Variable Temperature-Scanning Hall Probe Microscopy With GaN/AlGaN Two-Dimensional Electron Gas (2DEG) Micro Hall Sensors in 4.2–425 K Range Using Novel Quartz Tuning Fork AFM Feedback

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In this paper, 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 a wide bandgap 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 bandgap 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 $\sim 1 \times 1\ \mu$m, 10 mΩ/G at 4.2 K, 6.3×10^{12} cm^{-2} and 12 kΩ at room temperature and 7 mΩ/G, 8.9×10^{12} cm^{-2} and 24 kΩ at 400 K, respectively. A novel method of AFM feedback using QTF has been adopted. This method provides an advantage over scanning tunneling-guided 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. 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 (AFM), GaN/AlGaN heterostructure, Hall probe, quartz tuning fork (QTF), scanning Hall probe microscopy (SHPM).

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 choices as it provides means to perform sensitive, noninvasive, and quantitative imaging. The 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 the high temperature regime. The main reason behind the unsuitability of SHPM at high temperatures 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 bandgap and physical degradation of the materials.

In this paper, we have investigated the AlGaN/GaN heterostructures with two-dimensional electron gas (2DEG), which offer the advantages of a bandgap greater than 2.5 eV. This wide bandgap heterostructure offers the advantage of reducing the possibility of thermally induced intrinsic conduction and the existence of a high mobility of a 2-D 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 $2 \times 2\ \mu$m has been demonstrated using scanning tunneling-guided (STM) feedback at higher temperatures ($\sim$375 K). In this paper, fabrication and characterization of AlGaN/GaN heterostructures with dimension of $1 \times 1\ \mu$m 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$_3$N$_4$ atomic force microscopy (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 paper, the frequency response of the QTF 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 the QTF in a wide temperature range of 4.2 K–425 K, 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 metal–organic chemical vapor deposition (MOCVD) grown AlGaN/GaN on Si (111) 2-D electron gas

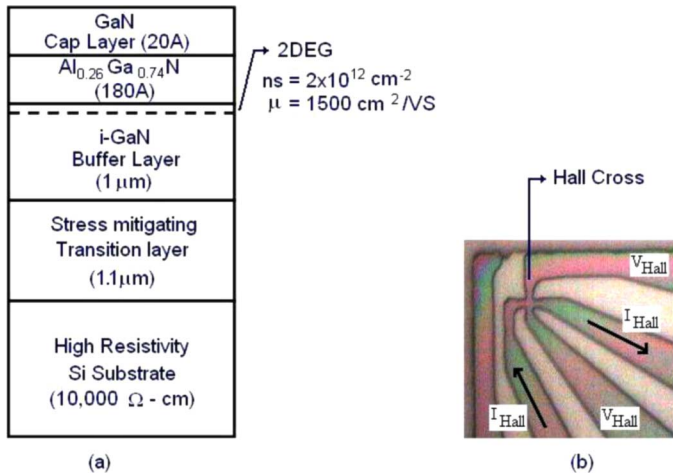[1]Color versions of one or more of the figures in this paper are available online at http://ieeexplore.org.

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

Fig. 2. Effect of temperature on the Hall voltage versus Hall current characteristics.

(2DEG) material [11]. The epistructure of the wafer, as shown in Fig. 1(a), consists of the following layers; 1) a 20-Å-thin layer of GaN cap layer for protection purposes; 2) 180 Å of $Al_{0.26}Ga_{0.74}N$ layer; 3) 1-μm-thick layer of undoped GaN, which forms a 2DEG at the AlGaN interface; 4) proprietary stress mitigating transition layer of 1.1 μm; and 5) high resistivity Si (111) substrate with a resistivity of 10 k Ω cm. The room temperature sheet carrier concentration and electron mobility of the 2DEG induced at the heterointerface were 2×10^{12} cm^{-2} and 1500 cm^2/Vs, respectively.

