

主辦單位: 台灣顯微鏡學會 協辦單位:

國立台灣大學 材料所 工業技術研究院 材料與化工研究所



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第四十一屆臺灣顯微鏡學會年會

110 年 12 月 11 日(星期六) 國立臺灣大學工學院綜合大樓國際演講廳

08:30-09:30	報到
09:30-10:00	開幕
	理事長致詞與會務報告
	會員大會
10:00-11:00	特邀演講(I) Untold cryo-EM stories behind the 2021 Nobel Prize of Physiology or Medicine
	2021 諾貝爾生理醫學獎的幕後功臣冷凍電子顯微鏡的故事 中央研究院化學研究所 章為皓 副研究員/合聘副教授
11:00-11:30	顯微鏡技術推廣(I) JEOL IT-800 系列 FESEM 介紹 捷東公司 應用部經理 洪英傑先生
11:30-12:00	台灣顯微鏡學會年會會員大會 Poster Session (地點:一樓大廳)
12:00-14:00	午餐時間 (備有午餐餐盒) Poster Session (地點:205,207,209,228 教室)
14:00-15:00	特邀演講(II) Computational Microscopy: Metallurgical Renaissance Inspired by Atom Probe Tomography 計算顯微術: 三維原子針尖斷層分析所掀起的冶金文藝復興 台灣大學材料科學與工程學系 顏鴻威 副教授
15:00-15:30	顯微鏡技術推廣(II) 讓材料說話—掌握先進製程的分析技術 関康科技 葛裕逢 經理
15:30-16:00	討論交流時間(一樓大廳備有點心茶點)
16:00-16:30	顯微鏡技術推廣(III) Spectra Ultra- The ultimate S/TEM for advanced semiconductor R&D Dr. Dong Tang, TEM Product Marketing Manager, Thermo Fisher Scientific
16:30-17:00	頒獎、閉幕

台灣顯微鏡學會

第十九屆理、監事當選名單

榮譽理事	蘇紘儀	儀祥投資有限公司	博士
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	楊哲人	國立臺灣大學材料科學與工程學系	教授
	李志浩	國立清華大學工程與系統科學系	教授
	劉康庭	友聯光學股份有限公司	董事長
	謝詠芬	閎康科技股份有限公司	董事長
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	溫政彥	國立臺灣大學材料科學與工程學系	教授
	張立	國立交通大學材料科學與工程學系	教授
	闕郁倫	國立清華大學材料與工程學系	教授
	陳金富	捷東股份有限公司	總經理
	薛景中	國立臺灣材料科學與工程學系	教授
	吳文偉	國立交通大學材料與工程學系	教授
	陳詩芸	國立臺灣科技大學材料科學與工程學系	教授
	林新智	國立臺灣大學材料科學與工程學系	教授
	李威志	E. A. Fischione Instruments	博士
侯補理事	陳福榮	香港城市大學材料科學與工程學系	教授
	章為皓	中央研究院化學研究所	副研究員
	陳俊顯	臺灣大學貴儀中心	主任
	謝達斌	科技部	政務次長
	劉全璞	國立成功大學材料科學與工程系	教授
	黃榮潭	國立臺灣海洋大學材料工程研究所	教授
	陳香君	國立臺灣大學生命科學系	教授
監事	鮑忠興	宜特科技股份有限公司	博士
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	陳志遠	國立臺北科技大學智慧財產權研究所	教授
候補監事	吴尚蓉	國立成功大學口腔醫學研究所	教授
	呂明諺	國立清華大學材料與工程學系	教授

110年年會特邀演講來賓

章為皓 副研究員/合聘副教授 Prof. Wei-hau Chang

演講題目:

Untold cryo-EM stories behind the 2021 Nobel Prize of

Physiology or Medicine

2021 諾貝爾生理醫學獎的幕後功臣--冷凍電子顯微鏡的故事現職:

中央研究院 化學所 副研究員 中央研究院 物理所 合聘副研究員 中央研究院 基因體中心 合聘副研究員 國立台灣大學 化工系 合聘副教授



中央研究院 化學所 副研究員 2012/01-中央研究院 化學所 助研究員 2003/10-2012/01 美國史丹福大學 博士後研究員 2001/02 至 2003/09

學歷:

史丹福大學 生物物理 博士 1993/09 至 2001/01 加州大學 生物物理 碩士 1992/08 至 1993/06 國立台灣大學 物理學系 學士 1985/09 至 1989/06



Abstract:

The Nobel Prize in Physiology or Medicine 2021 was awarded jointly to David Julius of UCSF and Ardem Patapoutian of Scripps Research Institute for their discoveries of receptors for temperature and touch. Our ability to sense heat, cold and touch is essential for survival and underpins our interaction with the physical world around us. In our daily lives we utilize these senses, but may not notice how temperature and pressure would initiate nerve impulses to generate the senses. This question at molecular level has been solved by this year's Nobel Prize laureates in Physiology or Medicine. In brief, Julius identified the heat sensor called TRPV ion channel as the receptor of capsaicin, a pungent compound from chili peppers that induces a burning sensation, in the nerve endings of the skin that responds to heat back in 1997, and Patapoutian exploited pressure-sensitive cells to discover a novel class of sensors called Piezo proteins that respond to mechanical stimuli in the skin and internal organs back in 2010. The discoveries of the key molecules and their function characterization are considered to be incomplete for the molecular understanding of how our nervous system senses heat, cold, and mechanical stimuli until direct visualization of the molecules are achieved. However, the TRPV and Piezo are channels are membrane proteins that getting 3D pictures of them are next to impossible due to the challenges in crystallization. Fortunately, those bottlenecks were suddenly removed in 2013 when the resolution revolution of cryo-electron microscopy (cryo-EM). Since cryo-EM did not need crystals, rapid structure determinations of TRPV and Piezo were made by the cryo-EM microscopists, friends or competitors of these two laureates, in five years (2014-2019) to complete the structure part of the investigation. In this lecture, I will first brief the TRPV and Piezo protein structures. Then I will explain the keys to cryo-EM resolution revolution

including imaging processing of large amount of data, followed by coverage on several research projects such as a membrane protein that converts methane to methanol and COVID-19 proteins done at Academia Sinica by using an automated cryo-EM (Titan Krios) to demonstrate the power of automated cryo-EM as standard equipment in making rapid discoveries at molecular level in biomedical sciences.

