Nuclear Instruments and Methods in Physics Research B xxx (2014) xxx–xxx

Contents lists available at ScienceDirect

Nuclear Instruments and Methods in Physics Research B

journal homepage: www.elsevier.com/locate/nimb

In situ characterization of thermal conductivities of irradiated solids by using ion beam heating and infrared imaging

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ARTICLE INFO

Article history:
Available online xxxx

Keywords:
Ion irradiation
Thermal conductivity
Infrared

ABSTRACT

We propose a method to characterize thermal properties of ion irradiated materials. This method uses an ion beam as a heating source to create a hot spot on sample surface. Infrared imaging is used as a surface temperature mapping tool to record hot zone spreading. Since ion energy, ion flux, and ion penetration depth can be precisely controlled, the beam heating data is highly reliable and repeatable. Using a high speed infrared camera to capture lateral spreading of the hot zone, thermal diffusivity can be readily extracted. The proposed method has advantages in studying radiation induced thermal property changes, for which radiation damage can be introduced by using an irradiating beam over a relatively large beam spot and beam heating can be introduced by using a focused testing beam over a relatively small beam spot. These two beams can be switched without breaking vacuum. Thus thermal conductivity changes can be characterized in situ with ion irradiation. The feasibility of the technique is demonstrated on a single crystal quartz substrate.

1. Introduction

The need to characterize thermal properties of thin films has driven the continuous development of various testing techniques. Among them, the laser flash method is one of the most common measurement types. This technique utilizes a high-intensity light source and is pulsed to illuminate the front surface. The temperature on the backside of the sample is measured to extract thermal diffusivities [1]. However, measuring specific heat is greatly challenging to the laser flash technique due to need to have standard samples and knowledge of the amount of absorbed energy. In another state-of-the-art technique, a laser beam is used to illuminate a sample surface after focusing, while the emitted thermal images from the sample are collected by an infrared (IR) camera. Since the IR camera can reach a lateral resolution of sub 100 µm, a high resolution temperate mapping can be achieved. Although previous work has shown that the lateral temperature distribution from a laser-heated central spot can be easily obtained from IR imaging, the laser-based technique has one disadvantage in that the exact power deposition is difficult to estimate since laser absorption efficiency of materials varies greatly [2].

We propose in the present study a new method to characterize thermal properties of thin films. In principle, a focused ion beam bombards a solid surface in vacuum and an IR camera records the temperature evolution when the beam is on and off. The camera is positioned near an IR transmissive window and is outside of the vacuum chamber. Through nuclear stopping and electronic stopping, the energy of the ion beam is completely absorbed by the film so the deposited power is precisely controlled. Furthermore, the longitudinal and lateral distribution of the deposited power, determined by projected range of ions and the beam spot size, are well known. This technique yields several advantages in that the heat flux can be well controlled in modeling to extract thermal diffusivities and thermal properties of the near surface region.

2. Experimental procedure

A single crystal SiO_2 (quartz) substrate of 0.5 mm thickness is used in the present study. The localized substrate heating comes from a 2 MeV helium ion beam, with beam current of 100 nA and beam diameter of 0.8 mm. An FLIR E60 IR camera with spatial resolution of 150 µm and recording speed of 30 frames per second is used. Since the IR camera cannot be located within the vacuum chamber, a vacuum-tight ZnSe window of 8–12 µm thickness is used. ZnSe is selected since it has excellent IR transmission

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http://dx.doi.org/10.1016/j.nimb.2014.02.101
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efficiency over a relatively large wavelength region. From 0.6 μm to 20 μm, the transmission efficiency is about 60–70%. Note that the wavelength of 9 μm corresponds to the radiation peak from a sample at 50 °C. For 2 MeV He ions in SiO_2, the depth of He peak predicted by SRIM code is 5.84 μm [3]. The temperature reading from IR and thermocouples are compared in a separate experiment in which the substrate is homogenously heated by an electric heater from the sample backside. The temperature of the front side is measured using thermocouples and IR camera, and the difference was found to be very small. In the temperature region of interesting to the present study, from room temperature to about 45 °C, the difference is less than 4%. Furthermore, a linear calibration relationship exists up to the highest testing temperature of 120 °C.

Ideally, thermal diffusivity can be extracted by assuming an adiabatic case in which thermal diffusivity, α, in one-dimensional diffusion, can be calculated by [1]:

$$\alpha = 0.1388\frac{d^2}{t_{1/2}} \tag{1}$$

where α is the thermal diffusivity, d is the distance of reference point from the heating source, and $t_{1/2}$ is the time required for the reference point to reach half of the maximum temperature rise.

However, for the present study with heating introduced by ion beam, discharging/sparking occurs due to charge deposition in an insulating substrate. The discharging creates short pulse-like artifact for IR imaging and leads to temperature fluctuations. We therefore further propose here to extract thermal diffusivities by using the temperature evolution data in the stage after the beam is removed. Since there are no heating sources at $t > 10$ s, the generalized heat equation is given by

$$\frac{dT}{dt} = \alpha\nabla^2 T \tag{2}$$

3. Results and discussions

Fig. 1 shows the temperature mapping obtained from SiO_2 sample as a function of time. The beam is turned on at time $t = 0$ s and is kept on until $t = 10$ s. At time $t = 0.5$ s, 3 s, and 10 s, heat is spreading from the ion beam spot. At $t = 10.5$ s, corresponding to 0.5 s after the beam is turned off, the heated region quickly cools down and becomes barely noticeable at time $t = 12$ s. The absolute temperatures are provided by the IR camera.

