Atomic Spectroscopy

Article

A Novel Approach for Fabricating Atom Probe Tomography Samples from the Thin Region of Electropolished TEM Disc

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Received: *September 03, 2024;* ***Revised:*** *October 18, 2024;* ***Accepted:*** *October 18, 2024;* ***Available online:*** *October 26, 2024.*

DOI: 10.46770/AS.2024.205

ABSTRACT: Atom probe tomography (APT) is a cutting-edge technology capable of imaging three-dimensional atom distribution and measuring chemical composition at the sub-nano scale. Since APT samples are tip-shaped and have specific size requirements, several manufacturing methods based on focused ion beam (FIB) have been developed. In order to precisely preserve the region of interests (ROI) into the tip and also satisfy the need of correlative characterization between crystallographic information and chemical information, we developed a new approach of directly fabricating APT tips from the twin-jet electropolished transmission electron microscopy (TEM) thin disc. This method has been successfully demonstrated on a Ni-B disc sample. After its nanocrystalline structure been observed by TEM, APT tips were prepared and analyzed from the selected region of the very-edge of the center hole. The distributions of major elements from the sample itself and induced by FIB processing were also discussed. This approach exhibits universal applications, which can not only feature in the correlation between TEM and APT analysis, but also benefit for the Ga-sensitive materials as only minimum FIB processing is required.

INTRODUCTION

Atom probe tomography (APT) is the cutting-edge three dimensional chemical analysis technique with near atomic resolution.[1,2] However its capability of characterizing crystallographic information is limited due to the limited detector efficiency and other artifacts such as local magnification and trajectory aberration.[3–5] Focused ion beam (FIB)-based methodology is a widely used technique which enables the site-specific APT sample preparation.[2,6] Still due to the extreme small interaction volume, many features yield poor or no contrast under the scanning electron microscopy (SEM) imaging as the thinning proceeds. Therefore to preserve a desired region of interest (ROI), such as grain boundary, interface or nano-particles in the analysis volume, is of great challenge.[7–11]

To satisfy the need of precise correlative characterization between crystallographic and chemical information, and the need of site-specific sample preparation, additional crystallographic analysis techniques are often introduced during the traditional FIB-APT experimental setups.[12–16] One common approach is to conduct transmission kikuchi diffraction (TKD) or transmission electron microscopy (TEM) to assist the APT tip sample preparation. These procedures involve the identification of the ROI on the bulk sample using SEM or electron backscattered diffraction (EBSD) prior to the FIB lift-out. During final sharpening, the annual milling is intermittently stopped and the TKD or TEM characterization is performed from time to time to guarantee the ROIs are correctly preserved in the final tips.[12–15] Besides the correlative characterization, the SEM/TEM images of the final tips could further be benefit for the later APT data reconstruction.[17,18] These experimental setups require special designed sample holders to adopt the tip-shape samples and to allow the sample transfer between SEM-FIB, TEM and APT.[19,20]

Recently a reversed method has been introduced by Baik *et al.*.[21] Instead of directly fabricating the APT tip, they prepared a thin TEM foil by FIB first. The TEM characterization was carried out in advance to gain the structural information and to locate a ROI. Later the wedge-cutting and annual milling were applied to modified the FIB lift-out thin foil into an APT tip with the ROI preserved. This method is of great benefit for those samples, in which the ROIs could not be identified from surface of the bulk sample and could be only determined under TEM with sufficient structural information. However, as the TEM foil was placed on a pre-sharpened pillar of Cu grid with less than 1 micron of the top diameter, the length of the TEM foil was limited to be less than 3-4 micron to minimize its internal stress and avoid fracturing. The selection of ROI was confined in a region with less than 1 micron in length which was right on the top of the pillar, otherwise there wasn't any supporting Cu grid in the bottom to form a tip. Furthermore, this method is only applicable on a FIB lift-out TEM foil, but not possible on the twin-jet electropolished disc.

