

## Functional Thin Films and Surfaces Room Palm 3-4 - Session MB3-ThM

### Low-dimensional Materials and Structures

Moderators: Tomas Kubart, Uppsala University, Sweden, Vladimir Popok, FOM Technologies, Denmark

8:00am **MB3-ThM-1 A Novel Platform for Topologically Protected Quantum Computation: Massively Parallel Self-Assembled Pentasilicene Nanoribbons**, *Guy Le Lay* [guy.lelay@univ-amu.fr], Aix-Marseille University, France

The novel platform, which we propose for the emergence of Majorana Zero Modes (MZMs), i.e., non-abelian anyons, which could be possibly braided for realizing topologically protected quantum computation, is based on massively parallel, high aspect ratio, spontaneously self-organized epitaxial nanoribbons (NRs) proximitized by a standard s-wave superconductor [1,2]. These highly perfect NRs are atom-thin pentasilicene nanoribbons (SiNRs) [3,4]. They could host distant MZMs at their extremities allowing for the creation of highly stable qubits preserved against external disturbances and environmental noise, hence, protected from decoherence. Clearly, the self-assembly of these defect-free SiNRs could be a distinct advantage over presently engineered or atom-by-atom constructed nanowires.

[1] G. Le Lay, M. Minissale, P. De Padova and A. Molle, *Il Nuovo Saggiatore* (2025), in press

[2] M. Minissale, P. Bondavalli, M. S. Figueira and G. Le Lay, *Journal of Physics: Materials*, 7, 031001 (2024)

[3] R. C. Bento Ribeiro *et al.*, *Scientific Reports* 13:17965 (2023)

[4] R. C. Bento Ribeiro *et al.*, *Phys. Rev. B* 105, 205115 (2022)

8:20am **MB3-ThM-2 Multilayers of Two-Dimensional (2d) Ti<sub>2</sub>B<sub>2</sub>CiX, Obtained from Selective Etching of 3DTiInB<sub>2</sub>**, *Rodrigo Ronchi* [rodrigo.ronchi@liu.se], Johanna Rosen, Linköping University, IFM, Sweden

Authors: Rodrigo M. Ronchi <sup>1</sup>, Emile Defoy <sup>3</sup>, Andrejs Petruhins <sup>1</sup>, Justinas Palisaitis <sup>2</sup>, David Portehault <sup>3</sup>, Jonas Björk <sup>1</sup>, P.O. Å. Persson <sup>2</sup>, Johanna Rosen <sup>1</sup>

With the rapid expansion of two-dimensional (2D) MXenes <sup>1</sup> efforts have been made to find other families of nanolaminated materials in both 3D and 2D. One such group is boron-containing compounds, known as MAB phases, where metal (M) and boron (B) layers are separated by layers of A-elements (A=Al, In, etc.). Due to their similarities with MAX phases, experimental attempts to etch these 3D materials into its 2D counterpart have primarily involved selective etching of the A-layers using hydrochloric <sup>2-5</sup> and hydrofluoric acids <sup>6,7</sup> and, more recently, Lewis acids/molten salts <sup>7-10</sup>.

Despite, the 2D Mo<sub>4/3</sub>B<sub>2-x</sub> boridene <sup>6</sup>, the subsequent experimental research has demonstrated that 2D metal borides are significantly more challenging to obtain than MXenes (metal carbides/nitrides) <sup>11</sup>. For instance, MoAlB phase was only partially etched to Mo<sub>2</sub>AlB<sub>2</sub><sup>2,4,8</sup>. Molten salt etching of Hf<sub>2</sub>InB<sub>2</sub> has resulted in complete oxidation to HfBO <sup>7</sup>, rather than forming a halogenated MBene. Additionally, unsuccessful acid etching trials have been reported for Fe<sub>2</sub>AlB<sub>2</sub> <sup>6</sup>, Mo<sub>5</sub>SiB<sub>2</sub> <sup>6</sup>, Ti<sub>2</sub>InB<sub>2</sub> <sup>12</sup> and Hf<sub>2</sub>InB<sub>2</sub> <sup>7</sup>. Further, while In atoms from Ti<sub>2</sub>InB<sub>2</sub> have been removed through a dealloying reaction <sup>12</sup>, TEM images and the *Cmcm* space group found suggests that the resulting TiB is a 3D material, instead of a 2D counterpart.