2) Device Fabrication: Micro-Hall probes with effective dimension of 1×1 μm 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 30 s. Four Hall sensors are microfabricated on a 5×5-mm chip at a time and they are diced to a size of $1 \times 1 \times 0.5$ mm. The SEM graph of the fabricated Hall probe is shown in Fig. 1(b).

B. Electrical and Magnetic Characterization

1) Electrical Characteristics: In order to investigate the electrical characteristics of Hall probes, an electrical connection has been established with 12-μm gold wire using an ultrasonic wedge bonder. Hall sensors have been characterized based on Hall voltage (V_H) versus Hall current (I_H) under different bias temperature conditions. As shown in Fig. 2, linear relations can be observed between V_H versus I_H characteristics with two different dynamic resistances (slope of V_H versus I_H curve, $r_H V_H / I_H$) values. At 300 K as shown in Fig. 3, low current regime ($I_H < 100\mu A$) r_{HLC} is 510 Ω and in high current regime ($I_H > 100\mu A$) it is found to be $r_{HHC} = 62\Omega$. It is speculated that this decrease in the r_H value is due to the fact that there might be an 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.
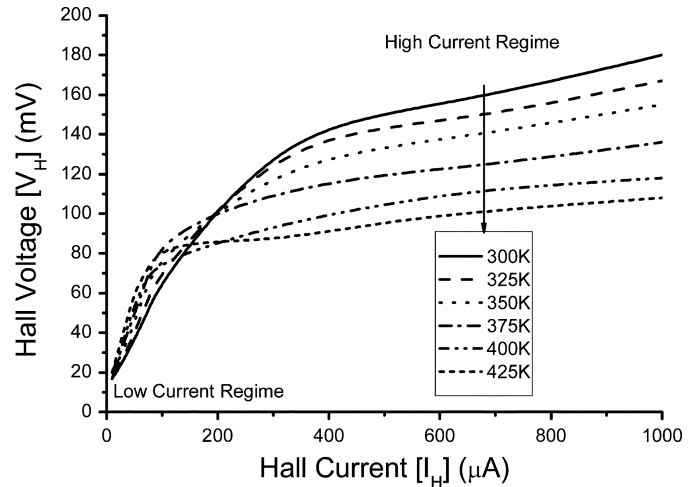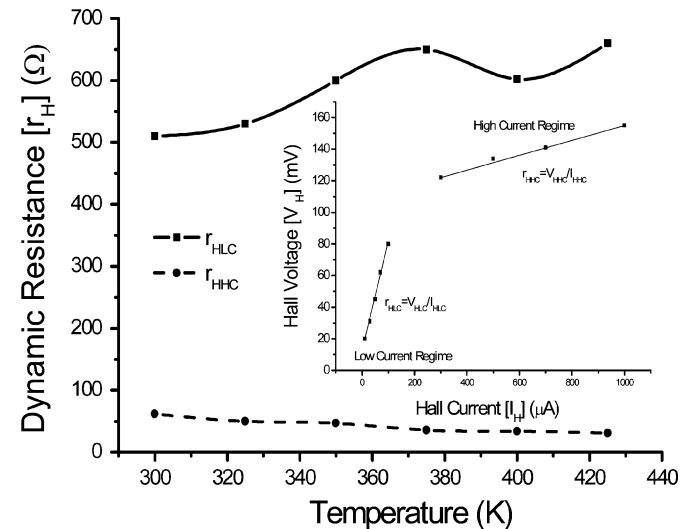

Fig. 3. Effect of temperature on the dynamic resistances r_{HLC} and r_{HHC}. The r_{HLC} and r_{HHC} are defined in the inset graph.

An increase in temperature from 300 K to 425 K 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.

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, the value of R_H also depends on the Hall current and two regimes can be formed as in the case of V_H versus I_H. At high Hall current bias, R_H is more affected by the temperature.

