Paper

Specimen preparation for three-dimensional atom probe using the focused ion-beam lift-out technique

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In three dimensional atom probe (3DAP) analysis, specimen preparation is of considerable significance. Specimens which are fabricated by the existing method are often ruptured from the part attaching the sample to the support needle because attachment force by deposition is not adequate. Specimen preparation methods which have stronger attachment force are required. In this study, we prepared some specimens using six types of cohesion methods in which the shape of contacting part between the sample and the support is different. We tested the strength of the specimens by applying the electric field to them, and searched the optimum specimen preparation method for 3DAP analysis using the results of comparison of physical strength and difficulty in fabrication of them. Then we concluded that specimen preparation method in which the sample was attached with the base needle by physical strengths of C-gas deposition in connected hole was the best way.

1. Introduction

As the coming of nanotechnology, the scale of electronic devices has been getting smaller and smaller. For example, commercialized devices are expected to be designed within 1-nm rules in the next decade. To analyze these devices, the three dimensional atom probe (3DAP) has attracted attention as one of the most powerful methods for three dimensional analysis on an atomic scale [1]. In 3DAP analysis, the needle-shaped specimen was required. In analysis of electronic devices, focused ion-beam (FIB) technique is used in the specimen preparation[2-4]. However, specimens are often ruptured from the attachment area between the sample and support needle because of high electric field stress during the 3DAP analysis. The physical cohesion methods [5], not welding methods [6], have been suggested to this problem. However, these methods were not adopted in the specimen preparation by FIB because of complicated procedure.

In this study, we prepared specimens using six types of physical cohesion methods [7] and welding method. Two kinds of deposition gas (C and Pt) were used in the FIB-chemical vapor deposition (FIB-CVD). We examined the strength of attachment area of specimens for the electric stress, and discussed the optimum specimen preparation method by FIB in 3DAP analysis.

2. Experiment

Six types of physical cohesion methods and welding method were used as shown in Fig. 1. Using FIB-CVD, Si chips lifted-out from Si base were attached to W needles prepared by electropolishing. Method (a) was welding method. Figure 2(a) shows scanning electron microscope (SEM) image of attachment method (a) before the gas deposition. In welding method (a), C or Pt gas was used. Methods (b)-(g) were physical cohesion methods. Si and W were attached by physical strengths of C-gas

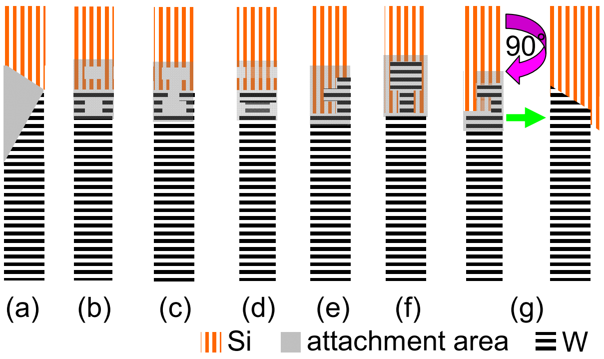

Fig. 1. Schematic images of specimens prepared by attachment methods (a)-(g).

deposition between samples and base needles in methods (b)-(d). In the methods (e)-(g), Si and W were attached by physical strengths of these joint. Figures 2(b) and 2(c) show SEM images of attachment methods (c) and (g) before the gas deposition, respectively. C-gas was used for deposition in methods (b)-(g).

Experiment I: Si chips with a cross-section area of about 3µm×3µm were cut-out from Si base (Fig.3.) and attached to W needles whose diameter at attachment area were 3µm. Size of attachment area were 4.5µm×3µm in all specimens.

Strengths of specimens were measured by applying high voltage to specimens in field ion microscope (FIM). The attachment strengths against electric field stress in these measurement condition in FIM were similar to that in 3DAP and we could observe the rupture of specimen. Applied voltage to the specimen was gradually increased until the specimen was ruptured or applied voltage attained the maximum (24 kV). The imaging gas (helium) pressure was in the order of 10^{-3} Pa. The specimen was cooled with liquid nitrogen.

Experiment II: The specimens were prepared in the same way as Experiment I and the apexes were needle-shaped by the annular milling method [8]. The taper angles of all specimens were between 15 and 35 degrees and the radiuses of curvatures were between 50 and 85 nm. The SEM image of an example of specimen is shown in Fig. 3. Attachment strengths measurements of these needle specimens were conducted using the FIM under the same condition (maximum voltage, pressure and temperature) as Experiment I.

