Reversible phase-transition control in nanometer-sized zirconium wires via pulse-voltage impression

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Abstract
Pulse-voltage-stimulated phase transition in nanometer-sized zirconium (Zr) wires was observed in situ by high-resolution transmission electron microscopy. Simultaneously, the variation in conductance during the transition between crystalline and amorphous phases was examined. The crystalline phase of a hexagonal closed-packed structure in the wires transformed into an amorphous phase while applying pulse voltages of 4 ns in width, and subsequently returned to the initial crystalline phase by the impression of pulse voltages of 5 ms in width; the reversible phase transition via voltage impression using shorter and longer pulse waves was observed. The average conductance per a unit area in the amorphous phases was decreased to 0.87 of the crystalline phases. The amorphous region in the wires expanded gradually by every pulse-voltage impression, whereas the conductance decreased stepwise in response to the gradual expansion. It was demonstrated that the conductance of the wires can be controlled in a stepwise manner via pulse-voltage impression, leading to the application of the nanowires to functional nanodevices.

1. Introduction

The refinement of electronic devices, such as transistors and diodes, has already reached a critical limit, and the future integration of circuits hinges on the size reduction of their junctions, i.e., wires and other contact structures [1–3]. Nanometer-sized wires (NSWs), i.e., wires having sub-10 nm widths, are possible candidates for this purpose [4–18]. In addition, NSWs reveal inherent electrical conduction properties different from those of larger wires, as exemplified by the quantization of conductance, which is applicable to multistage resistive switching and memories [2]. Thus, nanometer-scale size reduction brings in new functions into wires while promoting the integration; NSWs can be used as nanodevices.

Recently, the amorphization of pure metal NSWs via pulse-voltage impression has been investigated in connection with conductance variation [19–24]. The amorphous phases derived by this method are stable at ambient temperature, which is less common in amorphous metallic nanospheres prepared by spray freezing [25]. The stable amorphous metallic NSWs are expected to be applicable to nanometer-sized functional devices, such as non-volatile phase-change random access memories and switches [26, 27]. In particular, pure-metal NSWs have the advantage of the absence of precipitation even during long-term operation and fast phase transition; the NSWs are expected to become degradation-free high-speed memories [28–34]. However, the amorphization of pure-metal NSWs has been confirmed only for several elements of high-melting-temperature metals; limited researches have been performed [19–24, 28–31, 35–45]. In this study, we focused zirconium (Zr) with a melting temperature of 2125 K [46], for which amorphization in NSW structures has not been confirmed, and investigated the phase transition and the conductance variation in their NSWs via pulse-voltage impression.
2. Method

We used high-resolution transmission electron microscopy for in situ observation of Zr NSWs during pulse-voltage impression, as reported previously [13, 21–24, 45, 47–49]. Zr nanotips were prepared by milling of the triangle edges of thin Zr strips using the irradiation of 5 kV argon cation beams. The two nanotips were attached on two sample stages in a special-specification transmission electron microscope for in situ observation of picometer-precision nanotip-manipulation. The microscope was operated at an accelerating voltage of 200 kV. Zr NSWs were produced by the contact of the two Zr nanotips and subsequent elongation using piezodriving nanotip-manipulation while lattice-image observation. Pulse voltages of 4–5000 ns in width and 0.1–3.3 V in height were impressed to the NSWs. The structural dynamics during the pulse-voltage impression was observed in situ. The movies of observed lattice images were captured using a charge-coupled device camera for electron images. Simultaneously, the conductance of the NSWs during the process was investigated via a two-terminal method. The temperature of the sample stage was ambient. The pressure around the sample stage was 10⁻¹⁰ Pa.

3. Results

3.1. Amorphization of Zr NSWs

Figure 1 shows the lattice images of a Zr NSW during the one-time impression of a pulse voltage (see movie 1, which is available online at stacks.iop.org/NANOX/1/010050/mmedia in the supplementary data). The NSW appears in the center of each image. Before the pulse-voltage impression, the (011) lattice fringes with a spacing of 0.25 nm in Zr having a hexagonal closed-packed structure are observed in the NSWs, and the Fourier transform pattern of the image of the thinnest region, which is the wire-part region having the minimum cross-sectional width between two original nanotips, is a symmetrical spotty shape (figure 1(a)), implying that the phase of the initial state is crystal. Once a pulse voltage of 4 ns in width and 0.90 ± 0.05 V in height is impressed to the NSW, a pepper-and-salt image emerges in the thinnest region, and the Fourier transform pattern changes to an annular shape (figure 1(b)). Thus, the phase transforms to amorphous.

