affected by the synthesis of BaTiO₃ nanoparticles. The elemental composition and chemical states of BaTiO₃ nanoparticles in the thin films were checked by XPS measurement as compared with the bulk material. The optical absorption of BaTiO₃ nanoparticles was clearly observed and the absorption edge is estimated to be about 3.7 eV which is much larger than the bandgap of the bulk material.

The clockwise hysteresis was clearly observed in the capacitance-voltage characteristics of Au/BaTiO₃-containing SBA-15/p-Si capacitor and the hysteresis voltage width was increased and saturated as increased the applied voltage. These results suggest that the hysteresis was caused by ferroelectricity of BaTiO₃ nanoparticles. The properties of the BaTiO₃ nanoparticles will be discussed in the conference.

NM-WeP4 Structural Analysis of Hydroxyapatite Nanoparticles by Rietveld Refinement and Electron Diffraction, Magdalena Méndez-González, ESFM-National Polytechnic Institute of México; G.M. González, UPIBI-National Polytechnic Institute of México; M.M. Méndez, ESFM-National Polytechnic Institute of México

It was studied by Rietveld refinement and electron diffraction structural behavior of hydroxyapatite nano-particles, synthesized by chemical precipitation under normal temperature and pressure. Rietveld refinement using the data applied to XRD structural parameters of the nano-particles, its crystal size and the crystalline phases were obtained. Electron diffraction semi spherical and slightly elongated morphology, defined in relation to time and aging temperature used in the synthesis, distinguishing normal hexagonal prism morphology observed in natural hydroxyapatite crystals micrometer size was observed.

NM-WeP5 Fe₃O₄@Zn₃K₂[Fe(CN)₆]₂ for Cesium Removal from Radioactive. Aqueous Waste Solutions: A Mössbauer and XPS Study, Arely Cano, CICATA Legaria, National Polytechnic Institute, México; A.A. Lemus-Santana, CICATA Legaria, National Polytechnic Institute, Mexico; J. Tóth, L. Kövér, Institute for Nuclear Research (ATOMKI), Hungarian Academy of Sciences, Hungary; E. Reguera, CICATA Legaria, National Polytechnic Institute, México

Zn₃A₂[Fe(CN)₆]·xH₂O with A= Na⁺, K⁺, Rb⁺ shows a high ability for Cs⁺ removal from aqueous solutions by ionic exchange. These solids have a porous framework formed by six large ellipsoidal cavities per hexagonal unit cell with dimensions close to 15.5x11.1x7.9 Å. The neighboring cavities remain connected by elliptical windows of about 6.8x8Å. Within a given cavity two charge balance cations A⁺ are found. These cations are located close to N corners of the ZnN_4 tetrahedron [1]. For Cs^+ the ionic exchange is highly favorable due of its large volume [2], the Cs⁺ ion is found interacting with two N neighboring atoms. This explains that when Zn₃A₂[Fe(CN)₆]₂·xH₂O is immersed in an aqueous solution containing Cs⁺, the ionic exchange is quantitative. On the other hand, the iron oxides nanoparticles have attracted attention as simple, efficient and economical materials for pollutant removal, including heavy metals. Their physicochemical properties as large surface areas, surface reactivity and strong adsorption capability can be modified and enhanced to obtain effective adsorbents materials. In addition, the iron oxide behaves as ferrimagnetic or ferromagnetic material; such property can be used to allow the separation process using an applied magnetic field. This is the case of the system under study in this contribution. In this study, paramagnetic magnetite nanoparticles were synthesized and covered with

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a thin shell of Zn₃A₂[Fe(CN)₆]₂, then the shell was progressively growing at the surface. The nanocomposi tes obtained were evaluated for Cs⁺ removal from aqueous solutions. For evaluation, the nanocomposites were prepared with different iron oxide particles sizes and Zn₃A₂[Fe(CN)₆] thin shell . The structural study was carried out from XRD powder patterns and Electron Microscopy data.

The interactions at the interface between the core of magnetite nanoparticles and the zinc hexacyanoferrate shell were studied by X-ray Photoelectron Spectroscopy (XPS) and Mössbauer spectroscopy. The nanocomposites studied were prepared by two different successive adsorption cycles of shell growth, $Fe_3O_4@[Fe(CN)_6]^{4-}$ and Zn^{2+} . The quantitative analysis by XPS allows determine the shell thickness using an appropriate model and supported with Mössbauer data which allows the identification of species formed and their interactions at the interface.

