Structure Modification of M-AFM Probe for the Measurement of Local Conductivity

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***Abstract*- In order to realize the evaluation of electrical properties of materials in nanoscale orders, a method for the measurement of local conductivity was presented. A microwave atomic force microscope (M-AFM) probe in which microwave signals can propagate was fabricated. An open structure of a waveguide at the tip of the probe was introduced by focused ion beam (FIB) fabrication. The microwave measurement system consisted of the combination of a network analyzer working at 44.5 GHz and an AFM were used to measure the samples without contact. The amplitude and phase of the reflection coefficient of the microwave signal were measured to determine the electrical conductivity of non magnetic metals. The conductivity obtained by this method agrees with that measured by the high-frequency conductometry.**

I. Introduction

The development of the technique which is able to measure the electrical properties, such as conductivity, permittivity and permeability, in nanometer scale is far in the rear by comparing the development of other new scanning probe microscope. Electrical property is one of the most important basic properties, which has an influence on functionality of materials. Especially, the electrical properties of materials in a nanoregion are affected by not only the structure and composition of materials but also the mechanical factors of stress and strain related to the lattice oscillation. The measurement of the electrical properties in nanoregion is expected to applying the creation of nanomaterials, the development and evaluation of nanodevice, mechanism elucidation of living tissues and so on. To realize the purpose, the extensions of microwave measurement are some kinds of hopeful method to evaluate electrical properties of materials without contacting a sensor, because the response of microwave is directly relative to the electrical properties of materials. Thus, microwave microscope has been an interest to many researchers [1-4]. To evaluate the electrical properties of materials using microwave, it is necessary to keep the standoff distance between a microwave sensor and a sample constant, because standoff distance affects microwave signal strongly in the near field. It is difficult to distinguish the difference of microwave signal due to the properties of a measuring object or the change of stand off distance. Therefore, to evaluate the electrical properties of materials in nanometer scale and with high resolution, it is indispensable to control the standoff distance high precisely.

Ju and coauthors [5-9] have proposed a microwave atomic force microscope (M-AFM).This technique combines the characteristics of the microwave microscope and the atomic force microscope which has nanoscale spatial resolution and can keep standoff distance constantly by the atomic force between the tip and sample. Having these advantages, M-AFM is able to realize the evaluation of electrical properties as well as the measurement of topography of materials in nanometer scale. In the earlier research, using the fabricated M-AFM probe, the emission of the microwave signals from the probe tip by connecting the improved AFM holder to a network analyzer was detected [9]. Moreover, Hosoi and coauthors [10] confirmed the influence on the mechanical properties and the electric sensitivity of the M-AFM probe due to the difference of the coating films introduced by different coating methods. However, the measurement of local conductivity quantitatively has not been discussed. In order to evaluate the local conductivity of materials quantitatively, the microwave reflectometry was applied with M-AFM.

II. Probe Fabrication

A no doped GaAs wafer was used as the substrate of the probe in order to restrain the attenuation of microwave propagating in the probe. Wet etching was used to fabricate the probe because it is possible to obtain the desired structure by causing a side etching under the etching mask in contrast to dry etching. The fabrication method of the probe with GaAs wafer was studied in details by Ju and coauthors [5-10]. The M-AFM GaAs probe was fabricated by the following process: (a) Patterning of the etching mask for the tip fabrication; (b) Forming the tip of the probe by wet etching; (c) Patterning of the stencil mask for the waveguide on the top surface; (d) Coating Au film on the top surface; (e) Forming the waveguide by lift-off process; (f) Patterning of the etching mask for the cantilever fabrication; (g) Forming the cantilever of the probe by wet etching; (h) Patterning of the etching mask on back side for the holder fabrication; (i) Forming the holder of the probe by wet etching; (j) Coating Au film on the bottom surface to form the waveguide; (k) Forming the open structure at the tip of the probe by FIB fabrication. The fabricated M-AFM probe was observed with scanning electron microscopy (SEM).

