Robotic-based Selection, Manipulation and Characterization of 3D Microscale Particles with Complex Structures in SEM

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Abstract— **This article explores the challenges and solutions in the physical characterization of materials on the microscale using robots, with a specific focus on manipulating and characterizing micrometer-sized particles with different and complex 3D shapes and internal sub-micrometer structures. Particles considered as case-study in this paper are made of MoS_2 based materials generated within the contact interface during friction. These particles are of high interest for friction reduction and energy saving but they are dispersed randomly inside the contact and have complex sub-micrometer structures. Characterization hence demands precise manipulation techniques in an in-situ SEM environment. To address these challenges, existing commercial micro and nanomanipulation tools are integrated within a vacuum SEM chamber, and robotics strategies are investigated to enable the whole process from particle preparation, and manipulation setup definition, to effective MoS_2 particles characterization all in-situ SEM. A set of several complementary experimental investigations are done and involve force measurement and deformation estimation studies, leading to the first quantified results on MoS_2 particles. The work contributes to advancements in microscale manipulation and characterization and has implications for lubrication research.**

Keywords: microrobotics, micromanipulation, categorization, microscale, third-body, Molybdenum diSulfide (MoS_2).

I. INTRODUCTION

In recent years, the utilization of robotics within the Scanning Electron Microscope (SEM) environment has garnered significant interest due to its diverse and promising applications in various fields, including nano-electronics, instrumentation, and materials research [1], [2], [3]. The ability to perform a sequence of tasks, ranging from sample preparation, separation, and manipulation, to multimodal characterization, under the SEM presents a multitude of opportunities for scientific advancements and technological innovations. Among the compelling applications, the study of the third-body material stands out as an area of interest. Self-created inside the contact interface during friction, it drives lubrication and wear performances. Reliable characterization of its physical properties offers key information to help reduce friction, one of the most significant sources of energy losses [4]. It also opens up the route to far-reaching implications for future ecological and sustainable solutions.

Understanding and characterizing third-body particles' mechanical properties, including forces and deformations, are very compelling, given their relevance in addressing friction-related challenges. Several studies have demonstrated the potential of using a nanomanipulation platforms inside a SEM for motion control [5] and multi-physical characterization [6]. However, the complexity and dimensions of these particles (micrometer-sized non-identical 3D structures) necessitate advanced tools and robotic strategies to achieve a seamless sequence of tasks within the SEM environment.

The challenges posed by the manipulation and characterization of these microstructures are not only specific to this study but also extend to numerous other applications. This common problem calls for the development of robust and innovative strategies to facilitate a wide range of tasks in the SEM environment. It is in this context that this article aims to propose an approach to tackle the complexities of manipulating and characterizing micrometer-sized 3D particles, offering insights and solutions applicable across various fields of research.

Through the integration of existing commercial micro and nanomanipulation tools, along with advanced robotic strategies, this study endeavors to shed light on the potential of using robotics for the precise manipulation and analysis of materials with intricate 3D structures. By addressing the challenges of limited workspace, tool selection, and automation, this case study aims to contribute to the broader understanding of the third-body phenomenon while paving the way for future advancements in microscale materials research, friction studies, and lubricants.

II. MATERIALS AND METHODS

A. 3D microscale particles

In this case study, we specifically focus on investigating the particles generated during friction, with a particular emphasis on the third-body phenomena. Our investigation centers on three distinct types of particles: raw Molybdenum disulfide (MoS_2) grains, third bodies formed from pure MoS_2

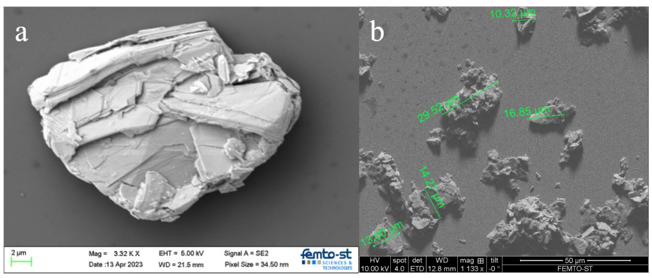

Fig. 1. a- MoS_2 grain, visualizing the nanolayers forming a microparticle. b- Different MoS_2 particles, scaling from 10 to 25 μm.

