Chapter 2 - Experimental Techniques

In this chapter, details of microcantilevers (MCs) used in the present work are given. Handling and cleaning of the MCs are discussed initially. Various experimental setups developed to detect the MC deflection and resonance frequency using Atomic force microscopy (AFM) head and Nano vibration analyzer (NVA) are detailed. This includes an environmental chamber used for water adsorption / desorption studies and the setup used for mapping the mode shape of MCs using NVA. Apart from this, the details about the synthesis of polymer solutions and the indigenously designed dip coating setup using three-axis micromanipulators are presented. Preliminary studies undertaken for identification of higher modes of MCs using AFM, NVA and FEM simulation are given. This chapter ends with the details of micro-Raman spectroscopy used for measuring residual stress in MCs.

2.1 Microcantilevers used in the present work

Four different MCs used in the present thesis are from commercial sources [1] and is made of single crystal n-type silicon with antimony doping. The MCs are labeled as MC1, MC2, MC3 and MC4 whose dimensions, natural frequencies and spring constant \((k)\) values are shown in Table 2.1. MC4 is an array of cantilevers consisting of two MCs with each having the dimensions as shown. Resonance frequency of these MCs was measured using AFM head and NVA (discussed later in this chapter). The spring constant \((k)\) is estimated using the resonance frequency and quality factor values, using Sader’s method [2]. The equation used for estimation of spring
constant \((k)\) is given by \( k = 0.1906 \rho_f W^2 L Q_f \Gamma_i (\omega_f) \omega_f^2 \) where \( \rho_f \) is the density of the fluid (medium in which the MC exist), \( Q_f \) is the quality factor of fundamental mode of MC, \( W \) and \( L \) are the width and length of the MC, \( \Gamma \) is hydrodynamic function and \( \omega_f \) is the fundamental resonant frequency in fluid. A typical scanning electron microscope (SEM) image and surface morphology of the MC used in this study is shown in Fig 2.1. The average roughness was estimated to be 0.85 nm on front side of MC.

Table 2.1 Details of four different microcantilevers used in the present study. The natural frequency was measured using AFM head and spring constant values are estimated using Sader’s method [2].

<table>
<thead>
<tr>
<th>Name</th>
<th>Dimensions</th>
<th>Resonance frequency ((f)) (kHz)</th>
<th>Spring constant ((k)) (N/m)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Length (L)</td>
<td>Width (W)</td>
<td>Thickness (T)</td>
</tr>
<tr>
<td>MC1</td>
<td>450 (µm)</td>
<td>40 (µm)</td>
<td>2.5 (µm)</td>
</tr>
<tr>
<td>MC2</td>
<td>225 (µm)</td>
<td>30 (µm)</td>
<td>3 (µm)</td>
</tr>
<tr>
<td>MC3</td>
<td>125 (µm)</td>
<td>35 (µm)</td>
<td>4.5 (µm)</td>
</tr>
<tr>
<td>MC4</td>
<td>500 (µm)</td>
<td>98 (µm)</td>
<td>1 (µm)</td>
</tr>
</tbody>
</table>

Figure 2.1 SEM and AFM images of MC1 used in this study. Average roughness of MC1 is about 0.85 nm
2.2 Handling and Piranha cleaning of the microcantilevers

Commercially, MCs are shipped in gel pack boxes as shown in figure 2.2 (a) in order to avoid moisture environment. Generally it is very difficult to handle these MCs. Usually MCs are attached to a small substrate wafer. Using a tweezer, one can hold the substrate chip and can move around it, as shown in figure 2.2 (b).

It is reported that a common shipping and packaging material like poly(dimethylsiloxane) (PDMS), causes a thin layer of silicone oil contamination on AFM cantilever tips [3]. Thin layers of contaminants may change the reactivity or adsorptivity of surfaces so that the desired modifications may not really be carried out on surfaces [3]. It is known that Piranha cleaning removes the organic contamination on MCs and a native oxide of about 5nm is grown [4]. Piranha solution, also known as Piranha etch, is a 3 :1 mixture of sulfuric acid (H$_2$SO$_4$) and hydrogen peroxide (H$_2$O$_2$) used to clean organic residues off substrates. Because the mixture is a strong oxidizing agent, it will remove most organic matter, and it will also hydroxylate most surfaces (add OH groups), making them highly hydrophilic (water compatible).

