Variable Temperature-Scanning Hall Probe Microscopy (VT-SHPM) with GaN/AlGaN Two-Dimensional Electron Gas (2DEG) Micro Hall Sensors in 4.2-425K range, Using Novel Quartz Tuning Fork AFM Feedback

R. Akram[1], M. Dede[1], and A. Oral[2]

[1]Department of Physics, Bilkent University, 06800, Ankara, Turkey
[2]Faculty of Engineering and Natural Sciences, Sabancı University, Orhanli-Tuzla, 34956 Istanbul, Turkey

In this report, we present the fabrication and variable temperature (VT) operation of Hall sensors, based on GaN/AlGaN heterostructure with a two-dimensional electron gas (2DEG) as an active layer, integrated with Quartz Tuning Fork (QTF) in atomic force-guided (AFM) scanning Hall probe microscopy (SHPM). Physical strength and wide band gap of GaN/AlGaN heterostructure makes it a better choice to be used for SHPM at elevated temperatures, compared to other compound semiconductors (AlGaAs/GaAs and InSb), which are unstable due to their narrower band gap and physical degradation at high temperatures. GaN/AlGaN micro Hall probes were produced using optical lithography and reactive ion etching. The active area, Hall coefficient, carrier concentration and series resistance of the Hall sensors were ~1µm x 1µm, 10mΩ/G at 4.2K, $6.3 \times 10^{12} cm^{-2}$ and 12kΩ at room temperature and 7mΩ/G, $8.9 \times 10^{12} cm^{-2}$ and 24kΩ at 400K, respectively. A novel method of AFM feedback using QTF has been adopted. This method provides an advantage over STM feedback, which limits the operation of SHPM the conductive samples and failure of feedback due to high leakage currents at high temperatures. Simultaneous scans of magnetic and topographic data at various pressures (from atmospheric pressure to high vacuum) from 4.2K to 425K will be presented for different samples to illustrate the capability of GaN/AlGaN Hall sensors in VT-SHPM.

Index Terms— Atomic force microscopy, GaN/AlGaN heterostructure, Hall Probe, Quartz Tuning Fork, Scanning Hall Probe Microscopy.

I. INTRODUCTION

THE recent interest in monitoring the localized surface magnetic field fluctuations with high spatial and magnetic field resolutions, at elevated temperatures, has made the scanning Hall probe microscopy (SHPM) [1] one of the best choice as it provides means to perform sensitive, noninvasive, and quantitative imaging. SHPM technique offers various advantages and complements the other magnetic imaging methods like Scanning Superconducting Quantum Interference Device Microscopy (SSM) [2], Magnetic Force Microscopy (MFM) [3], Magnetic Near Field Scanning Optical Microscopy [4] and Kerr Microscopy [5]. However, there have been few reports [6, 7] on magnetic imaging with Hall sensors at high temperature regime. The main reason behind the unsuitability of SHPM at high temperature is the use of compound semiconductors such as AlGaAs/GaAs and InSb for fabrication of Hall probes, which are unstable at elevated temperatures due to their narrow band gap and physical degradation of the materials.

In this study we have investigated the AlGaN/GaN heterosturcures with two dimensional electron gas (2DEG), which offer the advantages of a band gap greater than 2.5eV. This wide band gap hetero-structure offers the advantage of reducing the possibility of thermally induced intrinsic conduction and the existence of a high mobility of a two dimensional electron gas layer which greatly enhances the magnetic sensitivity of Hall sensors.