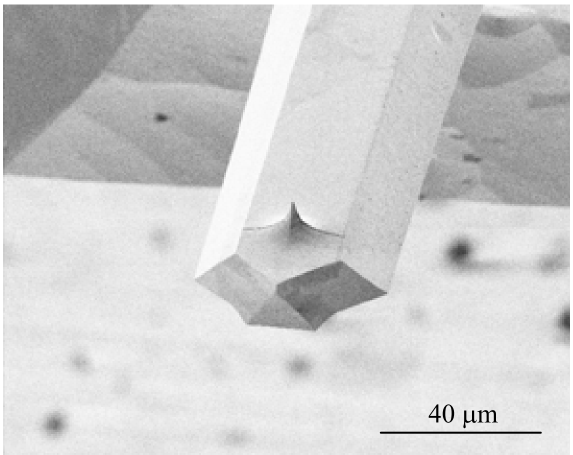

Fig. 1. An M-AFM probe introduced by FIB fabrication.

As shown in Fig. 1, a sharp tip having high aspect ratio of 2.0 was obtained. The tip is the 8 μm high, and the curvature radius of the tip is approximately 30 nm. A microslit was introduced through the center of the tip cross the cantilever by the FIB fabrication. The width of the microslit is approximately 100 nm.

III. MICROWAVE MEASUREMENT

The principle of the technique described here is based on the reflection of the microwave from materials. The measured reflection coefficient of the microwave signal can be expressed by considering the reflection only from the top surface as

$$\Gamma = \frac{\eta - \eta_0}{\eta + \eta_0}, \tag{1}$$

where

$$\eta = \sqrt{\frac{\mu}{\varepsilon - j\dfrac{\sigma}{\omega}}} \tag{2}$$

and

$$\eta_0 = \sqrt{\frac{\mu_0}{\varepsilon_0}}. \tag{3}$$

In the above equations, Γ represents the reflection coefficient, and η, σ, μ, and ε are the intrinsic impedance, the conductivity, the permeability, and the permittivity of materials, respectively and η_0, σ_0, μ_0, and ε_0 are those of free space, respectively. ω denotes the angular frequency, and $j = \sqrt{-1}$.

For non magnetic materials, considering $\mu = \mu_0$, and using the above equations, the reflection coefficient can finally be written as

$$\Gamma = X + jY = \frac{1 - \sqrt{\dfrac{\varepsilon}{\varepsilon_0} - j\dfrac{\sigma}{\omega\varepsilon_0}}}{1 + \sqrt{\dfrac{\varepsilon}{\varepsilon_0} - j\dfrac{\sigma}{\omega\varepsilon_0}}}. \tag{4}$$

By solving the simultaneous equations of the real and imaginary parts of (4) and eliminating ε, the conductivity of materials can be expressed as

$$\sigma = \frac{4\omega\varepsilon_0 Y(1 - X^2 - Y^2)}{[(1+X)^2 + Y^2]^2}. \tag{5}$$

For each material, the amplitude, $|\Gamma_m|$, and the phase θ_m, of the measured reflection coefficient, Γ_m, were obtained by microwave measurement as following equation,

$$\Gamma_m = |\Gamma_m| e^{j\theta_m}. \tag{6}$$

Γ_m is influenced by not only the electrical conductivity of metals but also the standoff distance, reflection generated at the aperture part of the probe, the connection parts between the probe and coaxial line, and so on. Therefore, to examine the correct value of σ, we must find the theoretical reflection coefficient Γ_t by calibrating the measured Γ_m.