Figure 2.2 (a) Commercially available gel pack box in which usually MCs are shipped (b) handling of the MCs using a tweezer.
In the present work, MCs were cleaned using a *Piranha* solution for 30 minutes and then rinsed immediately in triply distilled water for five minutes and finally dried in nitrogen. MCs were loaded into the experimental chamber immediately after *Piranha* cleaning. Contact angle measurements were performed using a standard contact-angle goniometer (M/s HOLMARc, HO-IAD-CAM-01, India) on MC chip before and after *Piranha* cleaning and are shown in figure 2.3. The contact angle was found to reduce from 95° to ~ 29° after *Piranha* cleaning. It is clear from this observation that *Piranha* cleaning makes the silicon surface hydrophilic.

![Figure 2.3](image)

Figure 2.3 Contact angle measurements on a) before and b) after *Piranha* cleaning of MCs. The contact angle was found to reduce from 95° to ~ 29° after *Piranha* cleaning.

### 2.3 AFM head

Static and dynamic measurements on MCs are performed using the laser photodiode arrangement of an AFM head (NT-MDT, Russia). Figure 2.4 shows the photograph of AFM head used and the schematic of measurement panel. This head consists of a laser ($\lambda = 650$ nm) beam which is focused on the MC and reflected back on a four quadrant photo diode (ABCD). MC was placed on a piezo actuator which is swept using a lock-in-amplifier. The laser beam of the AFM head is
reflected from the oscillating probe in the vertical direction. These oscillations of the probe result in oscillations of the laser beam spot with respect to the top and bottom halves of the photodiode. This induces a variable electrical signal, at the probe oscillation frequency, at the output of the registration system. The amplitude of this signal is proportional to the oscillation amplitude of the probe. Therefore the registration system measures the probe oscillation amplitude and converts it into an electrical signal. The variable component of the electrical signal can be applied to the input of Lock-In amplifier, RMS detector and Phase detector.

Figure 2.4  Photograph of AFM head and schematic of control electronics panel.
For vertical bending vibrations, the difference of AB and CD photo diode signal (DFL) was monitored and for torsional and lateral vibrations AD and BC difference signal (LF) was recorded as shown in figure 2.5. The device electronics is controlled by the commercial software “NOVA” program. Typical vertical and lateral bending vibrations of MC1 obtained using AFM head shown in figure 2.6 (a) and (b). First vertical bending (VB1) and Lateral bending mode (LB1) is obtained by monitoring DFL and LF signals. The direction of vibration of each mode is shown as inset. It may be noted that the vibration amplitude of VB1 is higher than the LB1 mode for a same piezo excitation.

Figure 2.5 The four quadrant photo diode system in AFM. Normal constituent corresponds to difference of AB and CD photo diode signal and it gives information about vertical vibrations. Lateral constituent corresponds to difference of AD and BC signals and it gives information about lateral and torsional vibrations.
Figure 2.6 First vertical bending (VB1) and lateral bending (LB1) modes of MC1 obtained using AFM shown in (a) and (b) respectively. Amplitude of vibrations is in nA. The direction of vibration for VB1 and LB1 is shown as inset.

For static measurements, the DFL signal was monitored with time. In this case, MC was not excited by piezo. Figure 2.7 shows a typical deflection response of MC with time when relative humidity is reduced from 50% to 6%.
Figure 2.7 Deflection response of MC1 with time when relative humidity is reduced from 50% to 6%.

Figure 2.8 Calibration curve obtained by performing force distance curve on hard Si surface in motion. From linear contact part, slope was found to be 20 pA/nm shown in inset. (b) Deflection direction of MC towards back during positive deflection signal of MC.
Deflection response (nA) can be converted into deflection (nm) by dividing with the calibration slope. To obtain calibration slope, force curve on a hard Si surface was measured by pushing the end of the MC to a defined distance upward by a piezoelectric crystal [5] and is shown in figure 2.8. Force curve is a measure of the photodiode current (I_{PSD}) versus height position of hard surface placed on the piezoelectric translator (Z_p). From the linearly increasing contact part (see inset of figure 2.8), deflection sensitivity \( \Delta I_{PSD}/\Delta Z_p \) was obtained and was found to be 20 pA/nm. Now, MC deflection (\( \Delta z \)) was obtained using the formula \( \Delta z = I_{PSD} / (\Delta I_{PSD}/\Delta Z_p) \). A positive deflection signal implies MC is bending in the direction of laser focusing i.e. towards back (see figure 2.8(b)) and vice versa.

