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Original Paper

Primary Measurement of Thermal Diffusivity of Thin Film by Photo Acoustic Tunneling Method

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Measurement system to analyze thermal diffusivity in longitudinal direction has been developed. The tunneling effect and the photo-acoustic effect were applied to the measurement system.

Key words: photo-acoustic, scanning tunnel microscopy, thermal diffusivity, thin film, tunneling effect

1. INTRODUCTION

Thermal physical properties of thin film have been important subject. Thermal diffusivity is one of the important thermal physical properties. Recently high sensitive measurement methods for thin film have been developed, such as the photo-acoustic method. This photo-acoustic effect is caused from absorption of chopped light. The photo-acoustic spectroscopy is to measure sound energy changed from thermal energy. The photo-acoustic effect has been applied to thermal diffusivity measurement since the middle of the 70's. Recently, a photo-acoustic microscope has been studied to observe damage exfoliation of thin films. Moreover, many modified methods are proposed, such as thermal deflection, thermal lens spectroscopy, optical calorimeter and so forth. Our developed system utilizes techniques of Photo-Acoustic effect and Tunneling effect. The tunneling effect, which is one of the famous results from quantum mechanics, has been applied to a scanning tunneling microscope (STM), since the tunneling current is highly sensitive to gap displacement. The current shows an exponential decrease with an increase of a gap between probe and surface of a sample. In STM system, the probe can move with nanometer resolution by using piezoelectricity device (PZT) and can trace atom surfaces without touching the sample. This system has improved the observation of atom ordered surface images.

In this study we have developed a new method to measure thermal diffusivity of thin film in longitudinal direction. The basis of the measurement system is as follows. When the bottom side of thin film was heated by light, the temperature difference between topside and bottom-side causes thermal stress. Since the thermal stress makes the plate bend, distance between the probe and the sample will change. The distance change will cause decrease of the tunneling current. The displacement between the probe and the sample can be estimated from the change of tunneling current. The thermal diffusivity in longitudinal direction will be estimated from the relation between time and bending displacement. This measurement system has the advantage of non contact and high sensitivity. We have measured copper, nickel plates. Each thickness of the plates is 0.1 mm, 0.3 mm and 0.5 mm. In this paper, possibilities of this system, termed photo-acoustic tunneling measurement are discussed.

2 . MEASUREMENT SYSTEM AND EXPERIMENTAL METHOD

2.1. MEASUREMENT SYSTEM

The measurement system, which we have developed, consists of five parts. A schematic figure of the measurement system is shown in fig 1. Details of each part of the system are explained as follows;

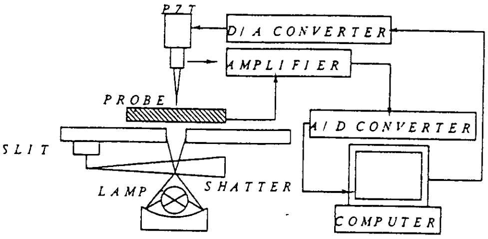

fig 1

(1) Atomic scale fine adjustment device A PZT piezoelectric device performs atomic scale fine adjustment between the probe and the sample. The PZT device was set in the vertical direction in the above of the sample. Since the spatial resolution of our fine adjustment device was 0.18 nanometer, the probe can be controlled within the tunneling current detectable region. The adjustable distance of PZT device was 1 micrometer.

(2) z-Direction micro stage The z-direction micro stage requires following specifications. The probe must be finely adjusted so that it does not touch the sample until it reaches the adjustable range of the PZT device. The z-direction micro stage consists of a movable stage and a micrometer, which performs fine feeding of the stage. The micrometer is dual type, which consists of 10 m/div. coarse adjustment and 0.5 m/div. fine adjustments. Fig 2 indicates a figure of the atomic scale fine adjustment device and the z-direction micro stage.

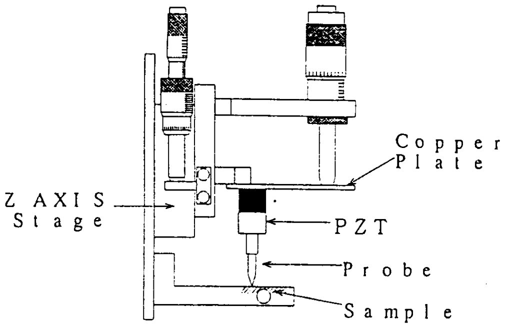

fig 2

(3) Vibration isolation The measurement system requires a vibration isolation in order to measure minute change of the gap. Therefore, the PAS unit was hung on a steel frame with rubber bands. Moreover, our system requires an electric shield, which was made of copper nets, to detect the nanoampere tunneling current.