Here, we present the synthesis of 2D multilayer Ti<sub>2</sub>B<sub>2</sub>Cl<sub>x</sub>, obtained from molten salt (ZnCl<sub>2</sub>) etching of Ti<sub>2</sub>InB<sub>2</sub>. Energy Dispersive (EDS) and Electron Energy Loss Spectroscopies (EELS) confirm that indium is fully removed and replaced by chlorine atoms from the salt, leading to an increased c-lattice parameter and a corresponding shift in X-ray diffraction (XRD) peaks to lower angles. Furthermore, transmission electron microscopy (TEM) shows the laminated atomic structure, with chlorine terminations of the stacked 2D sheets. These XRD and TEM results are consistent with density functional theory (DFT) calculations. *In situ* XRD experiments further reveal that the 3D to multilayer (ml) 2D transformation occurs without any intermediate phase. Furthermore, our DFT results provide insights into the reaction mechanism governing this transformation.

This work not only establishes the 3D MAB phases as 2D MBene precursors but also unlocks new possibilities for engineering of 2D multilayer metal borides using molten salt etching, facilitating controlled surface chemistry. This work paves the way for a new class of functional nanomaterials with

Figure, references and authors information: supplemental document

8:40am **MB3-ThM-3 Conformal Multifunctional Polymeric and Inorganic Aerogel-Like Oxide Thin Films for Optical and Energy Applications by Plasma Technology**, *Gloria P. Moreno, Triana Czermak, Jose Obrero, Francisco J. Aparicio, Juan Ramón Sánchez-Valencia, Ana Borrás, Angel Barranco* [angelbar@icmse.csic.es], Institute of Materials Science, CSIC, Spain

INVITED

The Remote Plasma Assisted Vacuum Deposition (RPAVD) process is a versatile methodology for fabricating functional nanocomposites from non-chemically polymerizable organic functional molecules and functional metal coordination compounds. This approach combines the physicochemical reactions inherent to plasma polymerization processes with the vapor deposition of functional molecules tailored to the specific demands of target applications. The resultant cross-linked polymer films exhibit insolubility and exceptional thermal stability. These films can incorporate a precise concentration of nearly any thermally stable, photo-functional molecule. The method exhibits scalability at the wafer level and complete compatibility with solvent sensitive and delicate substrates. Initially, this process was applied to developing thin optical films and photonic devices, encompassing optical filters, photonic sensing chips, and lasing media. Nevertheless, the film properties can be fine-tuned for various other functional applications, such as creating controlled wetting and ice-retarding surfaces, antimicrobial coatings, and high performance dielectric ultrathin films. We will also showcase very recent results about the encapsulation of nanostructures on surfaces and its role in encapsulating and modifying perovskite solar cells. Additionally, we will present recent results about the development of conformal aerogel-like oxide nanostructures by combining RPAVD and plasma processing of metal-containing plasma polymers. These aerogel-like oxide films have straightforward applications in photonics, omniphobic surfaces, solar cells, and smart coatings.

References

- 1.- F.J. Aparicio *et al.* *Advanced Materials* 2011, 23 (6), 761-765
- 2.- M. Alcaire *et al.* *ACS Applied Materials Interfaces* 2017, 9, 8948,
- 3.- M. Alcaire *Advanced Functional Materials* 2019, 29, 1903535.
- 4.- J. Idígoras *et al.* *ACS Applied Mater. Interfaces* 2018, 10, 1801076.
- 5.- J. M. Obrero-Pérez, *Advanced Energy Materials* 2022, 2200812.
- 6.- J.M. Obrero-Pérez *ACS Applied Materials Interfaces* 2024, 16, 39745

9:20am **MB3-ThM-5 Cluster-assembled Computers**, *Paolo Milani* [paolo.milani@mi.infn.it], University of Milan, Italy

INVITED

Self-assembled nanoparticle or nanowire networks have recently come under the spotlight as systems able to obtain brain-like data processing performances by exploiting the memristive character and the wiring of the junctions connecting the nanostructured network building blocks [1]. Recently it has been demonstrated that nanostructured Au films, fabricated by the assembling of gold clusters produced in the gas phase, have non-linear and non-local electric conduction properties caused by the extremely high density of grain boundaries and the resulting complex arrangement of nanojunctions [2,3]. Starting from the characterization of this system, it has been proposed and formalized a generalization of the Perceptron model to describe a classification device based on a network of interacting units where the input weights are non-linearly dependent. This model, called "Receptron", provides substantial advantages compared to the Perceptron as, for example, the solution of non-linearly separable Boolean functions with a single device [4]. Here I will present and discuss the relevant aspects concerning the characterization and implementation of nanostructured networks fabricated by supersonic cluster beam deposition of gold and platinum clusters for neuromorphic computing and data processing applications [5,6].

