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Developing New 2D Materials and Heterostructures for Printed Digital Devices

2D-PRINTABLE - Deliverable report

D2.2. – Functionalization of 2D materials beyond TMDs

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Project Scientific Abstract

The 2D-PRINTABLE project aims to integrate sustainable large-scale liquid exfoliation techniques with theoretical modelling to efficiently produce a wide range of new 2D materials (2DMs), including conducting, semiconducting, and insulating nanosheets. The focus includes developing the printing and liquid phase deposition methods required to fabricate networks and multicomponent heterostructures, featuring layer-by-layer assembly of nanometer-thick 2DMs into ordered multilayers. The goal is to optimize these printed networks and heterostructures for digital systems, unlocking new properties and functionalities. The project also seeks to demonstrate various printed digital devices, including proof-of-principle, first-time demonstration of all-printed, all-nanosheet, heterostack light-emitting diodes (LEDs). In conclusion, 2D-PRINTABLE will prove 2D materials to be an indispensable material class in the field of printed electronics, capable of producing far-beyond-state-of-the-art devices that can act as a platform for the next generation of printed digital applications.

Public summary

In this study, we explored novel chemical functionalization strategies for two distinct classes of materials: layered compounds (CrSBr, $Bi_2Se_2O_5$, VOCl, and PrOCl) and metal diborides (MgB_2, ZrB_2, and CrB_2). Our work contributes to advancing material design for applications in electronics, energy storage, catalysis, and protective coatings.

For the layered materials (CrSBr, $Bi_2Se_2O_5$, VOCl, and PrOCl), we focused on non-covalent functionalization using a range of surfactants, including Tween 20, oleic acid, oleylamine, cetyltrimethylammonium bromide (CTAB), and sodium dodecyl sulfate (SDS). Non-covalent functionalization enables the modification of material surfaces without altering their intrinsic structure, allowing for the tuning of properties such as optical characteristics through dielectric interactions. Additionally, this approach provided insights into the colloidal stability of these materials in various solvents, an aspect that has not been thoroughly studied for these compounds. This opens up new opportunities for developing stable dispersions of these materials.

For metal diborides (MgB_2, ZrB_2, and CrB_2), we investigated oxidation pathways and the impact of oxygen exposure on their properties. These materials are known for their excellent conductivity and high-temperature stability, but oxidation can lead to undesirable degradation of their performance. We identified the oxidation processes and developed strategies to prevent these transformations through controlled inert processing techniques. By understanding how these materials interact with oxygen, we can design more stable and durable metal diboride-based materials for applications that require resistance to environmental degradation, such as in high-performance electronics and coatings.

In both cases, we also developed optimized synthesis and processing techniques to fabricate these materials with high purity and controlled morphology, ensuring reproducibility and stability. This comprehensive approach of combining functionalization strategies with advanced material fabrication lays the foundation for future applications, enhancing the properties of both layered materials and metal diborides.

Our findings highlight the distinct advantages of non-covalent functionalization for layered materials and oxidation resistance strategies for metal diborides. By leveraging these techniques, we provide valuable pathways for enhancing material properties and performance in a range of cutting-edge technologies.

Instructions for filling in the title page and header/footer

1. On the title page:
    a. All items need to be filled out
    b. Where NAME is stated a person needs to be mentioned
    c. Where ORGANISATION is stated the **short** name for the organisation needs to be provided
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2. Complete the Footer
    a. In the Footer the Dx.xx Deliverable number followed by the title of the Deliverable
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    e.g., 2D-PRINTABLE D1.1_PU_TCD_v1.docx

Contents

1 Introduction

Exfoliation is a critical process in the development of two-dimensional (2D) materials, as it enables the isolation of individual nanosheets (NSs) from bulk materials. This process not only provides access to unique properties inherent to the 2D nature of these materials but also facilitates their integration into a wide range of applications, from electronics to energy storage. In this report, we focus on the non-covalent functionalization of 2D materials, particularly those produced by liquid-phase exfoliation (LPE) in WP1. The importance of exfoliation lies in its ability to produce high-quality, single- or few-layered nanosheets with well-defined surface characteristics, which can significantly enhance the properties of the materials, such as conductivity, mechanical strength, and surface reactivity.

