

NANOMECHANICAL STRAIN TESTING OF LOW DIMENSIONAL MATERIALS WITH MICRO-ELECTRO MECHANICAL SYSTEM (MEMS) CHIPS

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By
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Nanomechanical Strain Testing of Low Dimensional Materials with
Micro-Electro Mechanical System (MEMS) Chips

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September 2023

We certify that we have read this thesis and that in our opinion it is fully adequate,
in scope and in quality, as a thesis for the degree of Master of Science.

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ABSTRACT

NANOMECHANICAL STRAIN TESTING OF LOW DIMENSIONAL MATERIALS WITH MICRO-ELECTRO MECHANICAL SYSTEM (MEMS) CHIPS

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M.S. in Physics

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This thesis explores nanomechanical strain testing of low-dimensional materials. The limitations of existing experimental environments for testing low-dimensional materials are discussed. To overcome these limitations, the study proposes the use of self-designed Micro-Electro Mechanical Systems (MEMS) chips. The fabrication methods and advantages of MEMS chips are explained. The investigation involves studying few-layer 2H-MoS₂ crystals using MEMS chips. Device preparation techniques, such as mechanical exfoliation and all-dry transfer, are outlined. Raman measurements are conducted on MoS₂ to analyze its response to strain, specifically focusing on the E¹_{2g} mode Raman peak. The piezoelectric properties of MoS₂ are extensively discussed, including piezo voltage-resistivity and IV measurements. An interesting thermoelectric bipolar photocurrent response in MoS₂ is explored using scanning photocurrent microscopy (SPCM). Possible contributions of different mechanisms such as the Photothermoelectric Effect (PTE) or Flexo-photovoltaic effect of Bipolar Photocurrent response are discussed. The contribution of stress on the recently shown substrate effect is tested in this work.

The growth and transfer optimization of V₂O₃ crystals which have a strain-dependent phase transition properties is explored, with a focus on adapting growth to a conventional CVD chamber. Polymer-assisted transfer techniques and passivation methods are investigated. Raman spectroscopy results demonstrate a shift in the Raman spectrum, and completed V₂O₃ MEMS devices are showcased. In addition, some preliminary work upon V₂O₃ growth in different substrates (Mica) is tested. Finally, some small top-gate modulations of V₂O₃ exploiting Metal-insulator transition property is shown.

Overall, this thesis contributes to the field of nanomechanical strain testing

of low-dimensional materials by introducing MEMS chips as an effective experimental platform. The investigations provide valuable insights into the mechanism of the substrate-engineered photocurrent generation analysis of 2H-MoS₂. The thesis also provides a device fabrication methodology to study interesting phase transitions in strongly-correlated materials. These findings have implications for understanding the properties and potential applications of low-dimensional materials.



Keywords: Low-dimensional Materials, Micro-Electro Mechanical Systems (MEMS), 2H-MoS₂, V₂O₃, Scanning Photocurrent Microscopy, Raman, Piezo-Resistance, Photothermoelectric Effect (PTE), Flexo-photovoltaic Effect, Seebeck Coefficient.

ÖZET

MIKRO-ELEKTRO MEKANİK SİSTEM (MEMS) ÇİP İLE DÜŞÜK BOYUTLU MALZEMELERİN NANOMEKANİK GERİLME TESTİ

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Bu tez, düşük boyutlu malzemelerin nanomekanik gerilme testini araştırmaktadır. Düşük boyutlu malzemelerin testi için mevcut deneysel ortamların sınırlarını tartışılmıştır. Bu sınırları aşmak için, çalışma, özgün olarak tasarlanmış Mikro-Elektro Mekanik Sistemler (MEMS) çiplerinin kullanımını önermektedir. MEMS çiplerinin üretim yöntemleri ve avantajları açıklanmıştır. Araştırma, MEMS çiplerini kullanarak az katmanlı 2H-MoS₂ kristallerini incelemeyi içermektedir. Mekanik eksfoliasyon ve tam-kuru transfer gibi cihaz hazırlama teknikleri taslağa alınmıştır. MoS₂ üzerindeki Raman ölçümleri, özellikle E¹_{2g} modu Raman tepe noktasına odaklanarak, gerilmeye tepkisini analiz etmek için gerçekleştirilmiştir. MoS₂'nin piezoelektrik özellikleri, piezo gerilim-direnç ve IV ölçümleri dahil olmak üzere kapsamlı bir şekilde tartışılmıştır. MoS₂ içerisindeki ilginç termoelektrik kutuplu fotoakım tepkisi, taramalı fotoakım mikroskopisi (SPCM) kullanılarak araştırılmıştır. Çift kutuplu fotoakım oluşumuna farklı mekanizmaların olası katkıları, Fototermoelektrik Etki (PTE) veya Flexo-fotovoltaik etkisi gibi, tartışılmıştır. Stresin son zamanlarda gösterilen substrat etkisine katkısı bu çalışmada test edilmiştir.

V₂O₃ kristallerinin büyüme ve transfer optimizasyonu, standart CVD sisteminde büyümeyi uyarlamaya odaklanarak incelenmiştir. Polimer destekli transfer teknikleri ve pasivasyon yöntemleri araştırılmıştır. Raman spektroskopisi sonuçları, Raman spektrumunda bir kayma göstermekte olup, tamamlanmış V₂O₃ MEMS cihazları sergilenmiştir. Ayrıca, V₂O₃ büyümesi üzerine (Mika gibi) farklı substratlar ile yapılan ön çalışmalar incelenmiştir. Son olarak, V₂O₃'ün Metal-Yalıtkan geçiş özelliğini kullanarak bazı küçük üst kapı modülasyonları gösterilmiştir.

Genel olarak, bu tez, düşük boyutlu malzemelerin nanomekanik gerilme testine MEMS çiplerinin etkili bir deneysel platform olarak tanıtılarak katkıda bulunur. Araştırmalar, 2H-MoS₂'nin subtrat mühendisliğiyle fotoakım üretim mekanizmasına dair değerli öngörüler sunmaktadır. Tez aynı zamanda, güçlü etkileşimli malzemelerde ilgi çekici faz geçişlerini incelemek için bir cihaz üretim metodolojisi sunar. Bu bulgular, düşük boyutlu malzemelerin özelliklerini ve potansiyel uygulamalarını anlamak için önem taşır.



Anahtar sözcükler: Düşük boyutlu malzemeler, Gerilme testi, Mikro-Elektro Mekanik Sistemler (MEMS), 2H-MoS₂, V₂O₃, Taramalı Fotoakım Mikroskobu, Raman, Piezo-Resistance, Fototermoelektrik Etki (PTE), Flexo-fotovoltaik etki, Seebeck Katsayısı.

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Chapter 1

Introduction to Strain Testing in Low-Dimensional Materials

1.1 Overview on Strain Testing in Low Dimensional Materials

Strain control over low-dimensional materials has been investigated extensively because of the ability to manipulate their optical, electronic and magnetic properties with external strain[1]. Strain tests on two-dimensional (2D) materials especially transition metal dichalcogenides (TMDC) are good candidates in this context. Some TMDCs such that Molybdenum disulfide (MoS_2)[2], molybdenum ditelluride (MoTe_2)[3], Tungsten disulfide (WS_2)[4], etc have been tested in literature in large extent. Apart from 2D materials, Nanowires (NW) were a highly attractive research field in terms of their potential for being strain sensors regarding their strain-induced electrical and optical responses. In this research field, many creative experimental environments are exploited. As an example, Lungstein et al. studied the anomalous piezoresistance effect in Silicon NWs by a microelectro-mechanical loading module[5]. Greil et al. used 3-point strain apparatus to investigate strain-induced optical and electronic impacts on Germanium

NWs[6]. In addition, Amjadi et al. studied the strain-sensor properties of Silver NWs in a flexible sandwiched design with two PDMS elastomers[7]. In summary, testing the response of novel materials under stress is a significant investigation to find out possible applications of low-dimensional materials. In this search many creative and novel strain systems are introduced or developed.

1.2 Strain Engineering in Diverse Experimental Environment in MoS₂

MoS₂ has become a highly intriguing research subject due to its characteristic optical, electronic and catalytic properties even after the first time it is cleaved and isolated by Novoselov et al. [8]. MoS₂ is a Van der Waals semiconductor material built up as an S-Mo-S structure which may show up in different phases such as Hexagonal 2H-MoS₂ or Rhombohedral 3R-MoS₂[9][10][11]. The atomic structure of both 2H and 3R phases is demonstrated in Figure 1.1[12][11]. The variations in structure can greatly impact the electronic, optical, and mechanical characteristics of MoS₂'s distinct phases, rendering them vital factors in diverse applications like electronics, catalysis, and optoelectronics. Orientation of 2H and 3R phases contributes to photocurrent generation differently because of their centrosymmetric and noncentrosymmetric nature[13]. These orientations also vary the photocurrent generation response levels while applying stress.

MoS₂ exhibits remarkable changes in its properties due to reduced dimensionality when transitioning to the monolayer limit. The bulk form of MoS₂ has an indirect band gap of 1.29 eV[14]. By decreasing the layer number of MoS₂ steady shift in the indirect band gap from 1.29 eV bulk value over 1.90 eV is observed[14][15]. By following this trend, in the monolayer limit, the MoS₂ exhibits a transition from indirect to direct band gap[14]. These variations make monolayer MoS₂ very suitable application candidate and research subject.

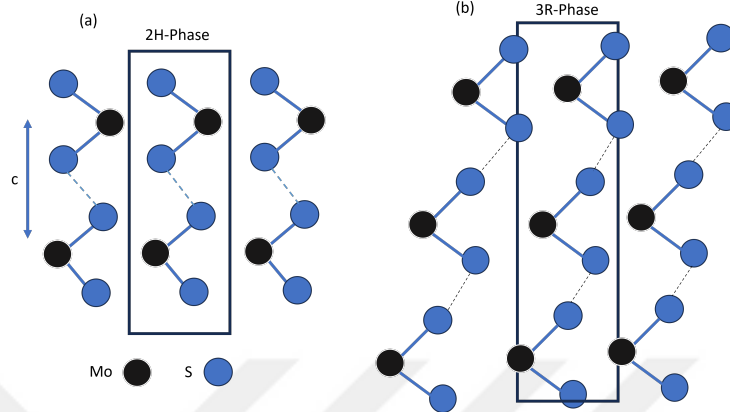


Figure 1.1: (a) 2H phase of MoS_2 : centrosymmetric (b) 3R phase of MoS_2 : non-centrosymmetric. Blue dots represent Sulfur, Black dots represent Molybdenum atoms

In recent studies, MoS_2 is tested mechanically for diverse experimental searches. Strain-dependent electronic, optical and magnetic variations are reported in a large extent[16][17][18]. In the literature, there are various kinds of experimental systems that can apply stress. Now, some examples of experimental environments from literature and their outcomes will be provided.

The majority of literature concerning mechanical testing utilizes the flexible substrate approach. As an example, Datye et al. studied the strain engineering in few-layer MoS_2 in a flexible substrate-polyethylene naphthalate (PEN)-apparatus[17]. A picture of the Flexible substrate is shown in Figure 1.2. In this work, they showed the enhancement of electron mobility by applying stress to the sample. The Gauge factor dependent on gate voltage is also calculated and the efficiency of MoS_2 as a strain sensor is shown[17].

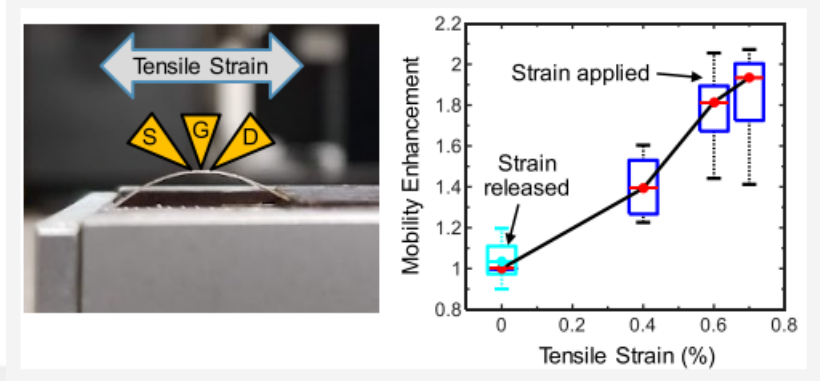


Figure 1.2: Strain apparatus of flexible substrate (PEN) and Mobility enhancement with respect to strain [17] Copyright © 2022 American Chemical Society

Although the majority of strain testing works exploit the flexible substrates, it may not be very suitable for the purposes of some experiments. As an example, Manzeli et al. used an Atomic Force Microscopy (AFM) probe to deform MoS_2 flake. While doing that they measured resistance which allow them to investigate the piezoresistive response of MoS_2 [19].

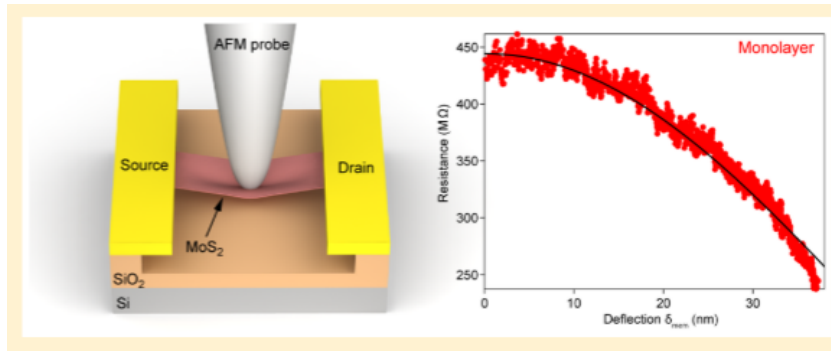


Figure 1.3: AFM tip approach to study Piezoresistive behaviour of MoS_2 with strain [19] Copyright © 2015 American Chemical Society

As a final example, Razeghi et al. suspended some MoS_2 crystals on etched holes to test different kinds of contributions of response such as Seebeck variation or strain contribution [20]. The conclusion from this work was substrate engineering can be applied to form thermoelectric junctions in materials with relatively

tunable Seebeck coefficient[20].

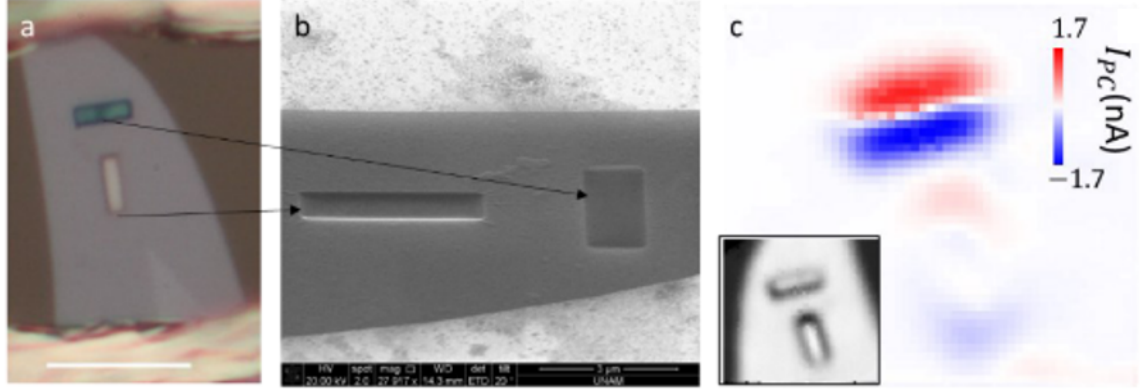


Figure 1.4: Photocurrent response from substrate engineered suspended MoS₂[20]
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From these examples, it can be understood that there is a large library of strain testing systems although some of them are more advanced or more suitable for specific purposes. Apart from this large library, new types of experimental strain apparatuses are introduced by various research groups.

1.3 Strain-Dependent Phase Transitions in Strongly Correlated Materials

Strongly correlated materials encompass a wide range of substances in which the interactions among electrons are notably strong, exerting a substantial influence on the characteristics of the material. These interactions give rise to various remarkable phenomena, including unconventional superconductivity, magnetoresistance, metal-insulator transition and distinctive magnetic arrangements, among others. In contrast to typical materials, which can often be comprehended using basic models treating electrons as isolated entities, the study of strongly correlated materials necessitates a more intricate methodology that takes into account the collective actions of electrons[21].

In this context, Vanadium dioxide (VO_2) is an archetype example of metal-insulator transitions which can be induced by internal strain. The strain-triggered phase transition in VO_2 involves the manipulation or modification of the way this phase transition occurs by imposing mechanical strain onto the material. In this scenario, strain pertains to altering the arrangement of the crystal lattice in VO_2 , achievable through diverse methods, including the exertion of mechanical pressure. By subjecting the material to strain, researchers can successfully adjust the temperature at which the phase transition takes place, granting them the capability to purposefully manage and adjust the material's characteristics[22]. Although, in general, phase transition of these kinds of materials is performed by doping which is a debated procedure, some experimental apparatus for applying strain on pristine form of material is introduced as well[23]. Figure 1.5 demonstrates a nanomechanical strain apparatus that can apply strain on suspended crystals.