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coating, and third bodies formed from MoS_2+Tantalum (Ta) coating. These third-body particles are created during friction, and while some are trapped within the contact interface, others are ejected outside. In our specific study, these third bodies are created under ambient air conditions. The third-body plays a major role in the lubrication and wear processes, lubrication efficiency indeed depends on its mechanical and physic-chemical properties. Pure MoS_2 coating failed to lubricate, while MoS_2+Ta sustained lubrication. Each of these particles exhibits distinct structures, and mechanical properties, which require individual characterization.

Raw MoS_2 grains in the microscale exhibit anisotropic lamellar structures, with layers stacked in a parallel arrangement, Fig. 1(a). The lamellar nature of these grains contributes to their lubricating properties, making them suitable for applications requiring low friction and wear. The alignment of the layers also plays an important role in influencing the mechanical behavior of raw MoS_2 during manipulation and deformation processes. Understanding the nature of these grains is essential for assessing their potential as effective friction-reducing materials in various industries. Additionally, the lamellar behavior of raw MoS_2 in the microscale opens up opportunities to explore its unique responses to force application along different orientations, providing complementary information to the existing literature [7], [8] on its mechanical properties.

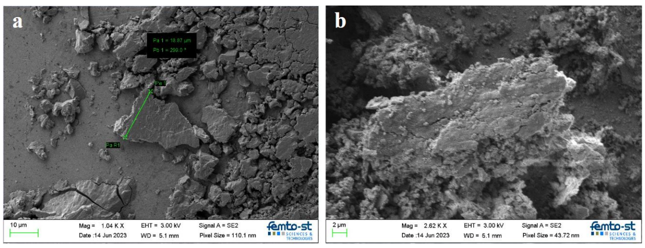

Fig. 2. a- Third-body particles created from pure MoS_2. b- Third-body particles created from MoS_2+Ta

The MoS_2-based third-body is obtained by submitting MoS_2 particles to friction. That resulted in the creation of a third-body with a unique structure, different from the original MoS_2. Although similar in shape to the raw MoS_2 material, the nanolayers disappear to the benefit of a relatively unified and continuous more homogeneous microstructured body, Fig. 2(a). The MoS_2+Ta based third-body is a composite material formed by submitting a coating comprised of MoS_2 and Ta to friction. This addition of Ta particles significantly alters the structure of MoS_2 [9], which eventually results in the creation of a unique third-body with granular microstructure. The composite material exhibits nanograins stacked together, forming a cohesive microscale body, Fig. 2(b).

This transformation to a third-body may have implications for the material mechanical properties, such as changes in its friction behavior, and lubrication performance. Investigating the structural modifications and mechanical responses of the pure MoS_2 third-body and the granular structure of the MoS_2+Ta third-body leads us to comprehend its potential as an effective friction-reducing material. To quantify the mechanical properties of these different particle structures, force-displacement measurements should be performed in different directions of interest.

Through the integration of advanced robotic systems and nanomanipulation techniques within the SEM environment, we seek to gain insights into the mechanical behavior of these particles under precise, directional, and controlled force application. By characterizing these different particle structures and quantifying their mechanical properties using the same system, this study aims to provide data for evaluating their performance as third-body materials and their potential applications in reducing friction and energy losses.

B. μROBOTEX platform

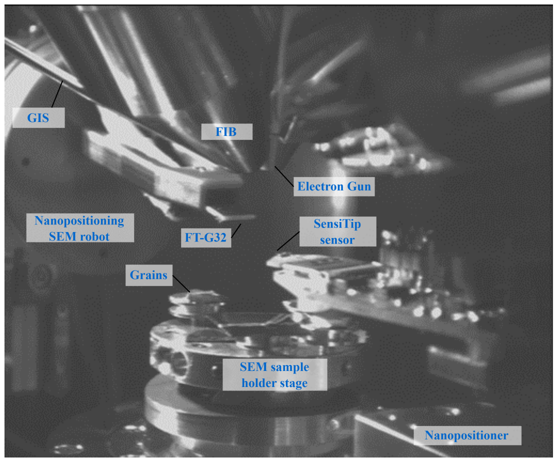

Fig. 3. Inside SEM vacuum room, different system view.