1.1 Non-covalent functionalization

Preliminary results from WP1 on the non-covalent functionalization of novel beyond-TMD 2D materials are presented here. These results, planned within T2.3, represent a pioneering step in identifying functionalization methods that are compatible with the exfoliated nanosheets, thus contributing to O2.1 and O2.2. Non-covalent functionalization is particularly important because it allows for surface modifications without altering the inherent structure of the material, preserving its intrinsic properties while enabling tailored modifications. This flexibility is crucial for achieving optimal performance in various applications, including sensors, catalysis, and energy devices.

The non-covalent functionalization of 2D materials is essential for understanding the interactions between nanomaterials and solvents, as well as between nanomaterials and surfactants. These interactions play a key role in determining the colloidal stability of the materials, which is a fundamental requirement for their successful dispersion in liquids, processing, and integration into functional composites. By studying these interactions in detail, we gain insights into how different surfactants and solvents influence the stability, dispersion quality, and overall behavior of the nanosheets. These insights enable us to design materials with specific interactions that are tailored to maintain long-term stability in solution, a critical factor for scaling up production and ensuring the reproducibility of nanomaterial-based devices.

Moreover, non-covalent functionalization opens the door to optimizing the functionality of 2D materials for specific applications. For example, the use of surfactants that induce specific electrostatic or van der Waals interactions can fine-tune the optical, electronic, or mechanical properties of the material, making them suitable for targeted applications. The ability to engineer the surface chemistry through non-covalent methods provides a versatile approach to modifying material properties while avoiding the potential drawbacks of covalent functionalization, such as structural defects or changes in electronic behavior.

By deepening our understanding of nanomaterial/solvent and nanomaterial/surfactant interactions, we can design and produce 2D materials that meet the specific needs of various industries, from electronics to environmental applications. This report outlines the key findings and methodologies developed within WP1 and T2.3, showcasing the potential of non-covalent functionalization as a powerful tool in nanomaterial design.

1.2 Covalent functionalization

1.2.1 Understanding Decomposition Pathways for Efficient Functionalization

One of the key challenges in the functionalization of 2D materials is ensuring that the desired modifications occur without undesired side reactions or degradation of the material. This is particularly important for covalent functionalization approaches, where the introduction of functional groups may inadvertently lead to the decomposition or alteration of the nanomaterial's structure, negatively impacting its performance. Understanding the decomposition pathways that can occur during functionalization is crucial for avoiding these issues and ensuring that the process is both efficient and selective.

Decomposition pathways typically arise when chemical reactions with the surrounding environment lead to the breakdown of the material or the functional groups being introduced. For instance, oxidation reactions or reactions with reactive solvents can lead to the formation of unwanted by-products that may affect the stability and integrity of the nanomaterial. By carefully studying these pathways, we can identify the conditions under which decomposition is most likely to occur and design functionalization strategies that minimize these risks. This understanding allows us to optimize processing conditions, select the right solvents and surfactants, and avoid conditions that could lead to material degradation.

Moreover, by designing more controlled and selective functionalization routes, we can tailor the properties of the nanomaterial without compromising its structural integrity. This is crucial for ensuring that the material retains its intended functionality in applications such as catalysis, energy storage, and sensing, where stability and performance are paramount.

1.2.2 Tailoring Chemical Interactions for Enhanced Functionalization

A deeper understanding of the chemical interactions between the nanomaterial and its surrounding environment is key to achieving precise and efficient functionalization. These interactions determine how the material behaves in solution, how it interacts with other molecules, and how functional groups are introduced onto its surface. By studying these interactions, we can design functionalization strategies that enhance the desired properties of the nanomaterial while avoiding unwanted side effects.

