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Research paper

Influence of ion beam scattering on the electrical resistivity of platinum hot films

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ABSTRACT

Platinum hot films have been used as precise resistance thermometers to measure the thermal conductivities of carbon nanotubes and graphene. Assisted by focused ion beam (FIB) irradiation, the influence of defects on phonon transport have been examined. However, wide lateral ion beam scattering may affect the electrical properties of hot films and cause uncertainty. In this letter, the effect of FIB irradiation on the electrical resistivity of platinum hot films was evaluated. To investigate this effect qualitatively, electrical resistivity measurement and FIB irradiation were alternated while changing irradiation positions and doses. Irradiated ions were found to travel further than 25 μm away from the directly irradiated area, resulting in an increase of electrical resistivity of the film according to total accumulated dose. The number of scattered ions was found to depend on the irradiated surface. An empirical equation describing the relationship between electrical resistivity and assumed ion density in the hot films was proposed. The obtained results enable us to accurately estimate the thermal or electrical properties of nanomaterials using hot-film sensors combined with nanofabrication techniques using FIB.

1. Introduction

Platinum (Pt) thin films are widely used to measure the thermal properties of nanomaterials [1–6]. It is well known that calibration of thin films plays an important role in these measurements because the values of their electrical and thermal properties strongly depend on their deposition conditions, geometric size, and defects. In addition, the properties of thin films are quite different from those of the corresponding bulk materials [7,8]. These deviations are attributed to the short electron mean free path or phonon mean free path in the thin films caused by surface scattering [9,10], and grain boundary scattering [8,11]. Recently, Hayashi et al. [12] used a Pt hot-film sensor combined with focused ion beam (FIB) irradiation on carbon nanotubes (CNTs) to unveil ballistic phonon transport. They artificially provoked phonon scattering at the region of the CNT irradiated with the FIB, and examined the dependence of thermal conductivity on the length of the pristine part of the CNT. Wang and co-workers applied this nanomachining technique for the thermal measurement of graphene on a gold hot film [13]. They irradiated graphene with a FIB to make nanoholes and tune its thermal conductivity. These two experimental reports demonstrate that the combination of hot-film sensors with FIB irradiation is a strong tool to experimentally investigate phonon transport in CNTs and graphene in situ. However, the effect of FIB irradiation is known to extend far from the directly irradiated area [14–16]. When a FIB is irradiated near a hot-film sensor, it may change the properties of the hot film. Consequently, it is necessary to understand how the electrical properties of hot films are influenced by FIB irradiation to obtain accurate experimental data.

There are two main reasons why a FIB exerts an influence far from its directly irradiated area [15,16]. One is the Gaussian distribution of a FIB. Liao et al. [15] observed the areas of graphene damaged by a FIB by Raman spectroscopy, and reported that the considerable lateral damage can be attributed to the Gaussian beam shape and unfocused gallium (Ga) ions in the FIB. The other reason is residual gas scattering in the vacuum chamber. Thissen and colleagues irradiated a FIB on graphene with residual gas at different background pressures and observed the damaged areas by Raman spectroscopy [16]. They found that the size of the damaged area decreased as pressure was lowered. However, although possible reasons for ion beam scattering have been specified, quantitative evaluation of the effect of a scattered ion beam on hot films has not been performed. In this work, we measured the electrical resistivity of Pt hot films exposed to a scattered Ga ion beam, and proposed an empirical equation that describes the relationship between ion density in the hot film and resistivity.

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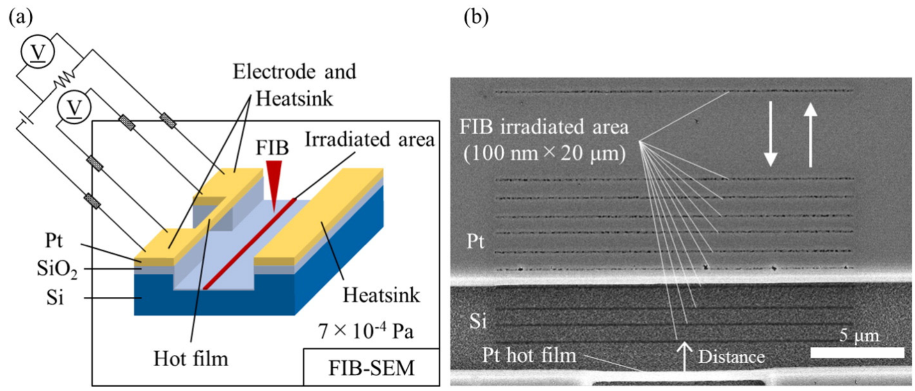

Fig. 1. (a) Schematic of experimental apparatus and FIB irradiation. (b) SEM image of a Pt hot-film sensor after FIB irradiation. The traces of FIB irradiation are observed parallel with the Pt hot film.