中文摘要

2021 年諾貝爾生理學或醫學獎授予加州大學舊金山分校的 David Julius 和斯克 里普斯研究所的 Ardem Patapoutian,以表彰他們發現了溫度和触覺感受器。我們 感知熱、冷和触覺的能力對於生存至關重要,並且是我們與周圍物理世界互動的 基礎。在我們的日常生活中,我們利用這些感官,但可能不會注意到溫度和壓力 如何啟動神經衝動來產生感官。今年的諾貝爾生理學或醫學獎獲得者在分子水平 上解決了這個問題。簡而言之,早在1997 年 Julius 通過發現辣椒素的受體蛋白, 找到 TRPV 離子通道為熱的傳感器,而 Patapoutian 於 2010 年利用壓力-敏感細 胞就發現了一類稱為壓電蛋白的新型傳感器,可以對皮膚和內臟器官中的機械刺 激做出反應。不過,我們對於感知熱、冷和機械刺激分子層次的理解不算完整, 除非實現分子的可視化。然而,TRPV 和壓電蛋白皆為膜蛋白,要用 X 射線結晶 學獲得它們的 3D 結構幾乎是不可能的。幸運的是, 2013 年發生了冷凍電子顯 微鏡 (cryo-EM) 的分辨率革命。由於 cryo-EM 不需要晶體,這兩位獲獎者通過與 cryo-EM 顯微鏡專家合作,使得在短短在五年內(2014-2019)多個 TRPV 和 Piezo 膜蛋白的原子結構快速被解決。 在本次演講中,我將首先簡要介紹 TRPV 和 Piezo 蛋白質結構。然後解釋冷凍電鏡分辨率革命的關鍵,包括大量數據的影像 處理,然後介紹幾個藉由中央研究院自動冷凍電鏡(Titan Krios)完成的研究項目, 例如將甲烷轉化為甲醇的膜蛋白和 COVID-19 蛋白,證明自動冷凍電鏡已經成為 生物醫學的標準配備,是在分子水平上提供快速發現不可或缺的神器。

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110年年會特邀演講來賓

顏鴻威 副教授 Prof. Hung-Wei (Homer) YEN

演講題目:

Computational Microscopy: Metallurgical Renaissance Inspired

by Atom Probe Tomography

計算顯微術: 三維原子針尖斷層分析所掀起的冶金文藝復興 現職:

國立台灣大學 材料科學與工程學系 副教授 國立台灣大學 工學院 國際事務執行長



經歷:

國立台灣大學材料科學與工程學系 副主任(任務型) 2019.8-2020.7

國立台灣大學材料科學與工程學系 助理教授 2014.8-2018.7

雪梨大學澳洲電子顯微鏡與顯微分析中心 原子針尖科學家 2012.9-2012.8

香港大學機械工程學系 訪問學者 2011.10-2011.11

臺大慶齡工業研究中心 博士後研究助理 2011.2-2012.8

學歷:

國立台灣大學 材料科學與工程學系 學士 2002-2006 國立台灣大學 材料科學與工程學系(逕讀) 博士 2006-2011 Abstract:

Computational Microscopy means applying computational algorithm to digital data or images to enhance capabilities beyond microscopes' intrinsic resolution, accuracy or functions. This promising field is enabled by high-speed, high-throughput and hightechnique computation in such a digital era of microscopy. A young protagonist of computational microscopy is Atom probe tomography (APT), a state-of-art characterization approach enabling atom-by-atom three-dimension information of materials inner space. Importantly, the data from atom probe tomography is completely digital and the information analysis is comprehensively computational. Today, this talk will start from an introduction to Computational Microscopy and atom probe tomography. Then, we will have an joyful journey into inner space of many advanced alloys and see new metallurgical principles with novel nano/microstructures. I would be happy to end this talk by our recent progress in atom probe tomography – Research Platform of Atom Probe for Emergent Crystalline Materials. In summary, atom probe tomography, providing unique three-dimension image of spatial resolution and chemical detection limit, can inspire new thinking and further drive new materials design. Hence, there is plenty room beyond the bottom when one enjoys mining into APT data.



第四十一屆臺灣顯微鏡學會年會研討會 <u>論文摘要子目錄</u>

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	¹ Department of Engineering and System Science, National Tsing Hua University, Hsinchu 30013, Taiwan.	12
	² National Synchrotron Radiation Research Center, Hsinchu 30076, Taiwan.	