Figure 2 shows the cross-sectional area of the thinnest region of the Zr NSW and the conductance during the amorphization in figure 1. The average conductance of the crystalline NSW before the pulse-voltage impression is 1.26 × 10⁻³ S and decreases to 1.12 × 10⁻³ S after amorphization; the conductance ratio of the amorphous state to crystalline state is 0.89.

3.2. The gradual expansion of the amorphized region and subsequent crystallization

Figure 3 shows the lattice images of a Zr NSW during the multi-time impression of pulse voltages (see movie 2 in the supplementary data). As with figure 1, before pulse-voltage impression, the (011) lattice fringes appear in the NSWs, and the Fourier transform pattern is a symmetrical spotty shape (figure 3(a)), showing that the phase of this state is crystal. A schematic of the NSW is shown in figure 4. While impressing the pulse voltages of 4 ns in width and 0.30 ± 0.05 V in height repeatedly, first, the image of the center part of the thinnest region changed to a pepper-and-salt image, and subsequently the observed area of the pepper-and-salt image expanded in a stepwise manner. In response to the gradual expansion, an annular shape pattern was added in the spotty Fourier transform pattern (figure 3(b)). After the impression of the 27 pulse voltages, the observed area of the pepper-and-salt images expanded to all of the thinnest region, and the spotty pattern disappeared in the Fourier transform pattern (figure 3(c)). In this process, the pepper-and-salt images changed at every pulse-voltage impression while the expansion, implying that the thinnest region melted at each energization. Thus, the amorphization proceeded stepwise. The lattice fringes emerged by impression of a pulse voltage of 5 ms in width and 0.30 ± 0.05 V in height, and the Fourier transform pattern changed to a symmetrical spotty shape; the phase returned to crystal after the amorphization of whole constriction region (figure 3(d)).

Figure 5 shows the cross-sectional area of the thinnest region of the Zr NSW, the conductance, and the conductance per a unit area during the phase transition in figure 3. Before pulse-voltage impression, the average conductance per unit area of the crystalline NSW at time a (ρ) is 5.0 × 10⁻¹³ S m⁻². After the impression of the three pulse waves, the conductance per unit area at time b (ρ) decreases to 4.2 × 10⁻¹³ S m⁻². After the subsequent impression of the 24 pulse waves, the conductance per unit area at time c (ρ) decreases to 3.3 × 10⁻¹³ S m⁻². Thus, as the amorphized region expands gradually, the conductance per a unit area decreases stepwise. Figure 6 shows the conductance per a unit area of the Zr NSW against the ratio of the amorphized area to a total area of the thinnest region. The conductance per a unit area decreases with increasing the area ratio. Then, the conductance per a unit area at time d (ρ) increases to 5.5 × 10⁻¹³ S m⁻² due to crystallization. The ratio of the conductance per a unit area at the initial crystalline state at time a to that at the amorphized state at time c (ρ̅ / ρ) is estimated to be 0.67.
3.3. The formation of amorphous nanotips after the fracture of Zr NSWs

Figure 7 shows the lattice images of the fracture of a Zr NSW due to pulse-voltage impression. First, two types of lattice fringes, i.e., the (0111) lattice fringes of 0.25 nm in spacing and the (0110) lattice fringes of 0.28 nm in spacing, are observed in the NSW (figure 7(a)). The crossing angle of these fringes ($65 \pm 1^\circ$) corresponds to the interfacial angle between the (0111) and (0110) planes ($64^\circ$). The cross-sectional width of the thinnest region is approximately 3 nm. The Fourier transform pattern of the region indicated by the white frame in the positive electrode is a symmetrical spotty shape (the insertion in figure 7(a)), also showing that the phase of this state is crystal. The NSW broke after the impression of a pulse voltage of 4 ns in width and $1.90 \pm 0.05$ V in height, followed by the formation of two separated nanotips (figure 7(b)). Pepper-and-salt images are observed around the edges of the two nanotips, and the Fourier transform patterns of the edges reveal annular shapes (the inserted figure in figure 7(b)). Hence, the amorphous nanotips are formed after the fracture of the NSWs due to the pulse-voltage impression.

