

ALD for Manufacturing

Room Evergreen Ballroom & Foyer - Session AM-MoP

ALD for Manufacturing Poster Session

AM-MoP-1 Numerical Analysis on Gas Flow Field for a Sustainable ALD Process Chamber, *Kyung-Hoon Yoo*, Korea Institute of Industrial Technology (KITECH), Republic of Korea; *G. Song*, KUMYOUNG ENG Inc., Republic of Korea; *C. Kim*, TNG Inc., Republic of Korea; *J. Hwang*, *H. Lee*, *S. Lee*, Korea Institute of Industrial Technology, Republic of Korea; *K. Lee*, SAMSUNG DISPLAY, Republic of Korea

In order to develop a sustainable ALD processing cluster tool for 300 mm wafers, it is necessary to establish a manufacturing technology for a high-productivity, high-efficiency ALD process chamber that reduces the intrinsic excessive consumption of energy and materials.¹ In the present study, as the part of countermeasure to the excessive consumption, a micro-gap ALD process chamber is considered for the optimized design with the process space volume decreased. The changes in the flow field of nitrogen in the process space of the process chamber with the gap sizes of 1 mm and 10 mm respectively are observed at 200 °C, utilizing computational fluid CFD numerical analysis. For the present nitrogen flow field with a base pressure of 1 Torr and a temperature of 200 °C, the Knudsen number $Kn < 0.1$ and Reynolds number $Re < 2300$ are evaluated, and consequently the continuity and momentum equations of a steady-state compressible laminar flow field are considered.^{2,3}

Acknowledgment

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References

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2. M. R. Shaeri, T.-C. Jen, C. Y. Yuan and M. Behnia, *International Journal of Heat and Mass Transfer* **89**, 468 (2015).
3. E. J. McInerney, 2017, *J. Vac. Sci. Technol. A* **35**, 01B138 (2017).

AM-MoP-2 Atomic Layer Deposition Reactor for Fixed-Bed Powder Processing with Inert Sample Transfer, *S. Andsten*, *J. Velasco*, *S. Larkiala*, Aalto University, Finland; *K. Salonen*, Elabs Oy engineering, Finland; *C. Gonsalves*, *J. Rask*, *J. Stang*, *V. Miikkulainen*, *S. Jääskeläinen*, *Riikka Puurunen*, Aalto University, Finland

Processing atomic layer deposition (ALD) coatings on porous high-surface-area particles is of increasing interest related to applications as heterogeneous catalysts. ALD on particles can be made in various reactor configurations such as fixed powder bed, fluidized bed, and rotating drum. Also reactors meant for thin film processing are used for porous particles, by placing the particles on a tray and allowing the gasses to flow over the bed and diffuse into the bed.

Although the fundamental ALD mechanisms are the same irrespective of the geometry of the substrate material, specialized particle coating reactors differ significantly from mainstream thin-film ALD reactors. Most important is to take into account the much larger reactant doses needed to saturate the surface with the adsorbed species. One gram of a high-surface-area often contains ~100 to 1000 m², compared to ~0.1 m² of a typical silicon wafer. The required amount of reactant scales directly with the surface area to be coated. Furthermore, porous high-surface-area materials can be estimated to have extremely high aspect ratios (HAR): mesopores with 5 nm diameter and particle size of 1 mm give an aspect ratio of ~100 000. Coating HAR substrates requires longer reactant exposures, allowing the reactant to diffuse into the structure. Of the various specialized particle coating reactors, the fixed bed reactors are the most simple to construct, and oldest in use: both historical development branches of ALD - atomic layer epitaxy (ALE) and molecular layering (ML) - employed such reactors.

In this work, we present a new ALD reactor design for coating porous high-surface-area particles in a fixed bed. The reactor reported in this work is aimed for fundamental laboratory-scale studies, allowing the coating of a few grams of porous high-surface-area material at a time. Loading and unloading of the sample inertly is possible, enabling the processing of air-sensitive substrates and the investigation of the adsorbed species without air contact. Pre-treatment can be made at temperatures up to 800 °C under controlled gas flow. ALD reactants are loaded in sources which can be

heated up to about 200 °C. Gaseous sources are included. The reactor is equipped with an afterburner and condenser for treating the unreacted reactant. Port for gas analysis e.g. via mass spectroscopy is foreseen.