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	³ Department of Mechanical Engineering, National Central University, Jhongli, 32001, Taiwan	



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	¹ Department of Engineering and System Science, National Tsing Hua University, HsinChu, Taiwan	36
	² Department of Materials Science and Engineering, National Taiwan University, Taipei, Taiwan	
	³ E.A. Fischione Instruments, Inc., Export, PA, USA	
	⁴ Taiwan Instrument Research Institute, HsinChu, Taiwan	
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	¹ Graduate Institute of Intellectual Property, National Taipei University of Technology, Taipei, 10608, Taiwan	37
	² Department of Research and Development, Gloria Material Technology Corporation, Tainan, 736403, Taiwan	
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Reconstructing the Three-Dimensional Structure of the Double Gyroid Polymeric Materials by Electron Tomography

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In order to explore the fine structure of double gyroid polymers, Polymethylmethacrylate-Poly(2-vinylpyridine) (PMMA-P2VP), we obtained the projection of the stained specimen at different angles by scanning transmission electron microscopes (STEM). The obtained images were pre-processed by alignment and background signal filtering. By combining the Fourier transform and phase retrieval iteration, a three-dimensional structure was successfully obtained. The internal fine structure of the sample in high resolution is able to find out whether the sample has a crystal-like structure, and also has a large potential for fine-structure determination.

Keywords

Electron tomography, Scanning transmission electron microscopes, Threedimensional structure

Asynchronous ptychography using multiple CPUs

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Ptychographic iterative engine (PIE) is a scanning coherent diffraction imaging (CDI) algorithm for extended objects. Overlapping illumination areas on the sample is the prerequisite to performing the reconstruction. Compared to conventional CDI, Ptychography promises extended field of view and superior convergence in phase retrieval, while the general issues are time-consuming computation and huge storage space. Here, we develop an asynchronous Ptychographic approach that significantly reduces the computational time by stitching multiple reconstructions from distributed CPU cores.

Atomic-Scale Electron Beam Induced Damage Observation of Chromium Sulfide Bromine through *In situ* Transmission Electron Microscopy

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CrSBr is one of the novel two-dimensional (2D) ferromagnets released in recent years. With large magnetic moments and spin polarization, CrSBr is also considered to be the promising candidate for future spintronic devices. However, the development is still in its infancy resulting in limited research in the literature. In this work, ultrathin CrSBr is prepared by scotch tape based mechanical exfoliation at first. Subsequently, Raman and X-ray photoelectron spectroscopy(XPS) analysis are conducted to obtain detailed information about chemical structure. The thickness of most exfoliated samples measured by atomic force microscopy (AFM) is about 1.6-6.5 nm corresponding to 2-8 layers. Notably, high resolution images of CrSBr atomic structure are seldom taken by transmission electron microscopy (TEM). It is remarkable that atomic-thick CrSBr is extremely beam-sensitive, which makes it easily damaged under e-beam irradiation. Nevertheless, e-beam damage opens an opportunity for studying the structural properties of CrSBr. The damage behavior, which is found to be consistent with the slip system of orthorhombic, is recorded and further analyzed through atomic-scale imaging. Besides, the observation of recrystallization process of CrSBr and formation and refilling of nanoholes is recorded via in situ TEM. These findings deepen the understanding of the fundamental insight in 2D magnets, and pave the way for next-generation spin-based electronics.



Figure 1. (a) Optical images of CrSBr. (b) AFM images of few-layer CrSBr. (c) HRTEM image of pristine CrSBr. (d-e) An irradiated area showing e-beam induced structural variation. (f) Stepped boundary of CrSBr after e-beam irradiation.

Direct structure-determination of nodavirus in solution by small-angle x-ray diffraction microscopy

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The structures of biological macromolecules are usually obtained from crystallography and cryo-electron microscopy. The requirements of sample preparation limit the understanding of their original state. Small-angle x-ray scattering (SAXS) is able to obtain structural information from biological samples in solution. However, analyzing the structure from the obtained one-dimensional(1D) data requires prior knowledge of the sample and cannot guarantee a unique solution. Coherent diffraction imaging (CDI) provides excellent uniqueness in 2D/3D phase retrieval while the resolution is limited by the poor signal-to-noise ratio during high-angle scattering. Here, we combine CDI and SAXS to directly image a 19-nm-sized nodavirus particle in solution at a 1.3 nm pixel resolution. 77,170 diffraction patterns are summarized from randomly distributed nodavirus particles. Without prior knowledge, the core-shell structure can be obtained from the diffraction intensity alone. The hollow density distribution of nodavirus particles revealed by our reconstruction is consistent with the structure determination from crystallography and cryo-electron microscopy. We believe that this work represents a new protocol for characterizing the structures of macromolecules in solution from small-angle x-ray scattering data.

Atomic-scale observation of biased monolayer MoS₂ via in situ transmission electron microscopy

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In situ observations *via* transmission electron microscopy (TEM) and STEM are powerful methods to examine the authentic behavior of atoms under electron beam irradiation [1, 2]. In this study, direct observation of structural evolution under biasing is revealed *via* powerful *in situ* transmission electron microscopy (TEM). The monolayer MoS₂ has been transferred onto specialized MEMS chips and patterned through EBL process to obtain the suspended MoS₂ device. Subsequently, *in situ* biasing process is recorded by TEM to provide the whole picture of evolution. The STEM imaging is used to deduce the exact atomic configuration of produced Mo nanograins, which represents the disintegration of MoS₂ and sulfur depletion, suggesting that joule heating is the cause of the evolution. Moreover, the intriguing long crack induced by thermal stress has been found in both *in situ* and *ex situ* biasing cases. The oriented edges and atomic structure are further investigated by STEM. We believe that insights of material damage can push the limit of material properties and broaden the range of MoS₂-based device applications.

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Figure 1. The high-voltage biasing of monolayer MoS₂ devices has been demonstrated through *in situ* TEM and aberration-corrected STEM to explore the mechanism of the material failure.