Experiment III: The GaAs chips were attached to the W base needles using attachment method (e) and (f). They were needle-shaped by the annular milling method. Atom probe (AP) analysis of these needles was performed. Analysis was performed by our homemade 3DAP instrument [9] with a pressure $<1\times10^{-7}$ Pa triggered with laser pulse (λ = 1064 nm, pulse width = 300 fs). The repetition rate of laser pulses was 5 kHz and the laser power was 2.5 or 1.25 nJ per pulse. The analyses were performed at room temperature.

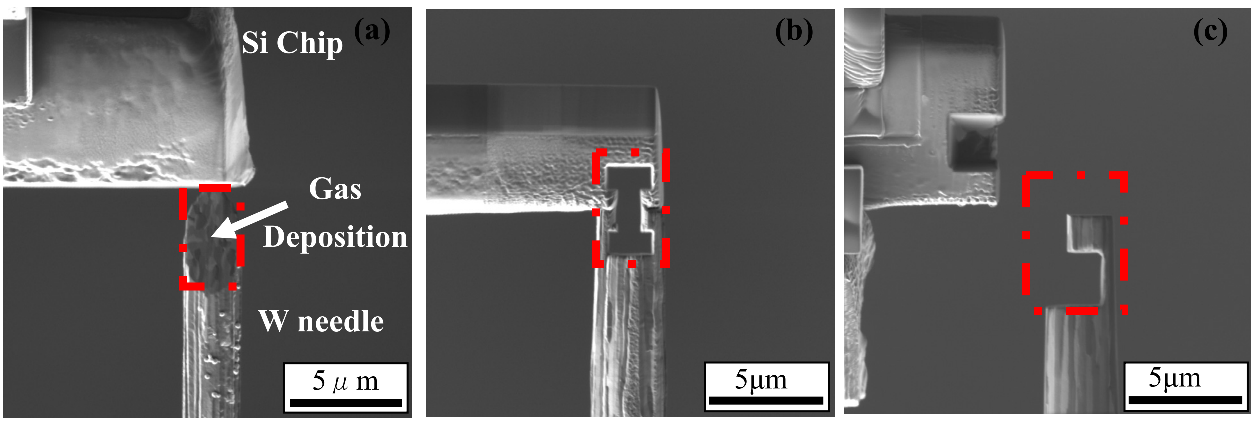

Fig. 2. (a) Specimens which was prepared using welding method (a). (b) Specimens which was prepared using physical strength of C deposition :method (c). (c) Specimens which was prepared using physical strength of joint between sample and base needle :method (g).

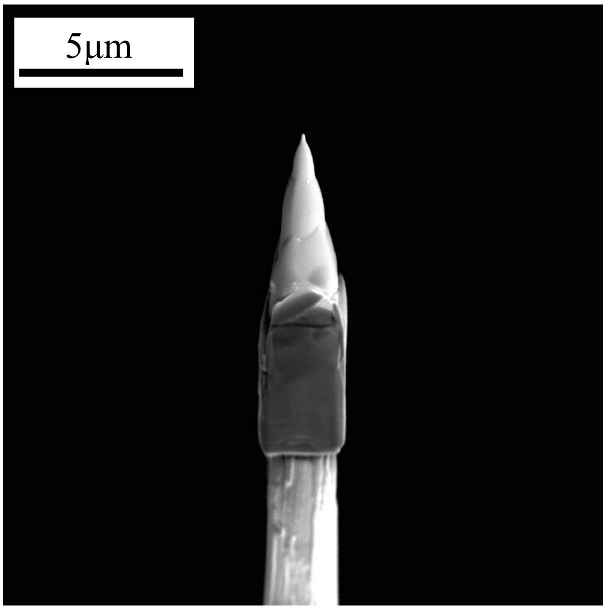

Fig. 3. Specimens which was needle-shaped by the annular milling method.

