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Strategies for fabricating atom probe specimens with a dual beam FIB

M.K. Miller[a,*], K.F. Russell[a], G.B. Thompson[b]

[a]Metals and Ceramics Division, Oak Ridge National Laboratory, P.O. Box 2008, Building 4500S, MS 6136, Oak Ridge, TN 37831-6136, USA
[b]Department of Metallurgical and Materials Engineering, University of Alabama, Tuscaloosa, AL 35487, USA

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Abstract

A FIB-based lift-out method for preparing atom probe specimens at site specific locations such as coarse precipitates, grain boundaries, interphase interfaces, denuded zones, heat affected zones, implanted, near surface and subsurface regions, shear bands, etc. has been developed. FIB-based methods for the fabrication of atom probe specimens from thin ribbons, sheet stock, and powders have been developed.
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Keywords: Atom probe; Focused ion beam; Ion milling; Specimen preparation

1. Introduction

Electropolishing has been the dominant technique for preparing needle-shaped atom probe field ion microscopy and atom probe tomography specimens. Several different electropolishing and a few chemical methods have been developed to fabricate and resharpen atom probe specimens from a wide range of metals and alloys [1–3]. However, there are many other materials that cannot be fabricated into suitable sharp needles with these methods. In addition, there are other types of samples, such as surface and multilayer films and site specific regions, where it is difficult, if not impossible, to electropolish the specimen such that the desired region of interest is in the analyzable apex region of the needle. Some lithography methods have attempted to fabricate specimens from these surface regions [4–12]. Although these methods are generally successful, they are extremely time consuming.

Ion milling with a low energy argon ion beam has been used for bulk and whisker materials [13,14]. In practice, these ion milling methods have generally been applied to removing surface films and other contamination. Precision ion milling in

*Corresponding author. Tel.: +1 865 574 4719; fax: +1 865 241 3650.
E-mail address: millermk@ornl.gov (M.K. Miller).

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conjunction with examination in an electron microscope has also been used to remove small amounts of material so that microstructure features, such as grain boundaries, may be located in the analyzable apex of the specimen [2,15–17]. However, this procedure is more generally performed with a pulsed electropolishing method [18–21].

Recently, focused ion beam (FIB) based methods have enabled transmission electron microscopy (TEM) specimens to be fabricated from a wide range of materials [22–26]. FIB-based methods have also been developed to produce atom probe specimens from multilayered thin films [6–11,27–33]. In this paper, some FIB-based methods are described to fabricate atom probe specimens from a variety of different types of starting forms including ribbon, thin sheet and powders and from site specific locations in bulk, surface and subsurface regions and thin film materials.

2. Experimental considerations

The goal of specimen preparation for the atom probe and field ion microscope is to produce a specimen with an end radius of less than 50 nm on a smooth shank with a taper angle of less than 5°. The apex region of the specimen should have a uniform cross section without any grooves or protrusions. It is important that the method used to prepare the specimen should not introduce any artefacts or change the microstructure or the solute distribution. The time required for the preparation of atom probe specimens is also a consideration as the latest generation of local electrode atom probes are capable of analyzing many specimens each day [34,35].

Unfortunately, ion milling with high-energy gallium ions generally introduces gallium into the surface layers of the specimen. Giannuzzi and Stevie have shown with the use of stopping range in materials (SRIM) calculations that the range of 30 keV gallium ions for many transition elements is ~10–30 nm for both incident, 0°, and high angle, 88°, beam configurations [25]. If the surface is not protected, gallium levels of up to ~30% have been measured with the atom probe [10,27]. As gallium atoms are larger than most transition elements, the gallium implanted into the material will impose an internal stress on the sample. This stress may cause premature failure of the atom probe specimen during analysis because of the hydrostatic pressure associated with the applied voltage. More importantly, the gallium introduced into the sample may intermix phases and regions of different compositions and may also turn crystalline regions amorphous [10,11]. However with proper procedures, these types of damage may be minimized to yield high quality atom probe specimens.

In FIB instruments, gallium is implanted into the specimen during imaging with the gallium ion beam. Therefore, dual beam instruments with a scanning electron microscope and a gallium ion beam column have been found to be beneficial for preparing atom probe specimens. The electron beam allows the specimen to be aligned to the instrument and examined with no ion damage to the specimen. In addition, the electron beam allows continuous monitoring of the ion milling process in real time. Real time monitoring is also helpful for end point detection so that milling can be terminated immediately when the apex of the needle is positioned at the desired location with the optimum end radius.

In order to protect the region to be analyzed during subsequent ion milling, a platinum cap may be laid down over the area of interest with either electron- or ion-beam deposition [6,27,36]. Platinum deposition with an electron beam or an initial electron beam deposition followed by ion beam deposition should result in the lowest possible level of gallium implantation during the deposition process. The thickness of this protective layer should be sufficient that the gallium that is implanted during milling operations is limited to this sacrificial cap. SRIM calculations indicate that 30 keV gallium ions have a stopping range of less than 10 nm in pure platinum (or tungsten). However, the deposited platinum contains high concentrations of impurities such as carbon and other elements. Therefore, a thicker layer is required in practice. A thicker layer is also advantageous in distinguishing the protective cap from the specimen by z-contrast imaging with the