For example, the choice of surfactant or solvent can greatly influence the surface charge, hydrophobicity, and overall chemical reactivity of the 2D material. Surfactants that interact strongly with the material’s surface can help stabilize nanosheets in solution, prevent aggregation, and promote uniform functionalization. The interactions between the surfactant and the material surface can also influence how easily functional groups can be introduced, whether through non-covalent interactions (e.g., van der Waals or electrostatic interactions) or covalent bonding. By tailoring these interactions, we can control the extent and specificity of the functionalization, ensuring that the right properties are imparted to the material for its intended application.

Additionally, by understanding how different surfactants and solvents influence the surface chemistry of the nanomaterial and their interaction with suitable electrophiles, we can optimize the functionalization process for different application needs, such as improving colloidal stability for various applications, for example enhancing conductivity for electronic devices. Fine-tuning the chemical environment surrounding the nanomaterial allows for a more targeted and efficient functionalization process, reducing waste and enhancing material performance.

1.2.3 Modifying the Electronic Structure of Nanomaterials

Covalent functionalization is a powerful method for modifying the electronic structure of nanomaterials, providing a way to tune their properties without permanently altering their fundamental characteristics. By introducing covalent bonds with specific functional groups, we can influence the charge distribution, electronic band structure, and overall reactivity of the material. These modifications can have significant effects on the material’s performance, particularly for applications requiring precise control over electronic properties.

For example, the attachment of electron-withdrawing or electron-donating groups to the nanomaterial’s surface can modify the local charge density, thereby altering its electronic behavior. This type of functionalization can change the material’s conductivity, work function, and bandgap, which are all crucial factors for applications that rely on specific electronic characteristics. The ability to fine-tune these properties through covalent bonds offers a pathway for creating nanomaterials with enhanced or tailored electronic structures.

Covalent interactions can also influence the density of states near the Fermi level, which governs the material's electrical conductivity and optical properties. For instance, functionalizing nanomaterials with groups that induce specific electronic states can improve charge transfer or increase the material’s reactivity toward specific reactions. This enables the creation of materials with highly controlled and predictable electronic behavior, opening the door to designing materials with exactly the right properties for their intended application.By carefully selecting the functional groups and controlling the conditions under which covalent bonds are formed, we can engineer materials with optimized electronic properties, ensuring that they exhibit the desired characteristics without compromising the integrity of the material’s structure.

2 Methods and core part of the report

2.1 Background

Sonication-assisted LPE has been used in the production of a range of novel non-TMD 2D materials, previously predicted theoretically (M3 and D1.2) and synthesized in the bulk form (M2; D1.3) in WP1.

2.2 Procedures

The non-covalent functionalization of novel 2D NSs has been studied by performing the LPE of bulk materials in the presence of additives selected from the group of cationic, anionic and non-ionic surfactants. The bulk materials were exfoliated in the solvents found effective for the exfoliation without the presence of any additives, i.e., isopropanol and chloroform in the present examples. The LPE was conducted for 6 hours and the vials with the exfoliated materials were then monitored for one week to evaluate possible signs of aggregation/sedimentation of the exfoliated material. For each material included in the experiments, for comparison purpose, a control sample was exfoliated in the respective solvent without any additives.

3 Results & Discussion

3.1 Results

Four materials with varying crystal structures and compositions were tested (CrSBr, Bi_2SeO_5, VOCl and PrOCl) along with five potential surfactants such as Tween 20, Oleic acid, oleylamine, cetyltrimethylammonium bromide (CTAB) and sodium dodecyl sulfate (SDS). It was found that CTAB is particularly effective in stabilizing NSs of CrSBr, suggesting that the surfactant molecules may be attached to cleavage Br planes of the crystal. On the contrary, this surfactant had no notable effect on any of the other materials in which the cleavage planes are expected to be halide-terminated. Oleylamine had visible positive effect on the colloidal stability of the exfoliated Bi_2TeO_5 and VOCl NSs, while some minor effect of SDS on the stability of PrOCl NSs was observed only within a couple of hours following the exfoliation.