### 2.4 Experimental set up for water adsorption studies on microcantilevers

A schematic diagram of the apparatus and photograph of experimental set up used for water adsorption studies is shown in figure 2.9 (a) and (b). For adsorption of water molecules, experiments are performed by placing the AFM head along with the MC, inside an airtight chamber purged with dry nitrogen (N_2) gas which reduces the Relative Humidity (RH) to 6% over a period of 2 Hrs. The flow rate of the gas inside the chamber was in the range of 100-500 ml min\(^{-1}\). For increasing the RH, N_2 is bubbled through DI water. RH in the chamber is measured using a standard RH meter with an accuracy of \( \pm 3.5\% \) RH. Shift in different resonant mode frequencies (VB, LB and TB) and deflection response during RH increase and decrease was also studied using this setup.
Figure 2.9 (a) Schematic diagram and (b) Photograph of the experimental setup used for water adsorption studies. Dry/wet N₂ gas is purged into chamber to reduce/increase relative humidity in the chamber.

2.5 Nano vibration analyzer

Apart from AFM head, Nano Vibration Analyzer (NVA) was also used to measure the natural frequency of MCs. The NVA (M/s SIOS, Germany) is a fiber-coupled laser interferometric vibrometer integrated in a precision technical microscope. It is
excellently suitable for measurements of dynamic properties and static displacements of microstructures, MEMS and microcantilevers.

The working principle of the interferometer measuring system is based on the application of a classic Michelson interferometer with plane-mirror reflectors utilizing the interference effect. The scale is based on the extremely exact wavelength of a He-Ne laser. All optical parts of the interferometer are located within the sensor head and balanced. The sensors are designed so that they can work on optically rough surfaces. This means that they do not require an additional reflector. The measuring beam emitted by the He-Ne laser passes through optics which focuses the beam on the object to be measured. The sensor head contains a miniature interferometer, which transforms the motion of the measurement reflector into an optical signal. This quantized, optical measuring signal is transformed in the sensor head into electrical signals, which are then transmitted to the modular supply and evaluation unit. There the signals are completely preprocessed up to the signal normalization with respect to amplitude and offset [6].

The block diagram and the photograph of the experimental setup used to measure the natural frequency of MCs using NVA are shown in figure 2.10 (a) and (b). MC was glued on a piezo actuator, which was excited using a function generator. The measuring laser beam ($\lambda = 632.8 \text{nm}$) was focused on the MC with a microscope objective of focal length of 35 mm and the back reflected signal interferes with the reference signal inside the interferometer. This optical signal is transformed into electrical signal using a photo diode, which is then transferred into the electronic unit where it is amplified and is fed into a PC interfaced. Position of the laser beam on MC was also monitored using a USB based digital camera with CMOS sensor
which is mounted on microscope and was connected to a separate PC. By varying the function generator frequency in steps of 100 Hz amplitude of vibration was recorded at each frequency and resonance frequency was estimated from the peak of the plot of frequency v/s amplitude and is shown in figure 2.10 (c). To perform this task a software routine in commercial software *InfasVIBRO* was written (see Appendix A 1). The advantage of this technique is one can obtain the mode shapes of the vibrating cantilever experimentally by scanning the MC area.

![Diagram](image)

**Figure 2.10** (a) Block diagram and (b) photograph of experimental setup used for estimating the natural frequency of the cantilever using NVA. (c) Resonance spectrum of MC1 obtained using NVA.
2.6 FEM analysis

Finite element modeling (FEM) analysis is performed using a commercial MEMS CAD tool (Intellisuite). FEM is a practical approach to simulate MEMS behavior under different types of loads such as mechanical and thermal. To estimate the natural frequency of MC, the cantilever of desired dimensions (MC1, MC2 and MC3) is designed by specifying the dimensions in the 3d builder module of the Intellisuite software. Then the same is imported to the thermo electromechanical (TEM) module and the simulations are performed by setting static and dynamic analysis. Mesh size is selected and then the material properties are selected from material database of the software. One end of the MC is fixed by boundary condition and the simulation was performed. In the simulation, MC with the asymmetric corner notches at the free end of the MC was taken into account.