During the experiments, the scanning sensor remains at high temperatures for long periods of time. We have investigated run time effect on V_H versus I_H and R_H versus 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.
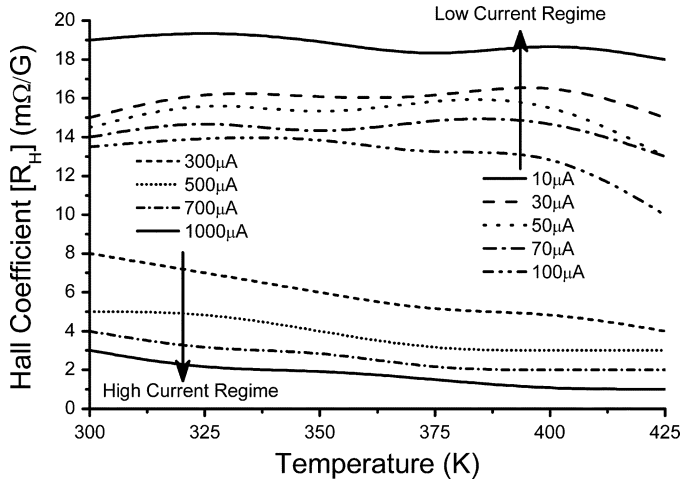
Fig. 4. Effect of Hall current on Hall coefficient versus temperature characterization.
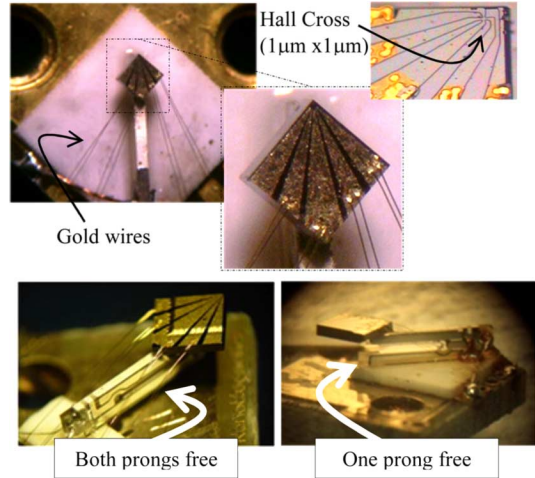

Fig. 5. (Top) Integration of Hall probe with QTF with gold wire for electrical contacts. (Bottom) QTF installation techniques, (left) when both prongs are free (free to move) and (right) when bottom prong is fixed to bottom.

III. SHPM

Our SHPM can operate under two different feedback schemes, namely, STM and AFM. As mentioned in Section I, in order to compensate for the drawbacks of STM feedback, especially at high temperatures, a novel method of QTF AFM feedback has been implemented.

A. QTF Hall Probe Microscope System

In this paper, 32.768 k Hz quartz crystals tuning forks with dimensions (l × w × t) of prongs 3.81× 0.34× 0.62 mm 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 nonmagnetic wiring. Furthermore, these QTFs are glued to a 10 × 10-mm printed circuit board sensor holder compatible with the scanning head of the commercially available SHPM system [12]. A Hall probe with chip size of $1 \times 1 \times 0.5$ mm has been mounted on the QTF 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 the Hall probe on a QTF.

In order to investigate the loading effect of the Hall probe chip on the resonance frequency of the QTF, detailed calculations have been done. These calculations were done based on the 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 31.7 k Hz (SHO model) and 21.2 k Hz (CBT model). The results are summarized in Table I.

Further, by using the computer simulation program (COSMOS), these QTFs were investigated for different mounting techniques as shown in Fig. 5. It has been observed under unloaded (no Hall probe attached to QTF) conditions when both prongs are free that we have two modes of operation, namely symmetric (both prongs are vibrating up and down in phase) with 16.581 k Hz, (close to CBT model) and Asymmetric (prongs are 180° out of phase while vibrating up and down) with 31.729 k Hz (close to SHO). The effect of fixing

TABLE I
CALCULATION RESULTS FOR QTF

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	$M_{SHO} = \dfrac{m_c M_t}{m_c + M_t}$ $= 0.73mg$		$M_{CBT} = M_t + 0.24 m_c$ $= 1.62mg$	
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]

one prong showed up as an elimination of the asymmetric mode and we get a single frequency of 15.642 k Hz.