(4) Probe The tunneling current probe requires nanometer order curvature tip. In the measurement system, 0.3 mm diameter electro-polished tungsten wire is used. In the electrolytic polishing process, 0.6N NaOH electrolyte was used. Load voltage between the probe and the electrode plate was 10 V. As the result, the probe, whose curvature showed 62.5 nm lengths, was served.

(5) Heating source An Infrared lamp was used for the heating source. The radiation was focused by a parabolic reflection mirror. The inner reflection mirror can focus the radiation twice as much as a condenser lens can. However, since scattering light cannot be avoided, an iron slit and an iron shutter were placed between the sample and the lamp.

(6) Electric system 0.03 V bias voltage was supplied between the probe and the specimen with commercial battery. 1nA current was amplified into 0.1V with an OP amp; OPA-128 (Burr-Brown Co.). Since the piezoelectric device performed 1 m deformation with 10 V voltage supply, the device was controlled by 5V output from 12 bit digital-analog (D/A) converter with resolution of 0.18 nanometer/bit. Sampling time was 0.5 ms.

2-2. EXPERIMENTAL

The experiment was carried out in the following series of processes.

1. A probe was cut into 20 (mm) length needle. It was fixed inside of a stainless pipe with little bending. The sample was set on a sample holder.
2. The probe was slowly fed closer to the sample with the coarse adjustment. At this time, the PZT device was made extension into the maximum length. When more than 50 nanoampere tunneling current was detected, the device contracted back to the minimum length. This is the way to adjust the appropriate gap between the probe and the sample with the coarse adjustment.
3. After the coarse adjustment, the gap was precisely tuned by the PZT device again. Until the tunneling current exceeds 50 nanoampere, the piezoelectric device was designed into extension. With the PZT device, angstrom order minute adjusting has become possible.
4. During the above two steps (2 and 3), it is important to keep the probe tip non-contacted from the sample. It is the essential factor to maintain the probe stable for repeatable measurement in a long period.
5. Light was radiated with a water cooled parabolic reflection mirror. The intensity of the light was controlled into constant with an appropriate slit. The tunneling current measurement was started after opening the shutter.
6. The tunneling current was recorded with every 0.5 ms. The result of the current was transformed into the displacement change between the probe and the sample. At the following paragraph, the mechanisms of the displacement will be discussed. Also, the reliabilities, potentials, problems and their counterplans of the system will be mentioned.

3. RESULT AND DISCUSSION

3-1. TUNNELING CURRENT CHANGE

In our system, the gap between the probe and the sample can be estimated from minute changes of the tunneling current. The current change was shown in fig 3.

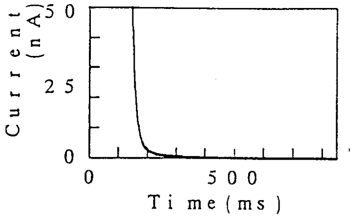

fig 3

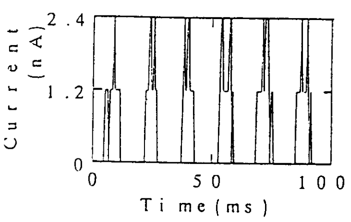

fig 4

The decrease of the tunneling current suggests the distance decrease between the probe and the sample. The tunneling current was sensitive enough to observe the finite displacement. Although the probe was sufficiently separated from the sample, minute current noise was observed. The current noise is shown in fig 4. Since this noise exhibits 60 Hz frequency, it is expected to come from an electric source.

3-2. DISCUSSION

We discussed the result of the tunneling current change in 0.5 mm of copper foil. The change of the tunneling current can be transformed into the change of displacement with the following formula;

,where V denotes bias voltage between the probe and the sample, D denotes electron density of state that is confirmed by the voltage, A denotes the constant; 1/mh, denotes work function, and d denotes distance between the probe and the sample. In the equation, a relation between the displacement and the tunneling current was expressed. Transformed displacement versus time is shown in fig 5.