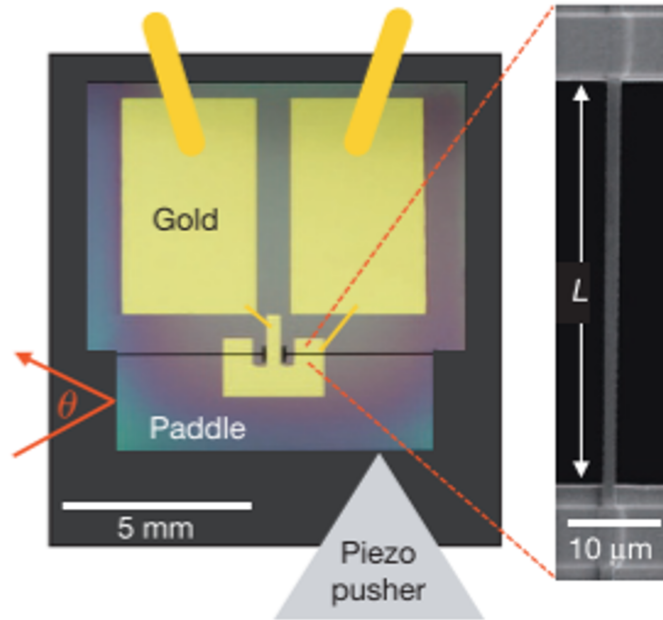


Figure 1.5: Strain apparatus for suspended VO_2 [23] Copyright © Springer Nature 2013

Finally, another intriguing material class that exhibits strain-induced phase transition is Charge Density Wave (CDW) systems. In CDW systems, electrons organize collectively in a regular arrangement, giving rise to the establishment of a CDW pattern. This arrangement can significantly impact the material's characteristics related to its electronic properties and movement of particles. For example, the emergence of a CDW can result in a repetitive alteration of the material's electrical resistance, which becomes evident as a unique pattern in measurements of particle transportation. Furthermore, CDW systems can manifest alterations in the structure of electronic bands, thereby influencing the material's electronic characteristics[24].

Applying strain to a CDW material has the potential to affect the arrangement of CDWs and disrupt the regular spacing of charge density. Consequently, this disruption brings about significant shifts in the material's electronic attributes and traits related to particle movement. The impact of strain on CDWs can encompass both nuanced adjustments and substantial transformations, often giving rise to complex interactions among the material's internal dynamics. Various studies to understand this strain-induced effect on CDWs are performed. One example is that suspending a 2H-TaS₂ and observing phase transition by interferometry[25]

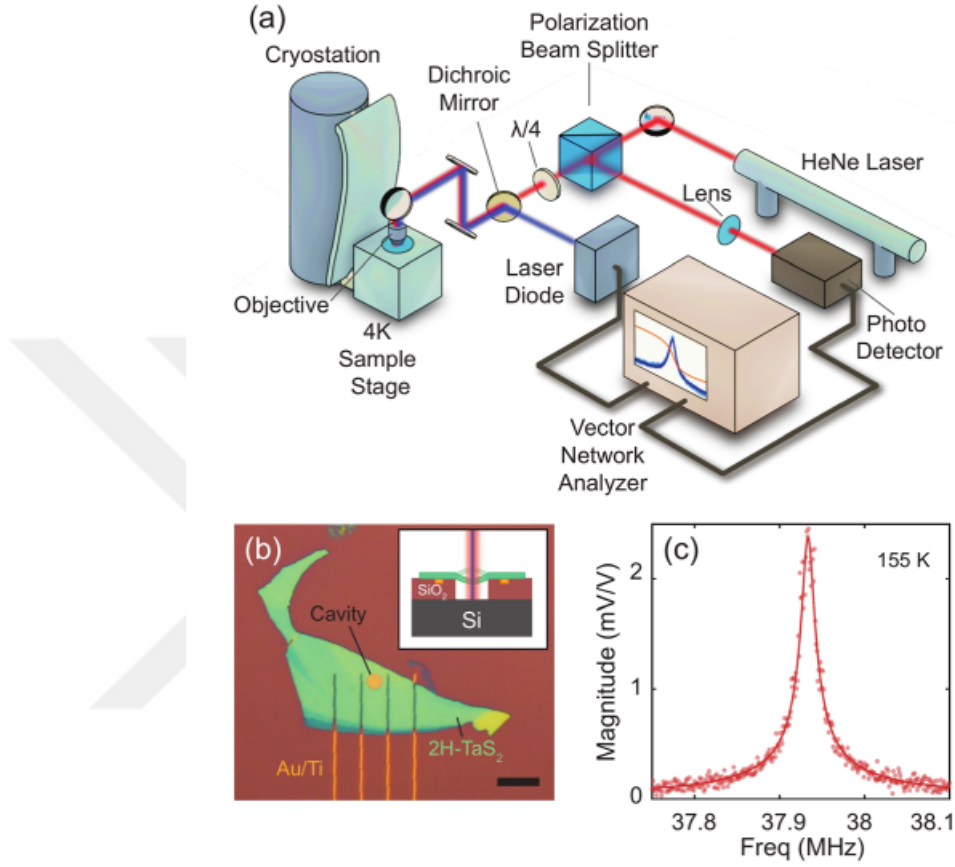


Figure 1.6: Suspended 2H-TaS₂ and interferometry setup[25]. © Copyright 2023 AIP

In short terms, strain testing environments are also very frequently used to attain an insight into how the phase transition mechanism works in strongly correlated materials.

1.4 Outline

This thesis, in general, includes an introduction and an investigation on an alternative strain apparatus called Micro-electro Mechanical System (MEMS) chips. MEMS chip approach will be introduced as a novel and unique platform for strain

testing experiments.

In the first part, the motivation of the project will be described briefly in terms of some drawbacks of conventional strain methods such as flexible substrates or Nanomechanical Strain Apparatus. Then, description of MEMS chip will be provided both verbally and schematically including its fabrication procedure. A small optical image demonstration of how MEMS chip works will be included in this part.

The third chapter will cover the MEMS chip application on exfoliated 2H-MoS₂ single crystals. Raman spectrum response, piezoresistive behavior and photocurrent response of MoS₂ with biaxial strain will be provided. Discussions on thermoelectric junctions caused by substrate variation will be expanded in this section.

The fourth chapter discusses the significance of controlling internal stress in materials undergoes phase transitions. An example which is Vanadium sesquioxide (V₂O₃) is provided as an example. It includes some V₂O₃ Chemical Vapor Deposition (CVD) growth optimization results. Then, it shows how to transfer crystals from the growth substrate to the channel of the MEMS chip and shows one complete device.

Chapter 2

Motivation and Description on MEMS Chip

As described in Chapter 1, various experimental studies such as strain engineering, mechanical testing, substrate engineering etc, are very significant to test the possible implementation of low-dimensional materials. Well-known experimental environments in this case can be exploiting flexible substrates such as polyethylene terephthalate (PET)[18] or using highly complicated Nanomechanical Strain Apparatus [23] setups. Even though flexible substrates are the most commonly utilized experimental systems in strain testing, many different experimental environments have been used to investigate interesting strain-dependent properties. This section will cover mainly some of these approaches and try to discover their advantages and disadvantages. After a short discussion in conventional setups, MEMS approach will be introduced and discussed how it can help to overcome these difficulties.

2.1 Advantages and Disadvantages of Conventional Strain Setups

Flexible substrates are one of the most frequently used experimental setups in strain-dependent measurements. It is a well-developed technique where a low dimensional material is transferred on a flexible substrate by either mechanical exfoliation or Polymethylmethacrylate (PMMA) assisted transfer approaches. Then, assuming the material has good adhesion with a flexible substrate, tensile and compressive stress can be applied by bending the substrate. Even though it can be a very useful and appropriate method, it still has some disadvantages. In certain situations, it is demonstrated that some failure modes can appear in flexible substrates which are not only suitable for practical applications but also misleads the data interpretation[26]. Figure 2.1 shows the transfer process of fabricated Si ribbons to the flexible substrate and it shows the 3 failure mode emerged while bending the substrate such as cracking, slipping and delamination[26].

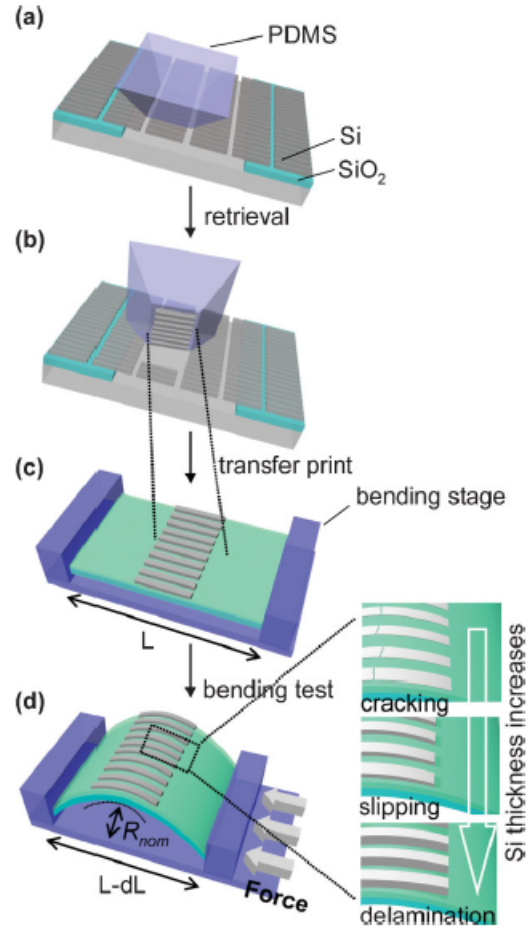


Figure 2.1: Failure Modes can be observed in various degrees of bending[15].
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Slipping and cracking effects are very frequently reported in the literature as varying the output of the experiment[27][26]. Figure 2.2 shows an SEM image that shows the effect of bending flexible substrate on silicon ribbons. It is clearly seen how ribbons cracked after bending[26].

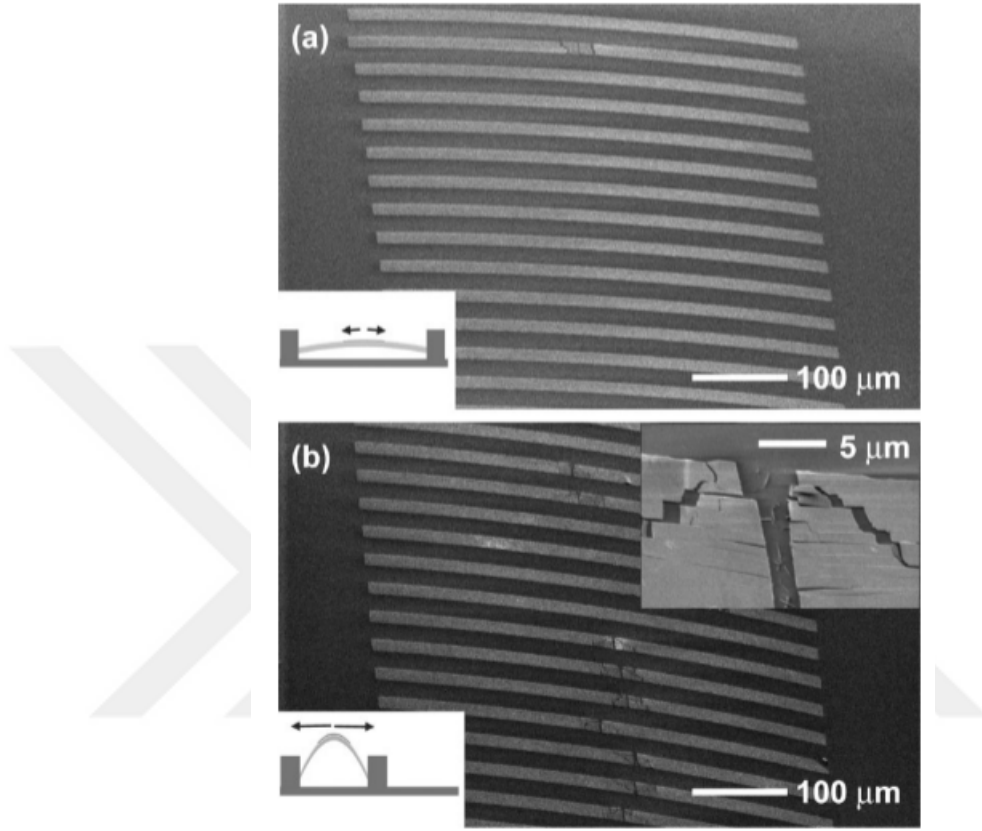


Figure 2.2: SEM images of Silicon Ribbons on slightly (a) and hardly (b) bended flexible substrate[20]. Copyright: 1999-2023 John Wiley & Sons, Inc

Apart from the flexible substrate approach some self-build Nanomechanical strain apparatus systems [23], different configured NEMS systems[3] and nanoindentation with AFM tip [28] are exploited for strain testing approaches. Adjusting gas pressure on suspended 2D materials also used to test optical property variations under stress[29]. These methods generally require big and cumbersome equipment or time and energy-consuming fabrication processes. An example of strain-apparatus is provided in Figure 2.3[26].

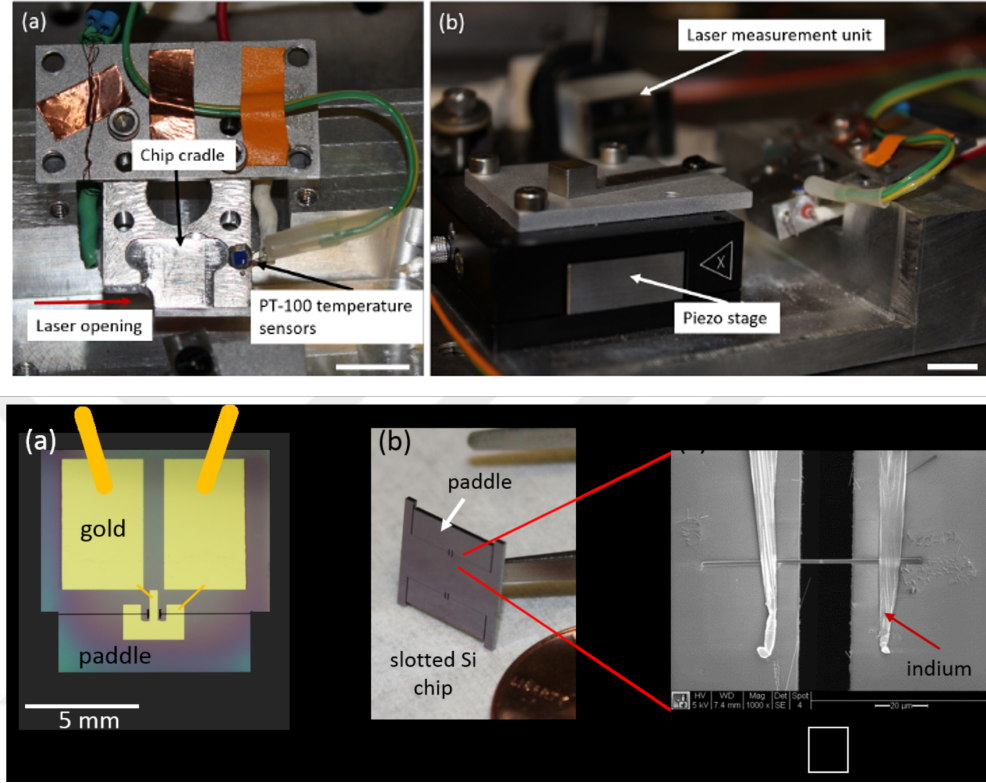


Figure 2.3: Nanomechanical Strain Apparatus[14]. Copyright © springer Nature 2013.

Apart from overcoming these drawbacks of conventional methods, the ability to alter electron-phonon scattering by the substrate engineering is recently observed[20]. The motivation of the thesis is to introduce unique, novel and substrate modifying experimental setup for strain testing. Self-designed MEMS chips in this regard are a very compact and efficient way of testing. The setup will be introduced in next section briefly.

2.2 A Brief Description of MEMS Chips

MEMS chip is a compact and straightforward experimental system to test low-dimensional materials mechanically. The system is comprised of an oxidized Si

substrate where approximately $10\text{ }\mu\text{m}$ width channel is etched by Inductively Coupled Plasma (ICP) dry etching. We collaborated with the Xfrontier Lab group in the manufacturing process. This process includes mask writer to prepare the desired pattern on Photoresist coated oxidized Silicon substrate. Then, Chromium evaporates on the substrate and the lift-off process proceeds to protect the rest of the substrate from etching. Then, the ICP process is applied to deep etch Silicon. The schematic demonstration and optical pictures in 20x and 100x pictures are provided in Figure 2.4.

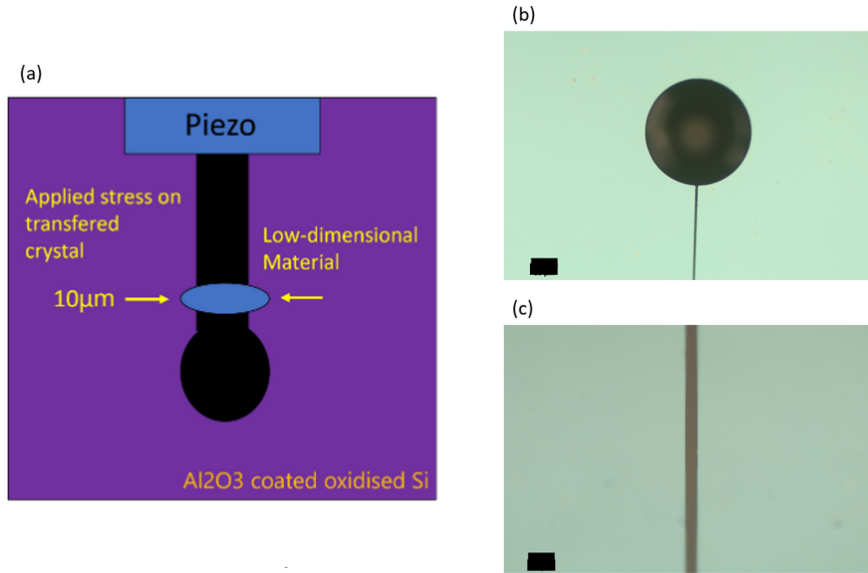


Figure 2.4: (a) Schematic of transferred crystal on MEMS chip. Optical images of MEMS chip under a microscope, (b) Scale bar is $50\text{ }\mu\text{m}$. (c) Scale bar is $10\text{ }\mu\text{m}$

After the fabrication Process of the MEMS chip, a low-dimensional material has to be suspended on the channel of the chip for testing. Transferring process may differ depending on the material we are working. If the subject material is Van der Waals TMDC which can be cleaved easily, simple mechanical exfoliation and all dry transfer is suitable for transferring. Nanowires can be fabricated by drop-casting around the channel with a thin pipette. If the concerned material is a Chemical Vapor Deposition (CVD) grown material, process requires

Poly-methyl methacrylate (PMMA) or some other proper polymer assisted wet-transfer process[30]. Details of device fabrication specifically for MoS_2 and V_2O_3 will be elaborated in chapter 3 and chapter 4. After transferring the crystal on the channel, fixing a piezo-actuator with glue and soldering allows us to control expansion and contraction of Piezo by applying piezo-voltage from the piezo controller (Figure 2.5), so that the channel as well. In the end, opening and closing the channel applies stress on low-dimensional material if it is properly fixed.