Simulation results after adding the Hall probe to the QTF show similar results as in the case of the free prong as we can observe the presence of both types of resonance frequencies: 17.138 k Hz (symmetric mode) and 31.4 k Hz (asymmetric mode). Under one-prong-fixed conditions we have a single frequency at 17.6 k Hz. 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,
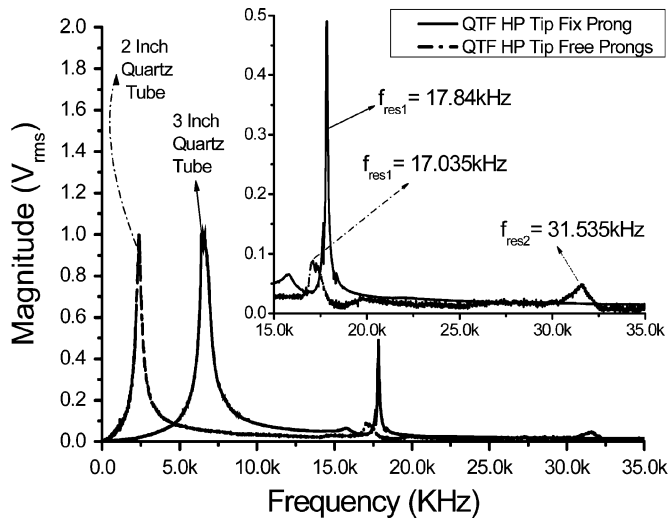
Fig. 6. Frequency response of 32 k Hz QTF with integrated GaN Hall probe under fixed-prong and free-prong configurations. The effect of the Quartz tube of scan head is also shown.

weight of the glue, and effect of ambient environment has not been included in the calculations.

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. The frequency response of the QTF has been measured using a simple I–V converter with a gain of 1.5×10^7 V/A, as we dithered the sensor assembly with the special excitation piezo integrated at the scanner.

From Fig. 6, it is clear that when both prongs are free we have two resonance frequencies around 17.035 k Hz and 31.535 k Hz, but when we fix one of the prongs we get a single resonance frequency around 17.84 k Hz. 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 k Hz 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-in-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 conditions 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 12 μm away from the corner of a deep etch mesa, which serves as a crude AFM tip. The sample is tilted ∼1°–2° with respect to the Hall probe chip, ensuring that the corner of the mesa is the highest point. As the combined sensor approaches 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
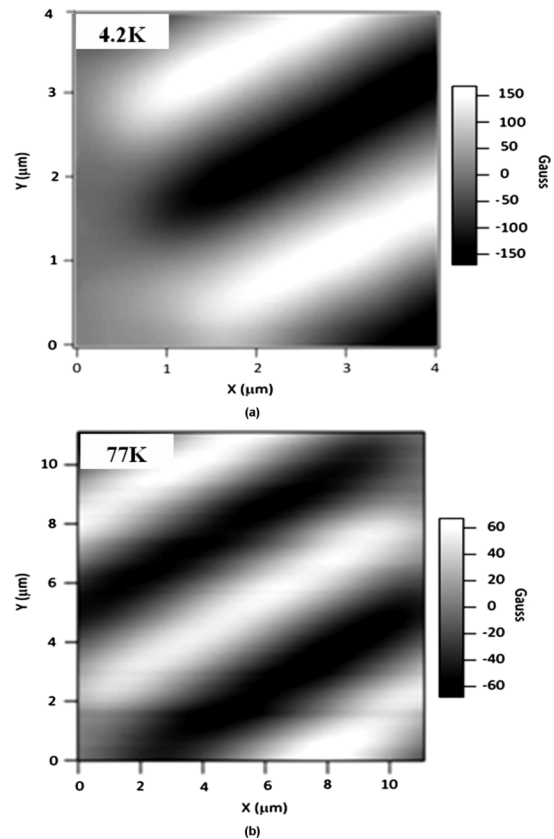

Fig. 7. SHPM image of hard disk sample at (a) liquid helium temperature (4.2 K) and (b) liquid nitrogen temperature (77 K). Scanning speed was 5 μm/s.