For the electropolished disc, as its thick region is often several of ten microns in thickness, it is possible to make an APT lift-out on the thick region of the disc. However, as this region is not electron transparent, no structural information can be obtained by TEM in advance, which makes it no different from a regular lift-out of a bulk sample. As the crystallographic information is only available in the thin region, in this work, we present a novel TEM-assisted APT sample fabrication approach to directly prepare an APT tip from the edge of the center hole of the electropolished TEM disc. This approach not only allows full crystallographic characterization by TEM in advance, but also facilitates subsequent APT analysis on the TEM-identified ROI, with multiple ROI choices possible. Furthermore, it requires minimum FIB processing, further reducing the Ga damage and contamination.

Fig. 1 TEM analysis of nanocrystalline Ni-B prior to the APT sample preparation.(a) TEM disc with the diameter of 3 mm prepared by twin-jet electropolishing; (b) TEM image of the edge of the center hole, in which the selected ROI is marked by red dash box; (c) bright field (BF) TEM image of the selected ROI in (b); (d) selected-area diffraction-pattern (SADP) of ROI; (e) typical high-resolution TEM image of Ni-B samples.

EXPERIMENTAL

Sample and selection of ROI. The sample we used is a nanocrystalline Ni-B film produced by direct current electrochemical deposition, using high-purity copper and nickel plates as the cathode and anode, respectively. The electrolyte contained 60 g/L nickel sulfate, 30 g/L boric acid, 0.2 g/L sodium dodecyl sulfate, and 1 g/L saccharin. Trimethylamine borane (0.1 g/L) was added as the boron source. Further details of the electrodeposition process are available in reference.[22] The sample was prepared by first mechanically removing the copper substrates and followed by annealing at 300 °C for 1 hour in an argon-protected atmosphere. A TEM disc with 3 mm in diameter was cut and then thinned using traditional twin-jet polishing with a solution of ethanol and perchloric acid with a volume ratio of 9:1 at -15 °C. The appearance of the TEM disc is shown in Fig. 1a. TEM observation was carried out using FEI Talos F200X G2. Part of the edge of center hole of this disc is shown in Fig. 1b, with the red dash box indicating where the ROI was selected. Fig.1c gives the bright field TEM image of the ROI with its diffraction pattern shown in Fig.1d. The average grain size of the ROI was estimated to be around 10 nm. The sample has a nanocrystalline structure, featuring grain sizes that ranging from less than 10 nm to 30 nm continuously. To give a better view of the nano-grain structure, a typical high resolution TEM image of the nanocrystalline Ni-B is shown in Fig.1e. Note that the high-resolution image was taken from another Ni-B sample, but not from the ROI. It is worth noting that this approach enables multiple choices of ROI selection along the edge of the center hole. The protruding area is preferred, as it requires less efforts to be sharpened, as shown in Fig.1b. The concave area is also possible, however more surrounding materials have to be removed to make the lift-out.

Fig. 2 The FIB procedure for preparing the APT tip from the TEM disc:(a) SEM image of the relocated ROI of the edge of the disc; (b) Pt deposition on the surface of the selected ROI marked by the red dash box; (c) welding the manipulator on the ROI; (d) cutting the side and the bottom of the ROI free; (e) lifting out the ROI with the manipulator; (f) rotating the manipulator by 90°; (g) side view and (h) top view of the ROI on a Si post; (i) annual milling for sharpening; (k) the final shape of the APT tip.

APT tip fabrication. The tip preparation was performed using Zeiss Crossbeam 350 equipped with the manipulator. After the TEM observation, the sample was transferred into FIB, with the selected ROI been identified using SEM, as shown in Fig. 2a. A thin layer of Pt (about 50 nm in thickness) was deposited on the surface of the ROI for protection. Meanwhile, the left side of the ROI was cut free from the disc, as shown in Fig. 2b. The manipulator was firmly attached on the ROI using Pt deposition, followed by the bottom side and the right side of the ROI been cut from the disc, as shown in Fig. 2c-e. After lift-out, Fig. 2f shows the manipulator rotated by 90° to make the surface of the ROI perpendicular to the sample stage. It is worth mentioning that a small angle between the surface of the ROI and the Z axis of the sample stage always existed due to the limitation of the manipulator. The bottom corner of the ROI was attached by Pt deposition on a flat-top Si post. The manipulator then was cut free from the ROI. Fig. 2g and h show the front view and the side view of the ROI left on the Si post to be sharpened. A few steps of annual milling were then performed to produce the tip-shape APT sample with desired top diameter and shank angle, as shown in Fig. 2i and k. Only small beam currents were applied in these steps, from 50 pA to 20 pA to reduce the Ga contamination. Finally, the low-kV viewing (5 kV, 20 pA) was used for clean-up to further remove the Ga damage.