![Figure 2.11 Mode shape of first vertical bending mode of MC1. Mesh used was 3nm. Normalized amplitude of vibration of mode is shown.](image)

The material and geometric properties used for the model are: Young’s modulus (E) = 106 GPa, Density (ρ) = 2328 kg/m³. These dimensions were adopted from the technical data sheet provided by the supplier [1]. After estimating the natural
frequency of the desired MC, mode shapes were obtained using Visual ease module. Figure 2.11 shows the mode shape of first vertical bending mode of MC1 obtained using FEM.

2.7 Functionalization of microcantilevers with polymers

2.7.1 Synthesis of PAAm and PVP polymer solutions

For addition of mass on MC, Polyacrylamide (PAAm) was prepared by chemical route [7]. Aqueous solution of acrylamide (AAm) is prepared by dissolving 0.6M of AAm and 2.5mM of potassium persulfate (KPS) in 15mL of water. The AAm solution is de-aerated by argon gas purging for 5 minutes. 36 μL of N,N,N',N'-Tetramethylethylenediamine (TEMED) is added to the AAm solution and vortexed it for 2 minutes. The AAm solution is allowed to stand for 10 minutes during the course of which AAm polymerized to polyacrylamide (PAAm).

For gamma dosimeter studies, Polyvinyl pyrrolidone (PVP) was coated on MCs. It was prepared by dissolving 30mg of PVP in 1 ml of Ethanol solution.

2.7.2 Dip coating set up

Figure 2.12 shows the experimental setup used to add the polymer mass on MC by dip coating. Both MC and polymer solution (in a container) were mounted on two different 3-axis manipulators to allow the precise movement of the both. Now, by carefully observing through a digital microscope (200x magnification), MC was slowly dipped inside the polymer solution (see inset of figure 2.10), kept for 20 minutes, removed slowly and was allowed to dry over night. The mass added on
MC was determined by the shift in the fundamental resonance frequency and was used to estimate the mass sensitivity.

![Figure 2.1](image)

**Figure 2.1** MCs are coated with polymer solution using this dip coating setup. MC and polymer solution in a container are mounted on two 3-axis micro manipulators, and by carefully observing through a digital microscope, MC was dipped into the solution (see inset), kept for 20 minutes and removed.

### 2.8 Preliminary studies on identifying the higher modes of vibration of microcantilevers

In the present thesis, the mass sensitivities of higher modes of MCs are compared. AFM head is used to obtain frequency spectra of MCs. The frequency sweep was done initially in the range of 5 kHz to 2 MHz with a step of ∼ 200 Hz, and after identifying the modes it was repeated with a high resolution step of 2 Hz at every mode. While doing this, piezo drive voltage was increased accordingly to excite the mode efficiently. Figure 2.13 shows the VB signal between 5 kHz to 1500 kHz of MC1 obtained using AFM head. Each peak in this figure represents a mode and are
numbered from 1 to 7 accordingly. By comparing FEM results, modes 1 to 7 are identified as VB modes VB1 to VB7. These are indexed in figure and some of mode shapes with the normalized amplitude are shown in inset. It may be interesting to note that the mode assignment is only based on frequency and no significance is ascribed to the amplitude of peaks in figure 2.13.

Figure 2.13 Experimental results of vertical bending (VB) signals of the microcantilever studied in the present work using AFM head. Seven unique modes of vibration are identified and assigned from FEM simulations (shown as stick). Some of the mode shapes are shown as inset.

It is seen that the peak between 300 kHz to 500 kHz has highest amplitude for a given piezo excitation in the MC as compared to other modes. This arises due to the complex resonance associated with the piezo actuator, spring clamp used to hold the MC and MC chip used in the present experimental setup. This complex resonance is not seen when the frequency spectra was obtained using NVA. Figure 2.14 shows the frequency spectra of MC1 obtained using Nano Vibration Analyzer (NVA). Initially the frequency sweep was done for individual mode of vibration with a step
of 100 Hz and finally all the modes of vibrations are plotted as the frequency spectra shown. It is clear from the figure that fundamental mode of vibration has large amplitude as compared to other modes of vibration for a given piezo excitation.

![Frequency spectra of MC1 obtained using NVA.](image)

Figure 2.14 Frequency spectra of MC1 obtained using NVA. The peaks in the spectrum corresponding to vertical bending modes. VB1 has higher amplitude of vibration as compared to other higher modes.