In reported work from another group [6], SPM with AlGaN/GaN based micro Hall probes with dimensions of 2µm x 2µm has been demonstrated using STM feedback at higher temperatures (~375K). In this work, fabrication and characterization of AlGaN/GaN heterostructures with dimension of 1µm x 1µm is presented, together with the integration of sensors to the quartz Crystal tuning forks. Although there are other methods like integrating the Hall sensor with piezoresistive [8] and Si_3N_4 AFM cantilevers [9], it is relatively difficult and cumbersome to microfabricate these sensors. Quartz crystal force sensors [10] have been used in Scanning Probe Microscopy (SPM) for a wide range of applications; since the quartz crystal tuning forks are very cheap, readily available and the force sensing is performed using a simple current to voltage converter. By using QTF AFM feedback we have also eliminated the necessity to have a conductive sample or coat the insulating samples with a thin layer of gold. Moreover, the use of gold thin film as an integrated STM tip is also eliminated. On rough surfaces this STM tip can wear off easily and damage to Hall sensor follows inevitably. In this work the frequency response of Quartz Tuning fork has been studied in a systematic way based on theoretical models, simulation results and experimental data. The quartz crystal AFM feedback SHPM is very simple to run, as it uses self sensing quartz force sensors. The reliable operation of the scanning Hall probe microscopy using Quartz tuning fork in a wide temperature range of 4.2-425K, with a better lifetime performance for the Hall sensor than STM tracking is also demonstrated for the first time.

II. Fabrication and Characterization

A. Fabrication

1) Wafer Specifications

The semiconductor wafers used are rotating disc MOCVD grown AlGaN/GaN on Si (111) two dimensional electron gas (2DEG) material [11]. The epistructure of the wafer, as shown in Fig. 1 (a), consist of the following layers; 1) a 20Å thin layer of GaN cap layer for protection purposes, 2) 180Å of $Al_{0.26}Ga_{0.74}N$ layer, 3) 1µm thick layer of undoped GaN, which forms a 2 DEG at the AlGaN interface, 4) proprietary stress mitigating transition layer of 1.1µm, and 5) High resistivity Si (111) substrate with a resistivity of 10kΩcm. The room temperature sheet carrier concentration and electron mobility of the 2DEG induced at the heterointerface were 2 x 10^{12} cm^{-2} and 1,500 cm^2/Vs, respectively.

2) Device Fabrication

Micro Hall probes with effective dimension of 1µm x 1µm have been fabricated using optical lithography in a class 100 clean room environment. Device fabrication process consists of three major steps which are: 1) formation of the mesa and active "cross" patterns by reactive ion etching (RIE) using CCl_2F_2 gas plasma; (2) thermal evaporation of Ti/Al/Ti/Au Ohmic contacts; and (3) rapid thermal processing (RTP) in a nitrogen atmosphere at 850°C for 30s. Four Hall sensors are microfabricated on a 5mm × 5mm chip at a time and they are diced to a size of 1mm × 1mm × 0.5mm. The SEM graph of the fabricated Hall probe is shown in Fig. 1 (b).

FIG. 1 HERE

B. Electrical and Magnetic Characterization

1) Electrical Characteristics

In order to investigate the electrical characteristics of Hall probes electrical connection have been established with 12µm gold wire using ultrasonic wedge bonder. Hall Sensors have been characterized base on Hall voltage (V_H) vs. Hall current (I_H) under different bias temperature conditions. As shown in Fig. 2, linear relations can be observed between V_H vs I_H characteristics with two different dynamic resistance (slope of V_H vs. I_H curve, r_H V_H / I_H) values. At 300K as shown in Fig. 3, low current regime ($I_H < 100µA$) r_{HLC} is 510Ω and in high current regime ($I_H > 100µA$) it is found to be $r_{HHC} = 62Ω$. It is speculated that this decrease in the r_H value is due to that there might be opening of new conduction channels by applying high current causing an increase in the number of parallel paths. This has been further investigated by increasing the temperature. An increase in temperature from 300K to 425K shows that r_{HLC} increases by 29.4% and r_{HHC} decreases by 50% which further supports the previously stated argument of increase in the number of channels by increasing Hall current.

FIG. 2 HERE
FIG. 3 HERE

2) Magnetic Characterization

Hall probes have been also characterized for their magnetic response by applying magnetic field with an external coil and by measuring the Hall Voltage to calculate the value of Hall coefficient, R_H. As shown in Fig. 4, value of R_H also depends on the Hall current and two regimes can be formed as in the case of V_H vs I_H. At high Hall current bias, R_H is more affected by the temperature.

FIG. 4 HERE

During the experiments, the scanning sensor remains at high temperatures for long period of times. We have investigated run time effect on V_H vs I_H and R_H vs. I_H characteristics under high temperature environments. The results showed no significant change in the values, suggesting a safe use of these Hall probes in scanning systems over a long time in harsh conditions.