(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 liftoff mode. In our scanning experiments, we have used the AFM tracking mode with a Δf (amount of frequency shift) = 10 Hz. Furthermore, in order to detect AFM topography and the error signal generated by the PLL, along with the magnetic field image, SHPM electronics are modified. Even though a relatively heavy mass is attached at the end of the tuning fork, we usually get a quality factor, Q, at 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.

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 temperatures, the sample space is filled with 1 Atm of Helium exchange gas at 300 K and cooled down to 4.2 K. The Hall sensor was driven with 500 μA dc current, the series resistance of the Hall sensor was 0.5 kΩ, 3 KΩ, and 15 kΩ at 4.2 K, 77 K, and 300 K, 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 4.2 K, 77 K, and 300 K, respectively. Fig. 7 shows the magnetic image and topography of the hard disk sample obtained in AFM tracking mode at 4.2 K, 77 K with a scanning speed of 5 μm/s and scan area of 17× 17 μm and 30 × 30 μm, respectively.
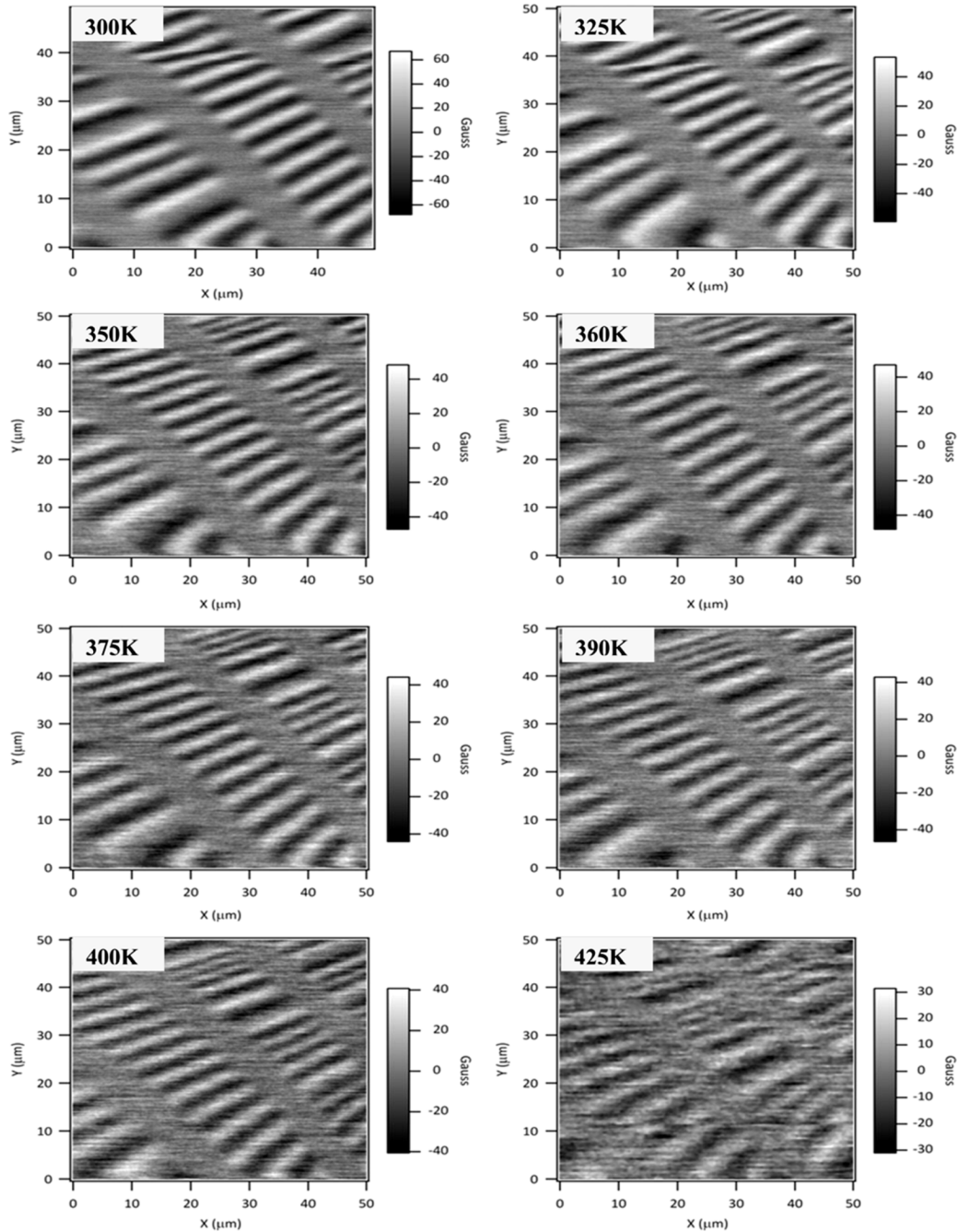
Fig. 8. SHPM image of hard disk sample at high temperatures. Scanning speed was 5 μm/s. Distortions at 425 K are most probably due to loss of adhesion between the HP and tuning fork.