Figure 2.5: Piezo-controller

In Figure 2.6, it is demonstrated how MEMS system works with an example of a prepared MoS_2 crystal. From the piezo-controller, voltage is applied so that piezo expand or contract where gap opens and close simultaneously. The effect on MoS_2 crystal is demonstrated in Figure 2.6. In the beginning, the crystal is bulged up around the channel in 0V piezo-voltage which is inevitable in dry-transfer method. When piezo-voltage is increased to 90V, bulged part straightens and the crystal is expected to experience stress. We should emphasize the correlation of piezo-voltage which is controlled by Piezocontroller (Figure 2.5) with gaps opening. From the rest of the thesis, the term of piezo-voltage will be referred to opening and closing of the gap of MEMS chip.

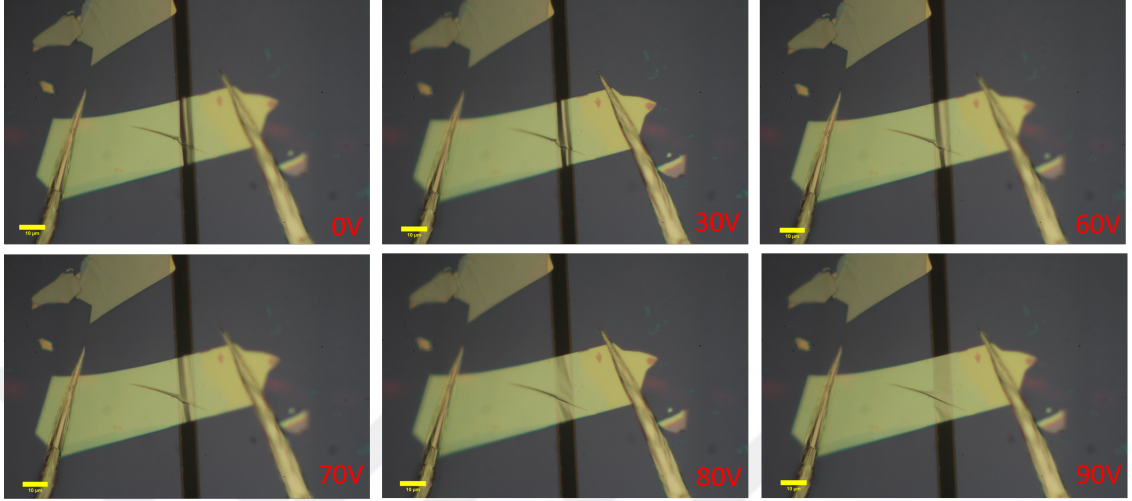


Figure 2.6: Optical image demonstration of crystal on MEMS chip from 0V piezo-voltage to 90V piezo-voltage

In the upcoming sections, fabrication processes will be elaborated and some results of applying stress will be provided.

Chapter 3

MEMS Chip Investigations on single material 2H-MoS₂ crystals

As described in the introduction, MoS₂ is a highly intriguing material for both academic and industrial purposes. Even though MoS₂ has been extensively studied in the last decades and a broad literature is formed, interesting phenomena remain. MoS₂ is a highly suitable material for mechanical testing experiments because of its high endurance to considerable strain without degradation[31]. In addition, its optical and electronic property variations depending on strain make it an intriguing material in strain measurements[31]. This section will cover our investigation of the stress-dependent behavior of MoS₂ with our new experimental setup introduced in the thesis. After a detailed device fabrication description, Raman spectroscopy, SPCM and Resistance measurements under stress will be provided.

3.1 Device Fabrication

Device preparation involves several steps. First, oxidized Silicon MEMS chips are coated with 50nm Alumina to passivate the substrate further, reducing leakage

with Atomic Layer Deposition. We found that at least 50nm Alumina is enough to suppress the background signal. To coat 50 nm Alumina, 471 cycle of layer coating is applied. After the passivation of MEMS chips, MoS₂ crystals are exfoliated on small pieces of Polydimethylsiloxane (PDMS) on a glass slide and they are checked to find relatively thin crystals. Then, PDMS on the glass slide is controlled and aligned with an approximately 10 μ m channel on the MEMS chip. After PDMS touches the chip, the heat stage is heated up to 50 °C degree. Then slowly PDMS is removed from the substrate and the crystal stays on the channel. The micromanipulator stage is provided in Figures 3.1.

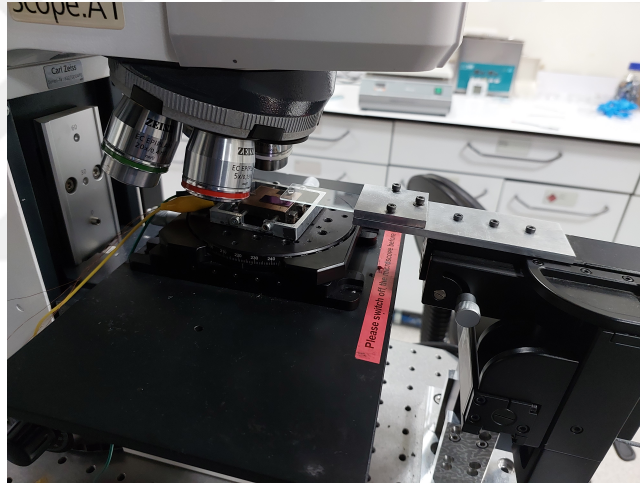


Figure 3.1: Micromanipulator transfer setup

In Figure 3.2 all-dry transfer procedure explained above is demonstrated by showing an optical image. In the first figure, a suitable relatively thin crystal on PDMS is shown. When PDMS is touched to the substrate, the color change is observed. This allows us to control the whole process.

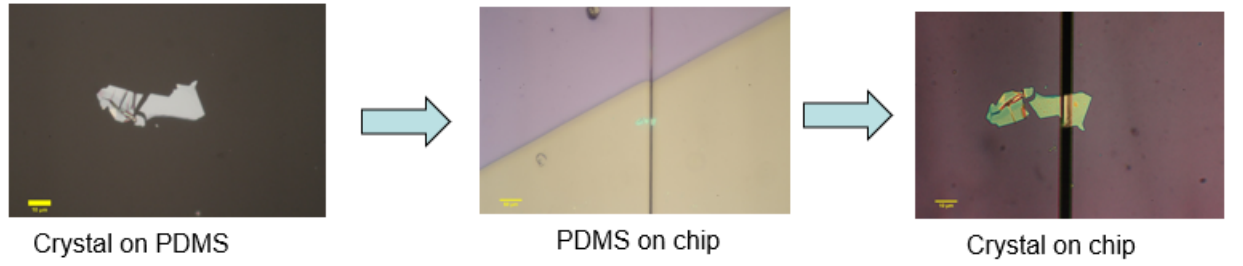


Figure 3.2: Optical image demonstration of all-dry transfer method

A challenge in the process is that since parts of crystals on the channel are suspended, they bulge up when disconnecting PDMS. Still, bulged portion can be straightened by opening the gap applying piezo voltage. In addition, since the bulged part experience strain by itself, experiment can be proceeded by releasing the strain instead of applying.

The next step is to place electrical contact to make a good Ohmic contact device which will be a high-quality electronic and optical device. In order to do that, drawing indium contacts with a tungsten needle connected to a micromanipulator is exploited. The tungsten needle setup is provided in Figure 3.3. Conventional device fabrication techniques with lithography and patterning is not preferred because of MEMS chip's fragility, which may not endure these harsh clean room processes. In addition, conventional methods may induce minor defects in the crystal which causes an increase in contact resistance at high temperatures led by Fermi-Level Pinning[32].

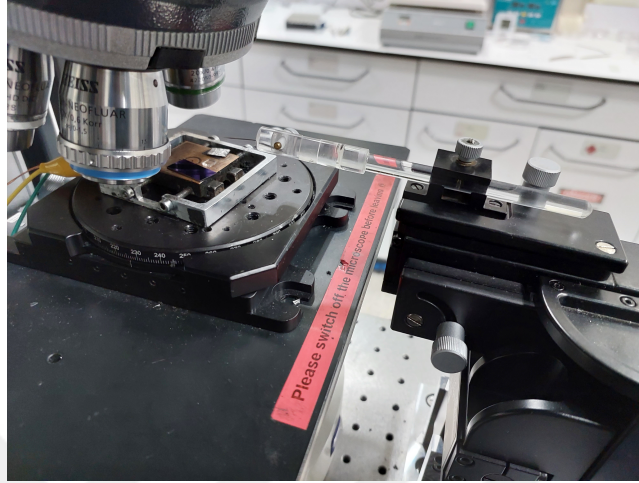


Figure 3.3: Setup of Tungsten Needle Micromanipulator

The indium contact procedure is listed below and demonstrated schematically in Figure 3.4. The biggest advantage of this device fabrication method is that it can be completed in only 10 minutes. This makes it a much more practical method compared to conventional methods.

1. Heat up the hot plate around 170 °C.
2. Approach with tungsten needle.
3. Touch the molten indium with tungsten needle.
4. Draw backward while lifting the tungsten needle.
5. Move to desired location
6. Place down the indium needle
7. Follow this steps to make second contact.

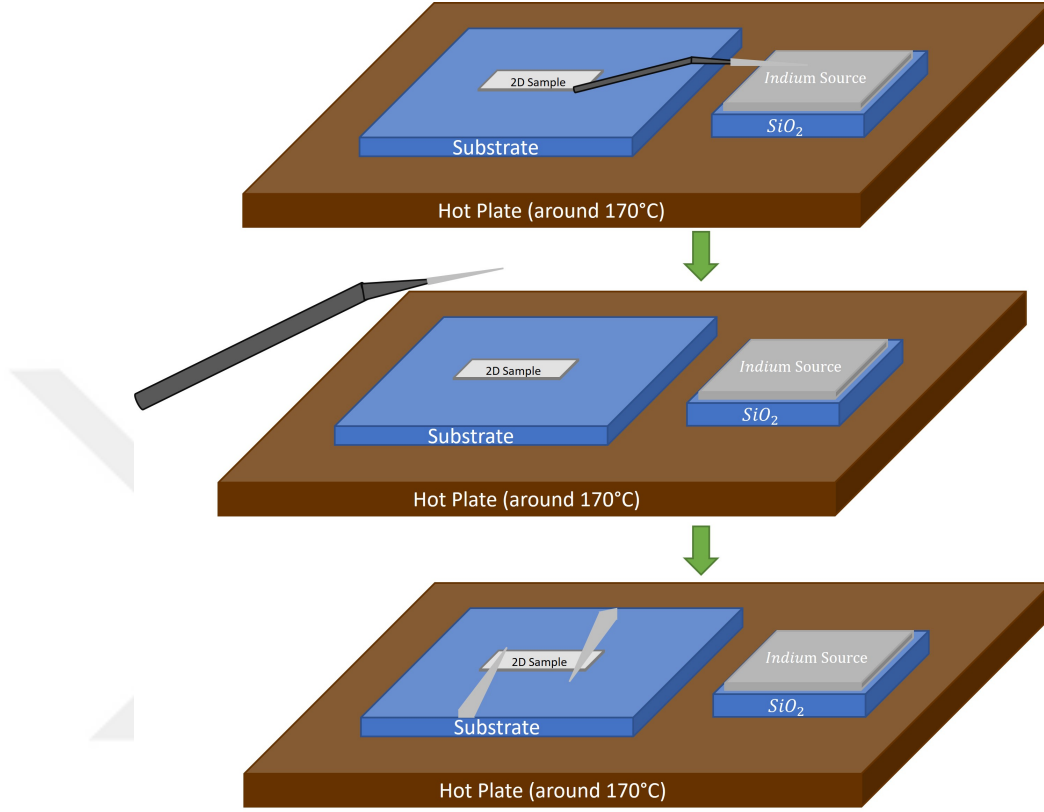


Figure 3.4: Schematic representation of Indium contact procedure

After indium contacts are placed, the device is checked under Voltage-Current (IV) measurements to ensure it has good Ohmic contact. An example of a prepared device and its IV measurements is provided in Figure 3.5.

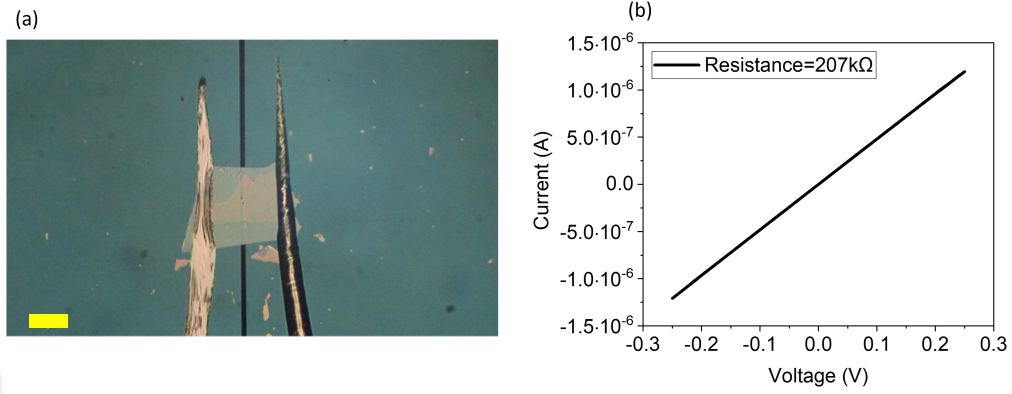


Figure 3.5: (a) An example of Indium contact MoS2 MEMS device. Scale bar is $50\mu\text{m}$. (b) IV behavior shows good Ohmic contact with approximately $207\text{k}\Omega$.

After ensuring good Ohmic contact, Piezo is placed on front of MEMS chip to complete device. A completed device is demonstrated in figure 3.6. The completed device (Figure 3.6a) is connected to piezo-controller and placed in measurement setup (Figure 3.6b).

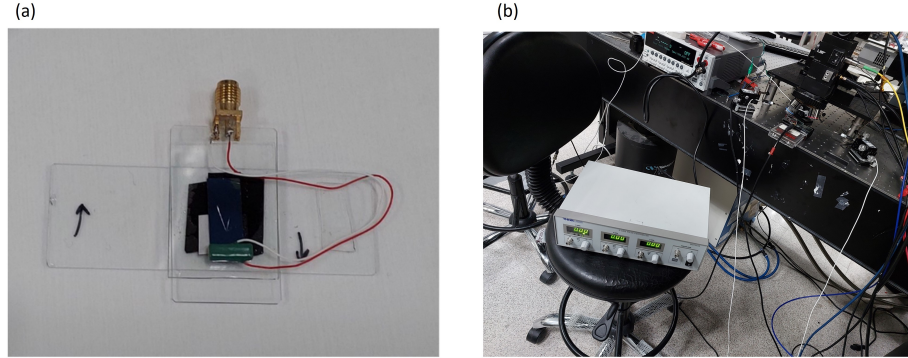


Figure 3.6: (a) Completed MEMS device (b) MEMS device connected to Piezo-controller under microscope. Resistance and SPCM measurements are taken in this setup

3.2 MoS₂ Raman Analysis Under Biaxial Strain

In order to understand whether the introduced MEMS chip approach can apply stress on a few-layer MoS₂ crystal, observation of in-plane Raman mode (≈ 385.3 cm⁻¹) is a suitable investigation. The strain effect on in-plane Raman mode of monolayer (E_1^{2g} mode) and few-layer MoS₂ has been demonstrated[33] and extensively studied for various thicknesses[13]. Since strain in MEMS system is applied in-plane, out of plane mode (A_{1g} mode) is not expected to be affected. Additionally, it is demonstrated that even suspending the MoS₂ crystal causes a redshift upon only in-plane mode. Schematic demonstrations of two characteristic modes of 2-H MoS₂ are shown in Figure 3.7 and an example of the difference in suspended and supported parts is shown. It can be seen that in-plane mode (E_1^{2g}) is much more shifted than out-of-plane mode (A_{1g}) as expected since the contribution of in-plane stress is more than out-of-plane (Figure 3.7d).