[1] A Vahl, G Milano, Z Kuncic, SA Brown, P Milani, *J.Phys. D: Appl. Phys.* 57 (50), 503001 (2024)

[2] M. Mirigliano, *et al.*, *Neuromorph. Comp. Eng.* 1, 024007, (2021).

[3] G Nadalini, F Borghi, T Košutová, A Falqui, N Ludwig, P Milani *Scientific Reports* 13 (1), 19713 (2023)

[4] B. Paroli *et al.*, *Neural Networks* 166, 634, (2023)

[5] G Martini, E Tentori, M Mirigliano, DE Galli, P Milani, F Mambretti, *Frontiers in Physics* 12, 1400919 (2024)

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[6] S Radice, F Profumo, F Borghi, A Falqui, P Milani, *Advanced Electronic Materials*, 2400434 (2024)

10:20am **MB3-ThM-8 Analysis and 3D Modelling of Percolated Conductive Networks in Nanoparticle-Based Thin Films**, *Stanislav Haviar [haviar@kfy.zcu.cz]*, University of West Bohemia, Czechia; *Benedikt Prifling*, Ulm University, Germany; *Tomáš Kozák, Kalyani Shaji*, University of West Bohemia, Czechia; *Tereza Košutová*, Charles University, Czechia; *Šimon Kos*, University of West Bohemia, Czechia; *Volker Schmidt*, Ulm University, Germany; *Jiří Čapek*, University of West Bohemia, Czechia

Thin films composed of copper oxide nanoparticles (NP) were synthesized using a magnetron-based gas aggregation source (MGA), with nanoparticle sizes controlled by varying the exit orifice diameter. The 3D model of the synthesized NP-based was constructed and assessed.

(i) Comprehensive characterization of the nanoparticle-based thin films was performed using SEM, TEM, SAXS, and XRD to determine particle morphology, size distribution, porosity and others.

(ii) The obtained experimental data served as inputs for generating virtual 3D microstructure models through a data-driven stochastic hard sphere packing algorithm, incorporating factors such as particle size distribution, porosity, and vertical density profiles.

(iii) These virtual structures were refined to account for oxidation-induced swelling and film roughness, enabling the simulation of realistic conductive networks.

(iv) A computational model incorporating a simplified adsorption mechanism was developed to simulate oxygen adsorption effects on surface conductivity, and finite element method (FEM) simulations were conducted to calculate the electrical resistivity of the modelled networks under varying oxygen partial pressures.

(v) The simulated resistivity values were validated against experimental measurements obtained via four-point probe resistivity techniques at 150°C under different oxygen concentrations, demonstrating both qualitative and quantitative agreement.

[1] Haviar; S., Prifling B.; Kozák et al. *Appl. Surf. Sci. Adva.* – submitted – 2024

[2] Shaji, K., Haviar, S., Zeman P. et al. *Surf. Coatings Technol.* 2024, 477

[3] Batková; Kozák, T.; Haviar, S.; et al. *Surf. Coatings Technol.* 2021, 417

[4] Haviar, S.; Čapek, J.; Batková, Š.; et al. *Int. J. Hydrogen Energy* 2018, 43

10:40am **MB3-ThM-9 Tailoring of Nanoparticle Deposition Rate and Film Structure Through Substrate Biasing: Enabling Sputtering-Based Synthesis of Novel Catalyst Materials**, *Dominik Gutnik [dominik.gutnik@unileoben.ac.at]*, *Florian Theodor Knabl*, Montanuniversität Leoben, Austria; *Prathamesh Patil*, CEST GmbH, Austria; *Christine Bandl*, Montanuniversität Leoben, Austria; *Tijmen Vermeij*, *Daniele Casari*, Empa, Swiss Federal Laboratories for Materials Science and Technology, Thun, Switzerland; *Michael Burtscher*, *Christian Mitterer*, Montanuniversität Leoben, Austria; *Christian M Pichler*, CEST GmbH, Austria; *Barbara Putz*, Montanuniversität Leoben, Austria