3.4. The ratio of conductance per a unit area between the amorphous and the crystalline states

Figure 8 shows the ratios of the conductance per a unit area of the amorphous to the crystalline states ($\rho_{amo}/\rho_{cry}$) in 14 Zr NSWs having wire widths of 2–12 nm. The ratio ranges from 0.66 to 0.98 with the average is 0.87 (the broken line in figure 8).

3.5. The amorphization voltages and crystallization voltages against the wire width

After the melting of the thinnest region of the NSWs, whether the amorphization or crystallization occurs is dependent on mainly the cooling rate, i.e., the heat distribution and the thermal conduction manner from the thinnest region. This manner is determined by the impression energy and the structure in which the energy...
impressed, i.e., the shape of the thinnest region and its neighboring two nanotips (electrodes), e.g., the minimum cross-sectional width, the length of thinnest region, the surface indices of the thinnest region and the two nanotips, the orientational relationship between the two nanotips, the convergent angle of two nanotips, and the defects and strain in them. In these factors, the minimum cross-sectional width leads to the conductance of the system, i.e., the impression energy for a certain pulse voltage, and we selected this as an argument of the pulse voltage of the amorphization and crystallization as follows.

Figure 9 shows the amorphization voltage against the minimum width in 40 crystalline Zr NSWs having the widths of 2–16 nm. For the fracture-less amorphization as shown in figures 1 and 3, the amorphization occurs at pulse voltages of $0.10 \pm 0.05$ V when the cross-sectional width is less than 12 nm. The amorphization voltage increases to higher than $1.10 \pm 0.05$ V when the width becomes wider than this value. For the amorphization of breaking nanotips, the amorphization voltage becomes higher than that of the non-destructive cases of the same width.

Figure 10 shows the crystallization voltages against the minimum width in 21 amorphous Zr NSWs having the widths of 2–16 nm. The crystallization occurs at pulse voltages of $0.10–0.30 \pm 0.05$ V when the width is less than 9 nm. As the width becomes larger than this value, the crystallization voltage increases to higher than $0.70 \pm 0.05$ V.

We selected the minimum width as the argument of the pulse voltage of the amorphization and crystallization. However, when the weights of the other shape factors contributing to the cooling rate increase, the state after melting, i.e., crystal or amorphous, is not governed by only the pulse voltage, as shown in figures 9 and 10; the energization of the same or similar pulse voltages resulted in both the amorphization and crystallization. When a NSW exhibits similar shapes, the prolongation of pulse energization resulted in the crystallization, as exemplified by figures 1 and 3.

4. Discussion

4.1. The amorphization

It is known that the amorphous phases of pure Zr was derived by slow cooling of liquid phases at high pressures [5]. However, in this study, the amorphization of pure Zr NSWs were observed in a vacuum (figures 1 and 3). Thus, the pulse-voltage impression, i.e., rapid cooling, brings about the amorphous pure Zr not at high pressures, and the amorphous phase was stable at ambient temperature. The amorphization via pulse-voltage
Figure 3. Lattice images of the expansion of an amorphized region in a Zr NSW due to the multi-time impression of pulse voltages (a)–(c) and subsequent crystallization due to one pulse impression (d) (see Movie 2 in the supplementary data). (a) Initial state of the crystalline NSW. (b) The NSW in which the lower side was amorphized after the impression of three pulse waves of 4 ns in width and 0.30 ± 0.05 V in height. (c) The NSW in which the total region was amorphized after the impression of 24 pulse voltages of 4 ns in width and 0.30 ± 0.05 V in height. (d) The crystallized NSW after the impression of a pulse wave of 5 ms in width and 0.30 ± 0.05 V in height. The NSW was supported by a positive electrode (nanotip) on the lower side (+) and a negative electrode (nanotip) on the upper side of the figure (−). The insertion in the upper right corner in parts from a to d is the Fourier transform pattern of the thinnest region indicated by the white frame.
impression occurs through melting due to Joule-heating and subsequent rapid cooling via thermal transfer through the two nanotips beside the NSW, i.e., semi-infinite thermal sinks [19, 20, 25, 37, 51–54]. The critical cooling speed for amorphization in Zr is estimated from molecular dynamics simulation to be $5.0 \times 10^{13}$ K s$^{-1}$ [42]. In this study, the damping time of the pulse voltages was approximately 3 ns. When we assume that the wire temperature decreases from over the melting temperature to ambient temperature in this period, the cooling