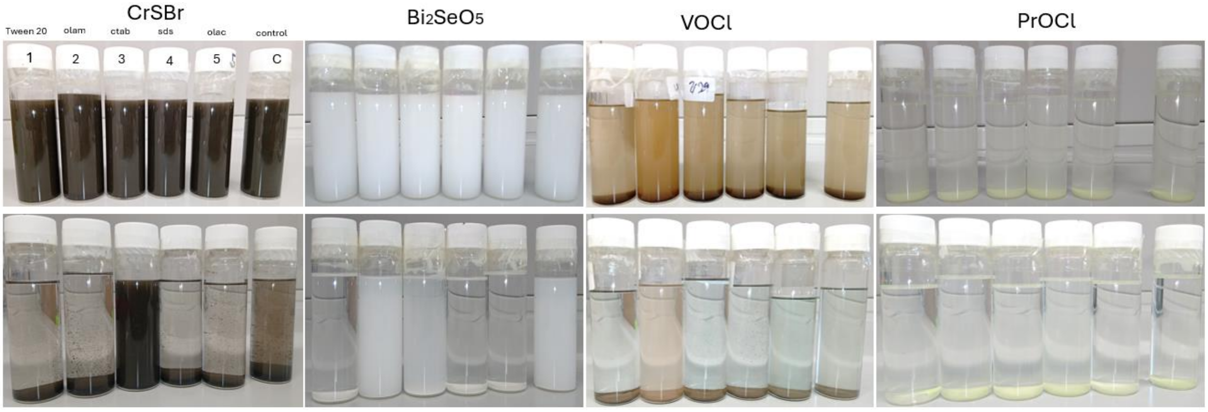

Figure 1. The suspensions of the exfoliated materials in the presence of surfactants immediately after exfoliation (top row) and after five days of sedimentation following the exfoliation (bottom row).

These observations highlight the complex nature of the interplay between surface terminations of the exfoliated NSs, types of surfactants and the surrounding solvent. Future work on this topic will include additional materials, all of which are being screened by BeD for electrochemical applications. In addition we evaluate the effect of additives on the process of the exfoliation itself (quality of the exfoliated material and quantity of the flakes satisfying mono/few layer thickness criteria) and contrasted with functionalization performed post-exfoliation.

We further demonstrate the exfoliation of metal diborides—MgB_2, CrB_2, and ZrB_2—into nanosheets under both ambient and inert conditions, providing critical insights into their exfoliation, stability, and functionalization. Metal diborides, being a family of layered non-van der Waals crystals with semimetallic properties, represent promising candidates for nanosheet-based applications. However, the tendency of these materials to undergo oxidation during exfoliation poses a significant challenge. The ability to control and mitigate this degradation is crucial for ensuring the retention of their intrinsic properties in solution-phase processing and for scalable fabrication of functional devices.

The exfoliation process was carried out via sonication-assisted liquid-phase exfoliation (LPE) in isopropanol (IPA), with two sets of samples being processed: one under ambient conditions and the other under inert conditions. Both exfoliation techniques yielded nanosheets with an average lateral size of approximately 350 nm, as confirmed through scanning electron microscopy (SEM). Despite the similarity in nanosheet size, significant differences were observed in the material's stability and oxidation states post-exfoliation.