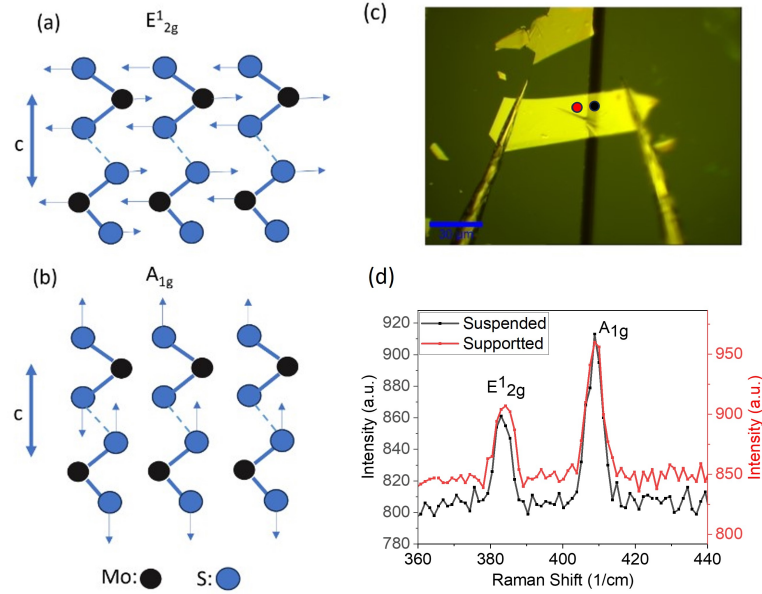


Figure 3.7: (a) in-plane (E_1^{2g}) mode of 2-H MoS₂ (b) out-of-plane mode of 2-H MoS₂ (c) MoS₂ crystal on MEMS chip. The points of Raman spectrum taken denoted with same colour code (d) Raman spectrum of suspended and supported parts

Then, we concentrated on behaviour of Raman modes when opening the gap. The expectation is that applying bi-axial stress while opening gap causes even more redshift in E_1^{2g} . To test this, we prepared a MoS₂ MEMS device with relatively wider indium contacts to reduce possible sliding effect. Figure 3.8 shows optical images of device for corresponding piezo-voltage from 0V to 150V which is opening the gap and each corresponding Gaussian fit Raman data. In the E_1^{2g} mode, until 50V an expected redshift is observed as peak center shifted from 383.86 to 383.39. After that, in 100V we saw that trend is reversed and peak shifted right to 383.62. If the optical image carefully observed, in 100V a crack can be seen which causes reversing the redshift trend. Following the crack, crystal started sliding and E_1^{2g} mode blueshifted. Another observation is that even until 50V, shift is not so dramatic and considering the thickness dependency of Raman shift[27], this sample may be excessively bulkish to be able to observe the shift. Apart from that, A_{1g} mode did not influenced by the process at all which is expected because it is out-of-plane mode where biaxial strain is applied. Center of peaks of Gaussian fit Raman data are provided next to each piezo-voltage measurement (Figure 3.8b and Figure 3.8c).

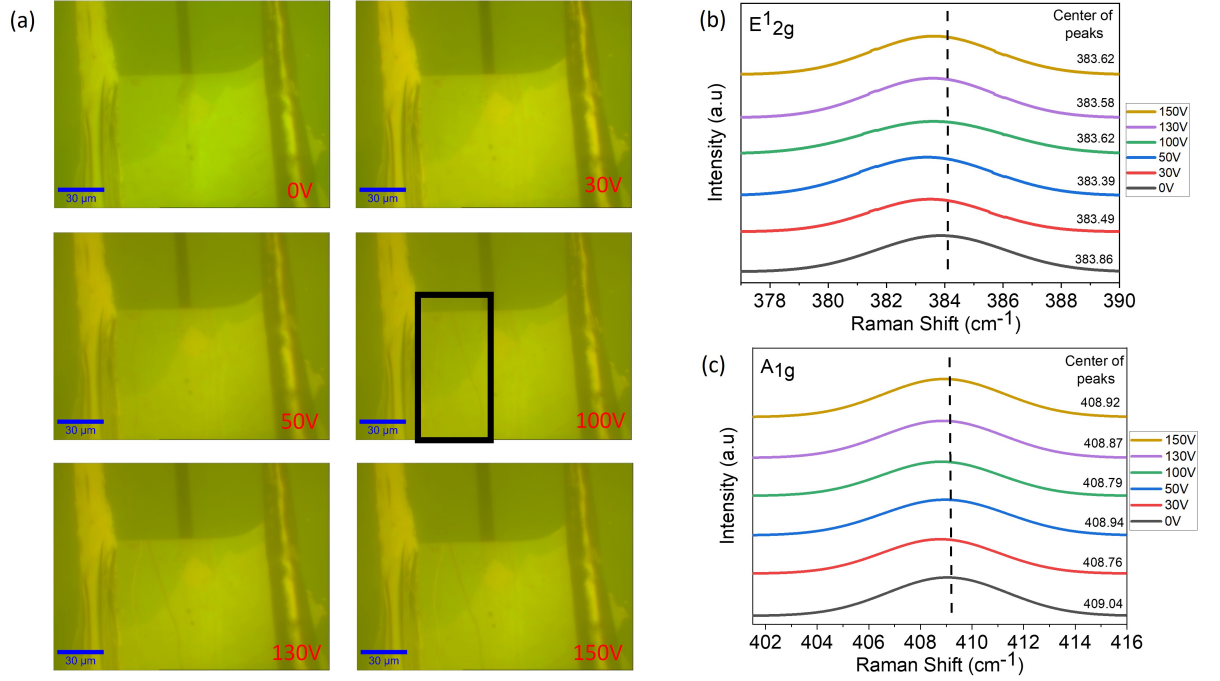


Figure 3.8: (a) Optical Images of MoS₂ MEMS device for each Piezo-voltage (b) The behaviour of E₁^{2g} mode by each piezovoltage (c) The behaviour of A_{1g} mode by each piezovoltage

A relatively thinner device is prepared and shown in Figure 3.9 for the sake of testing thickness dependency on shift. Raman measurement is taken from the crystal's upper thin part (Figure 3.8a), which is indicated with a green dot. Gaussian fits of Raman spectrum of E₁^{2g} mode for each piezovoltage where the gap is opening relatively is obtained. In 0V piezovoltage, crystal is already bended and it is experiencing stress. When the gap opened slightly and the crystal relaxed, we saw a shift in E₁^{2g} where it becomes stress-free in 10V piezovoltage. Following that opening the gap applies biaxial stress on the crystal so that shift on lowering frequency by -1.22 cm⁻¹ is observed. Behavior from 10V to 50V indicates a biaxial stress is applied without too much sliding effect. The optical images for 0V piezovoltage and 50V piezovoltage reveals that crystal is straighten and even experiencing further strain because of wrinkles on crystal (Figure 3.9c, 3.9d). Figure 3.9e summarize the behavior of E₁^{2g} mode where we can see a

consistent behaviour.

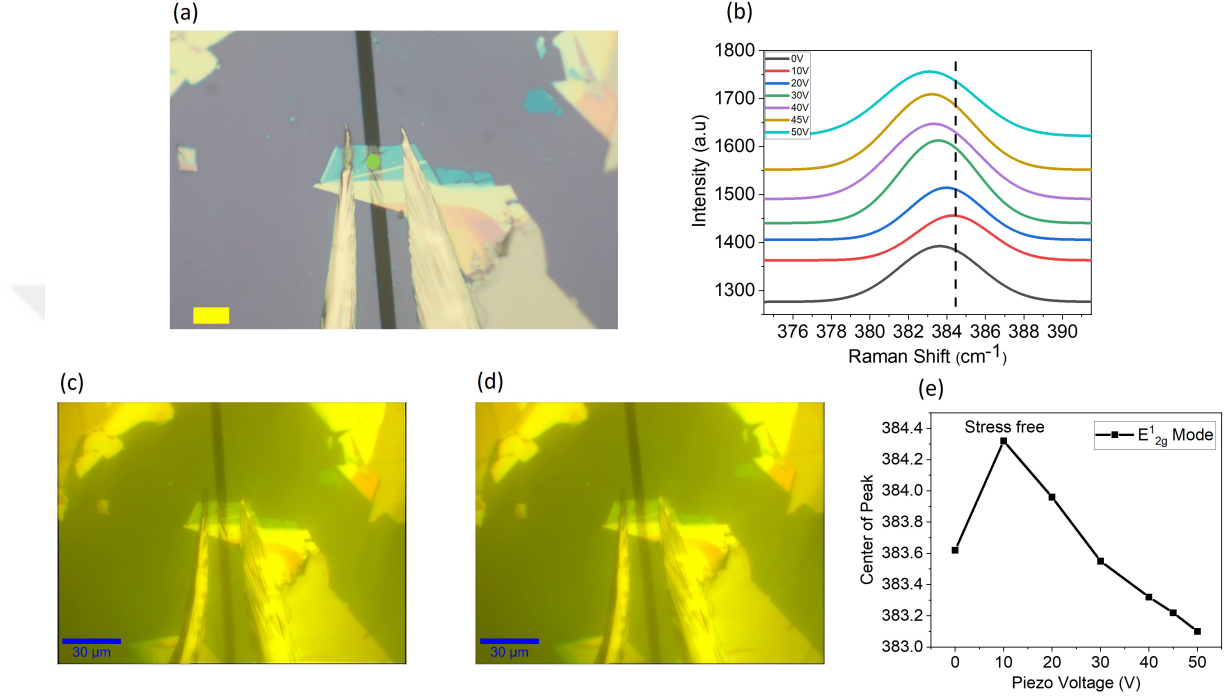


Figure 3.9: (a) Optical Image of MEMS device. Laser sent to the thin part demonstrated by green spot. Scale bar is 10 μ m (b) The behaviour of E₁^{2g} mode by each piezovoltage (c) Optical image on Raman microscope in 0V (d) 50V. (e) Shift behaviour of E₁^{2g}.

To demonstrate the shift more clearly, Raw data together with gaussian fits for 10V piezovoltage case and 50V piezovoltage case is plotted in Figure 3.10. It also includes the optical pictures for comparison (Figure 3.10a and 3.10b).

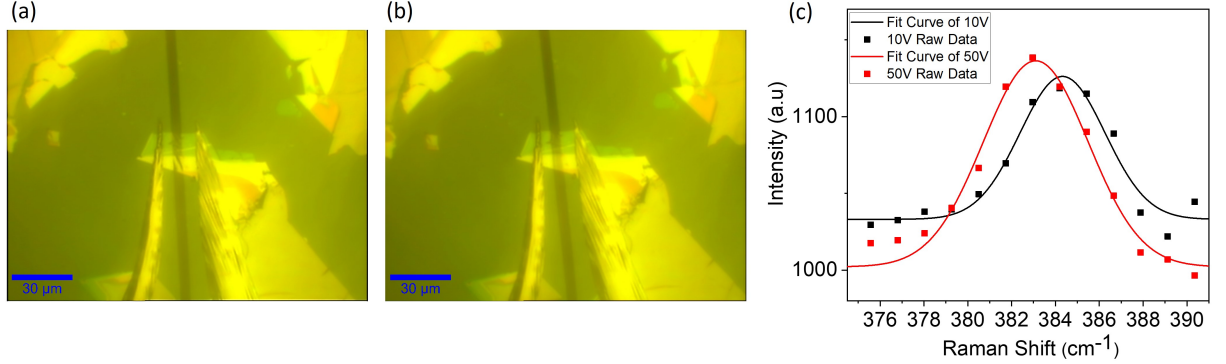


Figure 3.10: Optical Image of MEMS device in 0V piezovoltage (a) 50V Piezo-voltage (b). (c) Raman spectrum Gaussian fit data together with raw data taken from 0V and 50V

As a result, if a relatively thin crystal can be exfoliated for transferring on MEMS device, E_{2g}^1 Raman peak is responding biaxial stress. It is a good way to ensure that device is properly working and crystal is experiencing stress.

3.3 Piezoresistance Measurements

Since MoS_2 can withstand high strain, it is a well-suited material for Piezoresistance measurements. For the case of MoS_2 , mechanisms such as piezotronic effect and piezoresistive effect play roles in changing resistance with respect to stress level[34]. The piezotronic effect is an asymmetrical modulation in Schottky barriers whereas the piezoresistive effect is the bandgap change modulating entire resistance both strain-induced effects[34]. Nevertheless, both contribute to resistance change, observation of the piezotronic effect is easier near to the Monolayer limit[19] [34].

Below, figure 3.11 demonstrates resistance change with gaps opening. Current-Voltage (IV) variation for each piezo-voltage is indicating a slight decrease and then small increase in after 140V (Figure 3.11 a and c). Optical images reveals

that 140V is the point where crystal becomes straight and nearly stress free (Figure 3.11b). To understand the resistance behaviour better, we wrote a Labview program which controls the piezo-voltage while measuring resistance applying constant voltage (Figure 3.11d). Resistance measurements clearly show stress related resistance change which is symmetric indicating piezotronic mechanism does not play role here because of relatively thick crystal. We tried experiment by closing lights and we got a similar behaviour except an overall decrease in resistance because of diminishing contribution of photovoltaic effect.

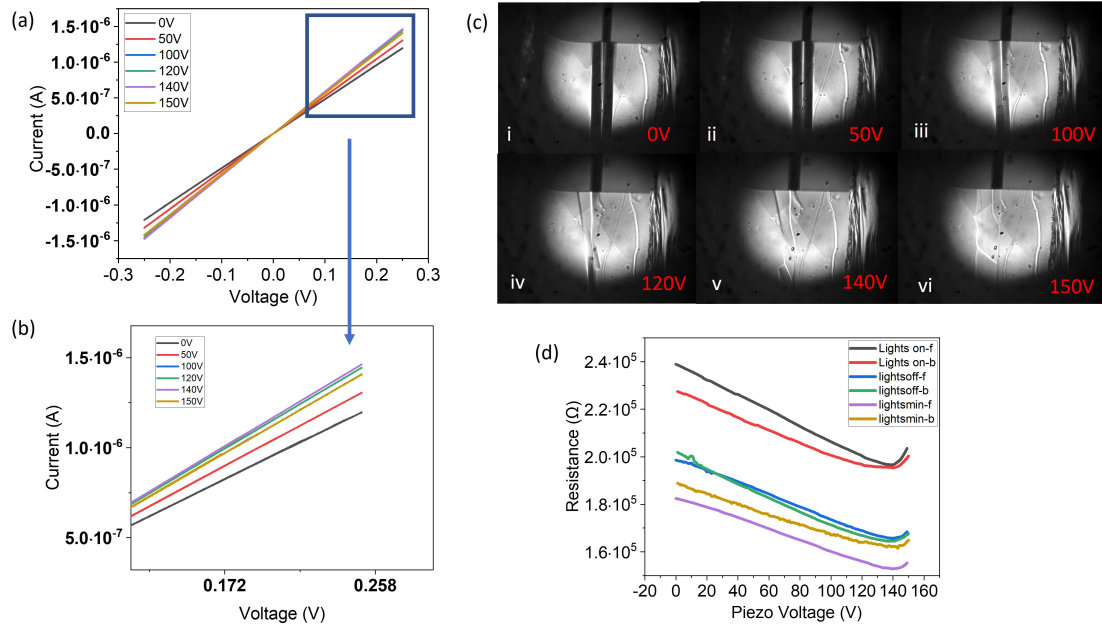


Figure 3.11: (a) IV behaviour for different piezo-voltages between 0V and 150V (b) Enclosed version of figure (a). (c) Optical Images of crystal with each piezo-voltage condition. At 140V crystal starightens which is stress free point. (d) Resistance behaviour with respect to Piezo-voltage

Piezoresistive behaviour can depend on various parameters as Crystal Structure[35], Band Structure[19][34], Carrier Concentration[36], Strain Magnitude[37][38]. Although near to monolayer limit, MoS₂ shows a negative piezoresistive behaviour[19] as resistance is decreasing by strain, in some configurations, it also shows positive piezoresistance[39] where its resistance increase by

strain. After expressing that piezoresistance is not a straightforward phenomenon since it depends on various parameters, all of our MoS₂ MEMS devices exhibit positive piezoresistive behavior.

Figure 3.12 demonstrates another example where actually opening gap was not enough to straighten the crystal. Optical images show that gap was opening but even in 150V piezo-voltage crystal has still bended around the channel (Figures 3.12a and b). This device has another situation where we are not applying stress but releasing it where it already feels. The effect can be seen in resistance behaviour as resistance is decreasing while opening the gap and reverse in opening the gap (Figures 3.12a and b). Resistance behavior is a good indication of releasing stress of crystal. Another evidence is the photoluminescence (PL) measurement for both condition. We found a slight shift toward right at 150V, indicating releasing stress (Figure 3.12d).

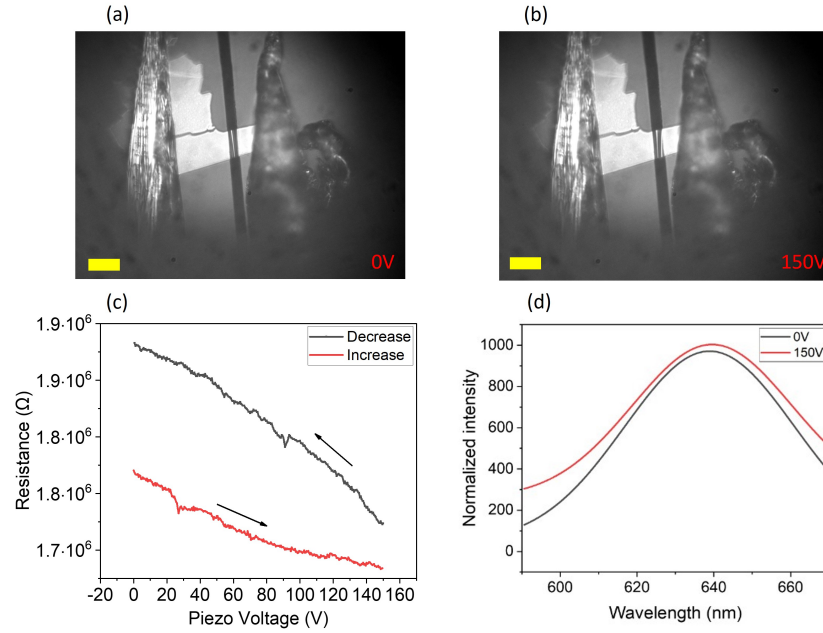


Figure 3.12: Optical Images at 0V (a) and 150V (b). Scale bar is 25 μ m. (c) Resistance with respect to Piezo-voltage. (d) PL spectrum at 0V and 150V piezovoltage.

The measurements upon piezoresistance demonstrate that the crystal is responding to gap opening as a positive piezoresistor. Although the entire mechanism of piezoresistance is not a straightforward subject and is still debated, in our case this measurement can be exploited to be ensuring we are changing the stress of low-dimensional material. Although it was hard to observe Raman shift by stress since some crystals were not thin enough, resistance measurements consistently indicate applying stress. It showed itself in all devices regardless of thickness.