III. Scanning Hall Probe Microscopy

Our scanning Hall probe microscope can operate under two different feedback schemes namely, STM and AFM. As mentioned in the introduction, in order to compensate the drawbacks of STM feedback especially at high temperatures a novel method of quartz tuning fork AFM feedback has been implemented.

A. Quartz Tuning Fork Hall Probe Microscope System

In this study 32.768kHz Quartz crystals tuning forks with dimensions (l × w × t) of prongs are, 3.81mm × 0.34mm × 0.62mm resulting in stiffness of 29 kN/m, have been used. In order to integrate these force sensors in SHPM for AFM feedback, they are extracted from their cans and their leads have been replaced with a non magnetic wiring. Furthermore these Quartz tuning forks are glued to a 10mm × 10mm printed circuit board sensor holder compatible with the scanning head of the commercially available SHPM system [12]. Hall probe with chip size of 1mm x 1mm x 0.5mm has been mounted on the Quartz tuning fork using low temperature epoxy for low temperature measurement while for high temperature scanning this bond has been reinforced by using super glue. Fig. 5 shows the assembly of Hall probe on a Quartz tuning fork.

FIG. 5 HERE

In order to investigate the loading effect of Hall probe chip on the resonance frequency of the quartz tuning fork detailed calculations have been done. These calculations were done based on two simplest models; Simple Harmonic Oscillator (SHO) and Cantilever Beam Theory (CBT). According to calculations based on these two models, the combination of HP with QTF gives a typical resonance frequency of 31.7kHz (SHO model) and 21.2kHz (CBT model). The results are summarized in Table 1.

TABLE. 1 HERE

Further by using the computer simulation program (COSMOS), these QTF's were investigated for different mounting techniques as shown in Fig.5. It has been observed under unloaded (no Hall probe attached to QTF) condition when both prongs are free then we have two modes of operation, namely symmetric (both prongs are vibrating up and down in phase) with 16.581 kHz, (close to CBT model) and Asymmetric (prongs are 180° out of phase while vibrating

up and down) with 31.729 kHz (close to SHO). Effect of fixing one prong showed up as an elimination of the asymmetric mode and we get single frequency of 15.642 kHz.

Simulation results after adding Hall probe to the QTF show similar results as in the case of free prong as we can observe the presence of both type of resonance frequencies; 17.138kHz (symmetric mode) and 31.4kHz (asymmetric mode) and under one prong fixed condition we have single frequency at 17.6kHz. These values are in close agreement with the calculated values from CBT (symmetric mode) and SHO (asymmetric mode) models. The slight discrepancy in the theoretical results, simulation and given specification is due to the simplicity of the model used where the effect of bonding wires, weight of the glue and effect of ambient environment has not been included in the calculation.

In order to confirm these results real probes, where GaN Hall probes have been attached to the tip of the QTF, have been prepared using both configuration (one prong fixed and both prongs free), as shown in Fig. 6. Frequency response of QTF has been measured using a simple i-v converter with a gain of 1.5×10^7V/A, as we dithered the sensor assembly with the special excitation piezo integrated at the scanner.

FIG. 6 HERE

From Fig. 6 it is clear that when both prongs are free we have two resonance frequencies around 17.035kHz and 31.535kHz but when we fix one of the prongs, we get a single resonance frequency around 17.84kHz. A slight discrepancy from the calculated and simulated results is probably due to the mass of glue and the position of the HP has not been included at the calculations and simulation. There is a peak around ~3 kHz or ~6 kHz, as seen in Fig. 6. This is due to the resonance frequency of the whole scanner assembly. We have used two different systems with 2" and 3" long scan piezo tubes.

Based on the above results, we can conclude that for stable operation one prong of the TF should be fixed. Under such condition we are close to symmetric mode and this can be easily explained by using the cantilever beam theory.