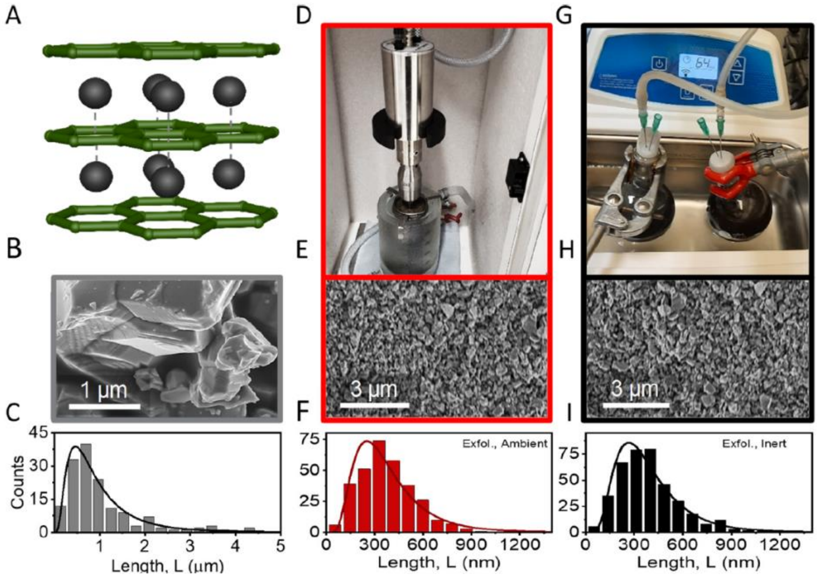

Figure 2. SEM comparison of metal diboride nanoplatelets made under ambient and inert conditions. (A) Hexagonal AlB2-type crystal structure, typical for layered metal diborides such as MgB2. Layers of negatively charged boron sheets (represented by a green hexagonal lattice) are sandwiched between Mg2+ ions (black spheres and square lattice). (B, C) Representative SEM image (B) and size distribution of MgB2 crystallites (C) in the powder used as a starting material. (D) Photograph of the setup used for exfoliation under ambient conditions. (E, F) Scanning electron microscopy (SEM) image showing an overview of drop cast nanosheets after exfoliation (E) using the setup shown in panel D and the corresponding size distribution of MgB2 nanosheets determined from SEM imaging (F). (G) Photograph of the setup used for exfoliation under inert conditions. (H, I) SEM image showing an overview of drop cast nanosheets after exfoliation (H) using the setup shown in panel G and corresponding size distribution of MgB2 nanosheets determined from SEM imaging (I).

The ambient-exfoliated samples exhibited pronounced oxidation, as evidenced by changes in optical absorbance spectra and energy dispersive X-ray (EDX) spectroscopy. A distinct peak at approximately 220 nm in the optical absorption spectra, typically associated with magnesium oxide (MgO), was observed in the ambient-exfoliated samples, whereas no such feature appeared in samples exfoliated under inert conditions. This indicates that ambient conditions introduce substantial oxidation to the material, whereas the inert environment preserves the pristine properties of the metal diboride nanosheets.

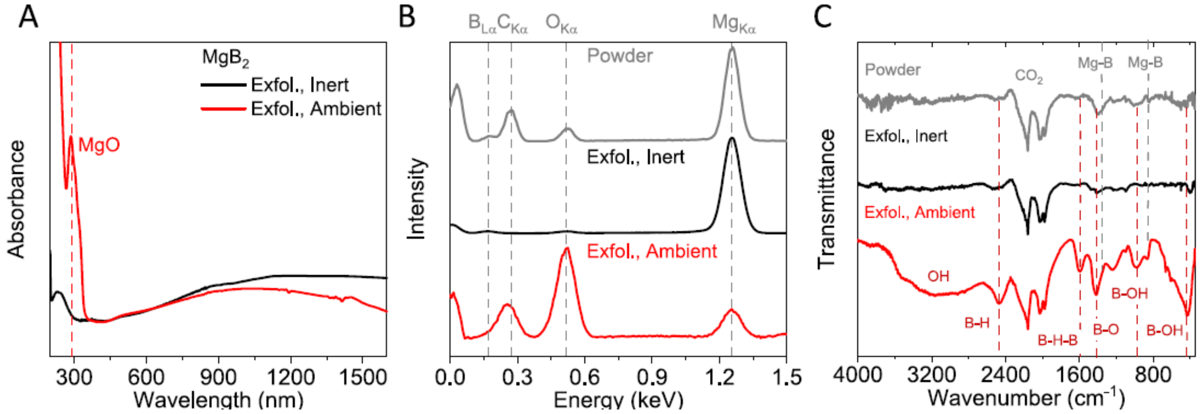

Figure 3. Spectroscopic analysis of MgB2 nanoplatelets prepared under inert conditions. (A) Absorbance measurements on nanosheet dispersions made under ambient (red) and inert conditions (black). An additional peak associated with MgO is found for the ambient sample. (B, C) EDX (B) and IR-transmittance (C) measurements for the starting material (gray) and nanosheets after exfoliation under ambient (red) and inert (black) conditions. The measurements suggest that both the starting and ambient-exfoliated materials contain oxide species, which is not evident in the inert exfoliated sample.