The μROBOTEX platform used in this study is a system that combines the capabilities of a Scanning Electron Microscope (SEM) with Focused Ion Beam (FIB) and Gas Injection System (GIS) technologies, Fig. 3. This powerful platform allows for both additive and subtractive approaches, enabling precise manipulation and fabrication at the microscale and nanoscale [10], [11].

The SEM provides high-resolution imaging, that to observe and analyze samples at the nanoscale level. The FIB technology complements the SEM by offering the ability to leverage a secondary view with a 54-degree angle at low power and perform material removal and patterning at the microscale with great precision at high power. Additionally, the GIS allows gas injection control, further enhancing the versatility of the platform.

The platform's sample stage offers 5 Degrees of Freedom (DoF), providing high flexibility for positioning and orienting samples during manipulation and analysis. The micro-nanopositioning robot boats 6 DoF, enabling precise and smooth multi-directional, long-ranged movements. Additionally, the nanopositioner contributes 3 DoF, facilitating precise movements at the nanoscale level for fine manipulation.

The combination of these advanced technologies and robots not only enables seamless coordination between imaging, manipulation, and characterization tasks but also serves

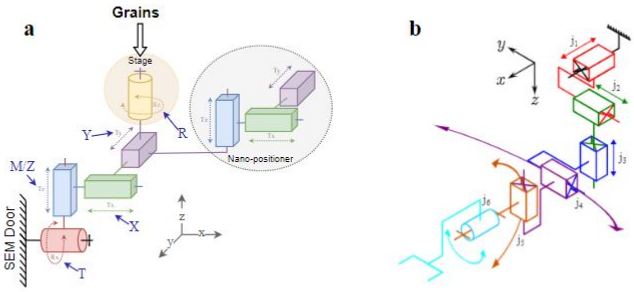

Fig. 4. a- Kinematic diagram showing the 5DoF sample holding stage and the 3DoF nanopositioner attached to it. b- Kinematic diagram of the SEM nanopositioning robot developed at the AS2M department [12].

to minimize the frequency of opening the chamber. This reduction is crucial to prevent particle loss due to pressure differences and saves valuable time that would otherwise be spent rechecking and restarting the entire system for manipulation purposes.

The kinematic diagram of the robotic systems, Fig. 4, inside the vacuum chamber of the SEM platform, illustrates how each component works to achieve the desired tasks. This cutting-edge platform is used to explore the mechanical properties of micrometer-sized 3D grains, enabling insights into friction and lubrication phenomena and advancing nanomanipulation techniques.

C. Robotic tools

In this study, several robotic tools were used to achieve precise and controlled manipulation of micrometer-sized 3D grains within the SEM environment. The first tool employed was a specialized tip for sample preparation, which allowed for the precise handling and positioning of the grains during the initial stages of the experiments.

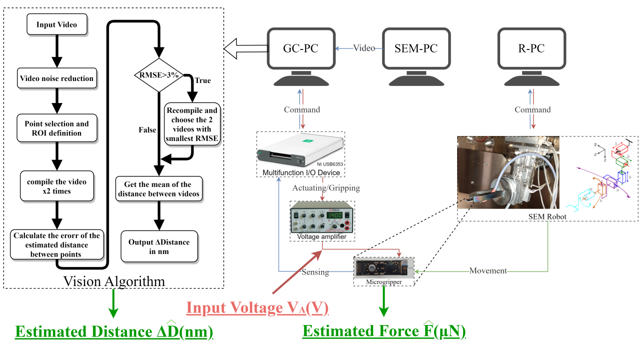

Fig. 5. Microgripper and vision control systems.

A microgripper (one finger actuated and one instrumented finger, ref. FT-G32) has been integrated with the SEM nanopositioning robot providing a seamless and coordinated approach to achieve complex manipulations at the micro and nanoscale levels, Fig. 5. This setup enabled us to grasp, move, and place the grains with accuracy.

