様　式　Ｃ－１９、Ｆ－１９－１、Ｚ－１９　（共通）

科学研究費助成事業　　研究成果報告書

令和　５年　６月２０日現在

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研究種目：若手研究

研究期間：2018～2022

課題番号：１８Ｋ１４１１９

研究課題名（和文）高感度単原子磁性検出法の開発

研究課題名（英文）Development of highly sensitive single-atom magnetic detection method

研究代表者

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交付決定額（研究期間全体）：（直接経費）　3,200,000円

研究成果の概要（和文）：私たちはTEMにおける電子渦巻きビームの作成に関する理論理解を深めました。フォーク型アパーチャーを使用して電子渦巻きを生成しました。TEMに位相アパーチャーを追加するには、電子光学を修正する必要があり、大きな予算が必要です。しかし、このプロジェクトのサポートを受けて、ドーピング、インターカレーション、相転移、超格子構造など、二次元材料の様々な側面を研究しました。過去5年間で、Nature、Nature Materials、Advanced Materials、ACS Nano、Nano Lettersなどの高影響力のジャーナルに論文34本を発表しました。これらの成果はこの資金援助のおかげです。

研究成果の学術的意義や社会的意義

The research on two-dimensional materials and electron vortex beams has potential social impacts in a variety of fields. The development of new materials with unique properties can lead to the creation of new technologies and products that improve the quality of life for people.

研究成果の概要（英文）：We have deepened our understanding of the theory behind creating an electron vortex beam in TEM. Fork-type apertures were fabricated to generate vortex electrons. Adding a phase aperture to TEM would require modifications to the electron optics and a larger budget to accomplish. Nevertheless, with the support of this project, we have studied various aspects of two-dimensional materials such as doping, intercalation, phase transformation, and superlattice structure. Over the past 5 years, we have published 34 papers, including articles in high-impact journals such as Nature, Nature Materials, Advanced Materials, ACS Nano, and Nano Letters, thanks to the support of this funding.

研究分野：材料・化学領域

キーワード：電子顕微鏡　EELS　2D materials

１．研究開始当初の背景

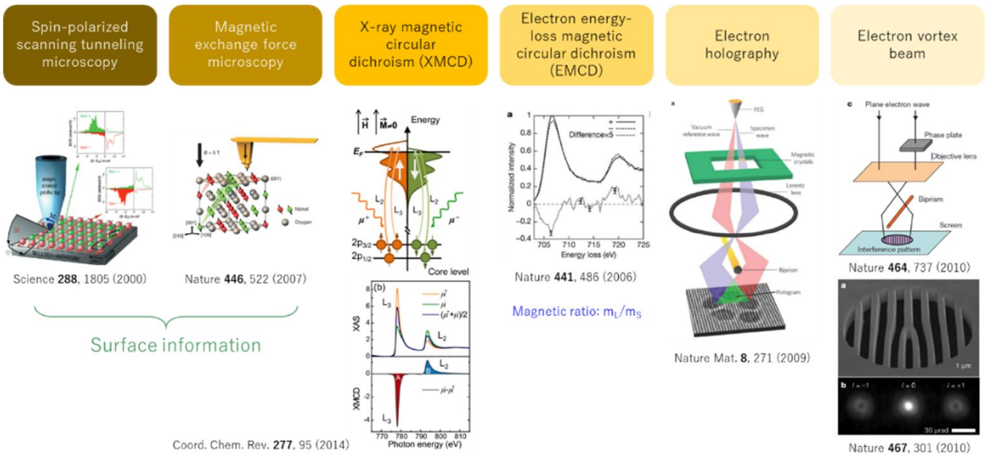
Information storage is of utmost importance in the current era of high-speed calculations and information explosion. Transition metals (TM) exhibiting ferromagnetism play a critical role in storing digital data by utilizing the orientation of their magnetic moments. However, the ongoing challenge in the manufacturing industry lies in the constant pursuit of shrinking the size of one-bit information storage to increase memory capacity.

To address this challenge, a proposed method for maximizing storage device capacity involves harnessing the potential of single TM atoms as individual storage units. This approach necessitates a comprehensive understanding of the magnetic properties exhibited by single atoms and nanoclusters. These magnetic properties primarily originate from the d-orbital electrons and bonding configurations of TM atoms.

