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Ultramicroscopy 107 (2007) 663–668
ultramicroscopy
www.elsevier.com/locate/ultramic

Measurement and estimation of temperature rise in TEM sample during ion milling

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Received 27 June 2006; received in revised form 27 December 2006; accepted 9 January 2007

Abstract

The actual temperature rise was measured during ion-milling process used in the transmission electron microscopy (TEM) sample preparation. Special probes were fabricated for the measurements, one with shielded, floating thermocouple mounted onto a 3 mm grid to compute the thermal load at the sample, and the other, a bare probe with a polymer coating to measure the maximum temperature attained. The temperature measured in the most commonly used ion-milling system reached up to 295 °C when the typical milling conditions, 6 keV ion-energy and an incident angle of 80°, were used. Based on the temperature profiles that were obtained by the shielded probe, two unknown parameters, the amount of heat deposited by the energetic ions/neutrals to the sample and the thermal conductivities between the materials, were estimated and used to compute the temperature rise in commonly adopted materials. The calculation showed that the temperature of the glass sample reached more than 300 °C under typical ion-milling conditions. The calculated value was confirmed with the experimental result of the crystallization of an amorphous Si on the glass under the typical ion-milling condition, which gave the same extent as annealing at 350 °C.

PACS: 61.16.Bg; 61.80.Jh; 81.05.Kf

Keywords: TEM sample preparation; Ion milling; Temperature

1. Introduction

Ion milling is the most commonly used technique for making samples transparent under the electron beam of transmission electron microscopy (TEM). In early models of ion-milling system, a single discharge chamber was used, where the cathode served as both the discharge and extraction electrodes. The thermal load deposited to the sample by the ion beam was not a serious factor to consider in the older models of ion-milling system, because the ion sources were not efficient in extracting ions and did not provide focused beams with high current densities. As improvements were made in the ion-milling system in order to increase the sputtering rate from the sample surface and to make the system more compact, the ion source of the ion-milling systems got furnished with a three-electrode system, which can produce as high as 10 keV ions. In addition, sometimes a magnetic field was applied to increase the plasma density and obtain a small ion-beam diameter in order to provide higher milling rate. As a result, the bombardment of high number density of ions and energetic neutrals onto a small sample stage inevitably introduces localized heating of the thin section of the sample. When the ion beam is focused to produce a diameter of approximately 1 mm with typical ion energy of 4–6 keV, the local temperature rise in the foils cannot reasonably be ignored. Even though the ion milling performed for TEM sample preparation is usually done at shallow incidence angles, a significant amount of energy is deposited in the sample. For example, TRIM simulations [1–4] showed that 5 keV Ar^+ ions incident at a glancing angle of 10° on carbon, copper and tungsten samples deposited 74%, 56%, and 51% of their energy as heat

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0304-3991/$ - see front matter
doi:10.1016/j.ultramic.2007.01.002

(at 5°, the corresponding values are 57%, 44%, and 45%), respectively. The temperature of the thin foil rapidly increases to levels that could change its microstructure, considering that the typical thickness of the samples used in TEM observation is in the range of 100–300 nm. The accurate estimation of the temperature is critical in order to exclude any artifact induced by the heating of the sample, since many challenging materials require growth or processing at low temperature and thin, strained layers can easily react with neighboring layers as the temperature rises.

Sample heating by the ion milling has been a well-known phenomenon and many reported about related issues, loss of grain boundary segregation [5], micro-structural change [6,7], estimation of the temperature using low-melting-point metals [8], and measurement using temperature probe [9–11]. The operational manuals of ion-milling system provide only a rough estimate of the thermal power, which can be as much as 300 mW at a high milling rate [12]. Even with wide spectrum of results, a versatile method of predicting the temperature rise from various materials has not been provided.

Ion sources inevitably produce energetic neutrals together with major population of ions, which make it difficult to deduce the extracted total energetic particles—ions plus energetic neutrals. If energy transferred to the sample is estimated only by the ion current reading, it always gives an underestimation of the amount of energy deposited. The best way of depicting the temperature rise is to measure the actual temperature using probes. The purpose of this paper is to provide the heating rate of the commonly used materials during the ion-milling process by quantifying the amount of heat deposited and thermal conductivities, which can be acquired from experimental measurement.