Metallic nanoparticles (NPs) exhibit intriguing properties as a consequence of their spatial confinement and their high surface-to-volume ratio. A topic rising in importance is the utilization of NPs as catalysts for energy conversion and storage. To facilitate more advanced use of NPs, a thorough understanding of their synthesis-structure-property relations is crucial.

In this study, the effect of different substrate biases on the deposition of size-selected Cu NPs, fabricated via Magnetron Sputtering Inert Gas Condensation (MS-IGC) in a so-called Haberland system, is analyzed. NPs nucleate and grow within the aggregation zone (usually pressures of 10 to 100 Pa), collect charge through plasma interactions and are accelerated by adiabatic expansion upon exiting the aggregation zone through an orifice. The charge they collect enables analysis and manipulation of nanoparticles through a Quadrupole Mass Spectrometer (QMS) before deposition on the substrate.

With this approach, Cu NPs with a diameter of 1.8 nm and 8 nm were filtered and accelerated towards the substrate with positive bias voltages of 0, 300 and 1000 V. In-situ QMS data reveals a significant increase of the NP-flux with higher biases, especially for smaller NP-diameters. Furthermore, changes in the morphology of the resulting thin films which were deposited for up to 45 minutes are observed with Scanning Electron Microscopy and changes in surface coverage and porosity are studied with X-ray Photoelectron Spectroscopy and Low-Energy Ion Scattering Spectroscopy.

Our results show that with rising bias voltages, the NP deposition rate estimated through QMS increases by 32% for the 8 nm diameter NPs, and the morphology of the resulting thin film shifts towards more densely packed structures, attributed to the higher energy of the NPs on impact [1]. An alternative method of NP synthesis in the form of hollow cathode sputtering will also be presented as a high throughput technique. With this technique, orders of magnitude higher NP deposition rates with position-dependent morphology can be obtained. These findings could facilitate the deposition of NP-based films with higher efficiency and with tailored morphology, making this technique more attractive for e.g. the synthesis of catalysts.

[1] Knabl, F., Gutnik, D., Patil, P., Bandl, C., Vermeij, T., Pichler, C. M., Putz, B., & Mitterer, C. (2024). Enhancement of copper nanoparticle yield in magnetron sputter inert gas condensation by applying substrate bias voltage and its influence on thin film morphology. *Vacuum*, 230, 113724. <https://doi.org/10.1016/j.vacuum.2024.113724>

11:00am **MB3-ThM-10 Tailoring Microstructure and Composition of Composite CuO/WO<sub>3</sub> Nanoparticle-Based Thin Films for Enhanced H<sub>2</sub> Gas Sensing**, *Kalyani Shaji [kalyanis@kfy.zcu.cz]*, *Stanislav Haviar*, *Petr Zeman*, *Michal Procházka*, *Radomír Čerstvý*, *Jiří Čapek*, University of West Bohemia - NTIS, Czechia

The conductometric gas sensors operate by modulating the electrical conductivity of the sensing material through adsorption-desorption reactions between the target gas and the sensor surface. Metal oxide semiconductors (MOS) are conductometric materials highly sensitive to oxidizing and reducing gases. In addition, composite MOS-based materials may further benefit from formed heterojunctions potentially significantly improving the sensitivity. Our focus is to develop advanced hydrogen-gas sensing materials composed of a mixture of p-type CuO and n-type WO<sub>3</sub> nanoparticles (NPs) with optimized microstructure of the film and volumetric ratio of CuO to WO<sub>3</sub> NPs in the film for enhanced H<sub>2</sub> gas sensing.