![Figure 4](image-url). Schematic of the NSW in figure 3. The wire part is crystal (a) and amorphous (c). The transition state from crystal to amorphous (b).
speed is estimated to be larger than approximately $10^{12}$ K s$^{-1}$, similar to the critical value. It is inferred that the NSW structure, i.e., an ultimate localized heating region connecting with semi-infinite thermal sinks beside it, realizes the critical rapid cooling for amorphization in pure Zr.
4.2. The gradual expansion of amorphized regions

The gradual expansion of the amorphized region is also observed in molybdenum (Mo) NSWs having widths of several tens nanometers, which is larger than that in this study [19]. Thus, gradual amorphization occurs in smaller regions in Zr. During the expansion, the entire thinnest region melted at each impression of a pulse voltage, and larger amorphous region was formed (figure 3). The cooling speed depends on the shape of NSWs, and the shape barely changed via the impression of shorter pulse voltages. When the cooling speed is similar, the larger the melting region increases, the larger the amorphized region expands. Higher heating energy is required to melt larger regions. The conductance per a unit area at the amorphous states is lower than that at the crystalline states; the amorphization does not contribute to the increase in heating energy, i.e., the expansion of the amorphized region. In contrast, as shown in figures 2 and 3, the minimum width, i.e., the conductance increased; Joule heating became intensified. Thus, the increase in the width at every pulse-voltage impression leads to the general expansion of the amorphized region. The expansion of the width becomes possible due to,

Figure 7. Lattice images of the fracture of a Zr NSW and the amorphization of separating nanotips due to the impression of one pulse wave of 4 ns in width and 1.90 ± 0.05 V in height. (a) Before and (b) after the one-pulse impression. The NSW was supported by a positive electrode (nanotip) on the left side (+) and a negative electrode (nanotip) on the right sides of the figure (−). The insertion in the upper right corner in parts a and b is the Fourier transform pattern of the region indicated by the white frame in the positive electrode.

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Figure 8. Ratios of the conductance per a unit area of the crystalline to amorphous states ($\rho_{\text{amo}}/\rho_{\text{cry}}$) in 14 Zr NSWs having the thinnest wire widths of 2–12 nm. The broken line indicates the average ratio (0.87).

Figure 9. Amorphization voltage against the thinnest width in 40 crystalline Zr NSWs. The filled red circles and unfilled orange circles indicate the amorphization due to one-time and multi-time impression using the 4 ns-width pulses, respectively. The brown crosses indicate the amorphization of the separated nanotips after fracture due to one-pulse impression.

Figure 10. Crystallization voltage against the thinnest width in 21 amorphous Zr NSWs. The unfilled blue triangles, the unfilled purple rhombuses, the unfilled green squares, the filled green square, filled blue circles, and unfilled right blue circles indicate the crystallization due to multi-time impression using the 4 ns-width pulse voltages, one-time impression using the 10 ns-width pulse voltage, one-time impression using the 1 ms-width pulse voltage, multi-time impression using the 1 ms-width pulse voltages, one-time impression using the 5 ms-width pulse voltage, and multi-time impression using the 5 ms-width pulse voltages, respectively.
for example, the neck growth of the constriction region. When the shape and the state, e.g., at compressive states or active electromigration states, of the constriction region are favorable for the atomic diffusion and condensation for neck growth at melting, the amorphization region persists to expand.

4.3. The crystallization
In NSWs of Mo, hafnium (Hf), and tungsten (W), the crystallization voltages of pulse waves are lower than their amorphization voltages; the crystallization occurs by weaker energization [19, 21–23]. In contrast, in tantalum (Ta) NSWs, the crystallization and amorphization occur via the same energization, i.e., the energization using pulse voltages of the same width and the same height [24]. In the present Zr NSWs, the amorphization and crystallization occurred via the impression of the shorter (4 ns) and longer (5 ms) pulse voltages of the same height (0.30 ± 0.05 V) (figure 3). We discussed this as follows.