Apart from VB signals, lateral (LB) and torsional bending (TB) signals are also recorded for MC1 using AFM head. Figure 2.15 shows the LF signals of MC1 between 5 kHz to 1500 kHz. LB signal apart from the peaks at the same position as VB signal, four unique peaks are identified and are numbered from mode 8 to mode 11. From FEM analysis, the peaks are identified as lateral and torsional bending vibrations. The appearance of vertical bending signals in torsional bending signals and vice versa (not seen here) is due to reasons like improper coupling of the piezo actuator vibrations to MC, geometric imperfection of MC and MC chip, etc., This also explains the very fact that why for a vertically excited cantilever, torsional bending modes of the MC arise [8].
Figure 2.15  LF signal of MC1 obtained using AFM. This signal shows both Lateral (LB) and torsional bending (TB) of MC1. The peaks 8 and 9 corresponds to LB1 and TB1 modes of MC1.

2.9  Mapping of mode shape of microcantilever vibrations

In this work, mode shapes of microcantilever are mapped experimentally using NVA. To map the mode shape of any vibration one needs both amplitude and phase information of the vibrating structure. The phase of the excitation signal from function generator was given as a trigger to NVA. With this, NVA records at each position the amplitude of vibration and phase difference between excitation and MC vibration signal. To perform this task a software routine in commercial software InfasVIBRO was written and is given in Appendix – A 2. Script controlled software enables the laser beam to scan the object surface along the length and width direction (XY stage) and provides the amplitude of vibration and phase information with a resolution of 2µm on the surface of MC. Initially vibration and phase was recorded along the length of MC for three different modes and are shown in figure
2.16. The solid line in this figure is the polynomial fit. It is clear from the figure that as we move from fixed end to free end along the length of MC, the amplitude of vibration changes and becomes almost zero at certain points including fixed end also at certain points it becomes almost maximum which depends on excitation frequency and mode number.

Figure 2.16 Mode shape of vibrations along the length of MC for its first three VB modes. The data is fitted to polynomial fit.

After the line shape analysis, more detailed scan in both XY direction was performed for various modes. The recorded data was imported into 3d imaging software for post processing. The first four vibrational mode shapes obtained by NVA are compared with the mode shapes of vibration obtained by FEM analysis of same dimensional MC shown in figure 2.17. It is clear from these figure that mode shape obtained experimentally matches well with simulation results. It is interesting to note that maximum amplitude recorded for fourth mode is only 0.4 nm.
Figure 2.17 Mode shapes of VB1, VB2, VB3 and VB4 of MC1 obtained using FEM analysis (simulation) and NVA (experimental). It may be noted that FEM results show normalized amplitudes, where as NVA results are real amplitude of vibrations.
The frequency values of MC1 for all modes of vibrations obtained using AFM head, NVA, Analytically and FEM analysis are compared and are given in table 2.2 and the difference between experiment and simulation is < 2%.

Table 2.2 Frequency values of MC1 for all modes of vibrations obtained using AFM, NVA (experimental), Analytically and FEM analysis (simulation).

<table>
<thead>
<tr>
<th>Modes</th>
<th>AFM (kHz)</th>
<th>NVA (kHz)</th>
<th>Analytical value (kHz)</th>
<th>FEM (kHz)</th>
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<tbody>
<tr>
<td>VB1</td>
<td>12.5945</td>
<td>12.030</td>
<td>13.46</td>
<td>12.5943</td>
</tr>
<tr>
<td>VB2</td>
<td>78.471</td>
<td>74.951</td>
<td>84.378</td>
<td>78.8322</td>
</tr>
<tr>
<td>VB3</td>
<td>219.36</td>
<td>209.901</td>
<td>219.36</td>
<td>220.479</td>
</tr>
<tr>
<td>VB4</td>
<td>429.272</td>
<td>409.302</td>
<td>462.98</td>
<td>431.514</td>
</tr>
<tr>
<td>VB5</td>
<td>708.084</td>
<td>677.490</td>
<td>764.55</td>
<td>712.387</td>
</tr>
<tr>
<td>VB6</td>
<td>1056.398</td>
<td>1008.591</td>
<td>1142.11</td>
<td>1062.7</td>
</tr>
<tr>
<td>VB7</td>
<td>1471.146</td>
<td>1409.507</td>
<td>1595.18</td>
<td>1482.06</td>
</tr>
<tr>
<td>LB1</td>
<td>199.446</td>
<td>--------</td>
<td>--------</td>
<td>214.925</td>
</tr>
<tr>
<td>TB1</td>
<td>313.458</td>
<td>--------</td>
<td>--------</td>
<td>314.046</td>
</tr>
</tbody>
</table>