Fig. 2 shows the signal flow graph of the reflection measurement circuit [11]. By using S_{11} , S_{12} , S_{22} and Γ_t, Γ_m is expressed as

$$\Gamma_m = \frac{b_1}{a_1} = S_{11} + \frac{S_{12}{}^2\Gamma_t}{1 - S_{22}\Gamma_t}, \tag{7}$$

where $a_1$and $b_1$are input and output of microwave signal, S_{11}, S_{12} and S_{22} are errors of the measurement system. By solving the (7), the true reflection coefficient can expressed as

$$\Gamma_t = \frac{\Gamma_m - S_{11}}{S_{12}{}^2 + S_{22}(\Gamma_m - S_{11})}. \tag{8}$$

Therefore, if S_{11}, S_{12} and S_{22} are determined, we can determine Γ_t from Γ_m by (8) and calibrate the microwave measurement system.

In order to examine the correct value of σ, the microwave measurement system must be calibrated. Three samples at which conductivity is known respectively are necessary to determine three unknown constants, S_{11}, S_{12} and S_{22}. By using three groups of the measured reflection coefficient data and the theoretical reflection coefficient data, (Γ_{m1}, Γ_{t1}), (Γ_{m2}, Γ_{t2}), (Γ_{m3}, Γ_{t3}), S_{11}, S_{12} and S_{22} are determined by calculating (7) as follows,

$$S_{22} = \frac{(\Gamma_{t1} - \Gamma_{t2})(\Gamma_{m2} - \Gamma_{m3}) - (\Gamma_{t2} - \Gamma_{t3})(\Gamma_{m1} - \Gamma_{m2})}{\Gamma_{13}(\Gamma_{t1} - \Gamma_{t2})(\Gamma_{m2} - \Gamma_{m3}) - \Gamma_{t1}(\Gamma_{t2} - \Gamma_{t3})(\Gamma_{m1} - \Gamma_{m2})}, \tag{9}$$

$$S_{12}{}^2 = \frac{(\Gamma_{m1} - \Gamma_{m2})(1 - S_{22}\Gamma_{t1})(1 - S_{22}\Gamma_{t2})}{\Gamma_{t1} - \Gamma_{t2}}, \tag{10}$$

$$S_{11} = \Gamma_{m1} - \frac{S_{12}{}^2\Gamma_{t1}}{1 - S_{22}\Gamma_{t1}}. \tag{11}$$

As described above, the conductivity σ_m can be obtained from (5) and (8).

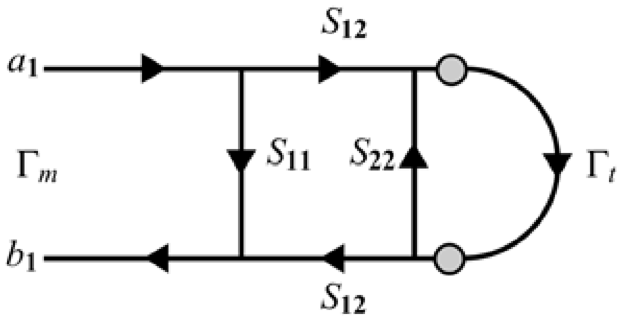

Fig. 2. Signal flow graph of reflection measurement circuit.

IV. EXPERIMENT

A. Microwave Measurement System

Fig. 3 shows the configuration of the microwave measurement system. The microwave measurement system connected to an AFM is shown in Fig. 4. A network analyzer which can generate a microwave signal at the working frequency of 10~67 GHz, was used to feed the microwave signal to AFM and to measure both amplitude and phase of the reflection coefficient. A coaxial line was used to connect the M-AFM probe with the network analyzer. In order to realize such connection, the coaxial line having the diameter of 1 mm was fixed on a probe holder which is used to set up an AFM probe for AFM measurement. The outer and inner conductors of the coaxial line are connected to the bottom and top surfaces of the M-AFM probe, respectively. Therefore, the microwave transmission line changes form the coaxial line to the parallel plate waveguide in the probe. By using this holder, the M-AFM probe can be set up repeatedly. Using the M-AFM probe, both amplitude and phase of the microwave signals were measured after approaching the tip of a M-AFM probe to a sample by the non-contact mode function of an AFM.