To accurately measure the forces exerted on the grains, even during the manipulations, two force-sensing methods were employed. First, the instrumented microgripper itself provided force-sensing capabilities, allowing for real-time force feedback during the manipulation tasks, reaching up to 70μN. Secondly, a piezoresistive force sensor was integrated onto the nanopositioner system, as shown in Fig. 6. This sensor provides precise force measurement capabilities with a larger and variable force range, reaching up to 5 mN, and offers a precision of less than 1 μN. This sensor is similar to the one used in the publication by Tiwari et al. [13]. To ensure additional safety during the experiments, a SmarAct X-axis rotator was also incorporated into the system.

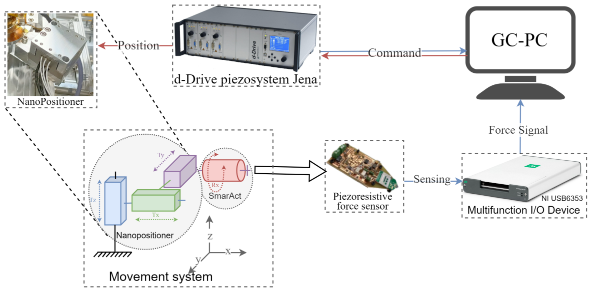

Fig. 6. Piezoresistive sensing system for controlled force application

The integration of these robotic tools and force-sensing technologies was used for the mechanical categorization of the micrometer-sized 3D particles displayed in the following sections. The combination of automated control and teleoperated manipulation with real-time force feedback allowed a smooth, controlled, and supervised view of forces and deformations experienced by the particles during the handling and manipulation processes.

D. Vision algorithm

In this study, a vision algorithm is developed to estimate the deformation of the micrometer-sized 3D particles while being gripped by the microgripper. By analyzing the changes in the particle's shape and dimensions, the algorithm provides valuable data on the deformation and mechanical response of the particles under force application. The algorithm utilized advanced image processing techniques to accurately track and quantify the changes in the particle's geometry from the video extracted by the SEM system, allowing us to obtain detailed force-deformation curves for different particles.

III. SAMPLE PREPARATION

The sample preparation and manipulation procedures were essential steps in the study of micrometer-sized 3D particles, raw MoS_2, MoS_2-based third-body, and MoS_2+Ta-based third-body. Initially, raw MoS_2 grains were in a dispersed state, randomly scattered within the workspace. To remediate this problem, we mixed the particles with ethanol (to enable a good spread of the particles) and an ultrasonic cleaning bath (to remove the nanograins that could be attached to the main particles of interest). However, the third bodies were directly extracted from the friction track vicinity (outside contact area on the sliding ball), placed on the SEM stage.

To begin the manipulation process, a metalized glass probe, refined using the FIB technology, Fig. 7(a), was employed to carefully detach the individual particles from the support. Very fine precision is required during this step to ensure that the grains are not damaged or altered during the detachment process.

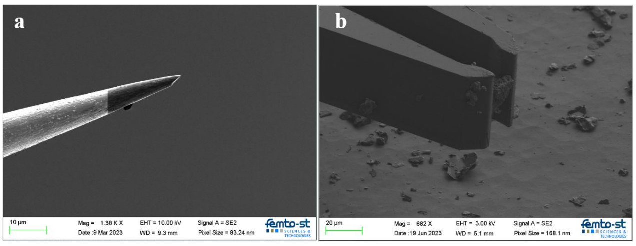

Fig. 7. a- Refined probe using the FIB. b- Manipulation of particles using the microgripper

Once detached, the individual particle was carefully tested by gripping force application without surface contact using the microgripper piezocapacitive tip and then transferred to a clean workspace, Fig. 7(b), for further manipulation and analysis. Next, the particles were adjusted to an exact gap location on the silicon-based support created using the FIB to anchor them, while applying force induced with the tip of the sensor, Fig. 8(b).