AM-MoP-3 Reverse Templating Effects of Low-Resistivity Ru Ald on Sputtered Ru, *Chenghsuan Kuo*, UCSD, Taiwan; *V. Wang*, UCSD; *R. Kanjolia*, EMD Electronics, USA; *M. Moinpour*, EMD Electronics; *J. Woodruff*, EMD Electronics, USA; *H. Simka*, Samsung Electronics; *A. Kummel*, UCSD

Ruthenium is a promising candidate to replace Cu as an interconnect metal due to its low resistivity in narrow vias and resistance to electromigration. In previous work, a Ru Atomic Layer Deposition (ALD) process using Ru(CpEt)₂ and O₂ was developed to produce films with bulk-like resistivities. However, the ALD exhibits poor initial nucleation with variable initial nucleation delay causing thickness control to be difficult and high surface roughness. In this work, the effects of depositing low resistivity ALD Ru on 2 and 10-nm sputtered Ru films are investigated to eliminate the initial poor nucleation while retaining low overall film resistivity.

AM-MoP-4 Thermal Evaporation Enhanced Atomic Layer Deposition for Far Ultraviolet Mirror Coatings, *Robin Rodriguez*, *J. Hennessy*, Jet Propulsion Laboratory (NASA/JPL)

UV instruments on NASA space missions such as Hubble Space Telescope (HST), the Far Ultraviolet Spectroscopic Explorer (FUSE), and the Galaxy Evolution Explorer (GALEX) have made groundbreaking astrophysical discoveries in areas as diverse as galaxy evolution, star formation, and molecular cloud chemistry. These spectrometers have all benefited from the use of Al mirrors that are highly reflective in the ultraviolet (UV). Aluminum is the only reflective metal that offers broad ultraviolet/visible/near-infrared response, making it highly relevant for use in all far ultraviolet (FUV, 90-200 nm) instruments. However, aluminum is very reactive and susceptible to oxidation, which can limit its reflective performance in the FUV. Ultra-thin coatings of metal-fluorides such as AlF₃, MgF₂, and LiF can be used to protect the Al surface while preserving its high reflectance in the FUV. Atomic layer deposition (ALD) provides unparalleled uniformity and thickness control, making it the ideal process for coating these curved mirrors and shaped optics. However, the Al mirrors are typically fabricated via a separate physical vapor deposition (PVD) process, requiring the mirror to move between one vacuum system to another and exposing it to air, which results in the immediate formation of the native oxide on the Al surface. Therefore, it is necessary to coat the Al surface with the metal-fluoride before exposing it to air.

Herein lies the need to have both processes, PVD and ALD, occur within the same vacuum environment. We report on the development of a custom, in-house built, thin film deposition reactor that is capable of doing thermal evaporation of Al and ALD of metal-fluorides within the same vacuum chamber. Sequentially combining both deposition techniques without breaking vacuum has the potential to enhance the performance of UV aluminum mirrors. It also has the potential to enable the fabrication of metal-dielectric bandpass filters with deeper FUV transmission bands. In this presentation we will describe our thermal evaporation enhanced ALD (TE-ALD) reactor and present preliminary characterization results of FUV mirrors and bandpass filters fabricated using this reactor.

AM-MoP-6 Technical Analysis and Solution of Critical Electrostatic Chuck Problem in High Temperature CVD Process through Estimation Model of the Johnsen-Rahbek Chucking Force, *Youngbok Lee*, *S. Han*, *S. Cho*, Samsung Electronics, Republic of Korea; *Y. Kim*, Samsung Electronics

In the state-of-the-art semiconductor manufacturing process, the deposition layer becomes thicker and contains higher stress, which requires higher chucking force and leads to various wafer chucking problems on Electrostatic chuck (ESC). ESC is the device utilized to fix the wafer by attractive force (chucking force) during the semiconductor manufacturing process such as chemical vapor deposition (CVD), etching and ion implantation. There are technical problems with the ESC during the processes such as Amorphous Carbon Layer (ACL) and mold CVD where excessively high voltage is currently required for increasing chucking force, which can lead to arcing problems and damages on wafers. Also recently, wafer backside defect problems such as cracks and scratches marks are arising during the CVD processes which is caused by excessive chucking pressure.