In situ TEM investigation of thermal-induced dynamic behavior and structural transformation of atomic-layer Cr₂S₃

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Two-dimensional magnetic materials have drawn considerable attention because of their potential for spintronic applications. Chromium sulfide(Cr_2S_3) is a ferrimagnetic material with special spin states and thickness-dependent conduction-type transition properties. However, the fundamental understanding of Cr_2S_3 is limited, and comprehensive study of structural properties is essential for pratical applications. Herein, facile chemical vapor deposition (CVD) has been used to synthesize ultrathin non-van der Waals Cr_2S_3 with thickness of ~1.9 nm. The morphology of Cr_2S_3 is observed to transform from triangle to irregular shape with the increase of precursor ($CrCl_3$) concentration. The phase transition of as-grown Cr_2S_3 is studied through direct thermal annealing under different pressures combining transmission electron microscope (TEM) characterization. To further investigate the mechanism, the advanced in situ heating techniques is employed to reveal the dynamic behavior induced by thermal effect. Moreover, energy-dispersive x-ray spectroscopy (EDS) are conducted to obtain elemental distribution and chemical constitution. Our work opens an opportunity for constructing Cr_2O_3 - Cr_2S_3 heterostructures, which are expected to be the building blocks for future electronic and spintronic devices.



Fig. 1 Atomic structure of as-grown Cr₂S₃. (a, b) Low- and high-magnification TEM images of Cr₂S₃ nanosheet. Corresponding FFT is shown in the inset of (b). (c) ADF-STEM image of as-grown sample. (d,e) Multislice simulated ADF-STEM image of R-Cr₂S₃ and T-Cr₂S₃ viewed down [001], showing highly similar structure and d-spacing. (f) Manually created atomic model of Cr₂S₃ viewed down [010]. (g,h) Manually created atomic model of R-Cr₂S₃ and T-Cr₂S₃ and T-Cr₂S₃ and T-Cr₂S₃ viewed down [110] respectively, showing different stacking sequences.

Investigation of Resistive Switching Mechanism in Single Tantalum Oxide Nanotube System with Two Memristive Functions

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One dimensional (1D) metal oxide structure is beneficial for confining the formation direction of conductive filament and scaling down the size of memory devices. However, the switching mechanism of 1D metal oxide is lacking, but urgently needed. In this work, the single Ta₂O₅ nanotube (NT) was fabricated as Ag/ Ta₂O₅ NT /Ag device and the capabilities of combining two memristive functions has been investigated – non-volatile unipolar memory and selector. According to the results of in-situ transmission electron microscope (TEM) and energy dispersive spectrum (EDS), a unique resistive switching mechanism has been established for the first time. The resistive switching behaviors included silver atoms doping, ions surface diffusion and oxygen vacancy. In addition, the crystallinity of Ta₂O₅ nanotubes was well controlled through rapid thermal annealing (RTA) and furnace. The relations between crystallinity and electrical properties were also revealed in details to explore the conduction mechanism in depth. Furthermore, the effect of grain size on the forming voltage was clearly identified. Most of all, the established mechanism revealed the dynamic behaviours of Ta₂O₅ NT in memory devices, which could be explored to wide range of nanotube materials for memory applications.



Figure 1. Characterizations of the Ag/Ta2O5 NT/Ag device after electrical measurement. (a) The I-V curves of the device. (b) Retention measurement at reading voltage of 0.1 V. Dark-field STEM images of the device near the (c) cathode, (e) anode and (d) the middle part after set process. (f) HRTEM image of a nanoparticle near the (e) anode. (g) HRTEM image and corresponding FFT-DP of blue square region in (f). (h) EDS mapping of the device near the (e) anode.

Atomic-Scale Investigation of Electromigration with Different Directions of Electron Flow into High-Density Nanotwinned Copper through In Situ HRTEM

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EM is an interaction between electron carriers and atoms, meaning that it arises from atomic behaviour. Both the number of twin boundaries (TBs) and the direction of electron flow are crucial factors affecting EM behaviour. In this study, EM phenomena with perpendicular and parallel electron flow were investigated by high resolution transmission electron microscopy (HRTEM). Twin planes could limit the growth direction of voids in the parallel case. The evolution of smooth surfaces into step-like surfaces resulting from the triple point retarding EM has been demonstrated. With the measurement of the resistance, it could be concluded that the specimen with electron flow per-pendicular to the TBs had better EM restriction. Columnar grains can confine the position of the voids. This study demonstrates the dynamic evolution of the surface structure, providing insight into the fabrication of interconnections in the integrated circuits industry.



Figure 1. Evolution of the free surface when the electron flow was parallel to the TBs.

Effect of Adding Trace Amounts of Ag on Precipitates Evolution in High Strength Al-Cu-Mg-(Ag) Aluminum Alloys

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AA2024 (Al-Cu-Mg) aluminum alloy is characterized by high specific strength and good fatigue resistance. However, it's still not sufficient to advanced applications like supersonic aircraft [1]. Adding small amounts of Ag can alternate the main strengthening precipitate from S to Ω phase. With constant thickness and high thermal stability, Ω phase not only improves the mechanical strength, but also enhances the elevated temperature properties [2].

In this study, the effect of Ag addition on the nano-precipitates evolution and mechanical properties were analyzed by HRTEM and EELS to realize crystal structures and distributions of S phase and Ω phase and quantitate the volume fraction of Ω phase, respectively. The results showed that the strength of aluminum alloy with Ag addition (Al-Cu-Mg-Ag) significantly improved around 20%. Except for the high volume fraction of Ω phase, the suppression of S phase is also an important part to enhance the strength. These two essential precipitates at different aging stages were fully studied in this research.

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Fig. 1. STEM image of precipitates morphology in 4A100h sample.



Fig. 2. HRTEM of S phase and its FFT images in Al-5.1Cu-1.0Mg aluminum alloy.