2. Experimental

Measurements were performed in the chamber of a Versa 3D (FEI, USA) equipped with a scanning electron microscope (SEM), FIB, and temperature-controlled stage, as shown in Fig. 1(a). A silicon (Si) substrate with a Pt hot-film sensor was set on the stage. This sensor was fabricated by the MEMS technique, and consisted of two Pt electrodes and heat sinks with a Pt hot film suspended between them, and another Pt heat sink located 5 μm away from the hot film. The dimensions of the six Pt hot films used in this study are summarized in Table 1. By repeating electrical resistivity measurements of the hot films using the four-probe method just after FIB irradiation scanning parallel to the hot film, we evaluated the effect of FIB irradiation on the electrical resistivity of the hot films. A Ga ion beam was raster scanned across an area of 100 nm × 20 μm at an accelerating voltage of 30 kV. Table 2 lists the three kinds of FIB conditions used in our experiments. Positions of FIB irradiated area were shifted with respect to the hot film, as illustrated in Fig. 1(b). Temperature and pressure were 300 K and less than 7×10^{-4} Pa, respectively.

3. Results and discussion

First, the FIB was irradiated at a position 500 μm away from the hot films (H1, H2 and H3). The position of the FIB irradiated area was gradually shifted closer to the hot films up to the shortest distance of 1 μm. Fig. 2 shows the measured electrical resistivity of the hot films. The gradual increase of electrical resistivity of these three hot films indicates that scattered ions reached a distance of more than 25 μm away from the directly irradiated area. The increase of electrical resistivity of the hot films, $\Delta\rho$ [Ω m], is defined as follows.

$$\Delta\rho = \rho_i - \rho_{i-1}, \tag{1}$$

where ρ_i [Ω m] is the electrical resistivity after ith irradiation, and ρ_0 [Ω m] is the intrinsic electrical resistivity of the film. As indicated in the inset of Fig. 2, a larger dose at a closer position results in a larger $\Delta\rho$. The $\Delta\rho$ curves show gaps at 5 μm because the irradiated material changed from Pt to oxidized Si. Pt atoms are bigger than Si atoms, so they have a bigger scattering cross section with the Ga ions in the FIB, resulting in larger $\Delta\rho$.

In the experiment using H4, the position of FIB irradiated area was shifted in the opposite direction of that using H1, H2 and H3, starting at a distance of 1 μm from the hot film. This is why the results of H4 show different tendency from the others in Fig. 2. Comparing H4 and the others at the shortest distance, $\Delta\rho$ of H4 is larger than that of H1 and even H2 and H3. At other distances, however, $\Delta\rho$ of H4 is smaller and close to zero at a shorter distance than the others. These results indicate that more Ga ions accumulated by previous irradiations result in smaller $\Delta\rho$ when the same doses are irradiated.

To investigate the relationship between electrical resistivity and Ga ion density in the hot films, each hot film was exposed to three different FIB irradiation doses at an identical position 1 μm away. The measured resistivity is shown in Fig. 3(a). Irradiated dose was transformed into Ga ion density in the hot films using reported results [16], which related scattered ion dose with distance from the directly irradiated area. The ion dose 1 μm away from the directly irradiated area was 1.5×10^{13} ions/cm^2, which was transformed into a Ga ion density in our hot film of 3.8×10^{24} ions/m^3. Assuming that the Ga ion density is proportional to the original ion dose of the FIB irradiation, the relationship between electrical resistivity and estimated density is presented in Fig. 3(b). The dotted curve is the fitting curve expressed as the function of the natural logarithm,

$$\rho = \rho_0 + \mathrm{X} \ln(\mathrm{Y}n + 1), \tag{2}$$

where ρ [Ω m] is the measured electrical resistivity after FIB irradiation, and n [ions/m^3] is the Ga ion density in the hot films. X [Ω m] and Y [m^3/ions] determined by the least-squares method were 5.75×10^{-9} Ω m and 8.53×10^{-25} m^3/ions, respectively. By substituting the total accumulated n into equation (2), calculated resistivity was compared with experimental results, as illustrated in Fig. 4. The electrical resistivity obtained from Eq. (2) is consistent with the experimental results for H4, but not with that for H1. This discrepancy for H1 is attributed to the

Table 1
Dimensions of Pt hot films.

Pt Hot film	Length[a] [μm]	Width[a] [nm]	Thickness[b] [nm]
H1	9.81	418	40
H2	9.75	458	40
H3	9.70	451	40
H4	9.65	549	40
H5	9.45	536	40
H6	9.55	577	40

[a] Length and width were determined by SEM observation.
[b] A film thickness meter was used to deposit an arbitrary thickness of Pt.

Table 2
FIB irradiation conditions.

Condition	Dose [pC/μm^2]	Beam current [pA]	Beam diameter [nm]
A	10.00	1.6	7.1
B	77.04	10.0	13.0
C	229.84	30.0	17.0