NM-WeP6 Subwavelength Color Printing with Wide Viewing Angle, HuiJoon Park, Ajou University, Republic of Korea

Color filter has been considered as an essential component of various display technologies such as LCDs and OLEDs, because the quality of image and resolution of those displays are significantly affected by the performance of color filters. Existing color filter systems, however, utilize colorant pigments or dyes, whose properties are easily affected by numerous factors including constant UV illumination, longstanding heat exposure and moisture, thus causing the performance degradation of display devices in a relatively short period of time. Furthermore, their low stability with respect to diverse processing chemicals and complicated patterning processes for creating individual pixels are also the biggest concern of the traditional color filter systems.

As an alternative to the present color-generating systems, a structural color filter scheme, which exploits a physical interaction of light with nano- and micro-structures (*e.g.* photonic and plasmonic resonances), has been widely investigated due to their potential advantages such as ultra-compactness, long-standing stability and simple manufacturability as compared to the conventional color filters.

Particularly, with the growing popularity of high-definition devices, the ability of attaining high spatial resolution by tightly localizing the light energy into neighboring gaps at the deep subwavelength scale, thus being able to create color pixels beyond the diffraction limit, has been another key feature of the structural color filter system. Therefore, in recent years, with the rapid developments of large-scale nanofabrication techniques such as nanoimprint lithography, colloidal self-assembly and laser interference lithography, substantial amount of attention has been given to the various nanostructure-based color filtering systems, which could provide unique and distinct possibilities for enabling a color printing with sub-diffraction resolution. However, there have been great difficulties in retaining optical resonances at the same wavelength over a wide angular range and simultaneously creating vivid full colors in a pixel unit through a one-step process.

Here, an angle-insensitive structural color filtering scheme based on strong interference effects in ultrathin subwavelength semiconductor gratings on metallic substrates is introduced. The proposed color filter devices, which are fabricated over a large area using nanoimprint lithography, produce distinctive colors that are easily tuned by altering a width of the nanostructured gratings even with the fixed thickness of the structure, thus allowing all the individual color pixels to be patterned via one-step fabrication.

NM-WeP7 Ultrasonic Assisted Preparation of Liposomes using Supercritical Carbon Dioxide, *Chiho Uemori*, Nagoya University, Japan; *W. Diono*, Nagoya University; *T. Kon*, Shalom Co.; *H. Kanda*, Nagoya University; *M. Goto*, Nagoya University, Japan

A liposome is a spherical vesicle having single or multiple lipid bilayers. Liposomes are generally composed of phospholipids. Lipophilic or hydrophilic compounds can be encapsulated in liposomes either in the phospholipid bilayer or in the inner aqueous phase. Liposomes are prepared conventionally by the Bangham method, the detergent depletion method, the reverse phase evaporation method, and emulsion method. Recently, supercritical carbon dioxide has been applied to produce liposome instead of organic solvent.

In this work, liposomes were produced utilizing supercritical fluid technology with ultrasonic mixing. Since no organic solvent was used in this process, solvent-free liposomes were prepared. As a lipid, we used sphingomyelin or phosphatidylcholine. Phospholipids and water were charged in high pressure vessel. Then, pressurized carbon dioxide was

pumped into the vessel. The vessel was placed in thermostatted water bath and applied ultrasonic power to mix phospholipids, water, and carbon dioxide phase in order to produce liposome structure. The obtained liposomes were identified as multi-lamella form by TEM analysis. The effects of operation parameters such as ultrasonic frequency, pressure, and temperature, particle size of liposomes were studied.

NM-WeP8 Method development and Determination of Trace Metal Impurities in Silicon Based Nanopowder in Solvent Mixture by Inductively Coupled Plasma Mass Spectrometry, Mohsina Islam, S. Liu, Chemtrace Analytical Testing and Solutions

In Semiconductor industry, the silicon based nanoparticles showed great promises in various applications like thin- film transistors for liquid crystal displays (LCD TFTS), solar cells and printed semiconductor materials because of its superior properties such as feasibility of surface functionalization, size dependent multicolor light emission and stability against photobleaching.