B. Scanning Results and discussion

A commercial Low Temperature-SHPM system [11] with some modifications for high temperature measurements is used to perform the scanning experiments. The Hall sensor is positioned 12μm away from corner of a deep etch mesa, which serves as a crude AFM tip. The sample is tilted ~1°- 2° with respect to Hall probe chip ensuring that the corner of the mesa is the highest point. As the combined sensor is approached to the surface, the resonant frequency of the sensor shifts due to tip sample forces. The sensor assembly is dithered at the resonance frequency with the dedicated split section on the scan piezo tube using a digital Phase Locked Loop (PLL) circuit. The frequency shift Δf, measured by the PLL circuit is used for AFM feedback to keep the sensor sample separation constant with the feedback loop. The microscope can be operated in two modes: AFM tracking and

lift-off mode. In our scanning experiments we have used the AFM tracking mode with a Δf (amount of frequency shift) = 10Hz. Furthermore in order to detect AFM topography and the error signal generated by the PLL, along with the magnetic field image, SHPM Electronics is modified. Even though a relatively heavy mass is attached at the end of tuning fork, we usually get a quality factor, Q, 150-220 even at atmospheric pressures. Despite more or less the planar geometry, the viscous damping is not a big problem due to high stiffness of the force sensor.

FIG. 7 HERE

We have imaged magnetic bits and topography of the Hard Disk at various temperatures to show the performance of the microscope with AFM feedback using QTF. In order to scan at low temperature the sample space is filled with 1Atm of Helium exchange gas at 300K and cooled down to 4.2K. The Hall sensor was driven with 500μA DC current, the series resistance of the Hall sensor was 0.5kΩ, 3KΩ and 15kΩ at 4.2K, 77K and 300K respectively. The resonance frequency and the quality factor were 17,780 Hz and Q~60, 17,630 Hz and Q~97, and 16,944 Hz and Q~305 at 4.2K, 77K and 300K respectively. Fig. 7 shows magnetic image and topography of Hard Disk sample obtained in AFM tracking mode at 4.2K, 77K with a scanning speed of 5μm/s and scan area of 17μm x 17μm and 30μm x 30μm respectively.

FIG. 8 HERE

In order to investigate the high temperature operation of these GaN micro Hall probes, a low noise heater stage has been embedded in the LT system. The results shown in Fig. 8 confirm the success of AFM feedback tracking system with micro Hall probes up to 425K. The observed distortions in the scanned images at high temperatures are considered to be not due to performance of the Hall probe but it is mainly due to the properties of the QTF and the problems related with the used glue and degradation of the sample. The resonance frequency and quality factor of the quartz crystal tuning fork changes with the temperature as shown in Fig. 9, however this change is still much less than the conventional silicon AFM cantilevers. Further investigation is under process to find out the methods to improve the thermal stability of the QTF AFM feedback and exploration of GaN Hall probe microscopy at even higher temperatures.

FIG. 9 HERE

IV. CONCLUSION

Micro-Hall probes with a Hall cross area of 1μm x 1μm with AlGaN/GaN heterostructure were fabricated and characterized for their application in variable temperature magnetic imaging. Electrical and magnetic characteristics can be divided into two regimes base on the Hall current value. In each regime Hall voltage vs. Hall current characteristics showed linear behavior, while the slope of V_H vs. I_H curve (dynamic resistance) was high in low current regime and low in high current regime while it was also a function of working temperature. Low current regime shows less effect on V_H

curves and has high R_H value but value of V_H is low. These Hall probes have been integrated with quartz tuning fork and successfully used in scanning Hall probe microscopy. Simulation and experimental study of quartz tuning fork with Hall sensor shows that the system works better if one prong of the QTF is fixed. Scanning Hall probe microscopy using Quartz crystal tuning fork AFM feedback with 1μm x 1μm AlGaN/GaN Hall probes has been successfully demonstrated for a hard disk sample for temperature range of 4.2K to 425K. We are continuing our efforts to achieve higher spatial resolution and even higher temperature applications of GaN Hall probes.