2.10 Micro Raman studies on microcantilevers

MCs used in the present work are commercially available and apriori knowledge of residual stress present in these devices is crucial for the studies undertaken in the present thesis. Several techniques are reported in the literature for measuring stresses in microscale structures. One commonly used method is X-ray diffraction [9], but the lateral dimensions of the film are required to be greater than the spot size of the beam. Other techniques, such as bulge tests, bending tests and resonant tests,
are utilized in micro devices, but are limited by their lateral spatial resolution which exceeds tens of microns [10].

Micro-Raman spectroscopy is an interesting alternative technique to study stress in micro systems. The combination of Raman spectroscopy with optical microscope systems enables the incident beam to be focused to a spot with a diameter of 1 μm. It is capable of analyzing the local stress on a micrometer scale, and is extensively used for local stress evaluation in silicon microstructures [10-14]. This method is non-destructive, fast and accurate enough.

The Raman signal originates from interaction of light (photons) with lattice vibrations (phonons in a crystal). When monochromatic radiation is incident on the surface of a solid can undergo transmission, absorption, and scattering. In general, two properties can be defined by the electronic distribution within a crystal lattice in equilibrium, namely, the dipole moment, and electric polarizability. These properties will change with deviation in equilibrium interatomic spacing of the lattice or time-dependent vibrational motions around these equilibrium positions. In Stokes–Raman scattering, the incident photons interact with vibrating crystal (phonon), which induces changes in the polarizability of the solid, causing the scattered photons to lose a quantum of lattice energy or phonon relative to the incident photons. Frequency (ω) of the excited phonons is given by [15]

$$\omega = \left[ \frac{1}{\lambda_i} - \frac{1}{\lambda_s} \right]$$  \hspace{1cm} (2.1)

Where λ<sub>i</sub> and λ<sub>s</sub> are the wavelength of the incident and scattered radiation, respectively. The quantity ω is called the Raman shift in units of cm<sup>-1</sup>. The Raman
spectrum contains information about the physical and chemical characteristics of the solid [15].

Mechanical stress and strain may affect the frequencies of the Raman modes. Stress or strain in Raman measurements can be obtained via so-called phonon deformation potentials (DPs), which are linear coefficients linking the change in the phonon frequency and the strain or stress in the material. For silicon, using the phonon deformation potentials of Chandrasekhar et al., [16] the stress (σ) is estimated as,

\[ \sigma \approx -518 \Delta \omega \]  

(2.2)

In the equation, \( \Delta \omega \) is difference between Raman shift measured with and without stress and expressed in cm\(^{-1}\) and \( \sigma \) is expressed in MPa.

Mechanical stress measurements of MC1 have been carried out using a Renishaw micro-Raman spectrometer. All spectra were excited with visible (514.5nm) laser light with power 2 mW and collected in the backscattering configuration. The spectra were recorded with a 3000 lines/mm grating. 50x objective was used to focus the excitation laser light on the right spot of the MC1. The exposed time was taken 1 s. First the experiment was performed on the substrate of the MC1 at room temperature (24°C). Experiments were performed at three different positions on MC1 namely free, fixed end and middle of the MC1 with same experimental condition. The experimental results of Raman peak shift and broadening due to laser heating at various positions on MC1 are studied. Effect of laser power on Raman shift at various positions on a free standing MC is studied. Raman shift along the length of the MC in free standing position, front and back resting on a substrate is also presented. Careful experiments were performed to measure this residual stress in three different configurations along the length of the microcantilever. Measured
Raman shift was converted into Stress using equation (2.2) with respect to Raman shift measured on the substrate.

Figure 2.18 shows the Raman shift (RS) at room temperature on the substrate of MC with Lorentzian fit. The observed RS and width are 520.95 cm\(^{-1}\) and 8.1685 cm\(^{-1}\) respectively which is close to ideal value.