In this work a novel analytical method was developed for the determination of trace metal impurities in silicon based nanoparticles dispersed in solvent mixture using Inductively Coupled Plasma Mass Spectrometry (ICP-MS). For this purpose silicon nanoparticles with metal dopants, e.g, phosphorous and boron dopant nanoparticles dispersed in various solvents like Isopropyl alcohol, PGMEA and Terpineol were studied. The sample was prepared by evaporation to solvent boiling point followed by Silicon dissolved and digestion with mixed acid solution. The trace metal (TM) residue was dissolved and reconstituted in dilute acid solution which is analysed through ICP-MS. Following the same method, a spiked sample was prepared with known TM level in the range of 3.0 to 6.0 ppb. The spike recoveries with this method is in the range of 75 to 125% for all elements. The dopant levels were measured as 0.05-0.3 wt.% for B and P following the dissolution and dilution of sample in mixed acid solution. The method detection limit as low as 0.1 to 10 ppb were achieved for different TM contaminants.

NM-WeP9 Optical and Morphological Characterization of ZnSe Nanoparticles Processed by Laser Ablation in Liquid, *Patricia Maldonado-Altamirano*, *R.Y. Ponce-Cano*, *L.A. Martinez-Ara*, ESFM-IPN, México; *M.A. Hernandez-Perez*, ESIQIE-IPN, México; *J.R. Aguilar-Hernandez*, ESFM-IPN, México

In this work we present results and analysis concerning the processing of semiconducting ZnSe nanoparticles obtained by laser ablation of diluted ZnSe powder in acetone. A Nd-YAG pulsed laser was used for ablation, tuned at the first harmonic, 1064 nm, 50 Hz frequency repetition during 60 minutes. The experiment was performed as a function of the laser power intensity. UV-Vis and Raman spectroscopies were used to characterize the ZnSe particles, whereas scanning electron microscopy, transmission electron microscopy and X ray diffraction were used to determine the morphology and size of the particles. According to the UV-Vis results it was confirmed a shift of the band gap towards high energy (blue shift) from 2.7 to 3.3 eV. Raman spectroscopy show the LO phonon at 250 cm⁻¹ and also the so called surface mode at 230 cm⁻¹, for the case of the smaller particles, approximately 10 nm, which was estimated through SEM and TEM images. The cristalline planes were also observed in the TEM images. X-Ray diffractograms confirmed that the particles show the wurtzite structure. A deep analysis of the results is presented and discussed.

NM-WeP10 New Method Of Synthesis Of Graphene From Cvd In Steady State, *Alejandra Moreno-Barcenas*, Centro de Investigacion y de Estudios Avanzados Del Intituto Politecnico Nacional, Mexico; *J.F. Perez-Robles, A. Garcia-Garcia*, Centro de Investigacion y De Estudios Avanzados Del Intituto Politecnico Nacional, Mexico; *L.M. Avilez-Arellano*, CINVESTAV Querétaro México, Mexico; *Y.V. Vorobiev*, CINVESTAV-Unidad Queretaro, Mexico

The Graphene is a material used in a wide range of applications due to its unique structure and excellent electronic, optical, mechanical and thermal properties. Currently, several techniques are being developed to obtain graphene, one of this techniques that has highlighted which chemical vapor deposition (CVD) technique is the most reported. The fastest progress in graphene methodology in recent years have been obtained by this way. In this sense, the aim of the investigation in graphene is to increase the graphene production by CVD with high quality and at low cost. However, usually to produce graphene, it is necessary to use high vacuum equipment and temperatures about 1800 °C, which represents a very high cost. In this paper a new method is proposed to obtaining monolayer graphene with good quality and low cost operation. Using acetylene and nitrogen as precursors by CVD is possible to obtained monolayer graphene by CVD, instead of methane and argon. Also a mix of nitrogen:hydrogen (90:10,

respectively) was used instead pure hydrogen. All the components are subjected to temperatures about 1000 °C and atmospheric pressure, on polycrystalline copper foil. The synthesis of the graphene is carried out in steady state, reducing the synthesis time around a minute. The amount of gas flow is determined taking into account the reactor volume per unit mass. The presence of graphene is shown by transmission electronic microscopy and Raman spectroscopy confirmed the monolayer of graphene.