Dynamic observation of garnet type all-solid-state battery lithiation/lithiation behavior by *in-situ* TEM <u>An-Yuan Hou</u>¹, Chih-Yang Huang¹,Yi-Tang Tseng¹, Hung-Yang Luo¹, and Wen-Wei

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Li-ion batteries (LIB) has been widely used in storage devices because of its excellent performance, long cycle life time and high energy density. All-solid-state LIB, garnet type Li7La3Zr2O12 (LLZO) electrolyte and LiCoO2 (LCO) cathode are common material replaced in traditional LIB because of their low electronic conduction, fast Li-ion conduction and higher thermal stability [1]. To realize the whole delithiation/lithiation process and volume expansion, *in-situ* transmission electron microscope (TEM) is a powerful tool to investigate the entire charging/discharging cycle. In this study, we applied positive and negative voltage to observe the dynamic delithiation/lithiation behavior and the process of crack formation in LCO/LLZO all-solid-state LIB. From high resolution transmission electron microscope (HRTEM) image, the d-spacing of LCO increased in 1.5V, proving its delithiation. On the contrary, the d-spacing decreased at lithiation process (-1.5V). These results could provide the information of charging/discharging mechanism in LCO/LLZO system.

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Figure 1. Pure cobalt precipitation in solid-state battery because of over-delithiation process

Atomic-Scale Investigation of Resistive Switching Behavior in CaFeO_x Perovskite Oxide RRAM Device

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In this study, we introduced novel material CaFeO_x to reveal the mechanism of oxygen vacancy transition in PV and BM structure. We grew the CaFeO_{2.5} on Nb-STO conductive substrate epitaxially, and deposited the Ti metal as the top electrode. The devices possessed great RRAM properties, high endurance, long data retention, steady high and low resistance state and uniform RRAM properties in each device. In addition, the device also demonstrated the multilevel resistance as changed the RESET voltage. In order to construct the switching mechanism, we use the high-resolution transmission electron microscope (TEM) to observe the topotactic phase change in CaFeO_x. In the low resistance state, the BM CaFeO_{2.5} would transform to PV CaFeO3 under the negative bias, which is SET process for the device. And atomic-scale STEM image showed that PV CaFeO3 also have epitaxial relationship with Nb-STO. When applied the positive bias, the device switched to high resistance state. The PV filament ruptured in half, and transformed to BM CaFeO_{2.5}. Furthermore, the EELS spectrum exhibited that this topotactic transformation was resulted from the oxygen vacancy transition, and induced the multiple valence state of Fe. This study not only firstly revealed the switching mechanism of CaFeO_X but also a new perovskite oxide option for the dielectric material in RRAM.



Figure 1. The initial state and electrical performance of CFO RRAM device.

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Effects of the Coherency of Al₃Zr on the Microstructures and Quench Sensitivity of Al–Zn–Mg–Cu Alloys

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Effects of the coherency of Al₃Zr on the microstructures and quench sensitivity of Al-8.3Zn-2.3Mg-2.4Cu (AA7055) alloys were investigated. These alloys were subjected to two cold-rolling ratios (i.e., 5% and 20%) in order to vary the coherency of Al₃Zr particles. The main objectives of this study were to reduce the quench sensitivity, refine the grain size, and retain the coherency of Al₃Zr for high-strength Al– Zn–Mg–Cu production alloys. Results reveal that with the increase in the cold-rolling ratio, the quench sensitivity of the Zr-containing alloys increases. At a high cold-rolling ratio, the interface between Al₃Zr particles and the matrix became semi-coherent after solution treatment. This semi-coherent interface led to the heterogeneous nucleation of solute atoms during quenching, which was the leading cause for the high quench sensitivity. By contrast, at a low cold-rolling ratio, the alloys did not exhibit any noticeable quench sensitivity. The Al₃Zr particles retained coherence in the matrix and did not afford heterogeneous nucleation sites.

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Figure 1. HRTEM images of Al₃Zr particles with a (a) coherent structure and a (b) semi-coherent structure, taken along the [110] L1₂ zone axis.

A Nano-scale TEM Study of η' Strengthening Phase in AA7075 Aluminum with different artificial ageing times and different contents of Cu

A-13

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Precipitate hardening is the major strengthening mechanism of 7075 aluminum alloy. According to the previous researches, η ' precipitates are responsible for the strengthening of the 7075 alloys during artificial ageing stage. Our study investigates the microstructure of the η ' at peak ageing of different artificial ageing temperatures. On the other hand, we compare the microstructures of 7075 aluminum alloys with 1.61 wt% Cu with that of 7075 aluminum alloy with 1.98 wt% Cu. Our TEM results indicate that at higher artificial ageing temperature, the size of the η ' and other precipitates at the peak ageing condition are larger, which is shown in Figure 1. Furthermore, the precipitate size of 7075 alloy with 1.98 wt% is smaller than that of 7075 alloy with 1.61 wt% Cu after 160°C artificial ageing, which is shown in figure 2. It suggests that the adding of Cu can restrain the coarsening of the precipitates.



Figure 1. 7075 alloy with 1.98 wt% Cu after peak aging at (a)100°C (b)120°C (c) 160°C



Figure 2. 7075 alloy with (a)1.61 wt% Cu (b)1.98 wt% Cu after artificial peak aging at 160°C

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Defect Structure and Morphology of Carbide in Lenticular Martensite of AISI440C Stainless Steel

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AISI 440C stainless steel contains high level of chromium and carbon that enable lenticular martensite to form. A lenticular martensite plate shows three regions mid-rib, extended twin and untwined region. Types of defect and the defect density are different in these three regions. Low temperature tempering process is carried out to investigate the relation between the carbide precipitation behavior and defect structure in these regions. TEM investigation are conducted to obtain the crystallography information of substructure and carbide precipitation. In this work three-regions precipitation of carbide is investigated. There are two kinds of carbides been observed, needle like and rod-like. Needle like carbide nucleated on twinning plane, while rod like carbide form due to dislocations [2]. The size distribution of the latter has strong correlation to dislocation density, thereby showing distinguishable morphology in tempered extended twin region and untwined region.