Throughout the manipulation sequence, the SEM micropositioning robots provided precise positioning while using the probe and the FT-G32 microgripper, ensuring that the particles were precisely controlled and moved to desired locations for various tests and analyses.

Overall, the sample preparation and manipulation procedures were conducted with meticulous care to ensure accurate and reliable results. The integration of advanced robotic tools and force-sensing technologies facilitated the precise handling and analysis of micrometer-sized 3D particles.

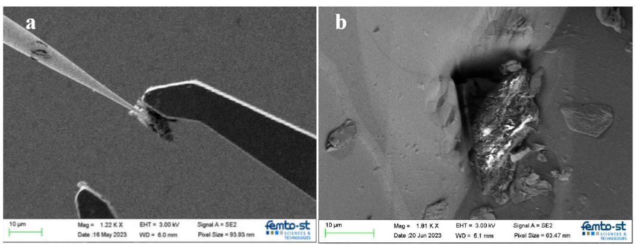

Fig. 8. a- The use of a fixed probe to remove the particle adhered to the gripper. b- MoS_2+Ta third-body positioned into the wall gap for force application.

Several challenges were encountered and addressed to ensure accurate and reliable results. The manipulation of micrometer-sized 3D particles posed unique difficulties, such as dealing with charging effects on the tools within the SEM environment which highlights the importance of grounding all the components of the workspace, and the occurrence of particle attachment to the robotic tools due to various phenomena, Fig. 8. Additionally, the precise positioning of the robotic tools was crucial to avoid unintended damage to the delicate components during the manipulation process. Moreover, the complexity of the 3D particles required careful planning and adaptation of the robotic strategies to achieve successful manipulation.

To overcome these challenges and enhance the efficiency of the experiments, automation played an influential role. Automating the manipulation tasks not only saved time but also reduced the risk of human error, ensuring reproducible results, especially for the specific orientation of the force application. The sample preparation and manipulation, coupled with the integration of advanced robotic tools and force-sensing technologies, allowed us the exploration of the micrometer-sized 3D particles' mechanical properties. Moreover, the successful automation of manipulation tasks highlights the potential of robotics in optimizing nanomanipulation processes.

IV. Results

The validation of the different robotic systems was an essential step in ensuring the accuracy and reliability of the results. To achieve this, several complementary tests were conducted on the instrumented microgripper, and the piezoresistive force sensor. The validation procedures aimed to verify the precision of force measurement, and the overall performance of the robotic tools within the SEM environment, while providing a reference about the behavior of the tools.

A. Validation

The validation phase of the microgripper (FT-G32) started with essential gripping tests to assess its repeatability. By performing gripping actions without any specimen, we evaluated the tool's behavior across multiple trials and verified its reliability for subsequent manipulations. Additionally, the derivative of the tool's behavior was examined under steady contact and no contact conditions, providing insights into its sensitivity and accuracy during gripping operations. To establish the accuracy and reliability of the microgripper, we employed a rigid silica glass bead as a reference material, Fig. 9. The glass bead served as a benchmark to compare the force response obtained from the gripper with the known properties of the reference material. This comparison allowed us to validate the accuracy of the gripper force readings and assess the performance of our vision algorithm for nanoscale measurements.

The vision algorithm's behavior was investigated under larger-scale movements, in the micrometer range, by following a specific point on the nanopositioner, showcasing its reliability across both nano and micro scales. The validation of the vision algorithm provided confirmation of its accuracy, establishing it as a robust solution for precise measurements at varying scales, and contributing to the effectiveness of our research in particle characterization.

The piezoresistive sensor underwent testing both before and after its implementation in particle manipulation experiments. A comparative test with a higher force scale FemtoTools sensor (ref. FT-S160) confirmed the piezoresistive'

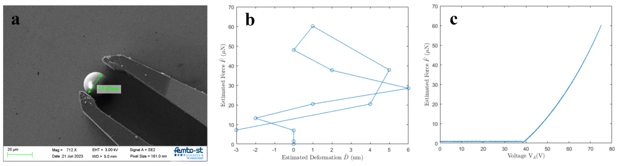

Fig. 9. Verification of the behavior of the microgripper (c) and the vision algorithm (b) by gripping a rigid Silica bead (a).

behavior and accuracy in force measurement, ensuring its reliability and precision. Post-experiment validation verified the sensor's stability and ability to maintain accurate force measurements even under challenging conditions during manipulation tasks. An empty test application provided essential baseline data for interpreting force measurements during subsequent particle manipulations. The validation of the sensor demonstrated its reliability and precision, proving its significance as a tool for accurate force analysis during particle manipulation within the SEM environment.

