In-situ thermoproperties measurement of Pd film deposited onto optical fiber prepared for photothermal reflectance detection

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Abstract. This article proposes an in-situ measurement method of the thermal diffusivity and the thermal conductivity of thin film during deposition inside the vacuum chamber based on a photothermal reflectance technique. Our new technique is simple and reasonable, although no adequate conventional instrument exists. A 2.5-mm-diameter FC-ferrule made from zirconium is contained in a vacuum and used to measure the thermoproperties of the thin film deposited on the end surface of the ferrule. The dependence of the thermal diffusivity and the thermal conductivity of the palladium film on the film thickness are demonstrated. These values are shown to be asymptotically adjacent to those of the bulk since the film is thicker. It is also described that the measurement error is increased, since the optical transmissivity of the thin film becomes to be high when the film is too thin.

1. Introduction

Photothermal measurement is recognized as a useful method for characterizing such thermoproperties as thermal conductivity and thermal diffusivity for controlling the quality of electronic materials. Among the several photothermal evaluation techniques that have been developed and commercialized, the photothermal reflectance [hereinafter modulated optical reflectance (MOR)] detection technique is widely recognized as effective for evaluating the thermoproperties of thin films [1]-[3].

To determine with high accuracy thermal conductivity and thermal diffusivity that depend on film thickness, in-situ measurement during the growth of thin film inside the vacuum chamber is significant to avoid chemical deterioration of the film surface by contamination and oxidization [4]. To the best of our knowledge, no conventional MOR instruments are adequate because they are not compact, adjustment-free, or mobile. Even though in-situ measurement using such instruments has been attempted, however, it was very difficult [4].

To overcome this problem, we recently proposed a “laptop” MOR instrument assembled with optical fiber components [5]. This instrument’s primary feature is that all the optical routes for pumping and probing the MOR as well as the two beam sources using a laser diode are composed of optical fiber components. Consequently, the above problems arising from the conventional MOR instruments’ technical shortcomings are completely solved.

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This article proposes a MOR technique using our instrument that is suited for in-situ thermoproperties measurement. First, we briefly describe the laptop MOR instrument. Then we show a DC diode sputter system specially designed for our MOR technique and emphasize the freedom of arrangement in the vacuum chamber of an MOR detector. Next the experimental data are demonstrated, and finally, our technique’s capability is certified.

2. Instrumentation system

2.1. Laptop MOR instrument

Figure 1 shows a block diagram of our recently developed laptop MOR instrument assembled with optical fiber components that addresses the technical shortcomings of conventional instruments. A laser diode module with a wavelength of 1.47 µm and a fiber output power of 110 mW was used as the light source of the pumping beam, and a super luminescence diode (SLD) module with a wavelength of 0.83 µm and a fiber output power of 1 mW was used for the probe beam. Both beams are unified using an optical fiber coupler (OFC) 1 for wavelength division multiplexing; OFC1 functions like a dichroic mirror. Since an OFC1 is used, the distance between the spot on the specimen’s surface illuminated by the pumping beam and one illuminated by the probe beam is constantly and automatically maintained at zero, realizing adjustment-free for initial setup.

A 1.47 µm was chosen as the pumping beam’s wavelength because 1.47 and 0.98 µm have been widely used as the pumping source’s wavelength for erbium-doped fiber amplifiers (for optical communication systems), so several high-power laser diode modules and their related optical components have been commercialized. Note that only selecting 0.98 µm is sufficient as the pumping beam’s wavelength to generate a plasma wave in the Si wafers. All the components, devices, and electronics shown in Fig. 1 are contained in an instrument case that is 400 mm wide, 250 mm deep, 60 mm high, and weighs approximately 1 kg: a laptop-sized instrument. Furthermore, it is only necessary to prepare a synchronous detection instrument for signal recovery (e.g., lock-in amplifier) with our MOR instrument.