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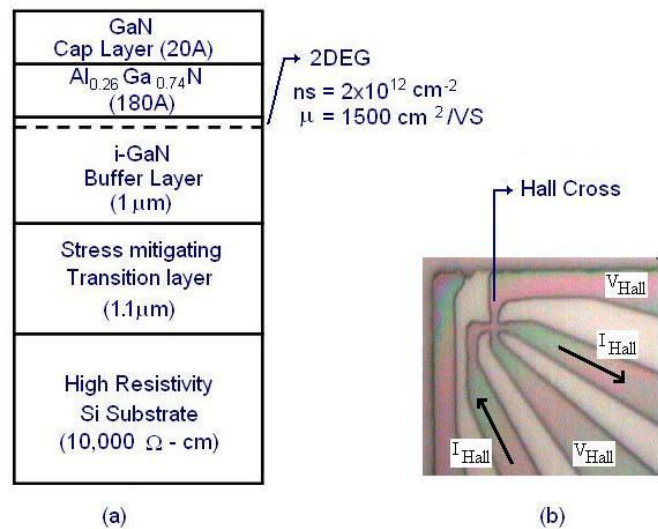
Fig. 1. a) Schematic diagram of the layer configuration of the AlGaN/GaN heterostructure used, b) Photograph of 1μm x 1μm Hall probe showing the STM.
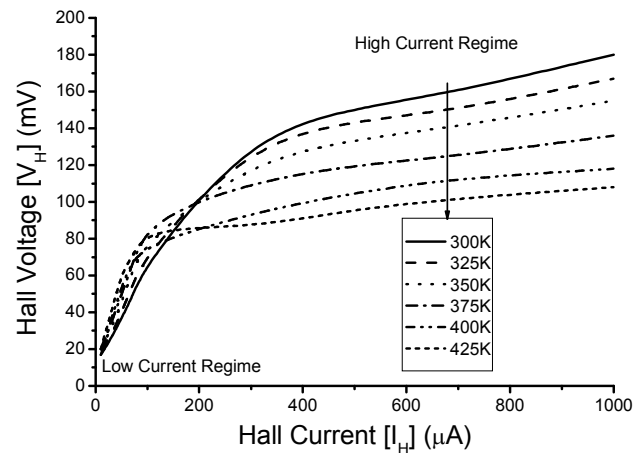

Fig. 2. Effect of Temperature on the Hall Voltage vs. Hall current characteristics.
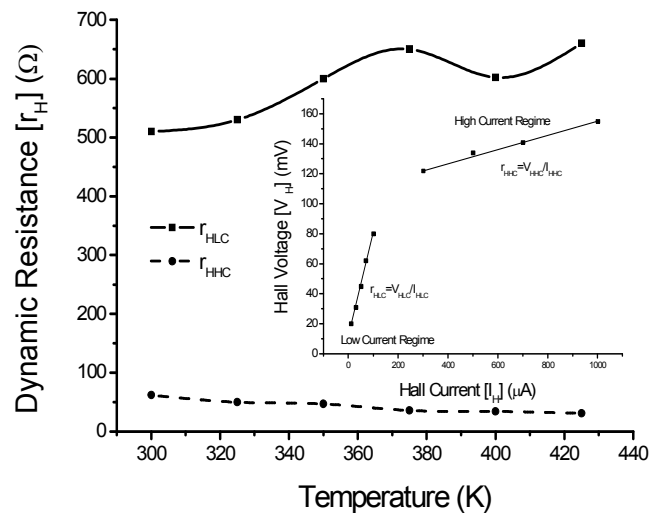

Fig. 3. Effect of temperature on the dynamic resistances r_{HLC} and r_{HHC}. The r_{HLC} and r_{HHC} are defined in the inset graph.

TABLE I
CALCULATION RESULTS FOR QUARTZ TUNING FORK

Dimensions of Quartz Tuning Fork				
Tuning Fork	Width	Length	Thickness	Mass of Cantilever
	0.34mm	3.81mm	0.62mm	2.13mg
Hall Probe	Width	Length	Thickness	Mass of Hall Probe
	1mm	1mm	0.475mm	1.11mg

Calculation		
Theoretical Models	Simple Harmonic Oscillator	Cantilever Beam theory
Effective Mass	$M_{SHO} = \dfrac{m_c M_c}{m_c + M_c}$ = 0.73mg	$M_{CBT} = M_c + 0.24 m_c$ = 1.62mg
Resonance Frequency	$f_{SHO} = \dfrac{1}{2\pi}\sqrt{\dfrac{k}{M_{SHO}}}$ = 31.7kHz	$f_{CBT} = \dfrac{1}{2\pi}\sqrt{\dfrac{k}{M_{CBT}}}$ = 21.2kHz