The NP-based thin films were synthesized using a magnetron-based gas aggregation source in Ar+O<sub>2</sub> gas mixture. First, effect of thermal annealing on the microstructure (i.e., NPs diameter, formed necks, porosity) of the films was studied since gas sensing materials are usually operated at elevated temperatures (up to 400°C). The CuO, WO<sub>3</sub> and their composite (1:1 volumetric ratio) samples were annealed at temperatures in the range 200 - 400°C in synthetic air and subsequently thoroughly investigated using various characterisation techniques such as SEM, XRD, XPS, and Raman spectroscopy. Significant changes in particle size were observed in the case of CuO-based material, while WO<sub>3</sub>-based and composite materials exhibited minor microstructural changes, even at elevated temperatures. Notably, at 400°C, the composite crystallized into a novel phase. Second, the volumetric ratio of CuO to WO<sub>3</sub> NPs in the films was optimized to maximize the response of the material. We demonstrate that a synergetic effect is reached when an optimum number of p-n heterojunctions is established in the material providing enhanced response of the composite film compared to the films formed by single-material NPs.

This study highlights the crucial role of thermal treatment in influencing NP microstructure, offering insights into stabilizing and tuning NP-based thin films for enhanced gas sensing. Additionally, the optimized ratio of CuO and WO<sub>3</sub> NPs within the composite improved H<sub>2</sub> sensing performance by promoting optimal p-n heterojunction formation, demonstrating that precise compositional control can significantly boost the sensitivity of nanostructured systems.

11:20am **MB3-ThM-11 Influence of Pretreatment and Deposition Parameters on Carbon Nanotubes Synthesized Directly on Oxidized Steel Substrates via Pulsed DC PACVD**, *Manuel C. J. Schachinger [manuel.schachinger@fh-wels.at]*, *Francisco A. Delfin*, University of Applied Sciences Upper Austria; *Bernhard Fickl*, *Bernhard C. Bayer*, Vienna University of Technology, Austria; *Andreas Karner*, *Johannes Preiner*, *Christian Forsich*, *Daniel Heim*, University of Applied Sciences Upper Austria; *Bernd Rübiger*, *Christian Dipolt*, *Thomas Müller*, RÜBIG GmbH & Co KG, Austria

Carbon nanotubes have recently attracted considerable attention due to their distinct qualities such as elevated strength-to-weight ratio, excellent thermal conductivity, high aspect ratio and special electronic and optical properties. However, the widespread use of CNTs is limited by their costly production, partly due to the laborious substrate-catalyst preparation involving expensive transition metals like Ni or Co, which must be sputtered and sintered to form sufficient growth sites on the substrate material. To avoid the costly and time-consuming pretreatment, it was shown that direct growth of carbon nanotubes on steel substrates is possible by

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application of a simple surface oxidation step prior to the synthesis process. The aim of this work is to optimize the oxidation pretreatment of the steel in a way that specific tailoring of the nanotube properties such as diameter, length and morphology becomes possible. To achieve this, cylindrical EN 1.4301 (AISI 304) steel samples were subjected to an oxidation step in air at atmospheric pressure for 15 s, 3 minutes and 15 minutes at 300, 400 and 500 °C, respectively. Subsequently, the synthesis process was carried out in the PACVD 40/60 system (RÜBIG, Austria) utilizing a unipolar pulsed DC discharge. Power density was varied between 50 and 100 W/m<sup>2</sup>. Ar, H<sub>2</sub> and C<sub>2</sub>H<sub>2</sub> gas concentrations were 67 vol.-% 32 vol.-% and 1 vol.-%, respectively. The pressure was 200 Pa and synthesis time was 1 h. The obtained CNTs as well as the oxidized steel surfaces after pretreatment were then analysed using SEM, EDS, TEM, AFM, XPS and Raman spectroscopy. SEM images showed the formation of a high-density forest of CNTs fully covering the steel surface for substrate-oxidation times greater than 15 s. Tube diameter increased with increasing oxidation times and temperatures from 20 to 200 nm. TEM revealed the formation of bamboo-like CNTs involving a tip growth-mechanism. Raman spectroscopy showed the characteristic D, G and D' peaks, with a large I(D)/I(G) ratio, indicating an elevated degree of disorder. AFM revealed significant RMS roughness and morphology variations of the oxidized steel surfaces dependent upon oxidation time and temperature, which were correlated with the nanotube length and diameter. In summary, it was possible to achieve CNTs with tailored properties only via the variation of the surface oxidation step prior to the synthesis, achieving a cost-effective production process that can easily be adapted to the specific requirements of the applicator.