3.4 Photocurrent Generation Analysis of Thermoelectric Junction

Photocurrent generation analysis provides extensive information on light-matter interaction. The mechanism behind the photocurrent generation can be attributed to many physical phenomena. The phenomena mainly depend on the electron-hole generation when photon interact with low-dimensional material. One mechanism called the photoconductive effect depends on the charge carriers which are electron-hole pairs generated by the absorption of photons with larger energy than bandgap. These extra charge carriers cause decreasing in resistance by more current passing through. Generally, it is hard to see this effect without bias voltage because there is no predetermined direction to move charge carriers. Another mechanism called photovoltaic effect is similarly correlated with electron-hole pair generation. In this mechanism, a built-in electric field formed by the Schottky barrier at the metal-semiconductor interface can separate electron-hole pairs which generate current[40]. Figure 3.13 is the simplified demonstration of Photovoltaic effect.

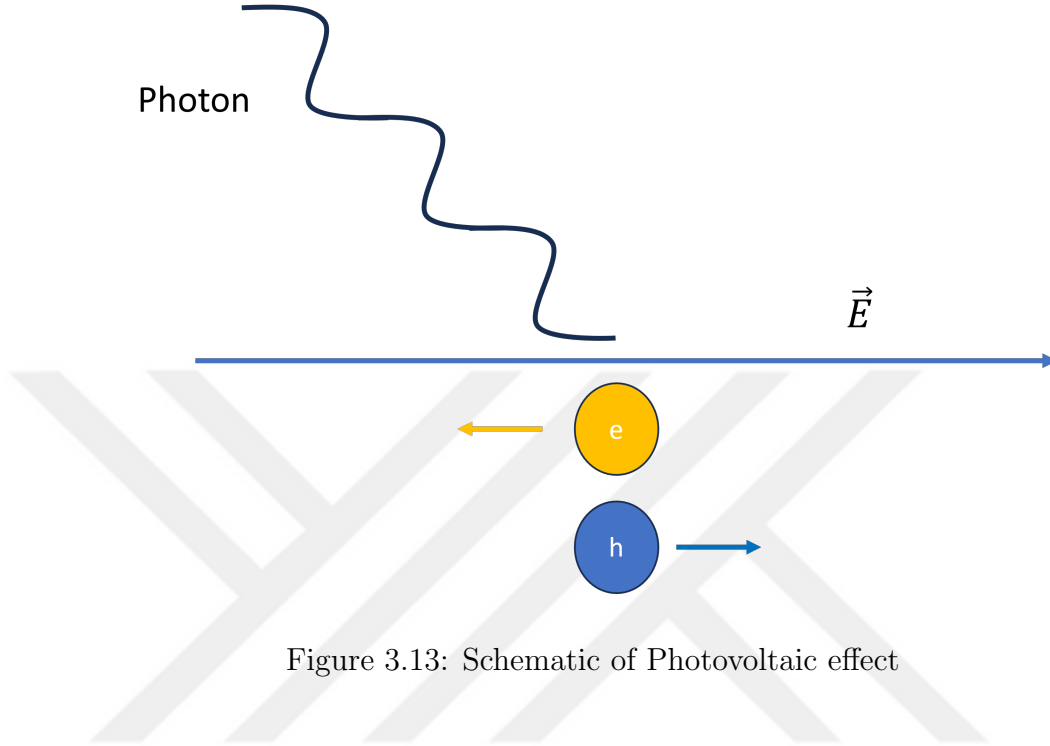


Figure 3.13: Schematic of Photovoltaic effect

Additionally, there are thermal mechanisms that can drive the charge carriers called the Bolometric effect and the photothermoelectric effect as well. In the bolometric effect, heating results a variation in resistance which can control the current passing through material[41]. The photothermoelectric effect is observed in junctions with different Seebeck coefficients which leads temperature gradient around the junction. This temperature gradient is connected to Voltage change linearly dependent on Seebeck coefficient as[40][42],

$$\Delta V = -S \cdot \Delta T \quad (3.1)$$

In our results, we are mainly investigating the Photothermoelectric effect. The substrate engineering as opening a channel causes a Seebeck coefficient variation and causes a bipolar photocurrent response. These results will be discussed elaborately in next sections providing outcomes.

Measurements are done with 532 nm laser wavelength and power is kept low around 20 μW to inhibit heating effect. The laser is chopped with a mechanical chopper before interacting with the sample. Chopping laser is necessary because it

enables lock-in amplifier to enhance only on-purposed chopping frequency. Then, laser-induced variation in device current can be measure by Lock-in amplifier. In the conditions of very week signals mostly occurs in metallic samples, a pre-amplifier (SR-570) is used to enhance signal before reaching lock in amplifier. Simply, it can be used to apply bias voltage. Schematics of SPCM setup is demonstrated in Figure 3.14.

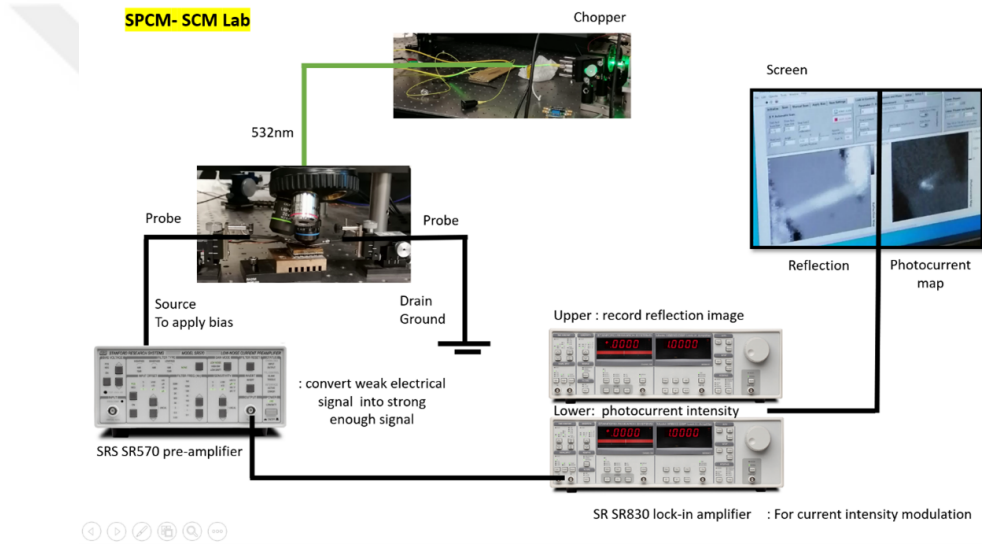


Figure 3.14: Schematics of SPMC setup

3.5 MoS₂ Thermoelectric Junction and MEMS Chip Testing

In low-dimensional materials, especially 2D materials, material properties can be strongly adjusted via correlation with substrate because of the big surface-to-bulk ratio[43][44][45][46][47][48]. Therefore, developing an understanding between substrate and material is crucial. Hence, thermoelectric junction in suspended single material MoS₂ is investigated based on the Seebeck coefficient difference between suspended and supported parts[20].

It is assumed that the complete Seebeck coefficient consists of energy-dependent diffusion (S_n), scattering (S_τ), and phonon-drag (S_{pd}) terms[49][50]. Here, the overall Seebeck coefficient can be written as $S = S_N + S_\tau + S_{pd}$ where S_N and S_τ terms can be derived from Mott relation[20]

$$S_\tau = -\frac{\pi^2 k_b^2 T}{3e} \left. \frac{\partial \ln \tau}{\partial E} \right|_{E=E_F} \quad (3.2)$$

and

$$S_N = \pm \frac{k_b}{e} \left[\frac{E_F - E_C}{k_b T} - \frac{(r+2)F_{r+1}(\eta)}{(r+1)F_r(\eta)} \right] \quad (3.3)$$

where k_b is the Boltzman constant, T is the temperature, τ is the relaxation time, E is energy, E_f is the fermi level, E_C is the conduction band energy, r is scattering parameter[20] and $F_m(\eta)$ is m-th order Fermi integral[51]. S_τ is 0 in equation 3.2 because τ does not depend on energy in 2D limit. The final term in the complete overall Seebeck coefficient can be defined as,

$$S_{pd} = -\frac{\beta v_p I_p}{\mu T} \quad (3.4)$$

where μ is electron mobility, β is a modifying parameter of electron-phonon interaction varying from 0 to 1, v_p is group velocity and I_p is mean free path of phonon[50]. In this term, 2 parameters, I_p and μ , vary by substrate presence or even impurities on the substrate. This condition causes a substantial variation in overall Seebeck coefficient[52]. Based on this discussion, substrate modification can cause a thermoelectric junction on suspended and supported MoS₂ single crystal.

Considering the terms contributing to overall Seebeck coefficient, S_{pd} is approximately calculated as $-100 \mu\text{VK}^{-1}$ for suspended MoS₂ and S_{pd} is approximately $-230 \mu\text{VK}^{-1}$ for MoS₂ on SiO₂ at room temperature. S_N term is very dependent to the presence of substrate as well. It is estimated between $-400 \mu\text{VK}^{-1}$

to $-200 \mu\text{VK}^{-1}$ assuming carrier concentration is changing from 10^{12} cm^{-2} for suspended to $3 * 10^{13} \text{ cm}^{-2}$ for supported[20]. Considering these, the overall Seebeck coefficient difference as a result of thermoelectric junction caused by substrate engineering is found,

$$\Delta S \approx 70 \mu\text{VK}^{-1} \quad (3.5)$$

This calculated value will be compared with the experimental outcome at the end of this section.

An example of a thermoelectric junction prepared on a MEMS chip is demonstrated in Figure 3.15. Optical image and reflection map reveal the bent part of MoS_2 on the channel which is the suspended part (Figure 3.15 a and b) at 25V piezovoltage applied. Observation of bipolar response around the suspended part in photocurrent map suggests thermoelectric junction (Figure 3.15c) similar to the results obtained on crystals on holes etched by Focused Ion Beam (FIB)[20]. Even though this response points out the presence of Photothermal effect (PTE), other mechanisms such as strain dependent modulations and flexo-photovoltaic effect may have a role in this result[53]. In this regard, MEMS chip is a very suitable experimental setup to test the contribution of strain because instead of the unintentional strain appearing around bulged up or down parts, it is possible to apply stress by controlling the gap of the chip. When 85V of piezovoltage is applied, it can clearly be seen that the crystal is stretched and under stress in the optical image and reflection map (Figure 3.15 d and e). SPCM map at 85V piezovoltage demonstrates a similar bipolar response with a slight variation in maximum photocurrent. This result might be the first evidence that dominating contribution comes from not strain-induced effects but substrate effect.

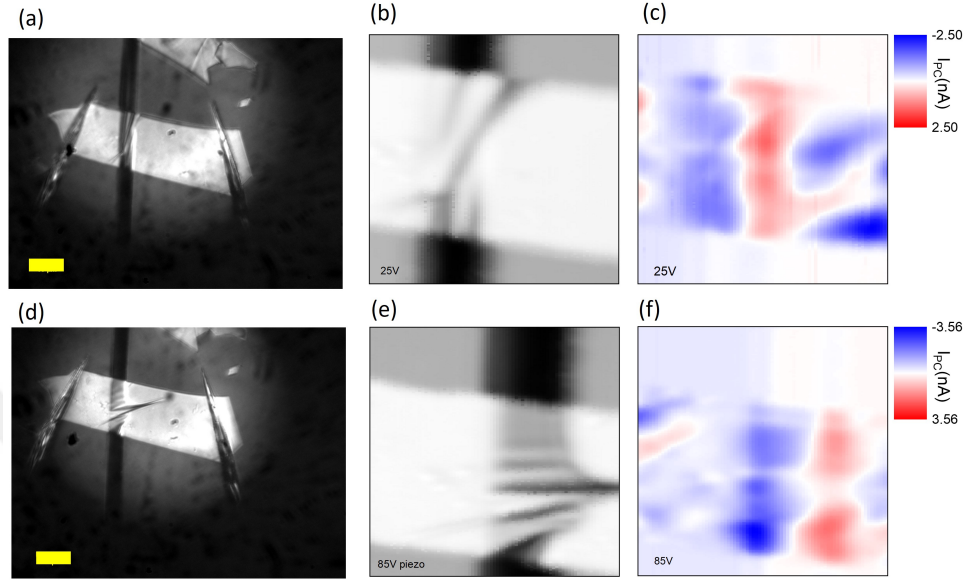


Figure 3.15: At 25V piezovoltage (a) Optical Image of MoS₂ (b) Reflection map (c) SPCM data. At 85V piezovoltage (d) Optical Image of MoS₂ (e) Reflection map (f) SPCM data. Scale bar 10 μm .

Although Figure 3.15 demonstrates the thermoelectric junction, some minor background signal from the substrate can be observed which might mislead the interpretation of the measurement. To inhibit this effect, we further coated our MEMS chip with thicker Alumina (80nm) to passivate the surface further. The response from the device is shown in Figure 3.16 which clearly eliminates the background signal with better passivation. SPCM results from this new device is demonstrated in Figure 3.16. It is the one that piezoresistance results in shown in the previous section (Figure 3.12). From photocurrent maps from 0V to 150V, the results of each measurement provide approximately the same outcome with small variations. This leads evidence of strain-related factors such that the Flexo-photovoltaic effect plays a minor role in this phenomenon. It gave a clue that still the mechanism that causes bipolar response is the photothermoelectric effect.

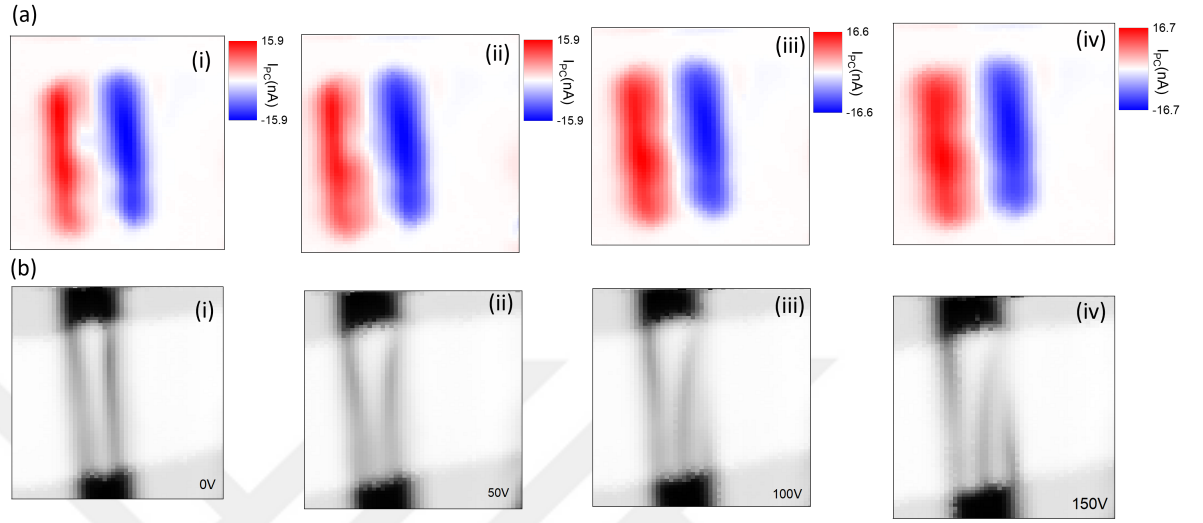


Figure 3.16: (a) SPCM mapping from 0V piezovoltage to 150V piezovoltage (b) Reflection maps of corresponding piezovoltages

In order to provide better insight, a line trace photocurrent behavior is plotted (Figure 3.17c). Black and Red lines denote the considered lines in the plot (Figure 3.17 a and b). This plot also suggests that the difference in generated maximum and minimum photocurrent are extremely close. This may reveal that the flexo-photovoltaic effect plays a minor role in this response.

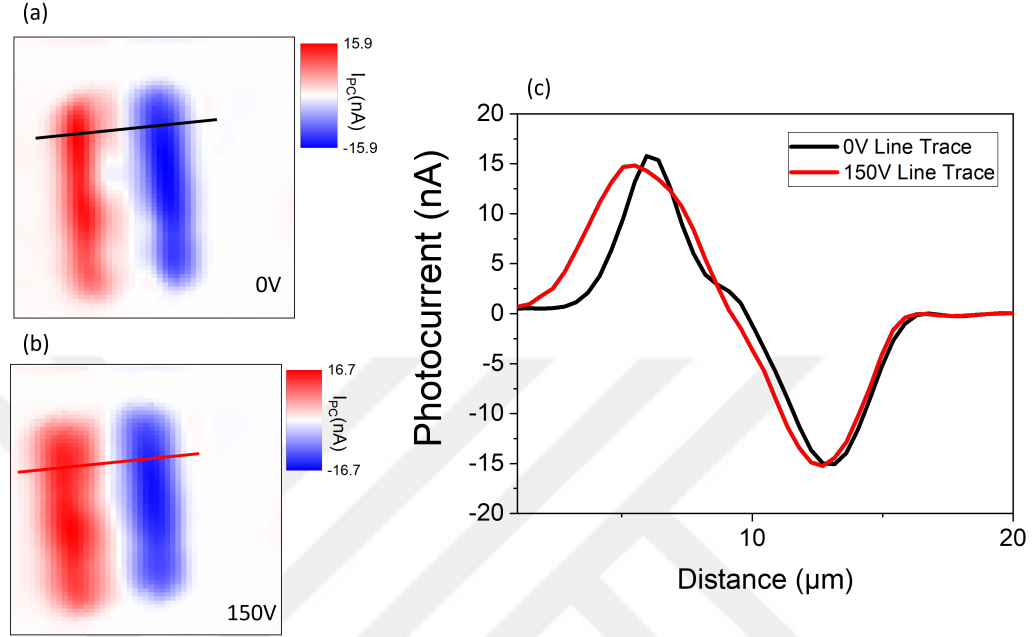


Figure 3.17: (a) SPCM mapping at 0V and (b) 150V. (c) Line trace to compare better. Black and Red lines in mapping denotes which part is to be considered.