To gain insights into the surface magnetic information of materials, techniques such as spin-polarized scanning tunneling microscopy [Science 288, 1805, (2000)] or magnetic exchange force microscopy [Nature 446, 522 (2007)] can be employed [Figure 1]. Additionally, recent advancements have introduced valuable techniques such as x-ray magnetic circular dichroism (XMDC) [Coord. Chem. Rev. 277, 95 (2014)] and electron magnetic circular dichroism (EMCD) [Nature 441, 486 (2006)]. By exploiting the selection rule, where electrons excited by different angular momenta exhibit different intensities in the spin up and spin down electron transitions from the 2p orbital to the 3d orbital, these techniques provide detailed information about the electronic and magnetic structures of nanoparticles, molecular materials, and single-molecule magnets.

Furthermore, the manipulation of electron beams through the electron holography technique [Nature Materials 8, 271 (2009), Nature, 464, 737 (2010), Nature 467, 301 (2010)] enables the generation of electrons carrying orbital angular momentum. This innovative approach finds broad applications in analyzing the local magnetic properties of nanomaterials. Leveraging the generation of electron vortex beams in scanning transmission electron microscopy (STEM) represents a powerful means to investigate the magnetic properties of single TM atoms and clusters.

By expanding our knowledge in these areas and harnessing advanced characterization techniques, we can pave the way for groundbreaking advancements in information storage, magnetic physics, and material science. This research has the potential to attract significant attention from various fields and lay the foundation for next-generation quantum storage devices and high-speed calculation units.



Figures 1. Methods to study the magnetic information of materials.

２．研究の目的

The primary objective of this project is to generate an electron vortex beam in a scanning transmission electron microscope (STEM) and utilize these vortex electrons to investigate the spin state of magnetic particles and single TM atoms through the electron energy loss spectroscopy (EELS) technique. By employing this advanced technology, we aim to develop a comprehensive understanding of the magnetic behavior exhibited by individual TM atoms.

This study is expected to yield high-impact experimental findings that will serve as a crucial cornerstone in the fields of atomic physics, magnetic physics, spintronics, and material science.

３．研究の方法

To achieve the generation of vortex electrons, our project begins by designing fork-type holograms. These holograms possess distinct geometries that enable the generation of electron beams with varying levels of angular momentum (refer to Figure 2). Our research delves into the intricacies of holographic reconstruction theory, allowing us to design the most suitable patterns for creating a holographic aperture that seamlessly integrates with our microscope setup.

By employing advanced computational techniques and simulation tools, we optimize the design of the holographic aperture to precisely control the phase and amplitude of the electron wavefront. This ensures the efficient conversion of a conventional electron beam into a vortex beam with well-defined orbital angular momentum. Through careful calibration and experimental refinement, we strive to achieve high-fidelity generation of vortex electrons, enabling us to explore their unique properties and application potentials. In addition to the holographic aperture design, we investigate the integration of the aperture within the scanning transmission electron microscope (STEM) system. This involves meticulous alignment and alignment adjustments to ensure optimal performance and reliable operation of the vortex electron generation. We also analyze the effects of aberrations and other imaging parameters to enhance the quality and stability of the generated vortex beams. The successful implementation of the designed holographic aperture in the STEM system opens up exciting possibilities for studying the spin state of magnetic particles and single TM atoms. By coupling the vortex electrons with electron energy loss spectroscopy (EELS), we can probe the magnetic properties and spin dynamics of these nanoscale systems with exceptional precision and sensitivity.

In the sample preparation phase, our first step involves isolating transition metal (TM) atoms within graphene. Graphene serves as an excellent platform for housing TM atoms due to its lack of inherent magnetism while providing a highly anisotropic environment. By utilizing electron energy loss spectroscopy (EELS) without the implementation of vortex beams, we initially investigate the spin state of TM atoms in graphene. This enables us to gain insights into the spin characteristics of TM atoms within the graphene lattice and explore their relationships with neighboring atoms. Subsequently, we introduce the holographic aperture into the microscope setup. This aperture plays a crucial role in generating vortex electrons with controlled orbital angular momentum. Through careful analysis of the vortex electron pattern, we fine-tune the size and positioning of the selective aperture. This iterative process allows us to achieve atomic resolution imaging while simultaneously obtaining EELS signals. By precisely adjusting the holographic aperture, we optimize the conditions for both high-resolution imaging and EELS analysis. This innovative approach enables us to obtain detailed structural information at the atomic scale while simultaneously probing the elemental composition and electronic states of the TM atoms within the graphene lattice. Our goal is to achieve atomic-level resolution imaging, which will provide crucial insights into the arrangement and interactions of TM atoms with their surrounding environment. Simultaneously, the complementary EELS signals obtained will contribute to our understanding of the electronic properties and chemical bonding involving the TM atoms in graphene. Through this comprehensive experimental strategy, we aim to unravel the intricate spin states, electronic properties, and local interactions of TM atoms within the graphene matrix. This research not only advances our knowledge of TM-graphene systems but also has broader implications for diverse fields such as material science, condensed matter physics, and nanotechnology.