Simulation		
Vibration Mode	Symmetric	Asymmetric
Both prongs are free	17.14kHz	31.41kHz
One prong is free	17.6kHz	0

Stiffness Constant (k) was calculated using $3\,\dfrac{E}{l^3}\,\dfrac{wt^3}{12} = 2.88 \times 10^4\,[N/m]$
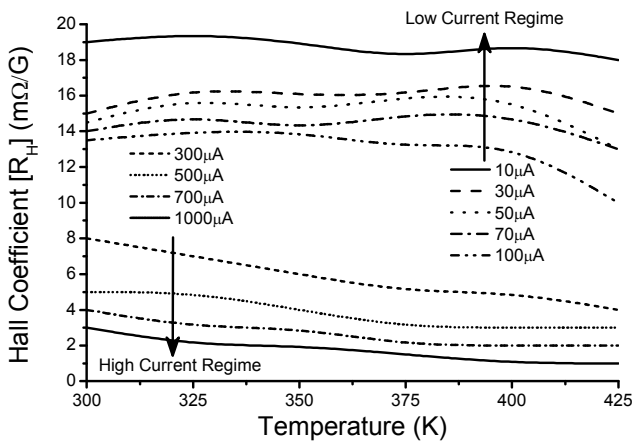
Fig. 4. Effect of Hall current on Hall coefficient vs. Temperature characterization.

Hall Cross
(1μm x1μm)

Gold wires

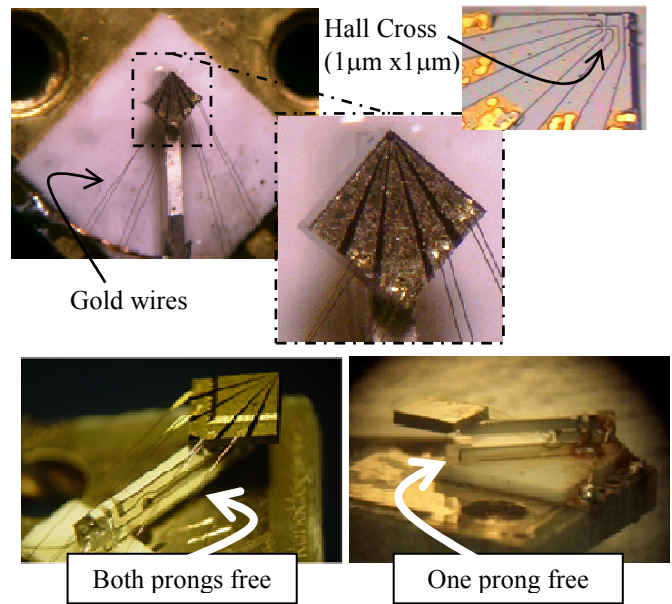Both prongs free   One prong free

Fig. 5. Top) Integration of Hall probe with Quartz Tuning fork with Gold wire for electrical contacts. Bottom) Quartz Tuning fork installation techniques, left) when both prong are free (free to move), right) when bottom prong is fixed to bottom.
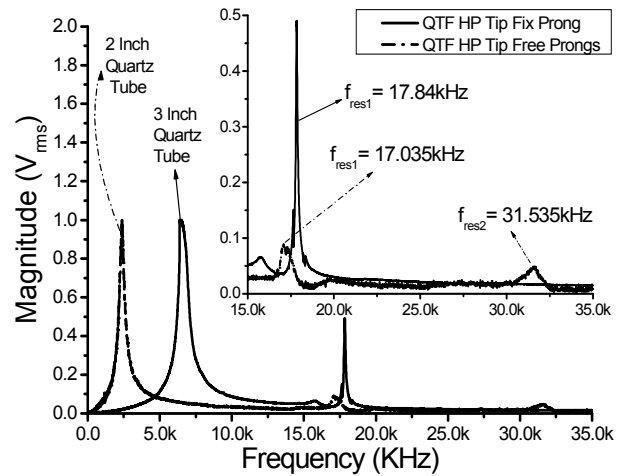