APT analysis. APT experiments were conducted using a local electrode atom probe (CAMECA LEAP 5000 XR). All analysis were carried out at a specimen temperature of 45 K, a laser pulsing energy of 35 pJ, a laser repetition rate of 125 kHz and a target evaporation rate of 0.4 %. The data reconstruction was performed

Fig. 3 The atom maps of the nanocrystalline Ni-B sample showing the distribution of Ni, B, C, Pt, Ga, and O.

using AP Suite 6.3, assisted by the tip shape from the SEM images taken after the final milling to guarantee the accuracy of the reconstruction. Due to the possible peak overlaps, for example the $C5^{+}$ and Ni^{+} isotope at 60 Da, $C5^{2+}$ and Ni^{2+} isotope at 30 Da, the reconstruction was separated into different parts according to their structures and the mass spectrum of each part was ranged accordingly.

RESULTS AND DISCUSSION

Figure 3 shows the atom maps of all major elements we detected in this sample, including Ni, B, C, Pt, Ga, and O. The Ni-B sample was successfully captured at the top part of the reconstructed volume, with the concentration measured to be 99.3 at.% Ni and 0.7 at.% B. The bottom part was the protection Pt layer on the surface of the ROI as well as the supporting Pt deposition between the ROI and the Si post, as the electron transparent thickness of the ROI was too thin to form the tip by itself. The C was a byproduct induced with the Pt deposition and the Ga was implanted by the FIB ion gun.

Surface oxidation. Fig. 4 plots the 1D concentration profiles along the red dash arrow placed perpendicular to the Ni-B/Pt deposition interface. An oxide layer was found in the interface, with the thickness of less than 5 nm. To give a clear indication of the O distribution, without any interference from peak overlap, only the peak at 16 Da was marked as O. Therefore, the O concentration of this oxide layer could be possibly underestimated.

The Pt and C distributions were not only found in the oxide layer, but also discovered in the surface region of the Ni-B, with the thickness of roughly 3-4 nm adjacent to the oxide layer. It is well known that when two phases which have large differences in their evaporation fields evaporating on the same X-Y plane, the ion trajectory aberration is inevitably induced and gives rise to the interface blur with the uncertainty of concentration measurement.[23,24] If we considered the Ni-B/oxide/Pt as a mulit-layer structure, this trajectory aberration occurred on both Ni-B/oxide interface and oxide/Pt interface. Therefore, the Pt and C distribution in the Ni-B as well as the B distribution in the Pt deposition were both undoubtedly risen from the trajectory aberration near the interface. Furthermore, as the oxide is often considered to have a higher evaporation field than the bulk material, the measured thickness of the oxide layer might be slightly overestimated due to local magnification effect.[23]

The Ga distribution across the oxide layer was mainly induced during the FIB lift-out process. Using SRIM-2013 software calculation, the penetration depth of Ga could be roughly

Fig. 4 (a) Atom map of Ni, B, and C of the tip, with one analysis cylinder placed perpendicular to the interface as marked by the red dash arrow; (b) 1D concentration profiles plotted within the analysis cylinder.

estimated. We assumed a multiple layer structure distributed as Pt/Ni-3 at % O/Ni-1 at.% B from top to bottom, and the thickness of each layer could be directly measured from the 1D concentration profile on Fig.4b. The ion energy was set to be 30 keV. The displacement energies of Pt, Ni, B and O atoms were set as 44 eV, 33 eV, 46 eV, 20 eV, respectively [25,26]. SRIM predicted a Ga penetration depth of roughly 35-40 nm. Taking into account that the thickness of the Pt layer was underestimated as the outside frame of the tip couldn't be collected due to the limited field of view, the actual distribution of Ga (25 nm from the Pt layer to the Ni-B matrix) was in rough coincidence with the SRIM calculation.