3.1.1 Impact of Oxidation on Material Properties

The degradation of material properties due to oxidation is a significant concern for the fabrication of functional thin films from exfoliated metal diborides. To study the stability of these materials, both the exfoliated dispersions and Langmuir-type deposited films were exposed to ambient conditions, and their degradation was monitored over time through optical and electrical measurements. For the dispersions, changes in the optical absorbance spectra suggested that oxidation occurred gradually over time, as indicated by the rise in the UV-vis peaks characteristic of oxidized magnesium species. Similarly, the Langmuir-deposited films exhibited time-dependent changes in their photospectroscopic profiles, with a marked degradation of the UV absorbance, confirming that exposed films were also susceptible to oxidation.

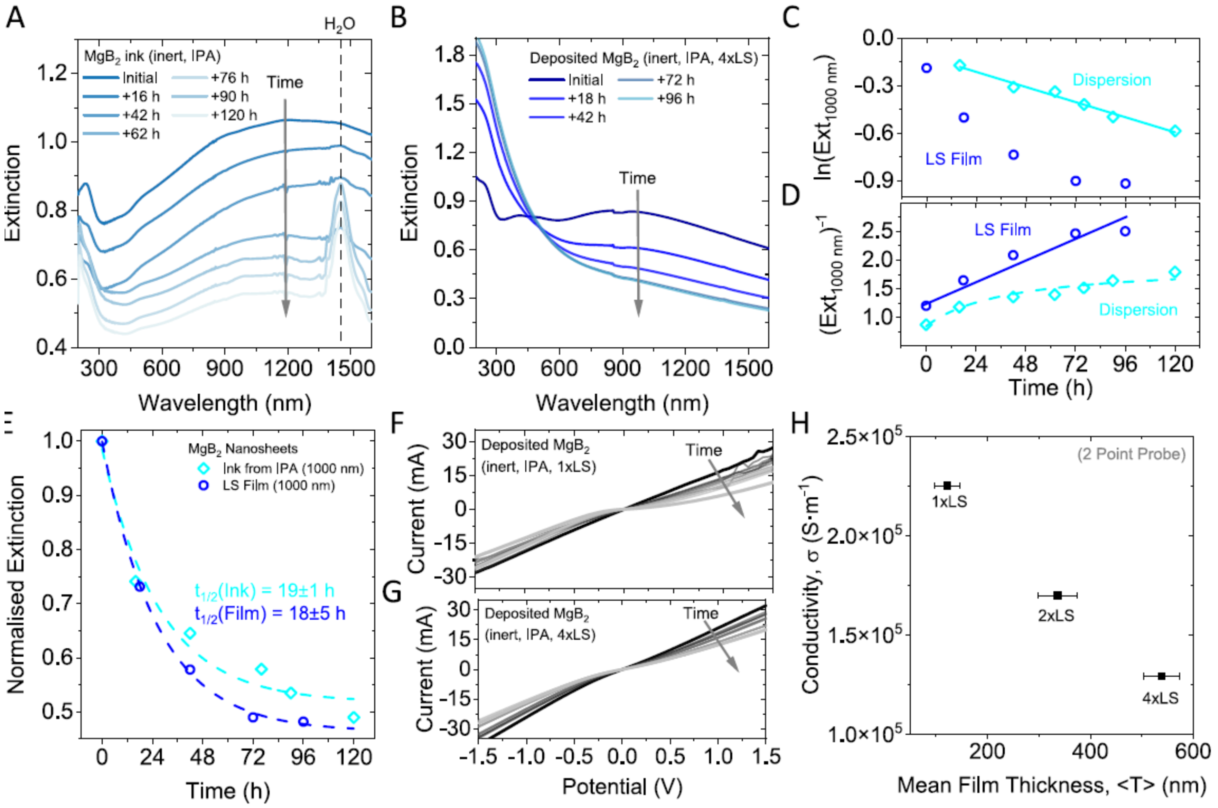

Figure 4. Nanomaterial stability. (A, B) Change in the extinction of liquid exfoliated MgB2 nanoplatelets in dispersion (A) and after thin film deposition on optical glass (B) as a function of time exposed to ambient conditions. In both cases, a systematic decrease in the overall response is observed for all wavelengths above 450 nm, while the signal below 450 nm undergoes changes in the spectral profile and shows an increased optical density in the UV region, which is indicative of oxide formation. Further, a peak that can be attributed to water forms over time in the nanosheet ink. (C, D) Kinetic plots for rate law analysis of the extinction measurements shown in panels (A, B). Panel (C) shows the change in the natural logarithm of the optical density at 1000 nm, and panel (D) shows the inverse optical density at 1000 nm as a function of time. While the lines indicate a reasonable agreement with a first-order rate law for the dispersed nanomaterial (C) and a second order rate law for the deposited material (D), the analysis is not unambiguous and is discussed in further detail in the SI. (E) Change in the extinction of the nanosheet ink and thin film at 1000 nm as a function of time. The data can be described by an empirical exponential fit, which allows determination of the material's half-life. The data for both individual data sets agrees well, and fitting suggests a nanomaterial half-life of 19 ± 1 h and 18 ± 5 h for the nanosheet ink and thin film, respectively. (F, G) I–V characteristics as a function of time for two sets of samples measured after deposition of a single layer (F) and after deposition of 4 layers (G). In both cases, similar systematic changes are observed: the initial ohmic response changes to a more rectifying transport behavior, which is consistent with the formation of an oxide layer. (H) Conductivity of the same MgB2 films measured as a function of the mean film thickness determined by profilometry at differentpoints across the substrate. A decrease in the conductivity is observed with increasing film thickness, which is counterintuitive, and may be attributed to surface oxidation of the nanomaterial during the processing steps.