The centrosymmetric nature of 2H-MoS₂ can explain this minor contribution. The strain effect depends on the directional polarization variation where the multilayer 2-H phase does not have[13]. For non-centrosymmetric structures on the other hand which is the 3R phase exhibit a strong strain-induced photocurrent generation caused by broken in-plane rotational symmetry. This symmetry-breaking causes in-plane polarization. In summary, the contribution of strain-induced effects such as the flexo-photovoltaic effect is negligible in centrosymmetric structured materials[13].

Although Figure 3.17 reveals a similar rate of photocurrent generation, there is a small shift in the positive Photocurrent part which means the location of response is changed as well by opening the gap. To understand this shift better, an image profile analysis is necessary. Figure 3.18 shows the image profiles of the 0V piezovoltage case. Image profiles of Photovoltage and reflection are plotted side by side (Figure 3.18 (a) and (b)). Then, reflection and PV along a line are plotted together (Figure 3.18 c). The reflection map reveals that there are

two buckles on the channel which will change in the 150V case. PV behavior gives evidence of bipolar response appearing on the suspended part and is not affected significantly by the buckle in the middle of the channel. This data is another piece of evidence for the dominant contribution to this phenomenon is the Photothermoelectric effect and strain-induced effects play a minor role here.

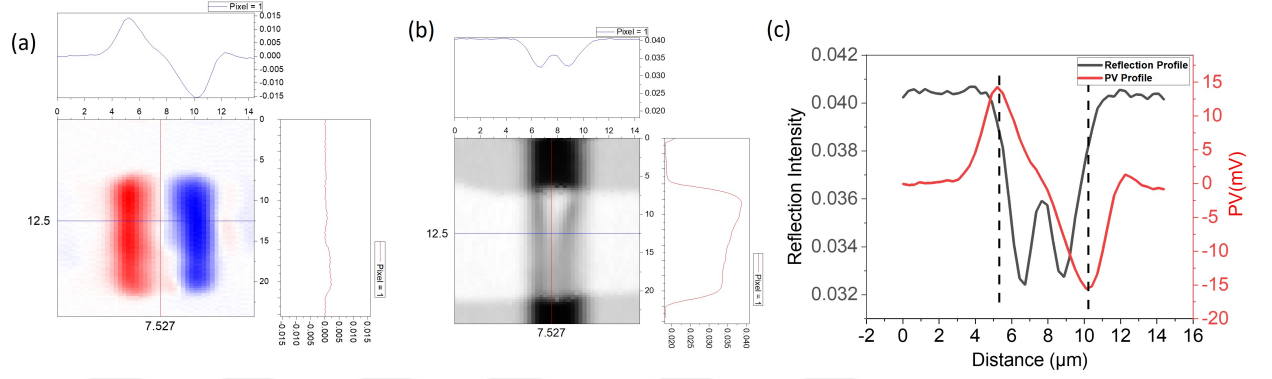


Figure 3.18: (a) Image Profile of SPCM and (b) Reflection Map (c) Reflection Intensity and PV profile.

It is also useful to make a similar analysis in a 150V case where the configuration of the crystal is changed. Figure 3.19 shows this data taken in 150V piezovoltage. One clear observation from the reflection map is buckles increased from 2 to 3 which should have an effect on strain on the crystal. However, PV behavior is still the same in the 0V case which means the response is not affected by strain change.

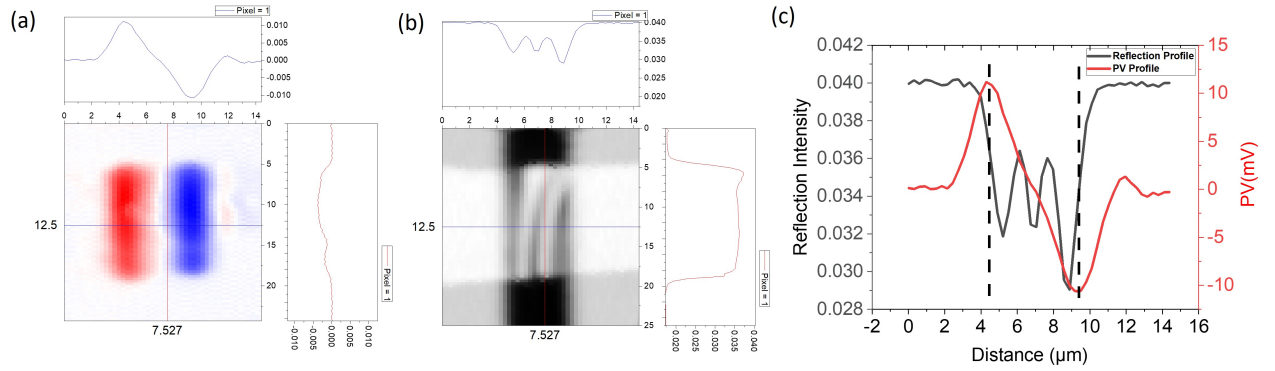


Figure 3.19: (a) Image Profile of SPCM and (b) Reflection Map (c) Reflection Intensity and PV profile.

Finally, the Seebeck coefficient from this SPCM map will be calculated and compared with equation 3.5 which is the theoretical result. We used equation 3.1 to calculate the Seebeck coefficient but first, we need the temperature gradient term (ΔT) in that equation. To find it we exploited the Raman Thermometry so that it is found how much heat difference is observed between suspended and supported parts. Raman thermometry states that for E_{2g}^1 mode there is temperature rise and shift relation such that $0.011 \text{ cm}^{-1}/\text{K}$ [54]. Since phenomena can be used to estimate ΔT by taking Raman from one from supported and one from the part that gives maximum PV signal. Figure 3.20 shows the difference in the Raman spectrum where optical images of the laser position are shown (Figure 3.20 (a) and (b)). Laser power is kept at $30 \text{ } \mu\text{m}$ which is the same with SPCM measurements. Calculation with respect to relation stated above gives ΔT as 6.527K .

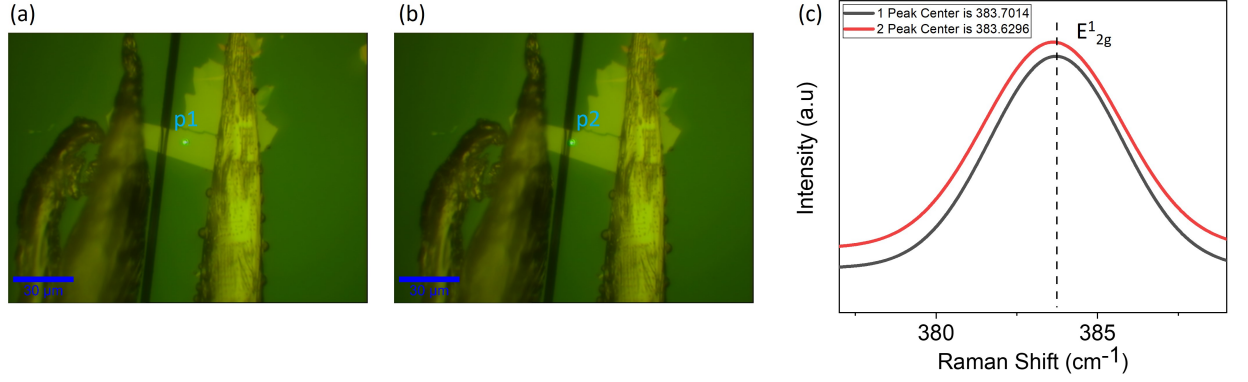


Figure 3.20: Optical images showing in which point Raman is taken (a) supported (b) Suspended. (c) Raman spectrum from both points.

Now, ΔS can be calculated as;

$$\Delta S \approx \frac{9.6mV * \sqrt{2}}{6.5 \frac{K}{\mu W} * 30\mu W} = 69\mu V K^{-1} \quad (3.6)$$

This result is very close to the theoretical calculation which gave $70 \mu V K^{-1}$. Maximum PV is multiplied with $\sqrt{2}$ because lock-in amplifier provides the rms value.

3.6 Gold Bottom Contact MEMS Chip Application

Generally, the Indium contact procedure which is explained elaborately above is utilized for device fabrication. However, another configuration which is obtained by depositing gold on MEMS chip to prepare a bottom contact device is prepared. The optical image and its Current-Voltage behavior are demonstrated in Figure 3.21.

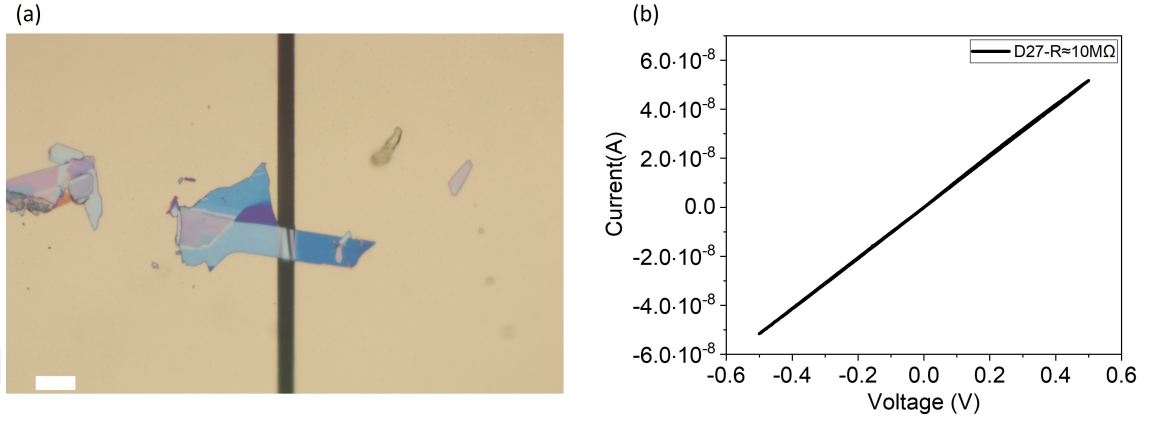


Figure 3.21: (a) Optical Image of Gold MEMS device. The scale bar is 10 m. (b) IV graph of the device. Resistance is approximately $10 \text{ M}\Omega$.

We performed SPCM with $30 \mu\text{W}$ Laser Power upon the gold bottom contact device to observe the response in this new configuration. A similar response is observed as the formation of a thermoelectric junction around the suspended region (Figure 3.22 (a) and (b)). Impressively, opening the gap by applying 150V piezovoltage affected the response by changing the location of the positive response (Figure 3.22 (c) and (d)). This interesting variation opened a requirement for closer observation of crystal.

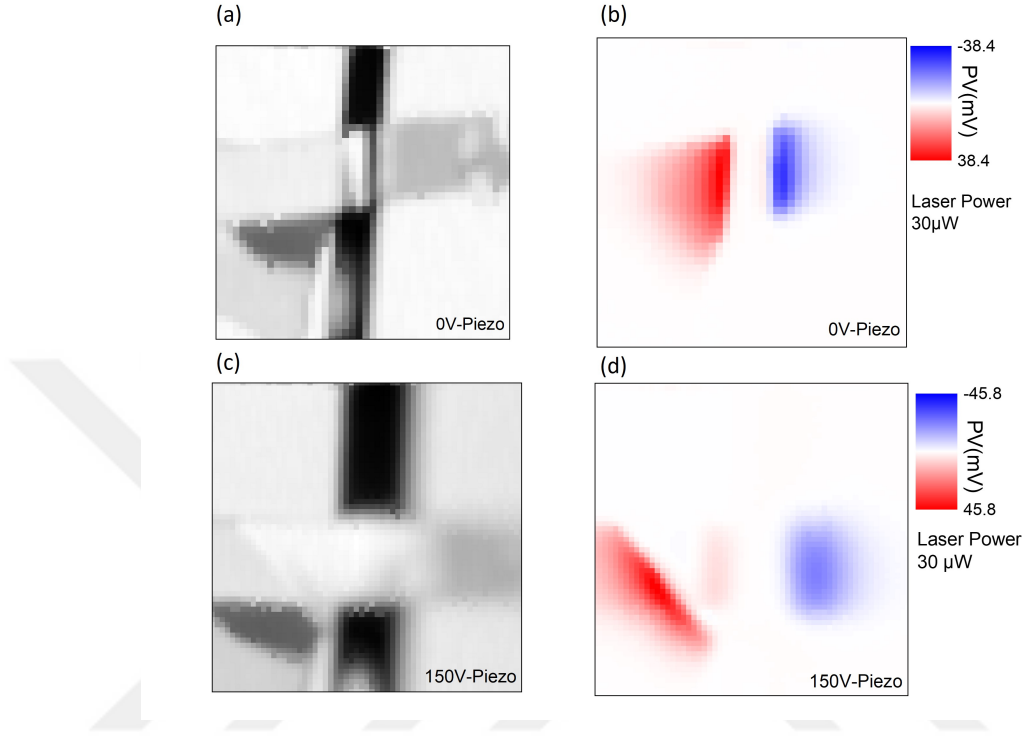


Figure 3.22: Reflection Map (a) and Photovoltage Map (b) of Gold Bottom contact MEMS device at 0V Piezovoltage. Reflection Map (c) and Photovoltage Map (d) of Gold Bottom contact MEMS device at 150V Piezovoltage.

Utilizing the ability to ground gold pattern device easier in Electron Microscopy imaging, we took some SEM images of the device while dynamically controlling the gap. This imaging allowed us to understand SPCM behavior much better. Figure 3.23 shows SEM observation of cases 0V piezovoltage (Figure 3.23a) and 150V piezovoltage (Figure 3.23b). The first observation is that there is a height difference between two sides of the channel which causes an inclined MoS_2 structure. It is intriguingly revealed that opening the gap disconnects MoS_2 and substrate. It levitates MoS_2 flake and we can observe that the suspended part moves which is explaining the movement of SPCM responses behavior. This is also good evidence to support the existence of the substrate variation effect.

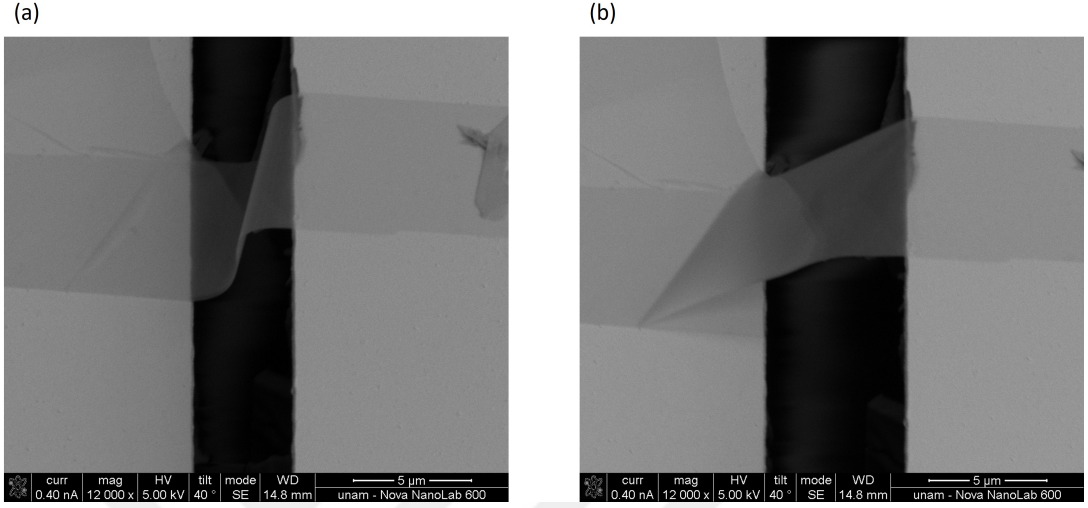


Figure 3.23: SEM image (a) at 0V piezovoltage (b) at 150V piezovoltage.

3.7 Polarization Dependent SPCM

Polarization-dependent measurement upon thermoelectric junction is also performed. The setup of controlling the polarization is demonstrated in Figure 3.24. The setup includes two linear polarizers and a quarter-wave plate (QWP) between them. Since the unpolarized light is not affected by QWP, it first enters the linear polarizer to attain a higher polarization extinction ratio. Then, QWP converts the incoming linearly polarized light to circularly polarized. The mechanism of QWP is to separate light into two electric fields by a quarter of wavelength retardation. Then, it can be allowed to adjust polarization with the final linear polarizer[55].

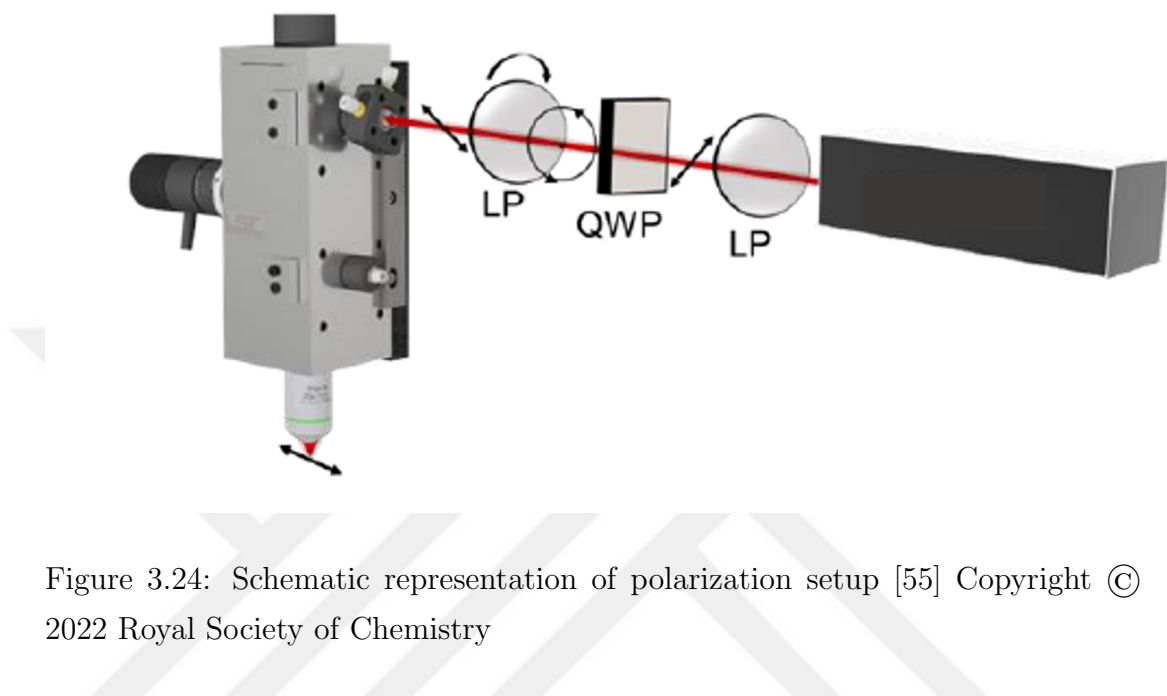


Figure 3.24: Schematic representation of polarization setup [55] Copyright © 2022 Royal Society of Chemistry

The Polarization dependent measurement performed on MoS₂ MEMS device and results are provided in Figure 3.25. The gap is opened enough to make crystal stress free so that stress contribution can be eliminated. Photocurrent maps reveal that the response is independent of polarization (Figure 3.25a ii-iv). Apart from mapping, maximum point measurements are done when the laser position is carefully aligned and tracked. Photocurrent versus polarization angle data shows polarization independence of junction with variation less than 10%.