B distribution. Judging from Fig.4b, B exhibits a concentration gradient from the oxide layer to the Ni-B. The peak of B is located on the Ni-B/oxide layer interface, to the left side of the O peak, with roughly about 1.5 nm apart. Such distribution could be resulted from several possible reasons. Firstly, as the evaporation field for B^{+}, B^{2+}, Ni^{+}, and Ni^{2+} are 64, 79, 35, and 36 V/nm, respectively, the temporary retention of B with the obvious higher evaporation fields would occur during the entire evaporation process, giving rise to a slightly increase in Z coordinates of all B atoms as well as the high B concentration near the Ni-B/oxide layer interface. Secondly, the high concentration of B at the top surface of the TEM disc could be attributed to the long storage time, as the twin-jet electropolishing was performed more than 5 years ago. Boron atoms have a thermodynamic tendency to segregate towards the Ni surface.[27] Although the diffusion coefficient of boron in the Ni lattice is low at room temperature,[28,29] the significant grain boundary fraction in our nanocrystalline sample facilitates faster diffusion along grain boundaries.[27,28] The diffusion coefficient of boron in coarse-grained Ni at room temperature (~25 °C) is estimated to be approximately 8.57×10^{-24} m^2/s[28,29]. Assuming an enhanced diffusion coefficient of 8.57×10^{-22} m^2/s (about two orders of magnitude higher than the coarse-grained counterpart) for boron in nanocrystalline Ni,[29,30] the estimated diffusion distance over 5.5 years is about 367 nm, which exceeds the typical thickness of the electron transparent region in TEM samples. This supports the plausibility of boron atoms migrating to the surface via grain boundaries during storage. Potential impact of Ga ion beam damage during sample preparation could be the third reason which also contributed to the observed heterogeneity in boron distribution from the oxide layer to the matrix.

Figure 5a shows the top Ni-B region with B iso-concentration surface at 0.98 at.%. The B iso-surface shows an inter-connected network feature. To give a clear view, a center slice of 5 nm of Fig. 5a was cut and shown in Fig. 5b. One cylinder was placed to penetrate three iso-surfaces perpendicularly to plot the 1D concentration profile along the red dash arrow, as shown in Fig. 5c. The positions where the cylinder intersected the iso-surfaces are marked as 1#, 2# and 3# on Fig. 5b, corresponding to the same positions indicated in Fig. 5c. Despite the existence of the B concentration gradient from the Ni-B to the oxide layer and other influences to the B distribution aforementioned, the B still clearly shows the segregation tendency to the iso-surface regions. It has been reported that B atoms tend to segregate at the Ni grain boundaries.[31] As the distances between the concentration peaks in the 1D profile correspond closely to the average grain size observed in the TEM image (Fig. 1c) and high-resolution TEM

Fig. 5 (a) Atom map showing the top region of the tip with the B-isosurface at 0.98 at.%; (b) center slice with 5nm in thickness of (a) for highlighting the B-riched interfaces, with one cylinder placed to penetrate three iso-surfaces; (c) 1D concentration profile plotted within the cylinder along the red dash arrow in (b).

image (Fig. 1e) of the present sample, it is highly likely that the B iso-surfaces plot out the nanocrystalline grain boundaries. Probably due to the sample's manufacture process, the distribution of B in the grain boundaries and the matrix is distinctly different from traditional alloys [32]. Still more experiments are needed to verify this point.