Fig. 6. Frequency response of 32kHz Quartz Tuning fork with integrated GaN Hall probe under fix prong and free prong configuration. The effect of Quartz tube of scan head is also shown.
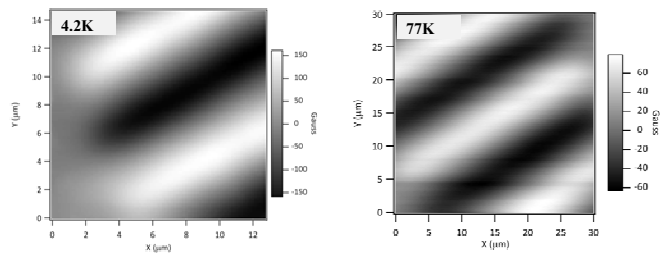

Fig. 7. SHPM image of hard disk sample at a) Liquid helium temperature (4.2K) b) Liquid Nitrogen Temperature (77K). Scanning speed was 5μm/s.
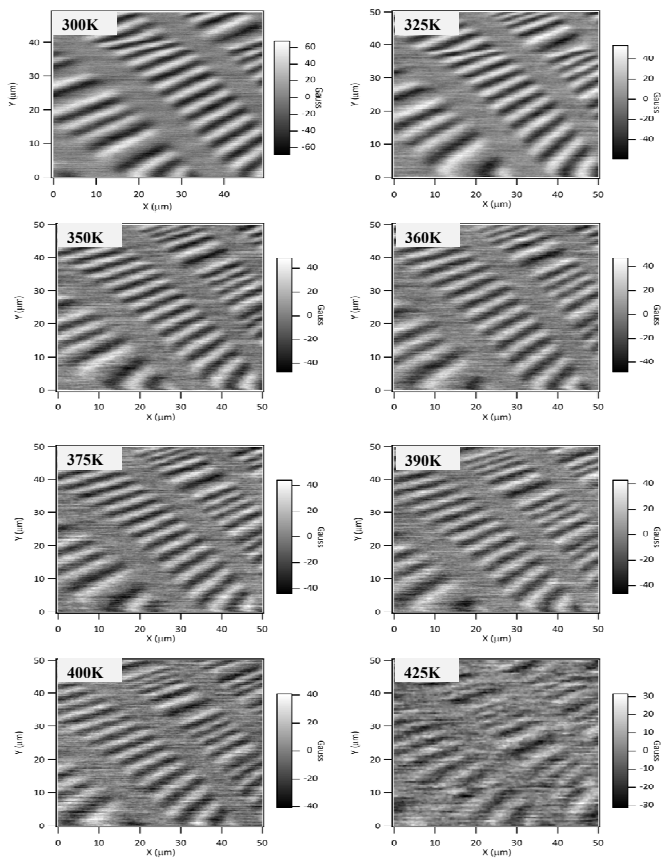
Fig. 8. SHPM image of hard disk sample at high temperatures. Scanning speed was 5μm/s. Distortions at 425K are most probably to be due to loss of adhesion between the HP and tuning fork.
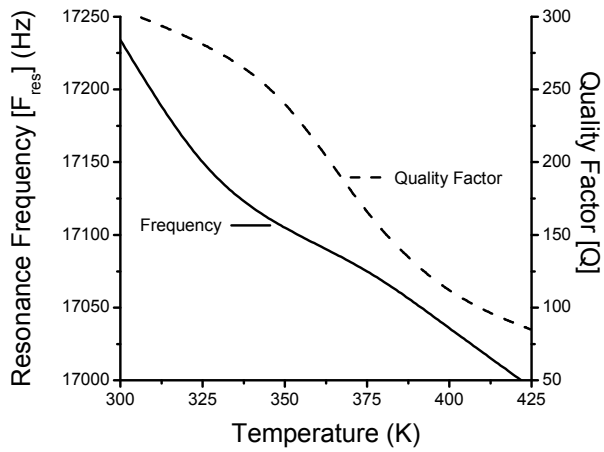

Fig. 9. Effect of working temperature on the resonance frequency and quality factor of the quartz tuning fork.