The decomposition of the nanosheets in ambient conditions followed a characteristic first-order kinetic behavior, as evidenced by the linear decrease in optical density with time. The nanomaterial's decomposition rate was quantified, and a half-life of approximately 18 ± 5 hours for the films stored in ambient conditions was determined. In contrast, the nanosheets prepared under inert conditions displayed a much lower rate of oxidation, demonstrating the efficacy of the inert exfoliation process in preserving the integrity of the metal diborides for longer periods.

3.1.2 Encapsulation and Stability Enhancement

To mitigate the oxidation of the nanosheets and extend their stability, a novel low-cost encapsulation method was developed. This technique involves the use of protective coatings that shield the nanosheets from direct exposure to oxygen, thus preventing further degradation. Encapsulated nanosheet films were shown to retain their electronic and optical properties for extended periods, providing a stable platform for further functionalization. This is particularly important for applications where long-term material stability is critical, such as in the development of printed electronics and other nanomaterial-based devices.

3.1.3 Chemical Functionalization Strategies for Enhanced Performance

Given the reactivity of metal diboride nanosheets in ambient conditions, understanding and controlling their interaction with the surrounding environment is critical for effective functionalization. We explored both non-covalent and covalent functionalization routes to tailor the electronic properties of the exfoliated materials. The use of surfactants and solvents in non-covalent functionalization helps stabilize the nanosheets in solution, improving their colloidal stability and enabling precise control over material interactions. Additionally, covalent functionalization offers a route to directly modify the electronic structure of the nanosheets, enhancing their conductivity and reactivity for specific applications.

The successful exfoliation of metal diborides under inert conditions and their subsequent functionalization sets the stage for developing highly stable, versatile 2D materials with tailored properties. By understanding the decomposition pathways, oxidation risks, and functionalization methods, we can design more robust nanomaterials that maintain their intrinsic properties and avoid side reactions during processing.