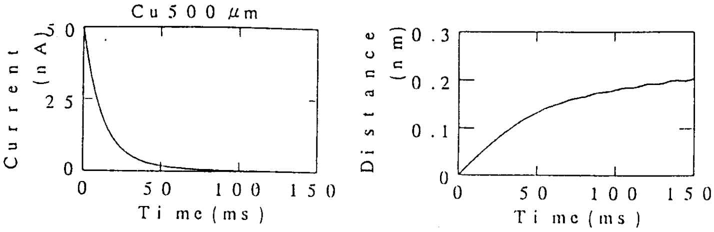

fig 5

At first, we will discuss the reasons for the displacement. When we assume that thermal expansion of the sample is the principal reason for the displacement, it will be much less than that obtained from tunneling current results. We measure the temperature of 0.5 mm thick copper foil by Chromel-Alumel thermocouple. Conditions of the experiment were as follows. Sampling time was 10 sec. Light was radiated from the bottom-side of the sample. The thermocouple was set on the sample with a digital-voltage meter. According to analytical calculation from the temperature increasing data, 0.5 mm thick sample was expected to extend 4.1 pm during 50 ms. When the calculated result was compared with the displacement from the tunneling current, it clearly suggested that the displacement of the experimental measurement be larger than that of analytical calculation. Therefore, in principal the sample displacement was caused by the other mechanisms such as the bending owing to thermal stress caused by temperature gradient.

Secondary, we discuss the displacement caused by the thermal stress. A distance change between the probe and the sample, which is due to the thermal stress, is described in a following model. By Temperature Profile method, a temperature increasing rate of the plate can be expressed. The temperature increasing rate of plate is approximated by a parabolic function within the thermal diffusion thickness in the plate, and in the other part temperature remains constant. We estimate time depended curvature change at the center of the plate. We put the initial at zero degree. Fig 6 shows the model to describe the displacement between the plate and the probe.

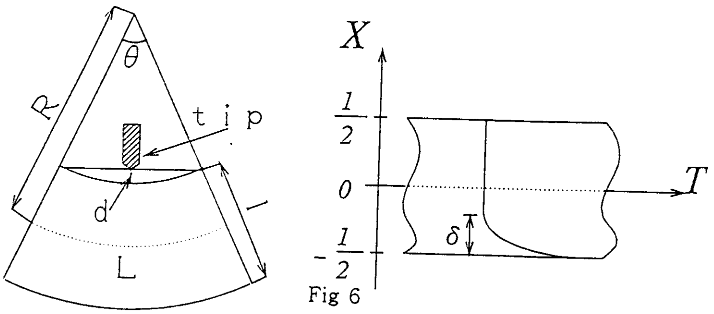

Fig 6

In the model, during the process, elastic strain energy; U during the process is expressed as follows;

$$U=\int_{-\frac{l}{2}}^{-\frac{l}{2}+\delta}\frac{E}{2}\left(\frac{x}{R}+\alpha T\right)^2dx+\int_{-\frac{l}{2}+\delta}^{\frac{l}{2}}\frac{E}{2}\left(\frac{x}{R}\right)^2dx$$

where R denotes the curvature of the plate, δ denotes thermal diffusion thickness, α denotes thermal expansion coefficient and l denotes thickness of the plate.

We differentiated elastic strain energy with respect to the curvature; R,

$$R=\frac{l^3}{2\alpha T_s\delta(2l-\delta)}$$

In fig 6, distance between the probe and the sample is expressed as following formula,

$$d=\frac{3L^2a\dot{Q}}{4l^3\ \rho c}t(2l-\sqrt{6Dt}\ )$$

where d denotes the gap between the probe and the plate, L denotes length of the plate, Q denotes inflow thermal quantity, denotes density, C denotes specific heat, D denotes thermal diffusivity, and t denotes time.

This formula indicates relation between distance and time. Comparing the experimental result with the analytic one, the estimated time, when the maximum distance change is exhibited, is one hundred times quicker than that of experiment. Therefore, the deformation of the thermal stress does not agree well with the experimental results in terms of the time at the maximum displacement. We have been continually considering the primary cause for the displacement phenomena in our system.

4. CONCLUSION

We have developed the measurement system to estimate thermal diffusivity of thin films with photo acoustic tunneling method. As the result of our study, the minute change of tunneling current was observed, and the displacement was detected with a resolution of 0.25 nm. This measurement system has the possibility to analyze thermal diffusivity of thin films after resolving some problems such as a fine adjustment, a light source and so on.

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