NM-WeP13 High Temperature Shift Reaction without Pre-reduction over Metal Substituted Spinel Ferrite Catalysts prepared by Assisted Sol-Gel Method, *Dae-Woon Jeong*, Changwon National University, Republic of Korea

The purpose of this study was to prepare directly the magnetite of spinel ferrite catalysts using glycine assisted sol-gel combustion method and to compare the catalytic activity of Ni, Co, Mn, Mg, and Zn substituted spinel ferrite catalysts for the high temperature shift reaction. Selected metal ions (Ni, Co, Mn, Mg, and Zn) were introduced into iron oxide (spinel lattice) and the resulting materials screened for catalytic performance for the high temperature shift reaction using waste-derived synthesis gas. The active phase of spinel ferrite catalysts was directly prepared by the glycine assisted sol-gel combustion method under a reducing atmosphere. Among the prepared nano catalysts, NiFe2O4 catalyst exhibited the highest CO conversion (X_{CO} =81%) with long term stability even at a very high gas hourly space velocity of 40,057h⁻¹. This result was mainly due to the inverse spinel structure and easier reducibility of NiFe2O4 catalyst. The properties of metal ferrite spinel have been analyzed by Brunauer-Emmett-Teller (BET) method, X-raydiffraction (XRD), and temperature programmed reduction (TPR).

NM-WeP14 Temperature Dependence of the PTFE Surface Morphology Irradiated by an Ar⁺ Ion Beam, Yuki Yamashita, I. Takano, Kogakuin University, Japan

Recently, the application of polytetrafluoroethylene (PTFE) spreads to various fields. PTFE is a macromolecular material that has the repetition of two fluorine atoms combined to a carbon atom. Because these atoms are strongly combined, PTFE has variously excellent characteristics such as a heat-resistant, an electrical insulation, a chemical stability, a low friction, etc. The significant characteristic among them is the high water-repellency which is well known as the coating of a cookware. The improvement of the water-repellency by modifying the PTFE surface morphology has been carried out by using the ion beam or the plasma treatment. In this case, the morphological change of the PTFE surface is caused by the heat and sputtering.

In the present study, the temperature dependence of the PTFE surface morphology irradiated by an Ar⁺ ion beam was investigated. Two kinds of PTFE were used. They are the adhesive tape type (T-PTFE; Nitto Denko Corporation.) and the sheet type (N-PTFE; NICHIAS Corporation). The irradiated ion species were Ar⁺ ions with 10 kV in an acceleration voltage at 40 μ A/cm² in a current density. The temperature of PTFE substrates was controlled by the both of liquid nitrogen cooling and the heater heating, and was kept with -50, -25, 0, 25 and 50 degrees. The surface morphology was observed by the laser microscope (OLS4500 OLYMPUS) and the SEM (JSM-5310LV JEOL). The modified surface was evaluated about the water-repellency which was measured by the water-contact-angle meter (Kyowa Interface Science Co, Ltd.) using the $\theta/2$ method. The surface chemical state was investigated using X-ray photoelectron spectroscopy (SHIMADZU Co.Ltd.).

In the same condition of the ion beam irradiation, the T-PTFE surface morphology changed by the T-PTFE substrate temperature. In the case of -50 degrees the surface change was hardly observed, while the needle-like structure was clearly observed in the substrate temperature of 50 degrees. It was considered that the surface morphology was strongly influenced by the substrate temperature than by sputtering of the ion beam. In addition, the atomic concentration measured by the X-ray photoelectron spectroscopy showed that fluorine atoms decreased and carbon atoms increased with a lower temperature, because the preferential sputtering was caused. On the other hand, oxygen atoms increased with a lower temperature because free bonds generated by the removed fluorine atoms from T-PTFE absorbed oxygen atoms in the atmosphere.

It was confirmed that the change of the PTFE substrate temperature in ion irradiation influenced the surface morphology and the atomic content.

NM-WeP15 Large-Area Multi-layer Hexagonal Boron Nitride, SooMin Kim, Korea Institute of Science and Technology, Korea Chemtrace Analytical Testing and Solutions

NM-WeP19 Photocatalytic CO₂ Reduction on Particulate Metal-ion-doped CeO₂, *Hideto Furuno*, *N. Saito*, *K. Sato*, *M. Nishikawa*, Nagaoka University of Technology, Japan

1. Introduction

In recent years, the issue of global warming caused by a greenhouse effect gas such as CO_2 has been becoming deeply. Fossil fuels are exhaustible resource, and CO_2 is released into the atmosphere by their combustion. In order to solve these problems, artificial photosynthesis using photocatalyst is one of promising approach. We have been studied photocatalytic overall water splitting into H_2 and O_2 on CeO_2 , and showed that proper metal-ion-doped CeO_2 has a sufficient ability to split water by UV irradiation.^[1] Although hydrogen is expected as the clean energy source, some problems such as storage and transport remains to be solved. On the other hand, CH_4 has attracted much interest as useful fuel because of their conventional chemical properties. In this study, we payed attention to convert CO_2 to CH_4 by artificial photosynthesis on Pt-loaded Sm-doped CeO_2 photocatalyst.