To prevent such issues in the ESC, it is helpful to estimate the chucking force at the various conditions. Therefore, we have established a rigorous estimation model of chucking force based on Johnsen-Rahbek type ESC. The proposed model reflects the emboss structure and dielectric coating on

Monday Evening, July 24, 2023

the ESC, and various layers of the wafer. The effect of the ESC geometry such as diameter, height and number of the emboss on the chucking force is adequately reflected on. Also, the model predicts the trend of change in chucking force with respect to the thickness/material of the ESC coating and layers on the wafer.

Based on the model, it is possible to verify how the chucking force changes according to the various conditions. Several technical proposals, such as changing the material of the ESC coating and wafer layers, are suggested in this research to increase the chucking force. Also, a strategy to relax the chucking pressure while maintaining the total chucking force same was suggested. Therefore, this proposed model can contribute to solve the chucking problems by giving the suggestions to increase the chucking force without the excessive chucking voltage. Moreover, the proposed methodology and analysis can be easily adapted to other processes and equipment that require extreme chucking performance in high temperature and thick layer condition.

AM-MoP-10 Multi Cycle and Material Deposition for Spatial Atomic Layer Deposition Process, Atilla Varga, M. Carnoy, M. Funding la Cour, M. Plakhotnyuk, I. Kundrata, ATLANT 3D, Denmark; J. Bachmann, Friedrich-Alexander Universität, Germany

Spatial Atomic Layer Deposition (sALD) offers a unique opportunity for localized deposition due to its physical separation and isolation of precursor and co-reagent dosing.^[1] While simple in theory, due to well-developed examples of sALD, in practice miniaturization of sALD requires substantial effort into the creation of suitable micro-nozzles.^[1] Uniquely, ATLANT 3D has developed proprietary sALD micronozzles, called microreactor Direct Atomic Layer Processing - μ DALP™.

The μ DALP™ process undergoes the same cyclic ALD process but is only done in a spatially localized area.^[2] The microreactor or micronozzle confines the flows of gases used for ALD within a defined μ m-scale centric area on the substrate, to deposit the desired material. Similarly, to spatial ALD, the creation of this monolayer then hinges on the movement of the substrate.^[1,2]

Since sALD and the μ DALP™ process are based on physical separation, it is theoretically compatible with any ALD material process however requires development as ALD processes are highly tool dependent.^[3] As such, the material capabilities can match traditional ALD and exceed other patterning techniques, such as lithography, which can be costly and time-consuming, especially for rapid prototyping required for innovation.^[4,5]

sALD using the μ DALP™ technology also vastly increases the efficiency and innovation potential of material and precursor development. Using a small amount of precursor (due to low flow rates required) multiple film thicknesses can be deposited onto a single wafer used to calculate a processes growth rate within only a few hours, compared to days for a traditional ALD process (Fig 1). Multiple depositions can also be performed at varying temperatures for the calculation of temperature dependent growth rate (for "ALD window"), and film characteristics all within a few hours on a single sample. The μ DALP™ process has also been used to demonstrate the selective deposition of different materials on the same substrate without the need for masking shown in Fig 2. By facilitating the more efficient development of ALD processes, μ DALP™ sALD can help to enable continued and more efficient growth of the ALD industry and the development of new and innovative technologies. Multi-material sALD also enables unseen potential for versatile patterning and complex geometry formation, applicable to efficient, iterative, and low-cost device and sensor development.

AM-MoP-11 Hike Furnace HCD SiN Matching TEL Furnace HCD SiN, Yuan Hsiao Su, Taiwan Semiconductor Manufacturing Company, Taiwan

In this work, we describe how Hitachi Kokusai (HiKE) vertical furnaces developed the novel thin film formation method, to provide the second tool type of FinFET hard mask silicon nitride (HM SiN) process that widely used in tsmc very large scale integrated (VLSI) devices fabrication line, The background is the first tool type Tokyo Electron (TEL) not able to provide formula furnace with end of maintenance (EOM).