Figure 1. Block diagram of laptop MOR instrument.
2.2. MOR detection head
The detection head illustrated in Fig. 2 is a 2.5-mm-diameter FC-ferrule made from zirconium and is used to measure the thermoproperties of thin film deposited on the ferrule’s end surface. This head is appropriate for \textit{in-situ} measurement of the inside of the vacuum chamber. Even if the film is deposited on the 2.5-mm-diameter surface, the actual net size of the detecting element is just a 6 \( \mu \text{m} \) diameter that corresponds to the core diameter of the fiber used; however, a few hundred \( \mu \text{m} \) must be estimated as the effective diameter to consider the thermal diffusion length.

2.3. Thin film deposition system specially designed for \textit{in-situ} MOR measurement
Figure 3 shows a DC diode sputter system specially designed for our MOR technique. In this system, a 20-mm-diameter palladium (Pd) pellet is used as the sputtering target, and the FC-ferrule (Fig. 2) is mounted on the substrate holder instead of the substrate. The chamber was evacuated by background pressure on the order of \( 10^{-4} \text{ Pa} \) by a turbo molecular pump. The distance between the sputtering target and the FC-ferrule was adjusted to a few ten mm to maintain the discharge stability, and the sputtering power was set to approximately 15 W by controlling the applied DC voltage and the argon gas pressure. The Pd film was deposited on a cross section of a 125-\( \mu \text{m} \)-diameter single mode (SM) optical fiber whose core diameter was 6 \( \mu \text{m} \), enabling \textit{in-situ} MOR measurement by feeding the optical fiber to the instrument shown in Fig. 1. This measuring system has freedom of arrangement in the MOR detector’s vacuum chamber.

3. Measurement results
Figure 4 shows the modulation frequency dependence of the detected amplitude MOR signal as a function of Pd film thickness. The film thickness was controlled by sputtering time at the constant incident power, and these measurements were executed using the sputter shown in Fig. 3 during deposition in a vacuum without ever breaking a vacuum; this is evidently \textit{in-situ} measurement. The solid curve and dotted curves drawn in Fig. 4 show the theoretical curves that are calculated by optimizing several parameters of the theoretical formula using the least squares method against the experimental results. Here, to establish the thermal wave interference between thin layers, a theoretical formula introduced by Almond et al. was used [6]. See ref. 5 for more details.

As seen in Fig. 4, the experimental data align almost perfectly with the theoretical curves when the film is thicker than 600 nm; however, deviation from the theoretical curve remarkably increases when the thickness is thinner for the following reason. First, the above theoretical formula can be applied effectively when each layer is assumed to be optically opaque. However, the optical transmissivity of the thin film becomes dramatically high as the film becomes thinner, especially for less than 0.5 \( \mu \text{m} \). Consequently, the numerical deviation between the experimental data and the theoretical curve is induced apodictically. On the other hand, this produces error in the estimated thermal conductivity and diffusivity.
Figure 5 shows the dependence of the estimated thermal conductivity and the estimated thermal diffusivity on the deposited Pd film thickness. These thermal parameters of thin film are asymptotically adjacent to those of the bulk (cited value from a data book [7] of thermoproperties published in Japan) since the film was thicker. On the other hand, the error in our measurement is enhanced in accumulation due to the above reasons since the thickness is thinner. We believe that the advantage of our technique can be demonstrated when the film thickness is over a several hundred nm.

4. Summary
This article proposed a new MOR technique suitable for *in-situ* and online thermoproperties measurement. This technique’s key point is attributed to an adoption of an adjustment-free laptop type MOR instrument and FC-ferrule as the MOR detector, allowing a facility for built-in inside the vacuum equipment. The estimated thermal conductivity and diffusivity of the Pd film dependence on film thickness were presented, and we conclude that measurement error is enhanced since the thickness is decreased due to the optical permeability of the thin film. Our technique provides the possibility of not only an optical thermoproperties measurement system for *in-situ* or online measurement and inspection for the semiconductor industry but also valuable data for resolving the physical properties of solid-state materials.

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