2. Experimental

Sm-doped CeO₂ was prepared by heating of Sm₂O₃ and CeO₂ mixtures at 1373 K for 16 h in air. Pt was deposited on the as-prepared Sm-doped CeO₂ surface by photodeposition method as a co-catalyst. Prepared Pt-loaded Sm-doped CeO₂ was characterized by X-ray diffraction (XRD) and field emission scanning electron microscope (FE-SEM). Photocatalytic CO₂ reduction was carried out by a hand-made closed gas circulation apparatus directly connected to a gas-chromatograph equipped with BID detector. Photocatalyst was dispersed in distilled water and irradiated by a UV-lamp. CO₂ gas was filled with vessel before light irradiation.

3. Results and discussion

In XRD measurements, the as-prepared Sm-doped CeO₂ showed welldefined fluorite crystal structure with significant peak shift corresponding to Sm doping into their lattice. SEM images indicated that Pt was highly dispersed on CeO₂ surface after photodeposition. As products for photocatalytic reaction, CH₄ and H₂ were detected without producing CO. It is interesting that CO that is a major product for CO₂ reduction was not detected. It is well known fact that Pt has an ability to adsorb CO strongly. These results indicate that Pt-loading plays an important role for CO₂ reduction.

4. Conclusions

Photocatalytic CO₂ reduction on Pt-loaded Sm-doped CeO₂ was investigated. Pt-loaded Sm-doped CeO₂ showed selective CH₄ production. This results indicate that metal-ion-doped CeO₂ become a suitable material for photocatalytic CO₂ reduction, and the combination between metal-ion-doped CeO₂ and co-catalyst will be an important factor for selective CH₄ production.

References

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NM-WeP20 Effects of Surface Acid Treatment on Mg-doped GaN for Photocatalytic Overall Water Splitting, *Takahiro Inoue*, *N. Saito*, *M. Nishikawa*, Nagaoka University of Technology, Japan

1. Introduction

 $\rm H_2$ has received attention as a next-generation energy alternative to fossil fuels. In the past four decades, photocatalysts have payed attention to energy conversion materials for water splitting into H₂ and O₂. Recently, we have discovered that Mg-doped GaN has an ability to decompose water when depositing RuO₂. ^[1] However, synthesized as-prepared Mg-doped GaN has an impurity phase such as MgO, and showed unstable activities at an initial stage of reaction, suggesting that an unstable layer soluble in water exists on as-prepared Mg-doped GaN. Therefore, it is necessary to remove them for improving the activity of photocatalytic water splitting. In this study, we investigated changes in photocatalytic activities by surface acid treatment.

2. Experimental

A precursor of Mg-doped GaN was obtained by co-precipitation method using $Ga(NO_3)_3$ and $Mg(NO_3)_2$ in alkaline solution. The precursor was heated at 1273 K for 15 h under NH₃ atmosphere. The obtained Mg-doped GaN was treated by stirring in 0.1 M HCl solution. RuO_2 was deposited by

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impregnation method in range from 1 to 5 wt% as a promoter. Photocatalytic water splitting was performed by closed gas circulation apparatus. A 300 W Xe lamp was used for photo-irradiation.

3. Results and discussion

As an impurity phase, an imperceptible peak for MgO was detected on asprepared Mg-doped GaN. MgO was completely removed by surface acid treatment. For photocatalytic water splitting, RuO₂-loaded as-prepared Mg-doped GaN evolved H₂ and O₂ with N₂ at an initial stage of reaction, then N₂ generation disappeared at the several runs of reaction, indicating that the outermost surface of the as-prepared Mg-doped GaN was dissolved in water by photo-irradiation. On the other hand, acid treatment gave stable and high activities for H₂ and O₂ evolutions compared to that of the asprepared Mg-doped GaN without N₂ production. The optimum amount of RuO₂ on Mg-doped GaN changed from 3.5wt% to 2.0wt% after acid treatment.

4. Conclusions

Effects of acid treatment on Mg-doped GaN for photocatalytic water splitting were investigated. This work indicates that removal of surface impurity phase and unstable layer on Mg-doped GaN is promising approach.

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