We develop an innovation HiKE low-temperature 500C process with good film property control of silicon nitride (SiN) formed by low-pressure atom layer deposition (LPALD), and it has been developed by using hexachlorodisilane (HCD, Si₂Cl₆). The potential scope of application to lead

the advanced production line extend from the conventional low-pressure vapor deposition (LPCVD) technique performed at 600C and produced by TEL formula vertical furnaces. The film formed by HiKE furnace shows excellent uniformity with the same refractive index 2.28 and silicon-nitride ratio (Si:N ratio), to lead the same performances of film properties and is advantageous for thermal budget and cost reduction.

In this study, HCD-SiN deposition characteristics, temperature dependence of the film composition and film properties under VLSI fabrication processes are reported, and the differences with the conventional LPCVD HCD-SiN are discussed.

ALD for Manufacturing

Room Regency Ballroom A-C - Session AM-WeM

Manufacturing

Moderators: Dr. Arrelaine Dameron, Forge Nano, Ganesh Sundaram, Veeco-CNT

8:00am **AM-WeM-1 Atomic Layer Technologies for III-V Nitride Epitaxy, High-K/Metal Gate, Ferroelectric Negative Capacitance, and Area-Selective Deposition**, *Miin-Jang Chen, C. Chou, T. Chang, W. Lee*, National Taiwan University, Taiwan **INVITED**

We report the recent progress from conventional atomic layer deposition (ALD) toward a variety of atomic layer technologies, such as atomic layer annealing, crystallization, densification, epitaxy, etching, etc. The topics in this presentation include (1) atomic layer annealing (ALA) for atomic layer epitaxy (ALEp) of GaN and AlN at a low deposition temperature of only 300°C, (2) Atomic layer tailoring for the realization of sub-10 nm, wake-up free ferroelectric $\text{Hf}_{0.5}\text{Zr}_{0.5}\text{O}_2$ thin films with high remnant polarization and low thermal budget, (3) transient negative capacitance in ferroelectric capacitors, (4) atomic layer crystallization and densification induced by substrate biasing for the enhancement of ferroelectric and dielectric properties, (5) ALA for high-K/metal gate, including the improvements in dielectric constant, leakage current, reliability, and the modulation of work function, and (6) atomic layer nucleation engineering (ALNE) for inhibitor-free area-selective ALD with high selectivity. The results demonstrate the promising potential of atomic layer technologies for the precise engineering and fabrication of nanoscale materials and devices.

8:30am **AM-WeM-3 Optimizing Vessel Design for Pulsed Delivery of Solid Precursors**, *James Maslar, V. Khromchenko, B. Kalanyan*, National Institute of Standards and Technology (NIST)

Solid precursors are widely employed in ALD processes and are often delivered to a deposition surface by entraining the precursor vapor in a carrier gas. Unfortunately, it can be difficult to reproducibly deliver solid precursors in this manner. This difficulty is often due to an inability to maintain carrier gas saturation in the precursor vessel head space, resulting in less than the maximum precursor amount being delivered. While this situation may not be an issue for an ideal ALD process (unless the total precursor dose is insufficient to saturate all surface reactive sites), this situation could negatively impact a non-ideal ALD process. The degree of carrier gas saturation depends on numerous factors including vessel design, precursor physicochemical properties, carrier gas flow rate, pressure, and idle time. (For many ALD processes, at least one precursor is idled – there is no flow from the precursor vessel - during a deposition cycle.) The goal of this work is to characterize the performance of different vessel designs for pulsed delivery of solid precursors and to identify relationships between vessel design aspects, precursor properties, and gas flow conditions that can be used to maximize the amount and reproducibility of precursor delivered. Computational fluid dynamics (CFD) were employed to simulate mass carryover for a range of vessel designs and conditions. For select cases, CFD mass carryover values were benchmarked to values measured using optical gas analyzers. The results of this investigation should improve the understanding of the relationships between vessel design, precursor properties, and gas flow conditions, thereby permitting a more informed selection of precursor vessel design for pulsed delivery of a particular precursor within a particular process parameter range.

8:45am **AM-WeM-4 Accurate Precursor Dose Delivery with Realtime Closed Loop Control**, *J. Ye, J. Ding, Guy Rosenzweig*, MKS Instruments, Inc.