Limitations and further improvement. As discussed above, the ROI of the Ni-B sample we selected using TEM was successfully preserved and analyzed by APT. This Ni-B sample has a nanocrystalline structure, which complicates the correlative characterization of each grain. Still, we were able to correlate TEM observation of the ROI with the grain size of roughly 10 nm, to the B distribution of the same scale in the APT reconstruction. It is known that for other samples with large grain size, if the ROI was selected to be a single grain boundary or a single precipitate, the correlation between TEM structural information and APT chemical information would be simpler and more straightforward. Still, we would like to use this complicate nanocrystalline structure to demonstrate how far we can push correlative observations and highlight the limitations of current correlative methods. To obtain the grain boundary information for each grain, high resolution TEM is necessary to be performed on the nanocrystalline structure, which requires an ultra-thin sample thickness, as shown in Fig. 1e.[33] Such sample is not suitable to be further modified into APT tips, as the ultra-thin ROI is extremely fragile. Therefore, for TEM samples, the ROI should have a certain thickness to withstand the further processing to form an APT tip, as shown by our Ni-B case. However, the thickness of these samples limits the observation of the structural information using conventional TEM. This is a dilemma that should be bared in mind. No matter which correlative method is applied, and whether APT tips are modified from FIB lift-out foils[21] or eletropolished discs, the correlation is limited as long as only limited structure information is available. It is worth to point out that, three dimensional TEM techniques are capable of characterizing the full grain structure of the nanocrystalline samples with large thickness.[34–36] Combining with those recent advances in TEM techniques, it is possible to push the TEM-APT correlative observation into a whole new level.

As previously the method to fabricate APT tips from FIB lift-out TEM foil has been proposed by Baik *et al.*,[21] we designed this novel approach to be a complement to allow the direct fabrication of APT tips from electropolished TEM discs. It demonstrates broad application on various kinds of materials, not only on metals and alloys, but for all materials which can be electropolished to be TEM discs. For a single grain boundary or precipitates with low density, this approach largely increases the chances of capturing these features in tiny APT tips, and is also capable of achieving the precise correlative characterization between TEM and APT. Furthermore, this approach is especially benefit for the alloys in which certain precipitates present under the in-situ heating TEM experiments.[37–39]

Since this approach only requires minimum annual milling steps under low beam currents, therefore it largely reduces the Ga contamination comparing with the traditional lift-out method. Still the Ga contamination from FIB processing to the very top surface of the Ni-B TEM disc was inevitable. Although the contamination was limited to a few nanometers, if the top surface is critical, we recommend adding an additional protective layer instead of Pt deposition. One should take extra care in the choice of these protective materials. Ideally, its chemical composition should differ from the ROI, but with a similar evaporation field to minimize trajectory aberration. The necessary deposition thickness to prevent the Ga penetration could be calculated from SRIM. For example, Ni and Cr are often used as protective material or embedding materials.[40,41] In the Ni-B case, Cr could be considered a preferred choice and more experiments are needed to further verify this point.

As discussed above, the trajectory aberration is almost inevitable for a multi-layer structure. Still, efforts can be made to minimize its influence as much as possible. Introducing a protective layer with a similar evaporation field is one option. Adjusting the angle between the interface and the Z-axis of the tip is another. It is well known that interface broadening is most severe when the interface is parallel to the Z-axis and nearly disappears when it is perpendicular. Due to the design of this approach, though it is impossible to fabricate the perpendicular interface, inducing large angle between interface and the Z-axis during FIB

processing could be a practical solution. Furthermore, extra care should be taken when interpreting the mass spectrum near the interface region.

CONCLUSIONS

In this paper, we present a novel approach of directly fabricating APT tips form the thin region of a TEM twin-jet electropolished disc. The approach has been successfully applied on a nanocrystalline Ni-B TEM sample. It also demonstrates the potential of universal applications on various kinds of samples, with the advantage of multiple choices of ROI from one TEM disc. For materials which are sensitive to the Ga contamination, such as Al alloys, this approach also proves to be very practical as it only requires minimum FIB processing and therefore largely reduces the Ga damage.

AUTHOR INFORMATION

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Notes

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ACKNOWLEDGMENTS

The authors acknowledge the financial support of the National Key R&D research project (2022YFB3707103), National Natural Science Foundation of China (52271058 and 52130107), China Postdoctoral Science Foundation (2024M753833) and State Key Laboratory of Mechanical Transmission for Advanced Equipment (SKLMT-ZZKT-2024Z04 and SKLMT-ZZKT-2024R03).

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