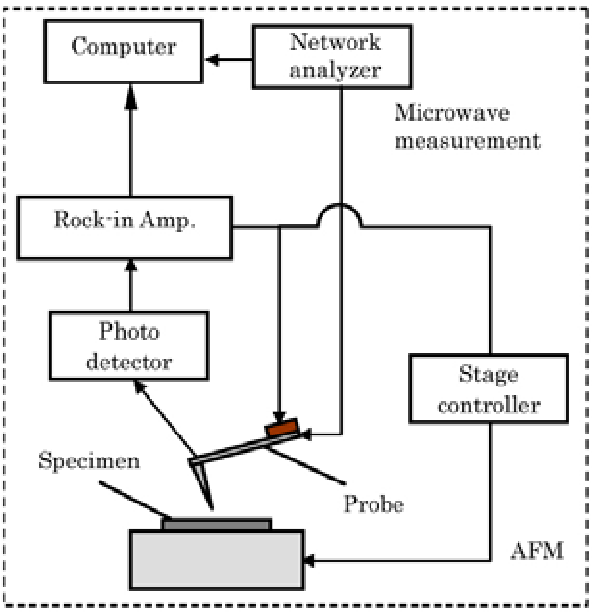

Fig. 3. Configuration of the microwave measurement system.

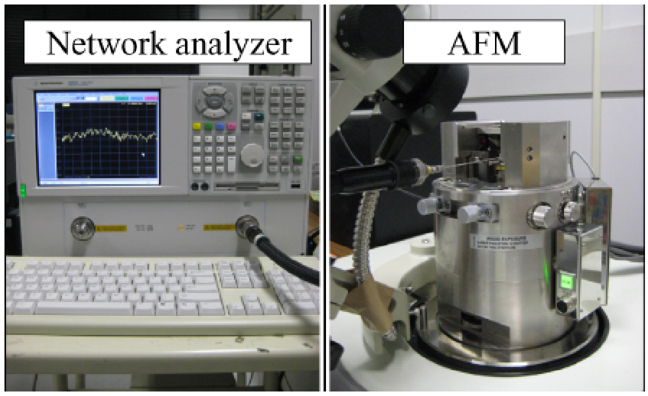

Fig. 4. Photograph of the microwave measurement system.

B. Measured Sample and Conditions

To calibrate the microwave measurement system and to evaluate the conductivity of materials, we prepared a sample which consists of four kinds of metals, cupper (Cu), aluminum (Al), brass (BS) and stainless steel (SST). Fig. 5 shows the SEM photograph of the used sample to measure the each conductivity. The sample consists of four kinds of metals which the height is 2 mm and the width is 0.5 mm, respectively. Table I shows the conductivity σ_t measured by high-frequency conductometry, respectively. Fig. 6 shows the amplitude of the reflection coefficient as a function of sweep frequency in free space. The working frequency was selected from sweep frequency having a low level of the amplitude of the reflection coefficient. This frequency is considered that the microwave signals are emitted from the tip of M-AFM probe much.

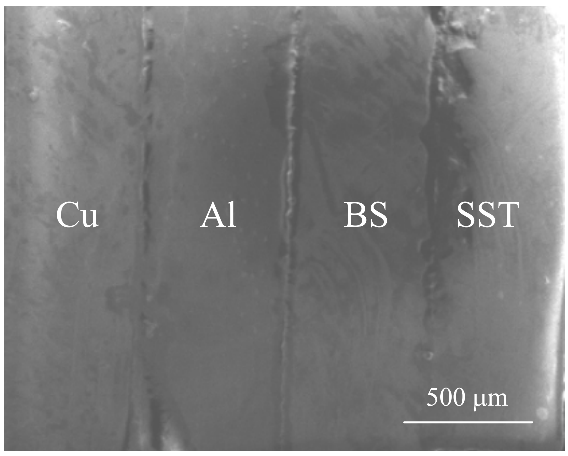

Fig. 5. The SEM photograph of sample which consists of four kinds of metals; the height is 2mm and the width is 0.5 mm, respectively.