Fig. 2 illustrates the radial temperature distributions extracted from the IR imaging. With increasing time, the profile spreading becomes wider, and the temperature of the central spot increases. Temperature evolution is well described by a Gaussian spreading. There is no steady state developed during the beam heating since the temperature of beam spot center continuously rises. This suggests that heat loss rate from boundaries through black body radiation is weak. Fig. 3 plots the extracted temperature profiles as a function of time after the beam is turned off. Since the heating source is removed at time $t = 10$ s, the temperature of beam spot center drops with further profile spreading.

Fig. 4 plots $\nabla^2 T$ as a function of its position. This second derivative is numerically obtained from the experimental temperature profile at $t = 10$ s when the beam is turned off. The curve has a negative peak in the center of beam spot and two slightly positive shoulders at the spot edges. These curve features are expected if the temperature profile follows a Gaussian-like distribution. The solid line in Fig. 4 is used to show the prediction from a perfect Gaussian profile. Fig. 5 plots the time derivative of temperatures, dT/dt, at the beam spot center. From 0 s to 10 s, there is one sharp positive pulse at the beginning. At the time beyond 10 s, there is a negative pulse associated with rapid cooling. Combining data from Figs. 4 and 5 (dT/dt and d^2T/dr^2 for the position located at the beam spot center and for the time $t = 10$ s), the thermal diffusivity is extracted. We obtain $1.7 \times 10^{-6}\ m^2/s$, which is reasonably close to the literature reported value ($3.4 \times 10^{-6}\ m^2/s$) [1].

The proposed method has great advantages in thermal property characterization of irradiated samples. An irradiating beam of large spot size can be used to introduce damage, while a heating beam of small spot size can be used to characterize thermal properties. These two beams can use the same ion species and can easily be changed without breaking vacuum. Thus the thermal property changes as a function of radiation damage levels can be systematically studied.

The feasibility of the technique is demonstrated in Fig. 6, which compares the temperature profiles at time $t = 10$ s for SiO_2 before and after 2 MeV He ion irradiation to a fluence of $5 \times 10^{16}/cm^2$. The beam spot for ion irradiation is intentionally enlarged to

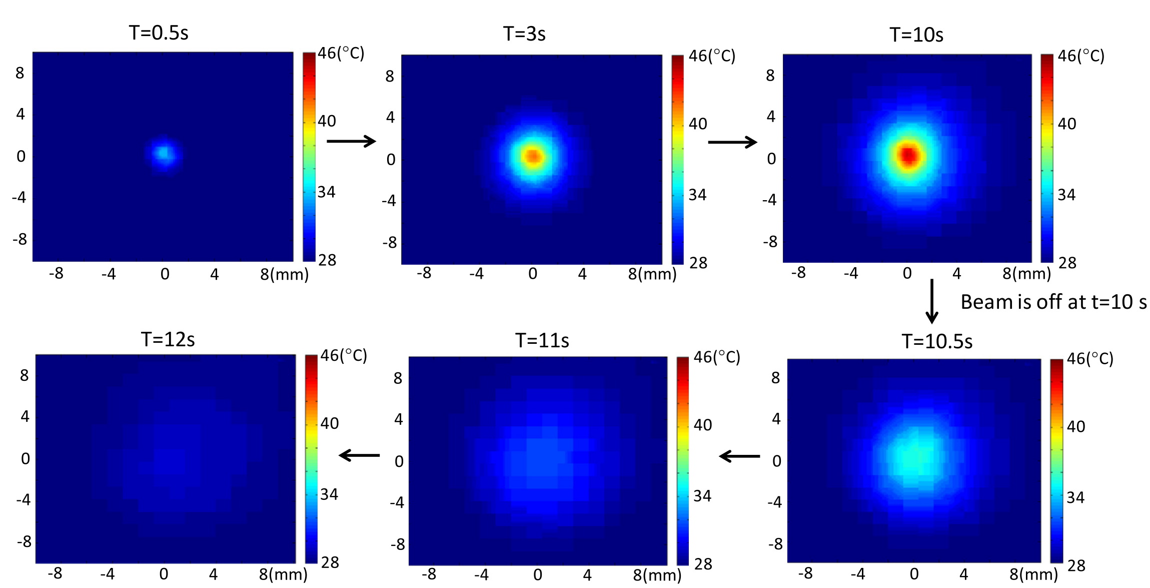

Fig. 1. Two dimensional IR temperature mapping of SiO_2 at different times. 2 MeV He heating beam is turned on at $t = 0$ s and off at $t = 10$ s.

Please cite this article in press as: N. Mondrik et al., In situ characterization of thermal conductivities of irradiated solids by using ion beam heating and infrared imaging, Nucl. Instr. Meth. B (2014), http://dx.doi.org/10.1016/j.nimb.2014.02.101