Atomic Layer Deposition (ALD) and Atomic Layer Etching (ALE) processes are the key technologies which are enabling the use of new materials and three-dimensional designs in advanced chip manufacturing. The ALD and ALE processes become a vital factor in the applications of *self-aligned patterning*, *3D NAND* and *FinFet*. Consistent precursor delivery is required for generating a stable and homogenous deposition. Unstable deposition will cause defects and create wafer to wafer and batch to batch variations. A consistent precursor dose delivery also further improves process throughput and cuts the waste of precursors by reducing overdosing.

The technical challenge of achieving precursor delivery consistency comes from two aspects: 1) The capability of measuring the precursor concentration in the delivery line in real time; 2) The capability of quickly adapting the precursor pulse shape to control the delivered dose at the pulse level.

MKS Instruments is researching and testing a device that integrates a precursor concentration sensing module and a precursor pulsing module. The device is expected to tolerate up to 200°C to accommodate most of the precursor delivery processes. The precursor concentration sensing module measures the concentration out of the source in real time and feeds the readings to the pulsing module immediately downstream. The pulsing module adjusts the delivery rate instantaneously based on the concentration and the amount of precursor has been delivered, such that the precursor dose delivered in each pulse is constant.

9:00am **AM-WeM-5 Fast and Efficient Large Format ALD**, *D. Lindblad, Matthew Weimer, A. Damerson, J. Ragonesi*, Forge Nano; *O. Snef*, Sundew Technologies

Manufacturing of large format objects, such as solar absorbers, optics, electrolyzers, and sensors, can benefit from a multitude of ALD applications, from conformal protective coatings to device-enabling films. However, largeformat ALD has struggled to maintain a foothold in manufacturing for various reasons. Simply increasing the reactor volume and precursor delivery amount does not solve the problem, instead, it tends to exacerbate the limitations. Low precursor efficiency and slow cycle times in these large volume chambers can make the application of ALD financially nonviable in all but the most price insensitive applications. Additionally, thickness uniformity can suffer on such a large scale to compensate for increasing precursor costs. To improve the field of large format ALD, Forge Nano has technology that can deposit uniform films, in thickness and composition, over a scalable area. Our standard wafer ALD reactor is designed for a 200mm diameter deposition area, yet we have demonstrated that, through the judicious placement of individual precursor dosing valve stacks (*Figure 1*), one can easily scale to a 525mm diameter, or approximately 7 times larger deposition area (*Figure 2*). Standard processes for Al_2O_3 , TiO_2 , and SiO_2 have been demonstrated at 125°C to have short cycle times and reasonable uniformity, less than 5% full range thickness, across the entire 525mm diameter area. A full comparison of qualified processes, between the 200 and 525mm diameter tools, will be discussed in detail, with specific examples shown in *Table 1*. This effective scaling has been accomplished with a unique chamber design and custom, proprietary, ALD fast-pneumatic valves (FPV). These valves can be constructed into sets of continuous precursor delivery stacks and are capable of actuating at sub-1ms speeds in environments up to 200°C. Coupled with a unique chamber design, these FPVs enable low precursor consumption and enable short purge times. This work demonstrates that increasing the number of delivery points, using our proprietary valves and chamber design, will allow for the scaling of large-format ALD at production capable speeds while maintaining efficient use of precursor.

9:15am **AM-WeM-6 Mechatronic Spatial Atomic Layer Deposition for Closed-Loop Process Control**, *Daniel Penley, T. Cho, A. Brooks, L. Ranshoff, H. Park, E. Herman, O. Trejo, K. Barton, N. Dasgupta*, University of Michigan, Ann Arbor

Close-proximity atmospheric-pressure spatial atomic layer deposition (AP-SALD) holds promise to address the large-scale manufacturing needs of interfacial engineering at the nanoscale. A variety of system designs have been demonstrated and, notably, this technique has been industrialized for batch passivation of solar cells. Typically, close-proximity AP-SALD systems have fixed geometric parameters such as the gap size and relative alignment between the depositor head and substrate. While many systems have been designed to alter these parameters manually, there are few examples where digitally-controlled sensors and actuators are used to actively monitor and adjust geometric process parameters in real time. Therefore, there is limited scientific understanding of the importance of tolerances to these adjustable process parameters.