B. Characterization of different particles

The vision algorithm allowed us to estimate the deformation $\Delta\hat{D}$ of the particles during force application using the FT-G32 microgripper, providing details on the mechanical behavior of the different materials, Fig.10.

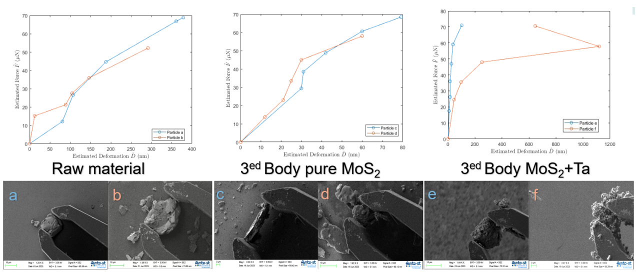

Fig. 10. Estimated force $\hat{F}$ vs deformation $\Delta\hat{D}$ of the different particles.

When analyzing pure MoS_2 particles, a deformation response of up to 400nm is displayed upon applying a force of 70μN. However, the particles consistently returned to their original size after force retraction, indicating high elasticity and resilience. In contrast, the third-body created from pure MoS_2 exhibited limited deformation of approximately 80nm and complete recovery to its initial state after force retraction, highlighting its exceptional rigidity. The third-body created from the MoS_2 and Ta mixture displayed a particularly intriguing finding. This material experienced substantial deformation of approximately 1200nm, significantly greater than other cases. Moreover, even after force retraction, the third-body retained its deformed state, suggesting permanent deformation. These observations appear consistent with the fact that lubricity from MoS_2-based coatings is achieved through the creation of third bodies that demonstrate ductile behavior (plastic flow) inside the interface [14]. Indeed, MoS_2-based coating failed to lubricate the contact, while MoS_2+Ta coating succeeded.

The comparison of results obtained from the vision algorithm hence provides an idea of the mechanical properties of these materials. The varying deformation responses and recoverability observed in the different materials shed light on their distinct mechanical characteristics. Further investigations and in-depth studies are necessary to fully explore the potential of these materials in practical engineering applications.

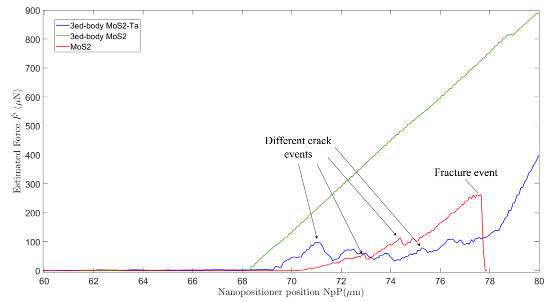

Fig. 11. Estimated force $\hat{F}$ from the piezoresistive sensor on the different particles, and observation of multiple crack events mainly from the raw MoS_2 and the third-body formed from MoS_2+Ta; and the fracture of the raw MoS_2.

Comparing the results from the piezoresistive sensor force application on different materials, we observed distinct mechanical responses for each material. The raw MoS_2 particles exhibited significant deformation with multiple crack events, the pure MoS_2 third-body displayed rigidity with minimal deformation, and the MoS_2 and Ta mixture third-body experienced fracturing and detachment of nanograins. The sensor-nanopositioner combination proved advantageous, offering precise force application and accurate measurements.