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Figure 1. Three-region carbide distribution in lenticular martensite tempered at 400°C

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Carbon@ Titania yolk-shell nanostructure with enhanced photocatalytic performance

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In this study, photocatalytic performance of carbon@ titania yolk-shell nanostructure was enhanced by tuning the synergetic effect of the titania shell and the residual carbon sphere. At first, carbon spheres with a diameter of 400 nm were used as templates for sol-gel method to deposit titania particles on it. The crystallinity of the shell and the size of the residual carbon sphere was controlled by altering the time as well as the temperature of the subsequent annealing process. Cross sectional TEM observations showed that as increasing the annealing time from 0 hour to 2 hour or enhancing the temperature from 400 °C to 500 °C, the size of carbon spheres shrank from 322 nm to 122 nm and finally disappeared. On the other hand, assynthesized TiO₂ shell is amorphous and uniform with thickness about 85.1 nm. The crystallinity of the TiO₂ particles was enhanced significantly after annealing. The size of TiO₂ particle was increased to 12.2 nm. XANES analysis results showed that the residual of carbon spheres in the TiO₂ hollow shell will lead to lattice distortion, more oxygen vacancies and trivalent titanium ions. This unique carbon@ titania yolk-shell nanostructures were used to study the photocatalyst performance and acetaminophen was chose as the target. The photocatalytic analysis studies revealed that the existence of carbon nanospheres will improve the photocatalytic efficiency. The mobile electron flew from Ti³⁺ to carbon nanospheres under both ultraviolet and visible light excitation was evident by in-situ XAS measurement. This process would prolong the life time of photon-induced electron and hole pairs, as a result increase the photocatalytic efficiency.

Three Dimensional Atomic Strain Mapping in a Core-Shell Nanoparticle

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Electron Tomography is an important technique to study 3D structures of materials at the atomic scale. We collected projections of a PtRuAu core-shell nanoparticle at 25 angles through scanning transmission electron microscopy. The 3D reconstruction is computed using a generalized Fourier iterative reconstruction (GENFIRE) algorithm. The 3D reconstruction allows us to trace coordinates of atoms from the 3D intensity map. By comparing the traced atomic coordinates with an ideal model, we measure the strain tensor in three dimensions. We also obtained the chemical species of each atom from the atomic position and the 3D reconstructed image. Then, we individually determined the crystal structure of the Pt shell and Ru core. The bulk Ru adopts a hexagonal closed pack (HCP) structure. However, we found that the crystal structure of the Ru core is face-centered cubic (FCC). The observation indicates that the structure of the atoms with the ideal model and the simulation in an ideal model, we revealed the dislocation in each direction. We found that the ($\overline{111}$) lattice planes shifted along [$21\overline{1}$] in this nanoparticle.

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Low-dose 3D reconstruction of the non-stained double-gyroid network polymer by scanning transmission electron microscope

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Staining the specimen to enhance the contrast of transmission electron microscope (TEM) images of polymers is an inevitable method that causes unavoidable artifacts. However, the dark field imaging in scanning transmission electron microscope (STEM) is a z-contrast pattern, the difference between nitrogen atoms or carbon atoms on the benzene ring of the polymer PS-P2VP can form the contrast on the STEM projections. Combined with the low-dose 3D reconstruction algorithm, Generalized Fourier Iterative Reconstruction (GENFIRE), we have established a set of non-stained polymer 3D reconstruction systems, which is competitive with the mathematical model of double gyroid PS-P2VP polymer.

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Figure 1. The STEM projection from -60 degrees to +60 degrees at 10 degrees

An Investigation of 465 Maraging Stainless Steel

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In this research, TEM, STEM were used to investigate the hierarchical structure, nano-precipitation and reversed austenite of 465 maraging stainless steel so as to understand the morphology, substructure and dispersion characteristics of nano-precipitation and reversed austenite in the hierarchical structure of 465 maraging steel. In addition, the microanalysis is used to study the preferred nucleation site of reversed austenite with different morphologies. Traditional stainless steel has excellent corrosion resistance. However, its low carbon content limits the strength of stainless steel. In order to solve this problem, 465 maraging stainless steel add trace alloying elements then conduct aging treatment to strengthen the stainless steel with the hierarchical structure of martensite, high density tangled dislocation and nano-precipitation. Furthermore, aging will also form reversed austenite at high-angle boundaries, thus, transformation induced plasticity (TRIP) effect could be introduced into the material to deal with the problem of strength-ductility trade off.

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Figure 1. 510°C - 4hr (peak aging). Nanotwin inside precipitation.

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Dislocation structure of massive ferrite in an Interstitial-Free Steel

A-19

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In this study, the hot-rolled IF steel with the composition of Fe-0.002C-0.002S-0.001N-0.1S-0.52Mn-0.04P-0.022Ti (wt%) was cut into tensile specimen using ASTM-E8 standard. These specimens were austenitized at 1200°C for 5 minutes. Then, these specimens were cooled to room temperature by water quenching, air cooling and furnace cooling, respectively. After tensile test, specimens were observed under TEM.

IF steels showed typical dislocation-slip structure. Dislocation cells and slip bands could be observed everywhere inside the deformed sample, as shown in Fig1.