In this study, we describe a customized AP-SALD system that enables mechatronic control of key process parameters. A showerhead depositor design delivers precursors to the substrate surface while linear actuators and capacitance probes maintain gap size and relative alignment through multiple-axis tilt and closed-loop feedback. Two precision motorized stages control the substrate velocity and positioning, and independent control of gas flow rates and pressure is facilitated by a fluid control system. Digital control of process variables with active monitoring is facilitated with a software control package. We demonstrate and validate the system by performing AP-SALD of TiO_2 . We probe the dependence of film quality and uniformity on gap size variance, relative alignment tolerances, and two-axis printing, with results supported by finite-element analysis. In the future, this mechatronic design will enable experimental tuning of parameters which can inform multi-physics modeling to gain a deeper understanding of

Wednesday Morning, July 26, 2023

AP-SALD process tolerances, pushing this technology towards manufacturing at the large scale.

9:30am **AM-WeM-7 Spatial Atomic Layer Deposition: A New Revolution in Ultra-Fast Production of Conformal Optical Coatings**, *J. Rönn, S. Virtanen, P. Maydannik, K. Niiranen, Sami Sneek*, Beneq, Finland

Since its invention in 1974, atomic layer deposition (ALD) has shown tremendous performance in depositing thin film structures for various applications in physical, chemical, biological, and medical sciences. Due to the unique layer-by-layer growth mechanism of ALD, thin films with exceptional uniformity, conformality and quality can be deposited not only on planar substrates, but also on the most complicated surfaces. In optical systems, these properties, often absent in traditional physical or chemical vapor deposition techniques, are of utmost importance when it comes to depositing thin films on complex geometries, such as integrated waveguides, highly curved lenses, or micro lens arrays. However, this comes at a price; traditional ALD suffers from relatively low deposition rates (<30 nm/h), which has greatly limited ALD's application in many optical systems where thin films with thicknesses comparable to the wavelength of light are often required.

In this work, we present a new-generation ALD technology that revolutionizes the production of conformal optical coatings: the rotary spatial ALD. In rotary spatial ALD, the substrate is rotated across successive process zones to achieve ultra-fast and high-precision thin film deposition. We present our latest results obtained with our novel C2R plasma-enhanced rotary spatial ALD system, including the fabrication of SiO₂, Ta₂O₅ and Al₂O₃ with deposition rates reaching >1 µm/h. We also show that these materials exhibit low surface roughness (<1 Å RMS), low optical loss (<10 ppm @ 1064 nm) and excellent non-uniformity (<2% over 200 mm), ultimately paving the way for ALD to breakthrough in the optics industry once and for all.

9:45am **AM-WeM-8 Spatial ALD of Iridium Oxide Electro-Catalyst Layers for PEM Electrolysis**, *C. Frijters*, SparkNano, Netherlands; *J. Shen, M. Ameen*, TNO/Holst Center, Netherlands; *J. Greer*, Air Liquide Advanced Materials, Germany; *N. Blasco*, Air Liquide Advanced Materials, France; *Paul Poedt*, SparkNano, Netherlands

Proton Exchange Membrane Water Electrolysis (PEMWE) is a commonly used technique to produce green hydrogen from water. A massive up-scaling of PEMWE installations is required in the coming decade to keep up with the foreseen demand for green hydrogen. State-of-the-art PEM electrolyzers make use of iridium-based electro-catalyst layers with iridium loadings of 1-2 mg/cm². The high cost and limited availability of iridium will limit the scalability of PEMWE if the iridium loading cannot be reduced.

Atomic Layer Deposition can be used to apply thin and highly conformal IrO₂ films on porous substrates with atomic-scale control of the amount of material that is deposited. When applied to PEM electrolyzers, this can be used to significantly reduce the loading of IrO₂. Recently, IrO₂ catalyst layers for PEM electrolyzers applied by Spatial ALD with iridium loadings well below 0.1 mg/cm² have been reported [1]

We will present plasma enhanced Spatial ALD of iridium oxide and metallic iridium films using a new iridium precursor developed by Air Liquide Advanced Materials. The electro-catalytic efficiency and stability has been characterized using RDE, showing that IrO₂ films of just a few nanometer demonstrate an excellent catalytic activity and stability. IrO₂ films have been conformally deposited on high surface area titanium porous transport layers (PTL's) as well as Nafion membranes. First demonstrations on full PEM stack scale show that Spatial ALD can enable iridium loads 10-100x lower than the state of the art while demonstrating an excellent stability in accelerated stress tests.