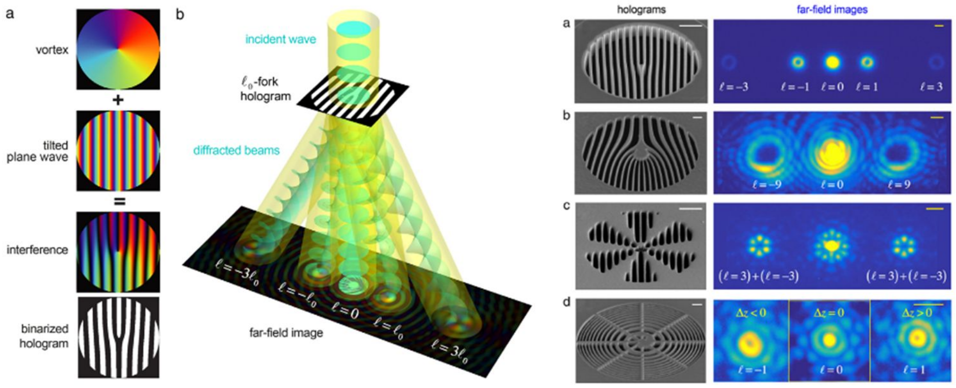


Figure 2. The relation of holograms and the generated pattern of the vortex electrons.

4．研究成果

We manufacture fork-type holographic apertures by depositing a 200 nm thick Pt thin film onto the surface of a single crystal NaCl substrate. The Pt film is carefully transferred to fully cover a 200 µm hole in a commercially available transmission electron microscopy (TEM) aperture. Subsequently, a holographic pattern is created on the Pt film using focused ion beam (FIB) etching techniques (see Figure 3).

The placement of the holographic aperture within the electron path of the microscope necessitates careful consideration of electron optics. Essentially, there are three different design configurations (as depicted in Figure 4):

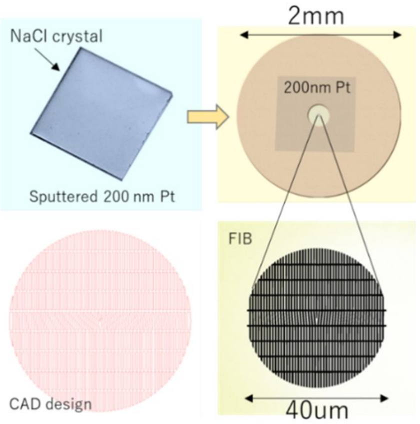


Figure 3. Design and fabrication of holographic aperture.

(a) Parallel Beam Configuration: In this design, a parallel electron beam is used to irradiate the sample in the TEM mode. To generate the vortex beam, a holographic aperture is placed above the sample. The electron beam passes through the holographic aperture, acquiring orbital angular momentum and transforming into a vortex beam. Subsequently, a selective aperture is employed to filter and collect the desired electron signals for EMCD analysis.