TABLE I
THE CONDUCTIVETY MEASURED BY HIGH-FREQUENCY CONDUCTOMETRY.

	Conductivity σ_t (S/m)
Cupper	5.22E+07
Aluminum	3.42E+07
Brass	1.57E+07
Stainless Steel	1.28E+06

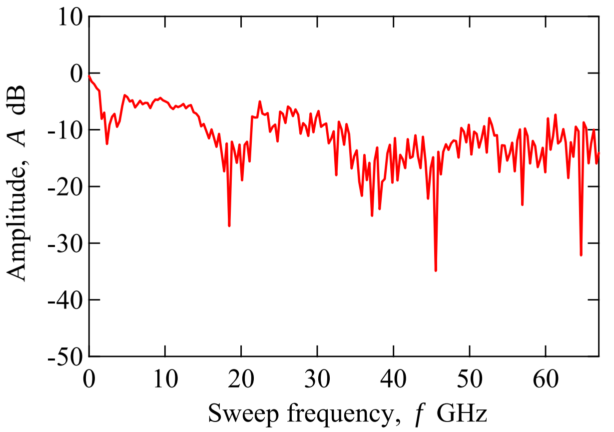

Fig. 6. The amplitude of the reflection coefficient as a function of sweep frequency in free space.

Therefore, for each material, both amplitude and phase of the reflection coefficient Γ_m were measured at working frequencies of 44.5 GHz to obtain the sensitively response of the microwave signals.

V. RESULTS

Fig. 7 and 8 show the results of the amplitude and phase of the measured reflection coefficient, respectively. These results indicate that the microwave measurement system can discriminate the conductivity of the metals. Fig. 9 and 10 show the amplitude or phase of the measured reflection coefficient as a function of the electrical conductivity, respectively. Using those values and (8), the theoretical reflection coefficient Γ_t can be determined. Finally, the conductivity of each material, σ_m, was obtained by using the calibrated the reflection coefficient, Γ_t. Fig. 11 compares the results of the conductivity, σ_m, obtained by the microwave measurement system and the conductivity, σ_t, measured by high-frequency conductometry. The conductivity of samples measured by microwaves agrees with that measured by high-frequency conductometry.

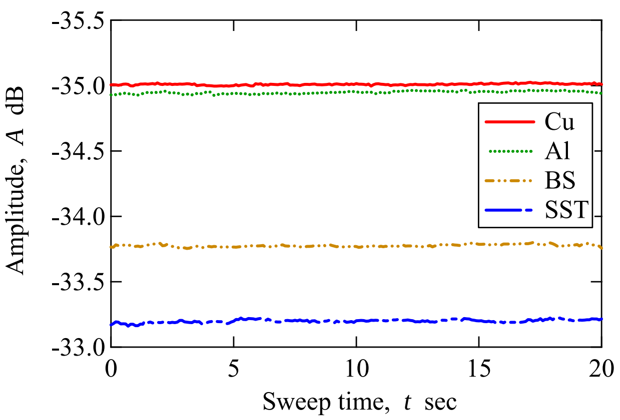

Fig. 7. The amplitude of the measured reflection coefficient.

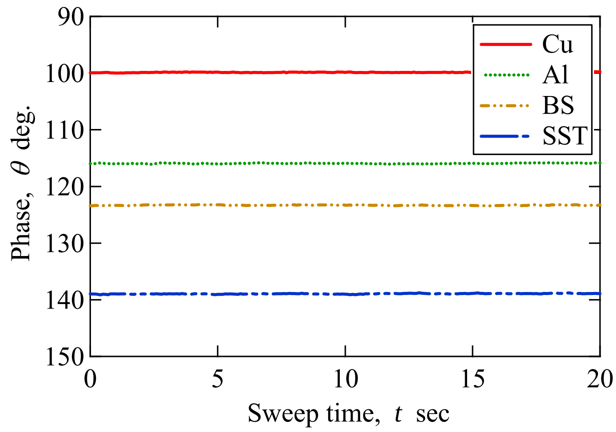

Fig. 8. The amplitude of the measured reflection coefficient.