In conclusion, the study highlights the significance of the exfoliation environment in determining the stability and functionality of metal diboride nanosheets. By adopting inert processing conditions, we can produce high-quality 2D materials with minimal oxidation, enabling the efficient functionalization of these materials for future technological applications.

These findings have recently been published in ACS Nano 2024, 18, 28596−28608

4 Risks and interconnections

4.1 Risks/problems encountered

If applicable (consider using table below to report risks – and solutions ! – encountered for the activities/tasks related to this deliverable)

Risk No.	What is the risk	Probability of risk occurrence[1]	Effect of risk[1]	Solutions to overcome the risk
1	Insufficient control of defect formation in 2D materials during exfoliation	2	2	Use of defective bulk materials (e.g., non-stoichiometric compositions); post-exfoliation defect engineering.
2	New materials are prone to degradation/oxidation in ambient conditions	2	2	Develop inert processing and device encapsulation; apply chemical defect passivation during exfoliation.

1) Probability risk will occur: 1 = high, 2 = medium, 3 = Low

5 Acknowledgement

The author(s) would like to thank the partners in the project for their valuable comments on previous drafts and for performing the review.

Project partners:

#	Partner short name	Partner Full Name
1	TCD	TCD THE PROVOST, FELLOWS, FOUNDATION SCHOLARS & THE OTHER MEMBERS OF BOARD, OF THE COLLEGE OF THE HOLY & UNDIVIDED TRINITY OF QUEEN ELIZABETH NEAR DUBLIN
2	UNISTRA	UNIVERSITE DE STRASBOURG
3	UKa	UNIVERSITAET KASSEL
4	BED	BEDIMENSIONAL SPA
5	TUD	TECHNISCHE UNIVERSITAET DRESDEN
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6 Appendix A - Quality Assurance Review Form

The following questions should be answered by all reviewers (WP Leader, reviewer, Project Coordinator) as part of the Quality Assurance procedure. Questions answered with NO should be motivated. The deliverable author will update the draft based on the comments. When all reviewers have answered all questions with YES, only then can the Deliverable be submitted to the EC.

NOTE: This Quality Assurance form will be removed from Deliverables with dissemination level "Public" before publication.

Question	**WP Leader**	**Reviewer**	**Project Coordinator**
	NAME (Organisation)	NAME (Organisation)	Jonathan Coleman (TCD))
1. *Do you accept this Deliverable as it is?*	Yes / No (elaborate)	Yes / No (elaborate)	Yes / No (elaborate)
2. *Is the Deliverable complete?* - *All required chapters?* - *Use of relevant templates?*	Yes / No (elaborate)	Yes / No (elaborate)	Yes / No (elaborate)
3. *Does the Deliverable correspond to the DoA?* - *All relevant actions preformed and reported?*	Yes / No (elaborate)	Yes / No (elaborate)	Yes / No (elaborate)
4. *Is the Deliverable in line with the 2D-PRINTABLE objectives?* - *WP objectives* - *Task Objectives*	Yes / No (elaborate)	Yes / No (elaborate)	Yes / No (elaborate)
5. *Is the technical quality sufficient?* - *Inputs and assumptions correct/clear?* - *Data, calculations, and motivations correct/clear?* - *Outputs and conclusions correct/clear?*	Yes / No (elaborate)	Yes / No (elaborate)	Yes / No (elaborate)
6. *Is created and potential IP identified and are protection measures in place?*	Yes / No (elaborate)	Yes / No (elaborate)	Yes / No (elaborate)
7. *Is the Risk Procedure followed and reported?*	Yes / No (elaborate)	Yes / No (elaborate)	Yes / No (elaborate)
8. *Is the reporting quality sufficient?* - *Clear language* - *Clear argumentation* - *Consistency* - *Structure*	Yes / No (elaborate)	Yes / No (elaborate)	Yes / No (elaborate)