In order to investigate the high-temperature operation of these GaN micro Hall probes, a low noise heater stage was embedded in the LT system. The results shown in Fig. 8 confirm the success of the AFM feedback tracking system with micro-Hall probes up to 425 K. 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 underway 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.

IV. CONCLUSION

Micro-Hall probes with a Hall cross area of 1×1 μm 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 based on the Hall current value. In each regime, Hall
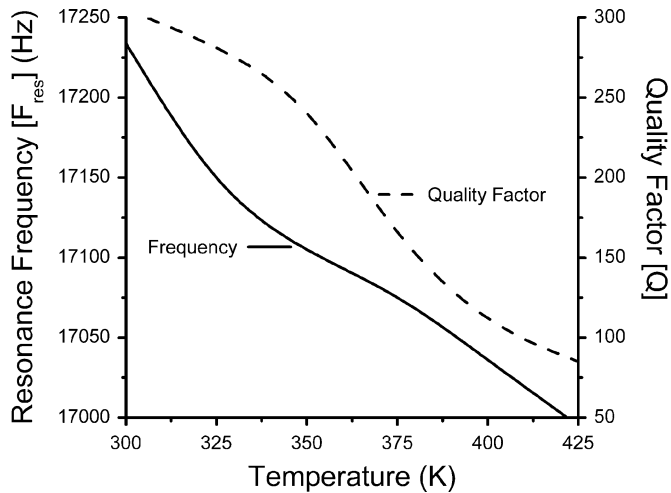
Fig. 9. Effect of working temperature on the resonance frequency and quality factor of the QTF.

voltage versus Hall current characteristics showed linear behavior, while the slope of V_H versus I_H curve (dynamic resistance) was high in the low current regime and low in the high current regime. It was also a function of working temperature. The low current regime shows less effect on V_H curves and has high R_H value but a value of V_H is low. These Hall probes have been integrated with a QTF and successfully used in scanning Hall probe microscopy. Simulation and experimental study of the QTF with Hall sensor shows that the system works better if one prong of the QTF is fixed. SHPM using a Quartz crystal tuning fork AFM feedback with 1×1-μm AlGaN/GaN Hall probes has been successfully demonstrated for a hard disk sample for temperature range of 4.2 K to 425 K. We are continuing our efforts to achieve higher spatial resolution and even higher temperature applications of GaN Hall probes.

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