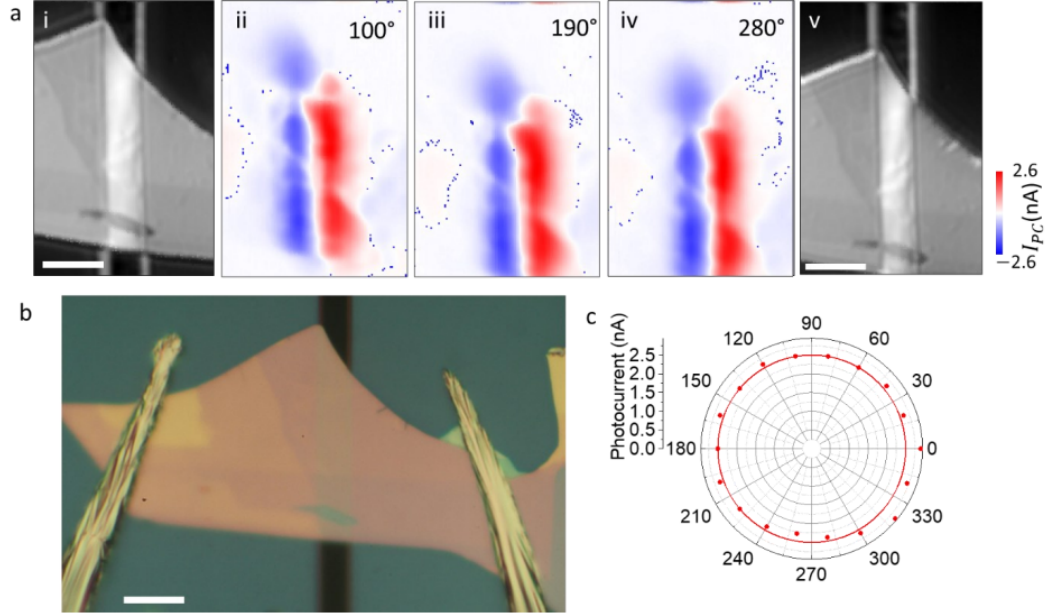


Figure 3.25: (a) shows reflection map (i) and photocurrent maps (ii-iv) for 90° rotation starting from 100° polarization. Since there is a small shift in the scan, we added reflection map of 190° and 280° (v). (b) shows the optical image of device. Scale bars are 10 μm . (b) Optical image of the device. (c) Photocurrent with respect to polarization obtained in various polarization angles. Deviation is less than %10.

Flexo Bulk-photovoltaic effect is majorly dependent on the polarization of incoming light[53]. Our crystal is very clearly independent of the polarization of the laser. This outcome also supports that the flexo photovoltaic effect does not have a role in this behavior.

Chapter 4

V₂O₃ Growth and Transfer Optimization for Future MEMS Chip Application

V₂O₃ is an archetypal Mott insulator which makes distinctive phase transitions depending on temperature and internal pressure[56]. It has 2 distinct phases depending on internal pressure: Paramagnetic Metal (PM) and Paramagnetic Insulator (PI) phases at room temperature. At low temperatures, Antiferromagnetic Insulator (AFI) phase can be observed. Temperature-dependent first order phase transition between PM and AFI phases is validated in many previous works[56][57][58]. The first order transition between PM and PI is performed by Chromium or Titanium doping procedures[59][60][61]. According to these works, an estimated phase diagram is prepared with predictions how doping affects the amount of internal strain. The phase diagram is provided in Figure 4.1. Apart from these, V₂O₃ has a critical point in its phase diagram where more than 2 phases are involved, making it a more intriguing material to investigate.

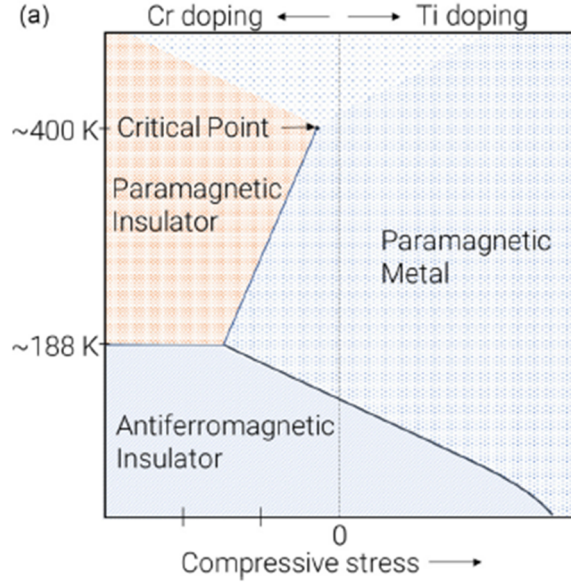


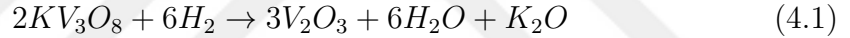
Figure 4.1: V_2O_3 phase diagram with respect to stress and temperature [54]
Copyright Phys. Rev. B (2019)

Although this phase diagram is provided, its accuracy is highly debated since stress parameters are generally controlled by Chromium and Titanium doping which also changes the material's nature. Nowadays, mechanism of metal-insulator transition of V_2O_3 is suggested as trigonal distortion in orbitals[57]. It claims that with a small trigonal distortion, t_{2g} orbital with triplet degeneracy splits to a_{1g} and e_g^π states which are singlet and doublet respectively[57]. This claim opens a new research field to study pristine V_2O_3 (without doped) in stress dependent apparatus. MEMS chips can be a good environment to investigate these phenomena. The following section will cover some work on V_2O_3 growth with a conventional CVD chamber and how to complete a MEMS device.

4.1 Growth and Transfer Optimization

V_2O_3 growth previously performed in real-time observation (RTO) chamber[62] with a specific recipe[56]. In this work, it is referred this recipe can be adjusted

to any conventional CVD chambers with some variations. This section will include this adaptation of how V_2O_3 is grown in a conventional circular tube CVD chamber. The growth mechanism requires Potassium Iodine (KI) and Vanadium pentoxide (V_2O_5) as precursors. Small granules of precursors are placed on a Sapphire substrate and placed in the center of the CVD chamber. The process started by increasing the temperature of the chamber. Around 680, KI granules melt and start reacting with V_2O_5 ending up Potassium Vanadate (KV_3O_8). After this intermediate compound is formed, it is further reduced by hydrogen introduction (40 sccm). When hydrogen is introduced, Argon flow rate is increased from 100 to 200 sccm. Hydrogen remains active until 850 °C and kept there 10 minutes. After 10 minutes, Hydrogen is closed and chamber is released to natural cooling. The reaction formula is provided in equation 4.1[56].



Selenium powders also placed on front of chamber. It has the role of providing Hydrogen Selenide (H_2Se) which is a better reducing agent. Figure 4.2 demonstrates the schematics of V_2O_3 growth in conventional CVD chamber. The outcome reveals clean and high-coverage crystallization.

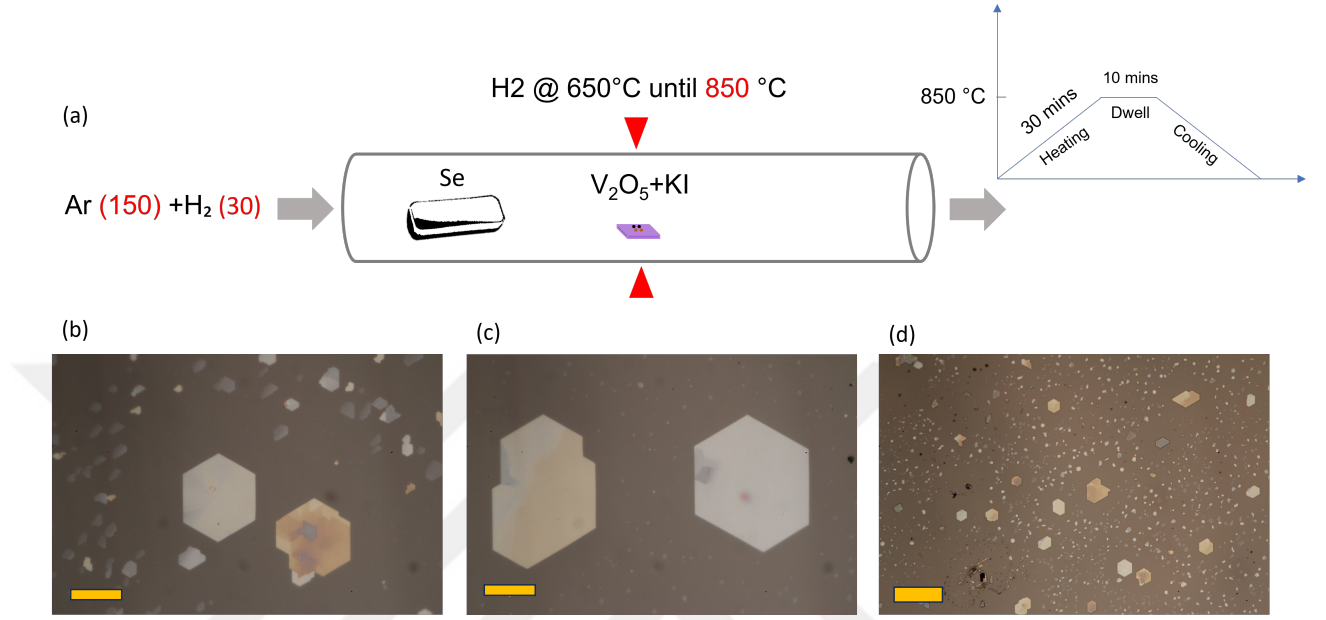


Figure 4.2: (a) Schematic representation of V_2O_3 . (a-b) Examples of growth crystals in different regions. Scale bar is 10 μm (c) high magnification optical image reveals high coverage. Scale bar 50 μm

To prepare a V_2O_3 MEMS device, as-grown crystals have to be cleaved out from the growth substrate which is challenging because of the epitaxial nature of the growth[56]. To solve this problem, apart from a better-reducing agent role, we exploited the passivation of sapphire ideas by first exposing bare substrate to Selenium by increasing temperature up to a bit more than the melting point of Selenium 230 °C[63]. The passivation procedure is summarized in Figure 4.3.

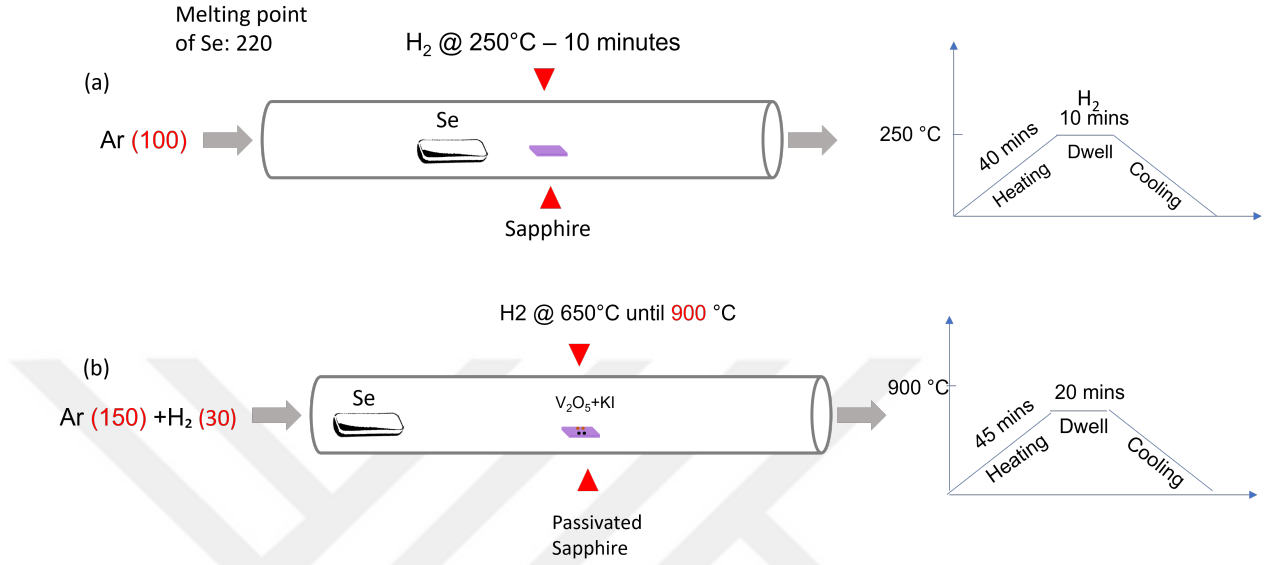


Figure 4.3: (a) First step: Passivation of Sapphire (b) Second Step: V_2O_3 growth on passivated Sapphire

Although verifying the formation of the Se passivation layer requires Transmission Electron Microscopy (TEM), we observed an increasing yield in our transferring attempts after the passivation procedure. The transfer recipe is schematically explained in Figure 4.4. Cellulose Acetate Butyrate (CAB) solution was first spin-coated by 800 rotations per minute (rpm) 50 seconds. Then, the polymer is dried at 160°C in 10 minutes. This procedure was repeated 3 times. Then, the substrate is immersed in water which removes the polymer film. CAB is chosen because of its hydrophobic nature which allows removing film easier. Finally, CAB film is fished with a PDMS and it is dissolved in Ethyl Acetate. Outcome reveals successfully transferred V_2O_3 crystals on PDMS (Figure 4.4b and c).

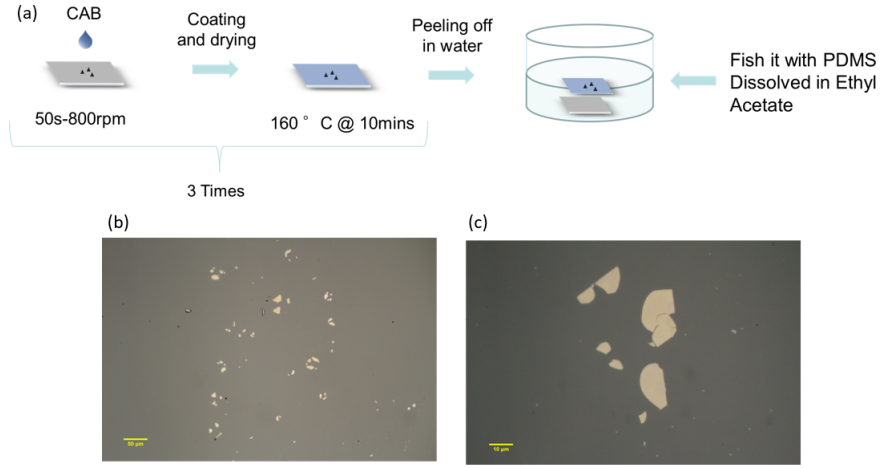


Figure 4.4: (a) Schematic representation of transferring recipe of V_2O_3 . Optical images of transfered V_2O_3 crystals at (b) x20, (c)x100

In figure 4.5, Raman spectrum from both as-grown and transferred crystals reveals a shift in Raman spectrum when it is transferred. The reason of the change in spectrum suggests a transition from PI to PM phase. Because of the Thermal Expansion Coefficient (TEC) difference between V_2O_3 and sapphire, as-grown crystals are growing with tensile stress in the natural cooling step[56]. When the crystal is transfered to another substrate, this stress is released and V_2O_3 go through PM phase. This observation is important for motivating MEMS work since it can be understood that internal stress can be controlled and have a role in phase transitions.

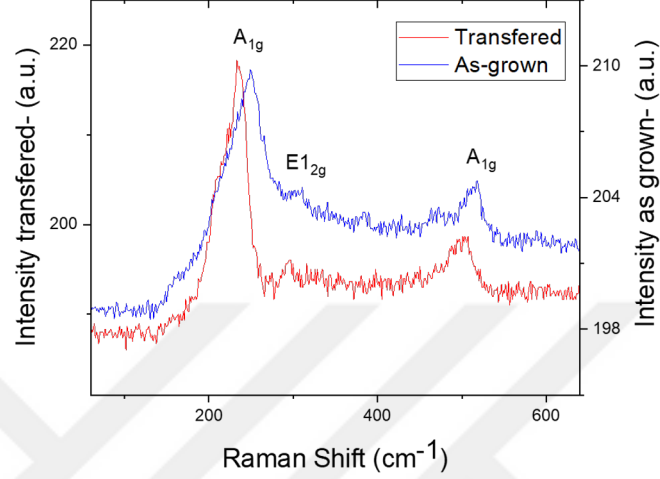


Figure 4.5: Raman spectrum comparison of as-grown and transferred V_2O_3 crystals

Raman spectrum data gives an evidence to ability to control V_2O_3 crystals internal stress. The next aim is to transfer crystals on MEMS chip. After successfully getting them on PDMS, it is necessary to transfer them on channel of MEMS chip by all-dry transfer method which is explained in section 3.1. Then, the transferred crystal has to be fixed with Platinum coating to inhibit the sliding effect with FIB. Figure 4.6 demonstrates a complete Platinum Clamped V_2O_3 device.