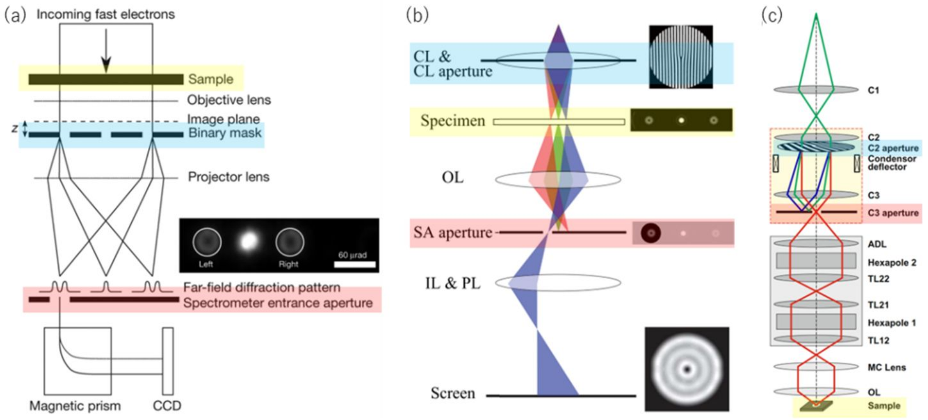


Figure 4. Three design configurations and modification methods for TEM electron optics that allow for the generation of electron vortex beams and the collection of EMCD signals.

(b) Convergent Beam Configuration I: A holographic aperture is placed at the top of the microscope, generating a vortex beam that is directed onto the specimen. Additionally, a selective aperture is utilized in front of the spectrometer to further control and filter the electron signals based on their angular momentum. This combined approach provides flexibility in tailoring the electron beam characteristics and collecting EMCD signals with specific angular momenta for accurate characterization of the sample's magnetic and electronic properties.

(c) Convergent Beam Configuration II: This design involves modifying the convergence aperture system of the TEM. A holographic C2 aperture is introduced into the system, typically positioned after the condenser lens. This aperture is responsible for generating the vortex beam by imparting orbital angular momentum to the electrons. Below the C3 condenser lens, a selective C3 aperture is added to filter the vortex electrons, allowing for the selection of specific angular momenta. The resulting vortex beam, with the desired angular momentum, is directed onto the sample for EMCD measurements.

By employing these different design configurations, we can tailor the electron beam characteristics to suit the specific experimental requirements. Each configuration offers unique advantages for studying the magnetic properties and spin states of magnetic particles and single TM atoms. The precise control of the electron beam's angular momentum through holographic apertures allows for detailed investigations of the interaction between electron spins and the magnetic structures of the specimens.

In this project, we have planned to implement design (c) in order to purify the irradiating electrons with specific angular momentum and apply them directly to single TM atoms. However, we recognize that modifying the aperture design poses significant challenges. It would involve altering the height of the microscope and potentially impacting the electron optics. This modification carries a high risk as it could degrade the current performance of single atom imaging and spectroscopy.

Considering these factors, we have realized that such a modification would require a substantial budget and it would be more prudent to initially attempt it on a spare microscope. Despite these constraints, our understanding of the electron vortex beam and EMCD experiments has significantly deepened. This knowledge will be valuable for future endeavors and may eventually pave the way for successful implementation of the desired modifications.

Throughout the course of this project, we have conducted extensive investigations into the spin states of individual Cr and Fe atoms in graphene. Our findings have revealed that when absorbed on graphene edges and divacancies (DV), Cr and Fe atoms exhibit a high-spin state. Additionally, we have observed that the spin state of the TM atom is influenced by its neighboring atoms. For instance, the spin state of a single Fe atom at a DV can transition from a high spin state to a low spin state if neighboring nitrogen atoms are present instead of carbon atoms (refer to Figure 5). These observations highlight the sensitivity of the TM atom's spin state to its local environment within the graphene lattice.

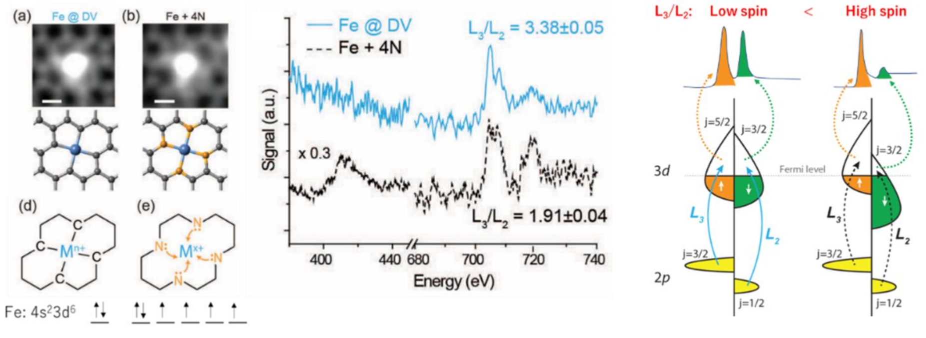


Figure 5. Spin state variation of Fe in graphene divacancy and in nitrogen defect.

