Ultramicroscopy ∎ (∎∎∎∎) ∎∎∎–∎∎∎

Contents lists available at ScienceDirect

Ultramicroscopy

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

Methodology exploration of specimen preparation for atom probe tomography from nanowires

Q1 Jiangtao Qu [a], Derek Wong [b], Sichao Du [a], Limei Yang [b], Simon Ringer [b,c], Rongkun Zheng [a,*,1]

[a] School of Physics, The University of Sydney, New South Wales 2006, Australia
[b] Australian Centre for Microscopy & Microanalysis. The University of Sydney, New South Wales 2006, Australia
[c] School of Aerospace, Mechanical and Mechatronic Engineering. The University of Sydney, New South Wales 2006, Australia

ARTICLE INFO

Article history:
Received 2 September 2014
Received in revised form
25 May 2015
Accepted 2 June 2015

Keywords:
Specimen preparation
Nanowires
Atom Probe Tomography
Focus Ion Beam

ABSTRACT

Semiconductor nanowires have been intensively explored for applications in electronics, photonics, energy conversion and storage. A fundamental and quantitative understanding of growth–structure–property relationships is central to applications where nanowires exhibit clear advantages. Atom Probe Tomography (APT) is able to provide 3 dimensional quantitative elemental distributions at atomic-resolution and is therefore unique in understanding the growth–structure–property relationships. However, the specimen preparation with nanowires is extremely challenging. In this paper, two ion beam free specimen preparation methods for APT are presented which are efficient for various nanowires.

1. Introduction

Due to their unique properties, semiconductor nanowires have attracted much attention recent years, and are believed to be the building blocks for next generation nano-electronic devices. Devices based on nanowires such as lasers [1,2], transistors [3,4], sensors [5,6] and photovoltaic solar cells [7,8] have been reported and are expected to be integrated with current state of the art silicon technology [1,9]. It is essential to control their synthesis process and understand the growth mechanism due to the intimate relationships between structure and properties including composition, morphology and doping distribution etc., which seems to be an impossible task with conventional characterization technique.

Capable of mapping 3D composition and position of identities in atom scale with single atom depth resolution and 0.2 nm lateral resolution, Atom Probe Tomography is regarded as one of the most powerful instrument to reveal chemistry and structure of materials [10–16], including semiconductors [17–19]. Freestanding nanowires on substrates have an ideal needle shape for APT analysis, but the difficulties of nanowires specimen preparation for APT lie in how to isolate a single nanowire and mount it in a correct orientation and position with regard to the local electrode in Atom Probe Microscopy. Several specimen preparation techniques have been developed for vertical nanowire. In 2008, Prosa and Smentkowski modified the standard Focus Ion Beam (FIB) lift-out techniques for TEM sample preparation to prepare APT specimens from nanowires [20]. Freestanding nanowires on substrate were firstly coated with nickel for protection. Thereafter, a wedge with nanowires was cut by FIB and attached to a micro-tip post using a micromanipulator. After that FIB and sulfuric acid were used to isolate a single nanowire and remove the protective material around it for analysis. This highly sophisticated approach is only applicable to nanowires that are moderate in length and densities because of the inefficiency in entirely embedding long nanowires with protective material especially when their densities are high. Therefore, the nether part of nanowire will be damaged or milled away by the subsequent FIB for isolating single nanowire. In addition, a special micro-tip post is required to support the specimen for this method.

In another methods [21], nanowires were firstly dispersed on a grid. Then a micromanipulator was used to pick up a single nanowire with e-beam Pt deposition and transfer it to a post with secure Pt deposition welding. Finally, the manipulator was carefully retracted until the initial small weld was detached. The difficulty of this approach is to precisely control the amount of the initial Pt deposition. If Pt deposition is too much, the nanowire would be pulled away from its upright position or even brake when retracting manipulator. If Pt deposition is too little,

Q2 * Corresponding author. Fax: +61 0293517682
E-mail address: rongkun.zheng@sydney.edu.au (R. Zheng).
[1] Australian Centre for Microscopy & Microanalysis, Madsen Building F09, The University of Sydney, NSW 2006, Australia.

http://dx.doi.org/10.1016/j.ultramic.2015.06.003

nanowires could not be lifted out because of the Van der Waals's force between substrate and nanowires.

Nanowires also have been synthesized on patterned post array [22] or grown epitaxially in low density by catalyst array [23] to suit APT. These two methods need to pre-defined nanowires before their growing and therefore are limited to specific ones, which are not applicable to various types of nanowires.

Nanowires can also be collected by truncated TEM grid scratching substrate. Afterwards a single nanowire in good orientation was targeted and picked up from bundle of them by manipulator and ready for APT experiment [24]. However, nanowires are prone to congregate on the edge of grid, which is quite time consuming to find a single nanowires in a good orientation for lifting-out. In addition, other nanowires are inclined to stick to the targeted nanowire because of the Van der Waals's force and therefore it is quite challenging to prepare sample with this method.

In this paper, we propose two FIB free methods of vertical nanowires specimen preparation for APT, which could be more efficiently applied to low-density and high-density nanowires respectively.

2. Material and methods

In this study, two types of vertical nanowires were used to demonstrate our specimen preparation methods respectively, and their APT experiments were both carried out in Cameca LEAP 4000XSi equipped with 355 nm laser pulse.