Fig1. Slip bands in a massive ferrite grain

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Investigating Resistive Switching Mechanism in Single Crystal PbHfO₃ Film via TEM

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Recently, binary metal-oxide based resistive random access memory (RRAM) has become one of the popular candidate for future application in memory system due to its excellent characteristics, such as high switching speed, and trusting stability. However, it is still invalid in the evidences of direct observation of the switching process to establish the mechanism. In this work, the dielectric layer PbHfO3 (PHO) films of 100 nm thickness have been epitaxially deposited on SrRuO3 bottom electrode and then deposited Au as top electrode. PHO shows great electrical properties, which performs high cycling endurance (more than 600 cycles), uniform distribution of both low resistant state (LRS) and high resistant state (HRS), and long retention time (over 104s). On the other hand, we investigated the switching mechanism through the X-ray photoelectron spectroscopy (XPS) and transmission electron microscopy (TEM) and scanning transmission electron microscopy (STEM). The high resolution TEM and STEM images demonstrate the PHO structure before and after the switching behavior. These results strengthen the switching mechanism and the detailed discussion of this switching behavior provide a novel aspect of the RRAM switching mechanism.



Figure 1. The initial state of the Au/PHO/SRO RRAM device with its EDS mapping and the electrical measurements. (a) Cross-section STEM image of pristine RRAM device. (b), (c), (d), (e), (f) and (g) EDS mapping. (h) High resolution TEM image of initial state of PHO/SRO shows the epitaxial deposition.

Specimen Preparation for STEM Tomography

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Most recently, it has been demonstrated that one can obtain 3D atomic structures within nanomaterials by high-resolution scanning-transmission-electron-microscope (STEM) Tomography. However, the distorted 3D reconstruction due to the missing images at high angles (i.e., missing wedge problem) is the most common challenge in this field. We generate rod-shaped TEM specimens using the milling and the lift-out technique of Focused Ion Beam (FIB) to solve the above issue. In this study, the preparation procedures and STEM images of the specimen will be presented.

An Investigation of 7075 Aluminum Alloy

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In this research, TEM, STEM were used to investigate the second phase, nano-precipitation of 7075 aluminum alloy so as to understand the morphology and dispersion characteristics of nano-precipitation in the matrix of 7075 aluminum alloy. In addition, the microanalysis is used to study the chemical composition of the second phase. Recently, sustainable development is an important issue to many industries, in order to improve the fuel consumption efficiency and reduce the carbon footprint in the next generation vehicles, the high strength and light weigh aluminum alloys are recognized as the one of the forward-looking materials. 7075 aluminum alloys are subjected to different aging treatments to compare the difference between each treatment. During the aging treatment, the nano-scaled microstructural evolution of precipitates such as GP zones, η' precipitates, and η precipitates are characterized by Transmission electron microscope (TEM). Last but not least, second phase is combined with several elements such as Al, Mg, Cu and Fe, it'll reduce the element concentration in Al matrix, which leads to the amount of precipitation decrease.

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Annealing twin and grain growth of 190µm AISI 316L stainless steel wires

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The AISI 316L stainless steel wire with a diameter of 190 μ m has been subjected to a tensile strain with heat treatments in order to obtain a "bamboo structure", which is a unique phenomenon that may occur when the ultra-fine FCC austenitic steel wire is annealed. In the present work, obvious grain growth and coarsening of grains with annealing twins were found under the observation of SEM/EBSD. At the same time, the twin relationship was studied with the aid of OIM analysis, as shown in Fig. 1. Although the current diameter is not thin enough to produce a bamboo structure, a case study for annealing twins in the treated specimens shows that among primary twins with an axis-angle pair of <111>/ 60°, the secondary twins with an axis-angle pair of <110>/ 39° can be revealed.



Figure 1. EBSD result of a 190µm 316L stainless steel wire annealed at 1200°C for 3 days and its misorientation profile along the black arrow.

Three-dimensional Semiconductor devices Tomographic Reconstruction by Scanning Transmission Electron Microscopy

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In this work, we employed an atomic resolution three-dimensional electron tomography(AET) technique to investigate the advanced process semiconductor devices, such as N7/N5 finFET, N3 MBC-GAAFET(multi-bridge channel-gate all around FET). We developed a standard manufacturing process to prepare these semiconductor devices into rod-shaped transmission electron microscopy(TEM) specimens less than 50 nm diameter using focused ion beam microscopy(FIB). Importantly, this rod-shaped TEM specimen has eliminated the missing wedge in 360° image acquisition by aberration-corrected STEM and tomographic reconstruction without losing information. Furthermore, with our group's mature three-dimensional reconstruction technology, we can turn this rod-shaped semiconductor specimen into a three-dimensional model to further elucidate the relationships between the structures and electric properties.

Keywords: electron tomography, transmission electron microscopy, focused ion beam

Precipitation behavior of η -Ni₃Ti in the Custom 465 stainless steel after isothermal aging treatment

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Custom 465 stainless steel was demonstrated to possess very high strength, good ductility, and excellent corrosion resistance at the same time, and thus attracted much attention in recent years [1]. Tiny-sized precipitates, namely η -Ni₃Ti, with a rod-like shape can be formed in custom 465 stainless steels after various isothermal aging treatments. The η -Ni₃Ti precipitates had a HCP crystal structure and adopted a Burgers orientation relationship, i.e., $\{011\}_{\alpha'}$ // $\{0001\}_{\eta}$; $< 1\overline{11} >_{\alpha'}$ // $< 11\overline{2}0 >_{\eta}$, with the martensite matrix [1]. It is found that the average diameter and length of η -Ni₃Ti precipitates became larger as the isothermal aging temperature and time increased, which implying the precipitation behavior of η -Ni₃Ti particles complied with the thermal activation mechanism.

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Fig. 1 (a) lower- magnification bright field TEM images and (b, c) dark field TEM images showing two different variants of precipitated particles randomly distributed within the martensite matrix of the Custom 465 stainless steel after isothermal aging at 600 °C for 4 hours.