During the duration of this project, additional experiments and research publications were carried out to investigate the properties of various other 2D materials. These investigations aimed to expand our understanding of the different 2D systems. As a result, the project has yielded a significant number of research outputs, with over 30 papers published on this topic. These publications have contributed valuable insights and findings to the field of atomic structures and physics, advancing our knowledge of nanoelectronic devices, material science, and the broader scientific community. The wide range of research conducted and the substantial number of publications demonstrate the comprehensive nature of the project and its impact on the scientific community.

５．主な発表論文等

〔雑誌論文〕　計30件（うち査読付論文　30件／うち国際共著　23件／うちオープンアクセス　5件）

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２．論文標題 Tunable Doping of Rhenium and Vanadium into Transition Metal Dichalcogenides for Two‐Dimensional Electronics	５．発行年 2021年
３．雑誌名 Advanced Science	６．最初と最後の頁 2004438～2004438
掲載論文のDOI（デジタルオブジェクト識別子） 10.1002/advs.202004438	査読の有無 有
オープンアクセス オープンアクセスではない、又はオープンアクセスが困難	国際共著 －

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３．雑誌名 Chemistry of Materials	６．最初と最後の頁 7301～7308
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３．雑誌名 Nanomaterials	６．最初と最後の頁 3218～3218
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2．論文標題 Embedment of Multiple Transition Metal Impurities into WS2 Monolayer for Bandstructure Modulation	5．発行年 2021年
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２．論文標題 Seamlessly Splicing Metallic SnxMo1-xS2 at MoS2 Edge for Enhanced Photoelectrocatalytic Performance in Microreactor	５．発行年 2020年
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掲載論文のDOI（デジタルオブジェクト識別子） 10.1002/adma.202003484	査読の有無 有
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１．著者名 Kim Gwangwoo、Ma Kyung Yeol、Park Minsu、Kim Minsu、Jeon Jonghyuk、Song Jinouk、Barrios-Vargas Jose Eduardo、Sato Yuta、Lin Yung-Chang、Suenaga Kazu、Roche Stephan、Yoo Seunghyup、Sohn Byeong-Hyeok、Jeon Seokwoo、Shin Hyeon Suk	４．巻 11
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３．雑誌名 Nature Communications	６．最初と最後の頁 5359
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１．著者名 Lee Gun-Do、Robertson Alex W.、Lee Sungwoo、Lin Yung-Chang、Oh Jeong-Wook、Park Hwanyeol、Joo Young-Chang、Yoon Euijoon、Suenaga Kazu、Warner Jamie H.、Ewels Christopher P.	４．巻 6
２．論文標題 Direct observation and catalytic role of mediator atom in 2D materials	５．発行年 2020年
３．雑誌名 Science Advances	６．最初と最後の頁 eaba4942
掲載論文のDOI（デジタルオブジェクト識別子） 10.1126/sciadv.aba4942	査読の有無 有
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２．論文標題 Isothermal Growth and Stacking Evolution in Highly Uniform Bernal-Stacked Bilayer Graphene	５．発行年 2020年
３．雑誌名 ACS Nano	６．最初と最後の頁 6834～6844
掲載論文のDOI（デジタルオブジェクト識別子） 10.1021/acsnano.0c00645	査読の有無 有
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１．著者名 Lin Yung-Chang、Ji Hyun Goo、Chang Li-Jen、Chang Yao-Pang、Liu Zheng、Lee Gun-Do、Chiu Po-Wen、Ago Hiroki、Suenaga Kazu	４．巻 14
２．論文標題 Scanning Moire Fringe Method: A Superior Approach to Perceive Defects, Interfaces, and Distortion in 2D Materials	５．発行年 2020年
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オープンアクセス オープンアクセスではない、又はオープンアクセスが困難	国際共著 該当する

１．著者名 Tsai Tsung-Han、Liang Zheng-Yong、Lin Yung-Chang、Wang Cheng-Chieh、Lin Kuang-I、Suenaga Kazu、Chiu Po-Wen	４．巻 14
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〔図書〕　計0件

〔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

〔国際研究集会〕　計0件

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