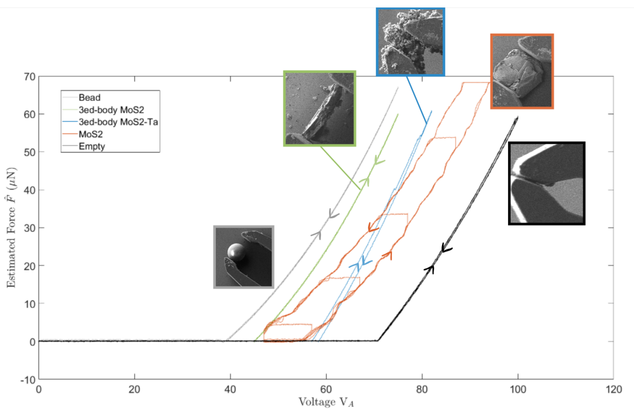

Fig. 12. Estimated force $\hat{F}$ vs input voltage V_A of the microgripper, empty and rigid bead gripping to set references, then lateral gripping on raw MoS_2, the third-body created by MoS_2 and the third-body created from MoS_2+Ta.

When applying longitudinal forces along the crystal layer, the raw MoS_2 particles exhibited rigid behavior. However, lateral force application on the edge of the stacked layers resulted in a distinct wavy pattern in the force curve with unique hysteresis, which exhibits elastic behavior with reversible deformation. We observed a reversible deformation response upon the application of force, as shown in Fig.10. Furthermore, the atypical behavior of the MoS_2 grain during lateral gripping, with a distinct wavy pattern in the force curve (Fig.12), may be linked to the reversible separation of MoS_2 layers, as observed by Tang et al.[8].

The pure MoS_2-based third-body displayed a more rigid response with minimal deformation under the applied forces. The force-voltage curve showed an almost linear behavior, indicating a lack of significant structural changes within the third-body configuration. The third-body composed of MoS_2 and Ta mixture exhibited substantially different behavior. Under lateral force application, this third-body experienced permanent deformation and multiple crack formations, leading to significant plastic deformation within the particles. The larger particles in the central region displayed a more rigid behavior.

In summary, the three materials demonstrated different mechanical responses under force application. The raw MoS_2 particles showed distinct behaviors longitudinally and laterally, indicating their anisotropic mechanical properties. The pure MoS_2-based third-body exhibited rigidity, while the third-body formed from MoS_2 and Ta displayed a range of behaviors depending on the location and size of the particles. These findings provide information into the mechanical properties of these materials and offer possibilities for better understanding their role in friction. Which paves the way for further application in various engineering scenarios.

These initial results show promising coherence and open avenues for further exploration and exploitation. Future investigations, though combined studies with numerical modeling for example [14], will contribute to a deeper understanding of the underlying mechanisms and potential applications of these unique nanoscale structures.

V. CONCLUSIONS

In conclusion, this article presents the exploration of the challenges and achievements in the manipulation and characterization of micrometer-sized particles with 3D structures having different geometries and complex internal submicrometer structures. Throughout the study, we integrated several complementary commercial micro and nanomanipulation tools within the vacuum SEM chamber environment. This integration allowed us to conduct precise and automated force-displacement measurements accompanied by carefully devised robotic strategies to enable the whole process from particle selection, manipulation, and characterization in-situ SEM. Several experimental complementary characterization tests have then been performed to compress particles perpendicularly but also along the main axis to the internal sub-micrometer layered structure. Different kinds of particles have also been investigated from raw materials to their third body which is obtained after friction. First quantified results have thus been obtained, compression forces are typically of several hundreds of micro-Newtons and deformations can reach one micrometer. Raw materials also showed different fracture events and a hysteresis-based behavior when compressed for the first time.

Overall, this article's primary contribution lies in the first quantified results on MoS_2 particles and their third-bodies, showcasing the utilization of advanced tools and robotics for simultaneous manipulation and characterization of materials with complex 3D structures at the micro and nanoscales.

ACKNOWLEDGMENT

This work has been partially funded by the DYNABOT Project under the contract ANR-21-CE10-0016, by Région Bourgogne Franche-Comté and the EIPHI Graduate School (ANR-17-EURE-0002) and the French RENATECH network through its FEMTO-ST technological facility. Authors are thankful to Pierre Roux for the design and fabrication of components.

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