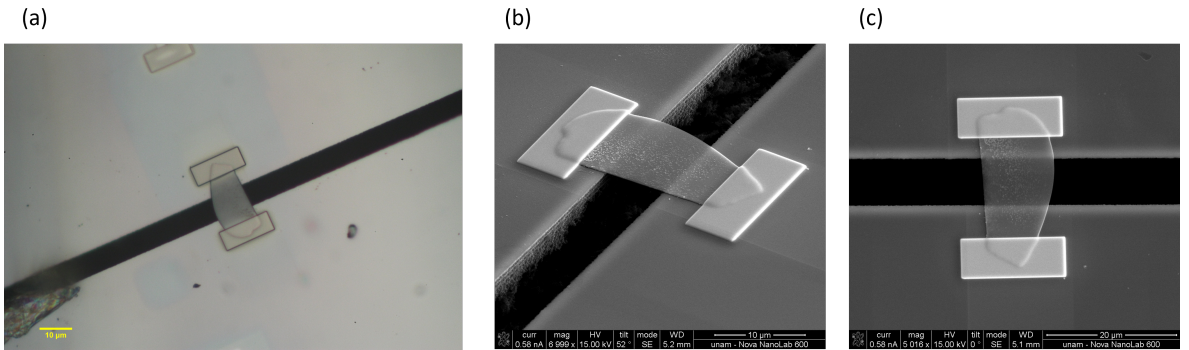


Figure 4.6: (a) Optical Image of Platinum Clamped V_2O_3 . (b), (c) SEM images of device with different directions.

4.2 V_2O_3 Growth on Mica Substrate

Because of the epitaxial nature of the growth and TEC differences between sapphire and V_2O_3 , it is challenging to cleave them up from growth substrate due to the stress on crystal. Even if it can be transferred, it is generally damaged. To overcome this difficulty, we also tried growth on a different substrate which is not previously tested. The Mica substrate is a good candidate in this case because of the absence of dangling bond on it[64]. The growth recipe is the same with sapphire attempts while only the substrate is changed. A schematic representation of growth and growth outcome is demonstrated in Figure 4.7. Although it is not very clean, some bulkish crystallization can be observed as an outcome of growth (Figure 4.7 b and c).

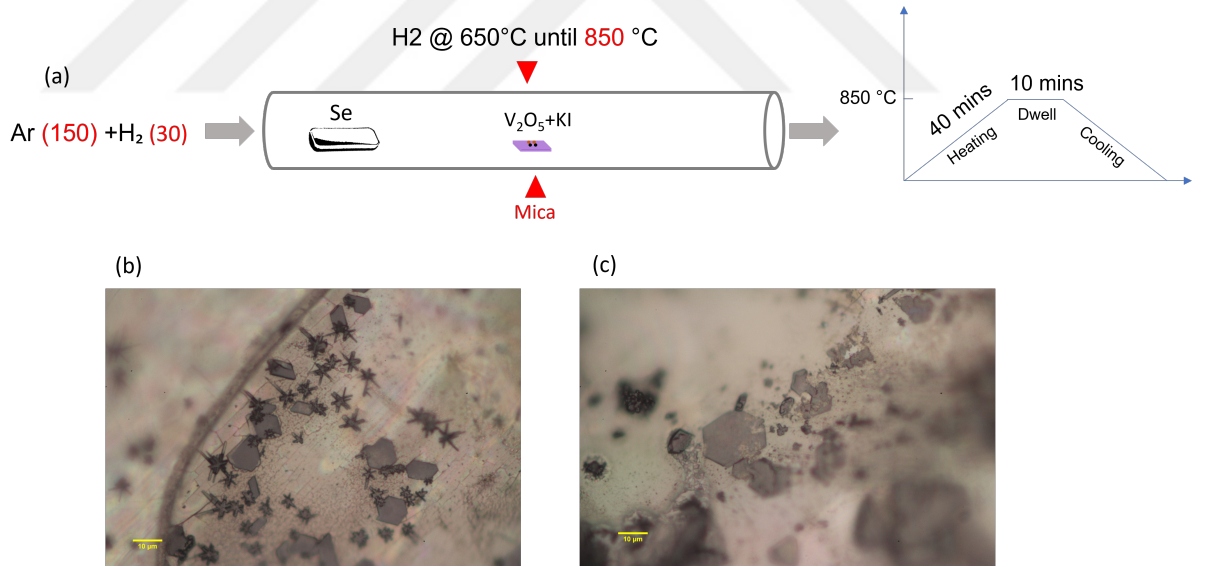


Figure 4.7: (a) Schematic representation of V_2O_3 growth on Mica. (b,c) Optical images of Growth outcome taken from different regions

After growth, we checked these mica-grown crystals in Raman to understand their characteristics. It is shown in Figure 4.8. Interestingly, characteristic peaks of Mica-grown and sapphire grown crystals showed a shift as similar to transferring comparison. This gives an insight that V_2O_3 crystals grown on Mica are in PM phase with smaller internal stress. Because of the absence of dangling bond,

they are growing with less stress than sapphire case.

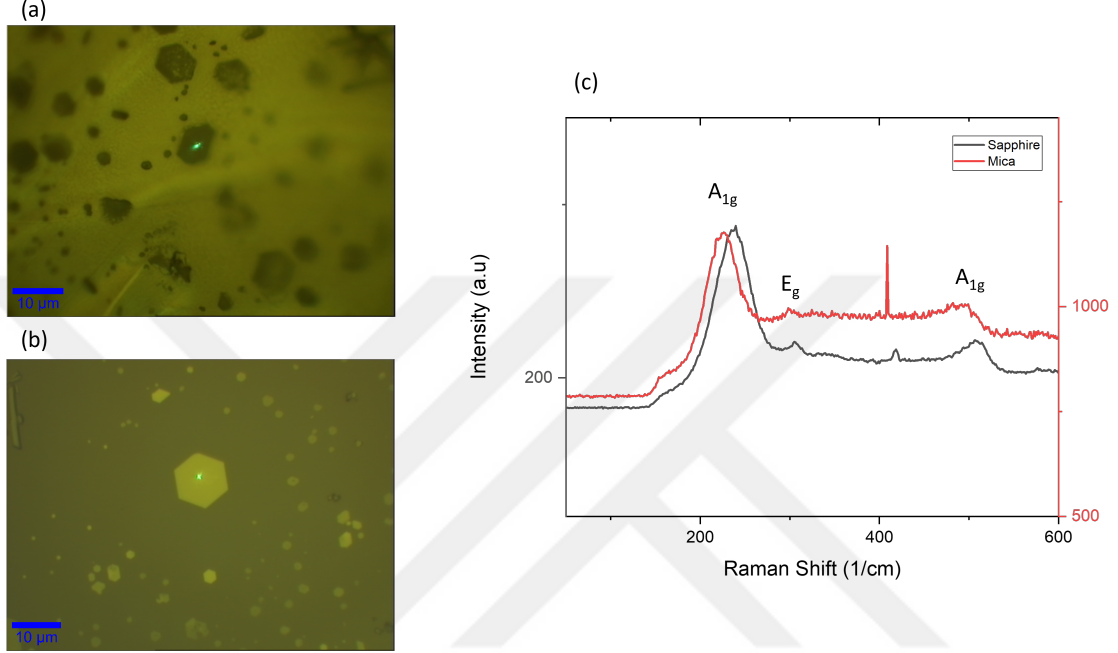


Figure 4.8: Optical image of Raman showing measured crystal on Mica (a) and Sapphire (b). (c) Raman spectrum comparison of these two cases.

To test the transfer performance of Mica growth, we performed transferring procedure similar to sapphire-grown cases. Schematic of transferring and outcomes are demonstrated in Figure 4.9. Although the yield of transferring attempt was much more than sapphire, growth crystals were extremely bulkish. Since these are much thicker than desired crystals, they are not very suitable to be tested in MEMS chip devices. Still, it is found that transferring them is much easier than sapphire and with an improvement in growth, the Mica approach can also be used for various research purposes.

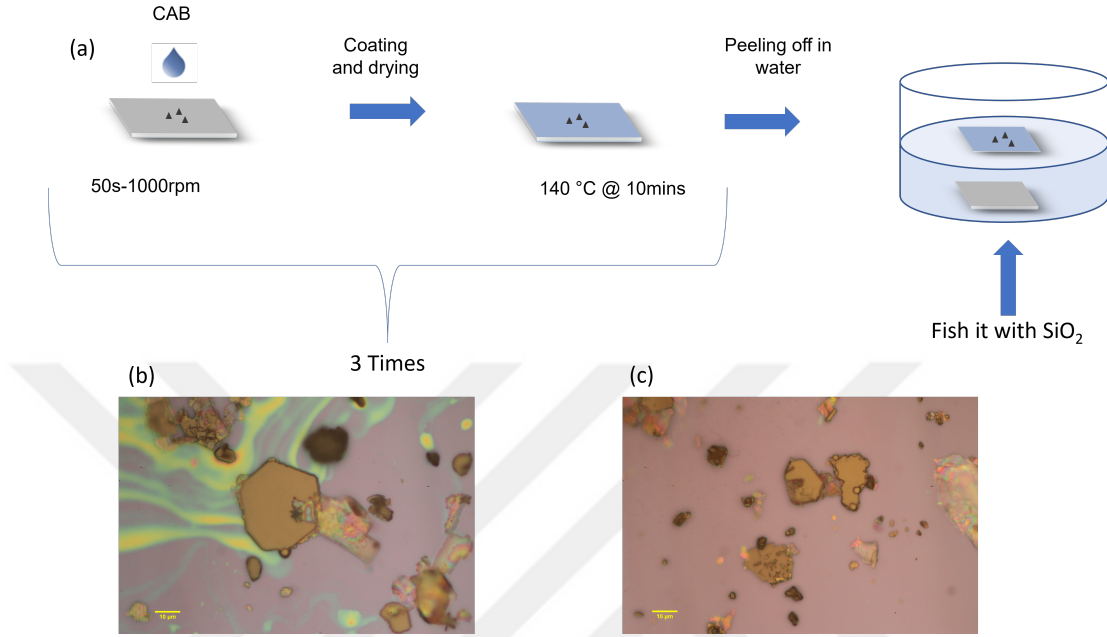


Figure 4.9: (a) Procedure of CAB assisted transfer method. (b)(c) Examples of transferred crystals on SiO₂

4.3 Gating Test of V₂O₃

Finally, we have some preliminary work to control MIT by Field Effect. We prepared a V₂O₃ gating device by placing dielectric material (h-BN) on top of it which is used as a top gate. Then, we place three indium contacts for being source, drain and gate electrodes. Figure 4.10 demonstrates the completed device and its response to gating. The outcome shows we can have a very small modulation even in this preliminary device.

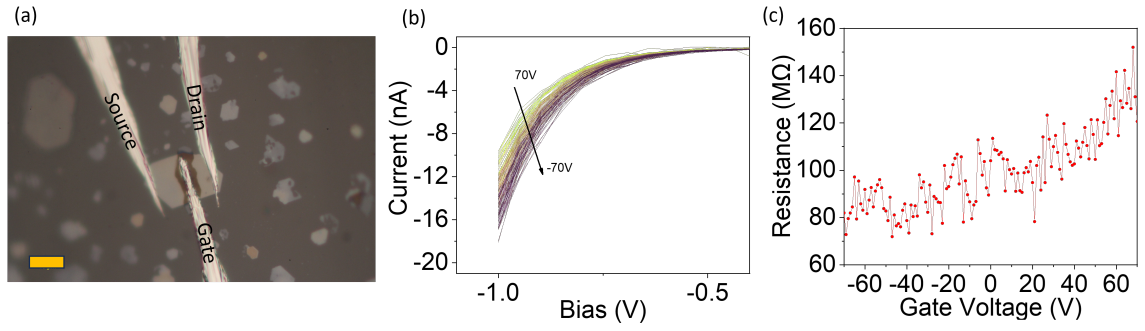


Figure 4.10: (a) Optical Image of 3-terminal device. h-BN is used as a dielectric material on top gate. Scale bar is 10 μm . (b) Current modulation with respect to gate voltage. (c) Small resistance variation by gating.

Chapter 5

Conclusion and Future Work

As conclusion, in this project a novel and unique experimental environment for mechanical testing is introduced as MEMS chips. In chapter 2, it is briefly discussed the drawbacks of frequently used conventional methods. It is explained why new experiment designs are required for this research field. Then, MEMS chip is described schematically and optical images of how it works is demonstrated.

In Chapter 3, MEMS chip application on MoS_2 as one of the most intriguing materials in this context because of its high endurance to stress is provided. Here, characteristic raman peak behaviours are investigated under stress. Following that, its piezoresistance properties are tested with MEMS. Finally, strain influence to thermoelectric junction which is formed by substrate engineering is explored via SPCM measurements. Although further experiments are required, evidences from SPCM maps suggests that strain has a small impact comparing to Photothermoelectric effect.

In chapter 4, device preparation of V_2O_3 which is a highly interesting Mott insulator is included. Its strain dependent phase transition properties makes it an ideal material to study in MEMS chip environment. An example of completed device is demonstrated in this chapter.

Since MEMS approach is a unique and novel method, it needs some further improvements. When placing piezo, the channel opens and closes unintentionally, damaging the material most of the time. Even though MoS_2 can withstand reasonably high stress, we lost some of our devices in this process. Similarly, V_2O_3 devices were damaged as well where they have a much laborious fabrication process. Additionally, some attempts to test other more fragile materials are failed because of this reason. An example of Silver Nanowire preparation is shown in Figure 5.1.

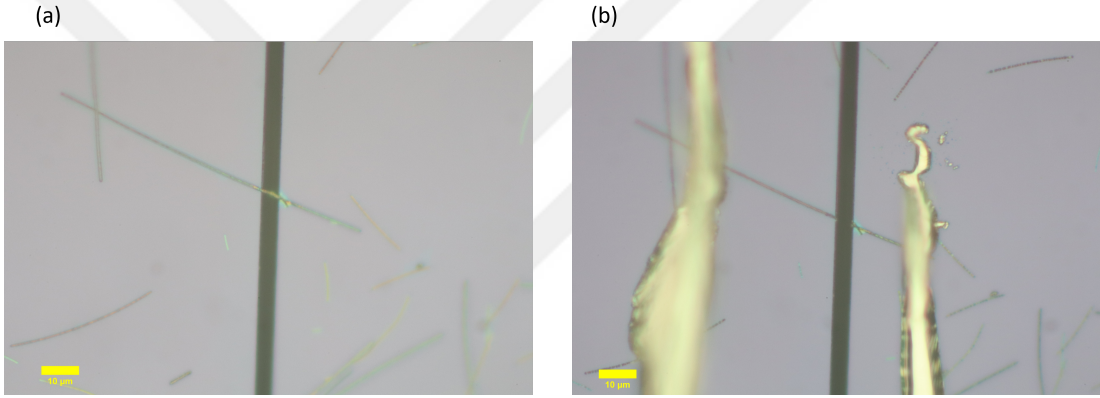


Figure 5.1: (a) Silver NW dropcast on channel (b) NW is broken after placing piezo

The Silver NW MEMS devices aimed to test the quantum confinement effect which can be an inciting subject for future experiments[65]. A properly working device has never been appropriately fabricated. To overcome this challenge, we designed a new chip carrier that will fix the channel before placing the piezo. For future applications, this chip carrier can be used to work with MEMS chips more efficiently. The design of the chip carrier is demonstrated in Figure 5.2.

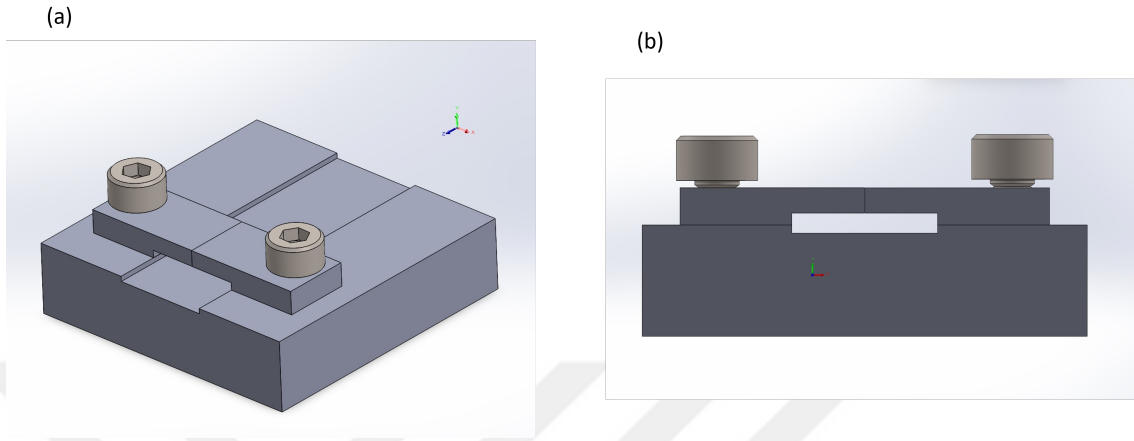


Figure 5.2: Design of the chip carrier shown in different perspectives

Overcoming the difficulties and naturally appearing drawbacks of MEMS chip will open a wide range of applications. Simply, this method can be applied to any materials which have strain-dependent optical, electronic or magnetic properties. Additionally, it can be used for defect engineering to insert vacancies in 2D materials such as Hexagonal Boron Nitride or Black Phosphorus.

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