The first type of nanowires is low-density InGaAs nanowires grown on N+ GaAs (1 1 1) substrate by metal organic chemical vapor deposition (MOCVD) [25]. At the beginning of the synthesis, the colloidal Au solutions were diluted with deionized water for the growth of desired low-density nanowires array. As shown in Fig. 1, the average distance between nanowires is beyond 38 μm.

GaAs substrates with freestanding low density InGaAs nanowires were firstly cleaved into many small pieces (below 2 mm^2) with diamond scriber. Then pieces in good condition were selected and stuck to copper stubs with conductive high vacuum silver epoxy. Some free standing InGaAs nanowires appropriate for APT could be easily found on each of the fragments. The distance between nanowires is over 38 μm to ensure only a nanowire is field evaporated. Thereafter, specimens are ready for APT experiment as shown in Fig. 2.

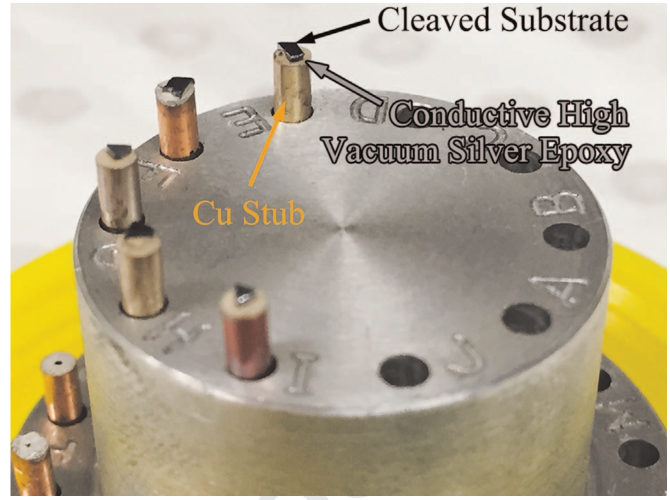

Fig. 2. Cleaved substrates are attached on Copper stubs with conductive high vacuum sliver epoxy.

However, this method is only applied to low-density nanowires. If the density of nanowires is high (two nanowires or more are spacing within 38 μm which is smaller than the size of local electrode), more than one nanowires may be field evaporated simultaneously as shown in Fig. 3(a). However, this phenomenon is quite rare for low-density nanowires. As shown in Fig. 3(b) the average nanowires spacing is over 50 μm, which is larger than the

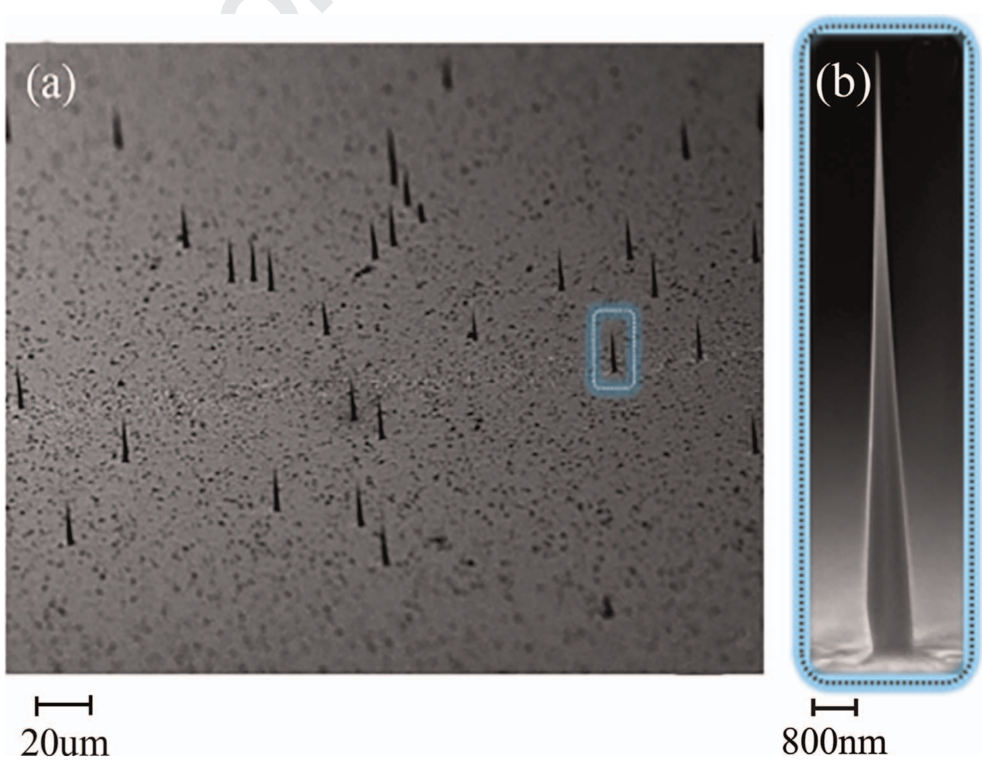

Fig. 1. SEM images of (a) low density InGaAs nanowire array, and (b) tapering shape single nanowire from the same substrate.

Please cite this article as: J. Qu, et al., Methodology exploration of specimen preparation for atom probe tomography from nanowires, Ultramicroscopy (2015), http://dx.doi.org/10.1016/j.ultramic.2015.06.003