Microstructure and Mechanical Behaviors of B2 Intermetallic Compounds with High-Entropy Sublattice

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In this study, the concept of high-entropy sublattice (HESL) was applied to develop new B2 intermetallic coumpounds (IMCs) by substituing Ni by Fe, Co, Pt and Pd in TiNi alloy. Here, it is named HESL-B2 alloy. Interestingly, it was found that the C14 laves phase, which had never been seen in TiNi alloy or its variants, is discovered. The influence of alloy composition was discussed for this new phase. Besides, these alloys possessed large compressive ductility because of the deformation twin formed during deformation. An excellent combination in strength (over 1900 MPa) and ductility (over 30%) was observed though C14 laves phase plays as cacking initiation sites. The Young's moduli of these alloys is between 42 and 63 GPa, which is close to human bones. They might have the potential to develop into biomedical materials.



Figure 1. {114} B2 deformation twins of compressed 0Pt ($\varepsilon = 2$ %) observed from TEM analysis. (a, d) Bright field image of the specimen. (b, e) Dark field images of mainly and second twins. (c, f) Electron diffraction patterns of twins.

Strength-Ductility Balance in High-Entropy Duplex Lightweight Steel Strengthened by Geometrically Necessary Dislocation Assisted Mechanical Twins

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A novel duplex high-entropy lightweight steel with chemical composition of Fe-26Mn-5.5Al-3Si-1C-2.5Ni-0.2Mo-0.1V (in wt. %) was investigated by transmission electron microscopy and crystallography map in this study. Microstructure of the steel is composed of face-center cubic (FCC) matrix and D0₃ intermetallic compounds (IMCs). The second phase strengthening effect of D0₃ IMCs by forcing the FCC matrix to form high density geometrically necessary dislocations (GNDs) at the interface presented. Moreover, the deformation twins were presented near the interfaces. This suggested the GNDs and stacking faults activated by D0₃ IMCs assisted the formation of mechanical twins. The hybridization of 2nd phase induced GNDs and GNDs assisted TWIP effect gives rise to the high work hardening rate, and excellent combination of strength (1300 MPa in ultimate tensile strength) and ductility (45% in total elongation). This work opens a metallurgical path for stronger, more ductile and lighter steels.



Figure 1 Deformed microstructure at strain state of 20% (a) bright field image at the interface (b) dark field image and the corresponding diffraction pattern from the white frame

The role of TiB on microstructure and lamella properties in heat-treated TiAl alloys

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This work aimed to elucidate the effect of titanium boride on microstructure and lamellar structure by scanning electron microscopy(SEM) and transmission electron microscopy(TEM) in TiAl alloys. The transformation mechanism of interaction between B27-type TiB and $\alpha \rightarrow \gamma$ transformation was studied by the lattice misfit in high resolution TEM image and crystallographic simulation. It was demonstrated that the existance of interface between TiB and primary α would act as a preferred site for growth of γ lamelle, which increased the mean thickness of γ lamelle. As a result, more fraction of localized ultrafine lamellar region generated caused by supersaturated of α lamelle. The boron addition seemed as a refining agent in casting and heat-treated TiAl alloys, but the effect on lamellar properties shouldn't be ignored.



Fig. 1 (a) TEM image of localized ultrafine lamellar region with low magnification (b) HRTEM image and FFT of γ nano-lamella



Denser region

Fig. 2 (a)(c) Bright field and dark field image of α_2 phase with and without boron addition, respectively. (b)(d) The distribution of lamellar thickness with and without boron addition, respectively.

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Optimization of strain characterization using STEM Moiré interference

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Strain distribution plays a very important role in the characteristics of semiconductor components, but there is currently a lack of standardized service mode for this issue. Many methods of strain characterization have been developed, including peak-pairs analysis (PPA), geometric phase analysis (GPA), nano beam diffraction (NBD), precession electron diffraction (PED) and scanning transmission electron microscopy (STEM) moiré interference images.^[1,2] Among these methods, the STEM Moiré interference can be performed with a wide range and high precision stress analysis. In this study, we try to optimize the image capture method of STEM Moiré. Traditionally, the interference images were captured through ADF detectors at some specific magnifications in the STEM mode. In this image capture mode, an interference image with high signal-to-noise ratio often takes 30 seconds to capture. We hope to shorten the overall acquisition time without reducing the image quality. The introduction of such a concept can reduce the strain calculation error caused by sample drift and environmental interference. Using the energy spectrum imaging function in the GMS software developed by Gatan, we can freely choose the analysis area. When a smaller analysis area was choosed, we can get the stress distribution value of the small area in a short analysis time. We can usually control the overall image capture time within 5 seconds. This analysis method can effectively reduce the stress deviation caused by sample drift. Although the analyzed area becomes smaller, we can still get a wide range of strain analysis results through multi-point nano-area strain analysis. Such stress analysis concepts and methods are expected to be applied to more advanced semiconductor devices.

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Figure 1 Synchronously capture STEM Moiré interference using spectrum imaging



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thermo scientific



Top: HAADF (DCFI) STEM image of GaN [212] at at 300kV taken on the Spectra 300 S/TEM showing 40.5 pm Ga-Ga dumbbell splitting and 39 pm resolution in the FFT on a wide gap (S-TWIN) pole piece.

Bottom: DyScO₃ specimen investigated with the Spectra 200 S/TEM. The combined ultra-high brightness of the X-CFEG, resolving power of the S-CORR and large solid angle (1.76 Sr) of the Dual-X detectors results in high signal to noise ratio, atomic resolution, raw and unfiltered EDX maps that can be collected with up to 90 pm resolution. Sample courtesy: Professor L.F. Kourkoutis, Cornell University.

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