We will also discuss how Spatial ALD of IrO₂ films can be up-scaled to mass production, with emphasis on efficiency or precursor utilization and precursor recovery for recycling.

[1]: <https://www.tno.nl/en/newsroom/2022/10/breakthrough-electrolyser-development/>

Author Index

Bold page numbers indicate presenter

— A —

Ameen, M.: AM-WeM-8, 4

Andsten, S.: AM-MoP-2, 1

— B —

Bachmann, J.: AM-MoP-10, 2

Barton, K.: AM-WeM-6, 3

Blasco, N.: AM-WeM-8, 4

Brooks, A.: AM-WeM-6, 3

— C —

Carnoy, M.: AM-MoP-10, 2

Chang, T.: AM-WeM-1, 3

Chen, M.: AM-WeM-1, 3

Cho, S.: AM-MoP-6, 1

Cho, T.: AM-WeM-6, 3

Chou, C.: AM-WeM-1, 3

— D —

Damerson, A.: AM-WeM-5, 3

Dasgupta, N.: AM-WeM-6, 3

Ding, J.: AM-WeM-4, 3

— F —

Frijters, C.: AM-WeM-8, 4

Funding la Cour, M.: AM-MoP-10, 2

— G —

Gonsalves, C.: AM-MoP-2, 1

Greer, J.: AM-WeM-8, 4

— H —

Han, S.: AM-MoP-6, 1

Hennessy, J.: AM-MoP-4, 1

Herman, E.: AM-WeM-6, 3

Hwang, J.: AM-MoP-1, 1

— J —

Jääskeläinen, S.: AM-MoP-2, 1

— K —

Kalanyan, B.: AM-WeM-3, 3

Kanjolia, R.: AM-MoP-3, 1

Khromchenko, V.: AM-WeM-3, 3

Kim, C.: AM-MoP-1, 1

Kim, Y.: AM-MoP-6, 1

Kummel, A.: AM-MoP-3, 1

Kundrata, I.: AM-MoP-10, 2

Kuo, C.: AM-MoP-3, 1

— L —

Larkiala, S.: AM-MoP-2, 1

Lee, H.: AM-MoP-1, 1

Lee, K.: AM-MoP-1, 1

Lee, S.: AM-MoP-1, 1

Lee, W.: AM-WeM-1, 3

Lee, Y.: AM-MoP-6, 1

Lindblad, D.: AM-WeM-5, 3

— M —

Maslar, J.: AM-WeM-3, 3

Maydannik, P.: AM-WeM-7, 4

Miikkulainen, V.: AM-MoP-2, 1

Moinpour, M.: AM-MoP-3, 1

— N —

Niiranen, K.: AM-WeM-7, 4

— P —

Park, H.: AM-WeM-6, 3

Penley, D.: AM-WeM-6, 3

Plakhotnyuk, M.: AM-MoP-10, 2

Poodt, P.: AM-WeM-8, 4

Puurunen, R.: AM-MoP-2, 1

— R —

Ragonesi, J.: AM-WeM-5, 3

Ranshoff, L.: AM-WeM-6, 3

Rask, J.: AM-MoP-2, 1

Rodríguez, R.: AM-MoP-4, 1

Rönn, J.: AM-WeM-7, 4

Rosenzweig, G.: AM-WeM-4, 3

— S —

Salonen, K.: AM-MoP-2, 1

Shen, J.: AM-WeM-8, 4

Simka, H.: AM-MoP-3, 1

Sneck, S.: AM-WeM-7, 4

Snef, O.: AM-WeM-5, 3

Song, G.: AM-MoP-1, 1

Stang, J.: AM-MoP-2, 1

Su, Y.: AM-MoP-11, 2

— T —

Trejo, O.: AM-WeM-6, 3

— V —

Varga, A.: AM-MoP-10, 2

Velasco, J.: AM-MoP-2, 1

Virtanen, S.: AM-WeM-7, 4

— W —

Wang, V.: AM-MoP-3, 1

Weimer, M.: AM-WeM-5, 3

Woodruff, J.: AM-MoP-3, 1

— Y —

Ye, J.: AM-WeM-4, 3

Yoo, K.: AM-MoP-1, 1