2. Experiments

In order to minimize the influence of ions in the ion beams during the temperature measurements, it is necessary to protect the surface of the thermocouples by isolating them from the external potential source, i.e. the ion beam. Two types of isolated temperature probes were fabricated to measure the temperature; one was a shielded K-type thermocouple and the other was a bare K-type thermocouple coated with M-bond 610 glue [13].

The shielded thermocouple consisted of a thin outer tube made of 304 stainless steel with K-type thermocouple inside, which was electrically floated. The outer diameter of the shielded thermocouple was 250 μm, and MgO powder was used to isolate them from the outer shield. The tip of the shielded probe was flattened to reduce the thickness, and then located at the center of a titanium disc using M-bond 610 glue mixed with carbon powder to fill the gap. A titanium disc had a slot cut at one end allowing for the probe to be squeezed in and fixed with additional glue. The thickness of the mounted probe was further reduced through the dimpling process. The final shape of the probe is shown in Fig. 1(a). The estimated thickness in the center was about 200 μm. Even though the thermocouple tip was separated from the outside sheath by the MgO filler material, it was found that its response to changes in temperature took less than a second when it was tested with ion beam on–off time intervals.

The other probe, called the bare probe, had a set of bare, 25-μm-diameter *K*-type thermocouple, which were mounted onto a 2×1 mm slit copper grid fixed using M-bond 610 glue, as shown in Fig. 1(b). It was found that the coating thickness was not uniform varying from several tens to several hundreds of μm, judging from the milling rate of the polymer. One of the characteristics of the bare probe was that, as the glue coating disappeared, the bare thermocouple was exposed to the ion beam and, consequently, the temperature reading became erratic. The bare probe responded to the ion beam faster than the shielded one, having a response time of much shorter than 1 s, but it had

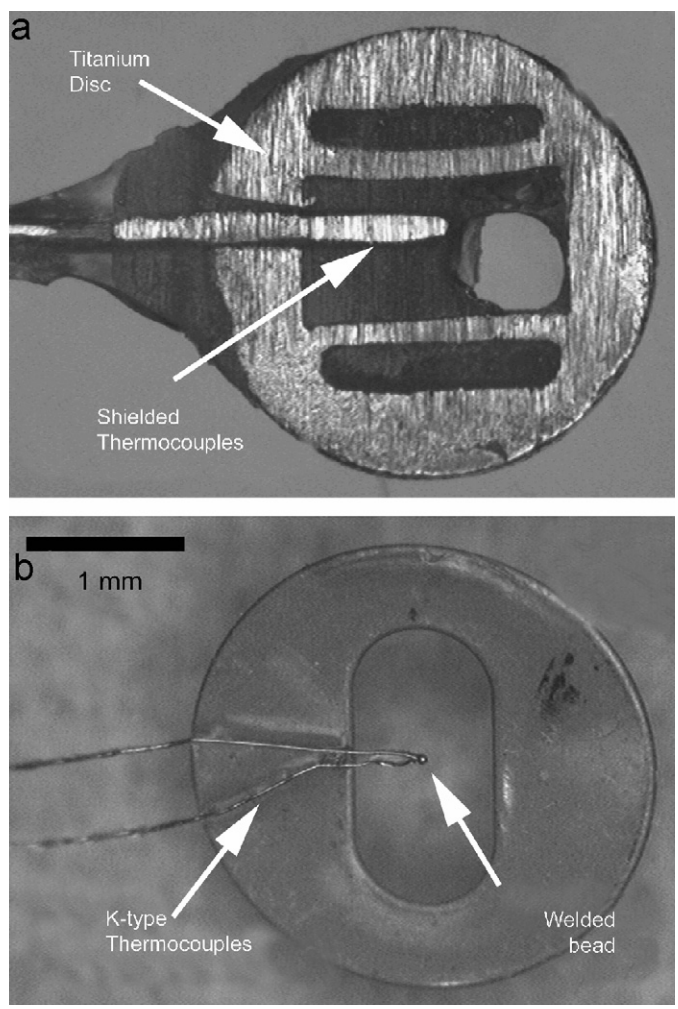

Fig. 1. Two types of probe used to measure the temperatures during the ion beam milling. (a) Shielded K-type thermocouple mounted on a titanium disc. The thickness at the center was about 200 μm. (b) Bare K-type thermocouple coated with M-bond 610 glue mounted onto a copper grid. The diameter of the welded bead at the joint was about 400 μm.