2.10.1 Effect of Laser power

Figure 2.19 (a) shows the variation of Raman shift on the substrate and at three different positions of micro cantilever namely fixed, middle and free end of the cantilever with increasing power. Figure 2.19 (b) shows these three different positions on a free standing MC during the experiment. From this figure following observations can be made. In general with increasing laser power there is a negative shift in Raman peak at all the positions indicating the rise in temperature due to localized heating. It is known when the crystal lattice is heated, the equilibrium positions of the atoms are displaced, resulting in an overall volumetric expansion of the lattice and a change in interatomic forces. These changes in the interatomic
forces modify the phonon vibrational frequencies which results in red shift in the resulting Raman phonon spectra [14, 17]. Difference in Raman shift is maximum of 6.87 cm\(^{-1}\) at free end of the cantilever for 40 mW laser power. This is because of small thermal mass of the microcantilever and also because the heat generated at free end can not readily dissipate into to the surround air. This is due to the large difference between thermal conductivities of silicon (150 W/mK) and air (~0.024 W/mK). Therefore the only way of removing the heat will be via the fixed end and substrate which can be considered as an infinite sink. This is evident from figure 2.19 where the observed shift is proportional to the distance from the fixed end. Based on these observations, for stress estimation in these devices 2mW laser power was chosen. It may be noted this is only the laser power at the source and sample will be exposed to about one fourth of it due to optical losses throughout the beam path [18]. At laser powers lesser than 2 mW, Raman count rate becomes very less and the stress profiles on the cantilever are not repeatable.

Figure 2.19  (a) Variation of Raman shift with increasing laser power at three different positions namely free end, middle and fixed end of the MC1 (b) Three different positions on a free standing MC1.
2.10.2 Residual stress in microcantilever

It is well known that the general uniaxial residual stress field in a thin film may be approximated to a polynomial [18, 19];

$$\sigma(z) \approx \sigma_0 + \sigma_z \left( \frac{z}{t/2} \right)$$  \hspace{1cm} (2.3)

Where $z \in [-t/2, t/2]$ is the coordinate along the thickness with origin on the symmetry plane. In this first approximation, $\sigma_0$ represents the constant mean stress which is symmetric about the mid plane and $\sigma_z$ represents the gradient stress and is anti-symmetric. The gradient stress is due to localized effects including atomic diffusion through thickness of micro cantilever, interstitial or substitutional defects and atomic penning [18]. The stress gradients contribute to axial load and bending moment, cause the structural stiffness change and out of plane deformation of micro cantilever [20]. The stress gradient profile responsible for the cantilever bending is obtained in the present work using micro-Raman. Careful experiments were performed to measure this residual stress in three different configurations along the length of the microcantilever. Measured Raman shift was converted into Stress using equation (2.2) with respect to Raman shift measured on the substrate.

Figure 2.20 shows the Raman shift and the estimated stress variation along the length of the microcantilever for three different experimental configurations i.e when it is front, back and back resting on substrate (as shown in inset) estimated. Following observations can be made from this figure. Stress is tensile and maximum at about 200 μm from fixed end on the front side, whereas on back side and back touching cases stress is compressive and the trend is opposite compared to front side. The opposite sign of stress on front and back side under no load
conditions indicate the cantilever bending induced by the presence of residual stress. At the fixed end the stress was maximum and was found to decrease when going towards free end. Although the trend is same, when compared to back side in back touching configuration stress reduces to 50 percent and might be due to stress relaxation as it is resting on a plane surface. This opposite trend also proves that the observed Raman shift is due to the stress and not due to local temperature rise due to probing laser. In that case, irrespective of the configuration, the Raman shift would have been more at free end compared to fixed end.

![Figure 2.20](image)

Raman shift along the length of the microcantilever in free standing position and resting on a substrate as shown inset. Stress calculated from Raman shift using equation 2.2 is shown in right axis of the figure. Arrow shows the laser probing direction and for these experiments 2 mW laser is used.

Raman spectroscopy is an effective technique to study the residual stress in microcantilevers with a good spatial resolution. Measured stress is tensile on the front side and is compressive on the back side. The opposite trend on front and back side clearly proved the microcantilever bending due to the presence of residual stress.
It is pointed out that while using micro Raman for stress measurements in microcantilevers, lowest laser power must be used to avoid the local heating induced Raman shift especially at free end.

2.11 References

1. Tipless micro cantilevers, M/s Appnano, USA and Duo 500 from M/s micromotive microtechnology, Germany
6. Laserinterferometric vibrometer Series SP-S and LSV user’s guide