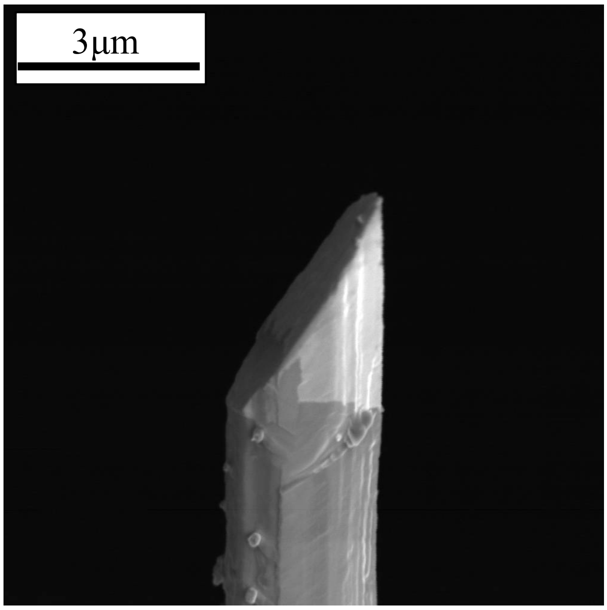

Fig. 4. Specimens which was ruptured by detachment of a Si chip from a W base needle: method (a).

3. Results and discussion

Results of Experiment I, II, and III are shown in Table. 1. In the Experiment I, specimens which were prepared using the attachment methods (a)(Pt) and (b)-(g) were not ruptured even when maximum voltage of 24kV was applied. In the attachment method (a)(C), Si chips with the attachment area were away from W base needle when 10.5kV was applied, shown in Fig.4. This result indicated that the adhesion force of C-gas was weak. The stress from the electric field applied to the attachment area of this specimen under this condition was calculated as 6×10^6 Pa. From the fact that the stress calculated from the electric field is ~10^7Pa in 3DAP analysis, it is assumed that method (a)(C) was not adequate for 3DAP analysis.

In the Experiment II, FIM images were begun to be observed at applied voltage between 3.0 and 3.8 kV in all measurements.

In the method (a)(Pt), the results were split up into two cases in several measurements. In the first case, the specimens were not ruptured at the maximum applied voltage. In the second case, the specimens were ruptured in the same phenomena as the result in the attachment method (a)(C). Although we could not definitely determine its reason, the different interfacial condition between Pt-gas and W or Si in each specimen seemed to do something with it. It was hard to recognize what split into two cases because the specimens in two cases apparently had the same welding condition. Besides, the stress from the electric field applied to the attachment area of this specimen in the Experiment II was 3-4$\times10^7$ Pa. This means that this specimen was ruptured when

Table. 1. Review of attachment method. RA:rupture from attachment area. RW:rupture of W base needle.

Attachment method	(a) (C)	(a) (Pt)	(b)	(c)	(d)	(e)	(f)	(g)
Experiment I applied voltage(kV)	10.5	24	24	24	24	24	24	24
Experiment II applied voltage(kV)		RA or 24	RA or 24	RW or 24	RA	RW or 24	24	RW or 24
Experiment III						○	○	

the stress nearly equal to that in 3DAP analysis was applied to. These showed (a)(Pt) was not adequate for 3DAP experiments.

In the methods (b)-(d), the results were split into several cases. In the method (b), the results were the same as that in the method (a)(Pt). In the method (c), some specimens were not ruptured in the maximum applied voltage and others were ruptured by the break of W base needle. From these results, it was suggested that attachment area of the specimens in the method (c) were stronger than base W needles. To compare the method (b) and (c), because gas flowed into connected hole in Si and W in the method (c) better than two separate holes in the method (b), attachment force between C deposition and W or Si in the method (c) was stronger than that in (b). In the method (d), to mill adequate gap that gas flows in was of great difficulty. Therefore the attachment force by the method (d) was not sufficient and specimens prepared by (d) were ruptured when about the half of maximum voltage was applied.

In the methods (e) and (f), the results were the same as that in the method (c). Attachment area of these specimens was stronger than W base needle against electric field stress.

In the Experiment III, 5 specimens were prepared and 4 were able to be analyzed. Only one needle specimen was ruptured by breaking of a base W needle. It showed that needle specimens prepared by using physical cohesion was effective in AP analysis from these results.

From the above results, it was found that the specimens fabricated using the methods (c), (e)-(f) were strong enough for the electric field stress. To calculate the stress from the electric field applied to attachment area of these specimens, it was found that they could bear over 1×10^8 Pa. From comparison of procedure in these methods, we concluded that the specimen preparation method in which sample was attached with base needle by physical strengths of C-gas deposition, (c) was most convenient one for the specimen preparation in 3DAP.

4. Conclusion

Specimens prepared by the seven attachment methods were compared from the point of view of physical strength against the stress and difficulty in fabrication. Adhesion force of welding methods was weak for 3DAP analysis. The optimum preparation method was the method in which sample was attached with base needle by physical strengths of C-gas deposition in connected hole, (c).

Acknowledgments

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