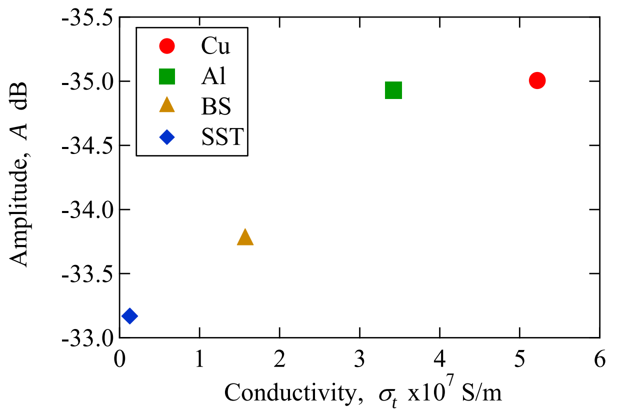

Fig. 9. The amplitude of the measured reflection coefficient as a function of the electrical conductivity.

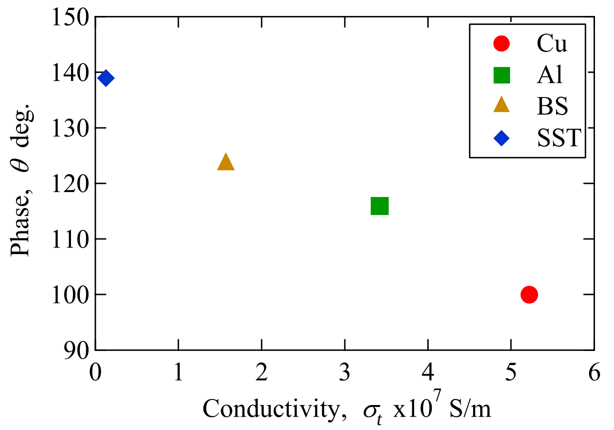

Fig. 10. The phase of the measured reflection coefficient as a function of the electrical conductivity.

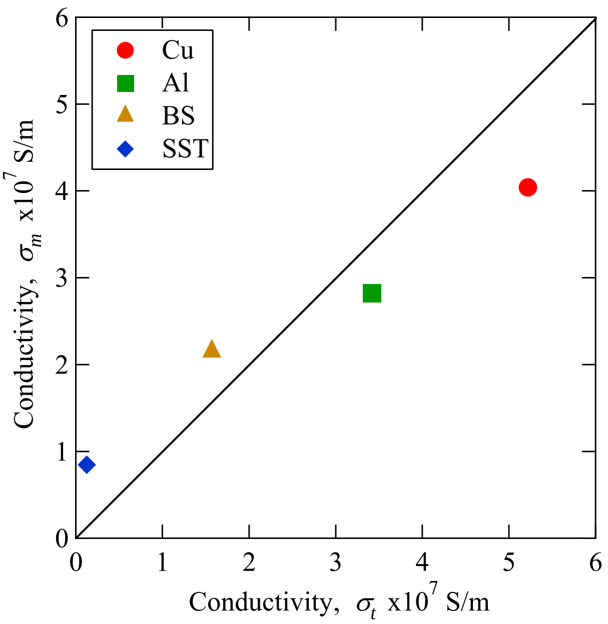

Fig. 11. Comparison of the conductivity, σ_m, obtained by the microwave measurement system and the conductivity, σ_t, measured by high-frequency conductometry.

VI. CONCLUSION

M-AFM probes were fabricated on the GaAs wafer by using photolithography and wet etching process. The method to evaluate the local conductivity quantitatively of metals by microwaves was demonstrated. To obtain correct values of conductivity of material using M-AFM, a method to calibrate the microwave measurement system was introduced. The conductivity obtained by the proposed method agrees with that measured by the high-frequency conductometry. The method has the potential to determine local conductivity of metals in nanoscale.

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