11:40am **MB3-ThM-12 The Influence of Magnetic Field on the Cluster Growth in a Magnetron Sputtering Gas Aggregation Source**, *Joao Coroa*, Teer Coatings Ltd, UK; *Giuseppe Sanzone* [[giuseppe.sanzone@teercoatings.co.uk](mailto:giuseppe.sanzone@teercoatings.co.uk)], Teer Coatings Ltd., UK; *Tibor Höltzl*, Furukawa Electric Institute of Technology, Hungary; *Hailin Sun*, Teer Coatings Ltd., UK; *Ewald Janssens*, KU Leuven, Belgium; *Jinlong Yin*, Teer Coatings Ltd., UK

Clusters produced by physical methods in gas phase have yet to see widespread adoption due to low deposition rates, despite the benefits that they could bring to many applications. It's demonstrated in this study that unbalancing the magnetic field configuration of a magnetron within a sputtering gas aggregation source significantly enhances dimer formation and subsequent cluster growth. Based on experimental results obtained with four magnetic field configurations, along with ab initio simulations, we discussed various scenarios for dimer formation and proposed that the contribution of ArPd<sup>+</sup> is essential for an increase in cluster throughput. We analysed the resulting plasma spatial distribution and demonstrated that the selected magnetic field configuration significantly influences the lifetime of ArPd<sup>+</sup> particles. When their lifetime is long, more ArPd<sup>+</sup> can react with another metal atom (Pd) and form other stable complexes (Pd<sub>2</sub>, Pd<sub>2</sub><sup>+</sup> or ArPd<sub>2</sub><sup>+</sup>), a critical first step in cluster growth, increasing cluster throughput by a factor of 150-fold. The proposed mechanism might be material-independent as other metal-argon dimers (ArCu<sup>+</sup>, ArTi<sup>+</sup> and ArCo<sup>+</sup>) have also been reported in the literature.

12:00pm **MB3-ThM-13 Tracking the Evolution of Ag Nanoparticle Solutions Upon Atmospheric Exposure Using a Combined Spectroscopic Approach**, *Héloïse Lasfargues* [[lasfargues@mch.rwth-aachen.de](mailto:lasfargues@mch.rwth-aachen.de)], *Lilli Charlotte Freymann*, *Jochen M. Schneider*, *Clio Azina*, RWTH Aachen University, Germany

With nanoparticles (NPs) finding increasing use in various fields such as the biomedical industry or catalysis, sputtering onto liquids (SoL) has attracted interest over the last decade as a single step method for NP production, requiring only a target and a host liquid. Beyond NP properties like size and shape, colloidal stability is of high importance and is primarily determined by the combination of NP material and liquid. The current understanding of NP stability in solutions produced by SoL being limited, further research is needed to thoroughly describe the complex interactions occurring between NP and liquid host. In this context, silver (Ag) NP solutions were produced by magnetron sputtering onto canola oil and their stability under atmospheric exposure was investigated by combining infrared (IR), UV-visible (UV-vis) and X-Ray photoelectron spectroscopy (XPS) measurements, with transmission electron microscopy observations (TEM). A color change from dark brown to light orange was observed within 35 days of atmospheric exposure of the as-synthesized solutions. This color change was accompanied by the formation of hydroperoxides, as revealed by IR spectroscopy and XPS. The observation of peroxides signals oil oxidation, suggesting that the latter was promoted by the presence of Ag NPs upon oxygen incorporation. In terms of size, more than 90% of the NPs were < 5

nm in diameter in the as-synthesized solutions, with an average size of 2.9 ± 2.3 nm. Upon atmospheric exposure the proportion of NPs > 5 nm in diameter increased by ~100% after 35 days, indicating that the NPs continue to grow in the solution. In addition to size variations, TEM analysis suggests the formation of Ag-Ag<sub>x</sub>O Janus-type NPs in large proportion and therefore partial oxidation of the produced NPs upon atmospheric exposure. These observations were correlated with UV-vis measurements, where a red shift of ~ 15 nm of the localized surface plasmon resonance and an absorbance decay of the solutions after 35 days was detected. Finally, the comparison of XPS spectra of the pure oil with as-synthesized and aged NP solutions revealed the formation of carboxylate groups (-COO<sup>-</sup>) and their interaction with Ag in the near-surface volume probed.

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