The impression of the longer pulse voltages leads to the increase in the temperature and the expansion of the heating region of NSWs. In this study, for NSWs having similar shapes, the prolongation of pulse energization resulted in the crystallization. The cooling speed in pulse-voltage impression decreases when the nanotips beside a NSW are heated and thermal transfer becomes slow. The Joule heat due to pulse-voltage impression depends on the conductance of NSWs and energizing time. The conductance is determined by the wire shape [55]. As shown in figure 3, the conductance during the impression of the shorter (4 ns) and longer (5 ms) pulse voltages was similar (1.33–1.37 × 10⁻³ S). Hence, the Joule heat becomes larger as the pulse width increases, resulting in the increment in the temperature of the nanotips. Thus, it is inferred that the cooling speed for the longer pulse voltage decreased and the crystallization occurred at the same voltage as the amorphization voltage.

4.4. The amorphization and crystallization voltages
The amorphization voltage was 0.3 V for Zr NSWs having wire widths up to approximately 12 nm and increased with the width for wider NSWs (figure 9). Similarly, the crystallization voltage was the same voltage for the NSWs having the widths up to approximately 10 nm and increased with the width for larger NSWs (figure 10). In the amorphization, NSWs once melt; the temperature needs to increase to higher than melting temperatures. For the crystallization, the temperature of NSWs also needs to rise at least to crystallization temperatures. In particular, in Zr NSWs, the energization comparable to the amorphization is needed to cause the crystallization as discussed in 4.3. The amount of the Joule heat to cause such transitions increases with the wire width. Thus, the wire temperature reaches such transition temperatures via the impression of 0.3 ± 0.05 V for the widths less than 10–12 nm, and larger pulse voltages for thicker Zr NSWs. When a critical pulse voltage for phase transition is found for a certain wire width, the amorphization and crystallization are controllable by the selection of the pulse width, as discussed in 4.3.

4.5. The amorphization of nanotips formed after the fracture of Zr NSWs
The amorphization of the broken surfaces (figures 7 and 9) is also observed in W NSWs [19, 23]. In Zr NSWs, the amorphization voltages for broken nanotips are higher than that for fracture-less amorphization (figure 9). The temperature of the thinnest region increases with pulse voltage. In addition, the pulse current stops promptly at fracture. As a result, faster cooling occurs at fracture in comparison with that at fracture-less cooling, which cooling speed is determined by the damping speed of the pulse voltage. Thus, it is deduced that the amorphous phases at the nanotip edges were formed at faster cooling rather than that for fracture-less amorphization, leading to higher degree of amorphization in the nanotips.

4.6. The conductance control of Zr NSWs via pulse-voltage impression
The average ratio of the conductance per a unit area at the initial crystalline states to that at the amorphized states was estimated to be 0.87 (figure 8). This value is similar to the ratio for W and Ta NSWs (0.82 and 0.85, respectively) and larger than that for Mo and Hf NSWs (0.62 and 0.72, respectively) [21–24]. In this study, the amorphized region expanded gradually for every pulse-voltage impression (figure 3), and the conductance of the NSWs decreased stepwise (figures 5 and 6). Thus, it was demonstrated that the conductance can be controlled stepwise via pulse-voltage impression. Also note that the amorphous phases were stable at ambient temperature. These results imply that Zr NSWs are applicable to nanometer-sized functional devices, such as non-volatile phase-change random access memories, switches, and other memory nanodevices [32–34, 56, 57]. In particular, the conductive filaments formed via the ordered arrangement of clusters of lead sulfide and silver (Ag) are employed to achieve enhanced memristor performance for mimicking biosynaptic behaviors [33, 34]. For the conductive Ag filaments, the applied pulses with a width of hundreds of nanoseconds are beneficial to realize fast learning and computing [33], whereas for the present Zr NSWs, the conductance control was performed using the pulses of 4 ns–1 ms widths (figure 9). The amorphization voltage was 0.20 ± 0.05 V for the NSWs having 8 nm width, much smaller than that for NSWs comprised of Ta, Mo, W, and Hf, in which amorphization were
confirmed (0.5–2.5 V) [21, 22, 24, 49]. In addition, the volume resistivity of Zr is the highest among the metals used for amorphous NSWs [58–61]. These features contribute to the reduction of energy consumption and resultant higher integration when Zr NSWs are applied to functional devices [26, 56].

5. Conclusion

The structural dynamics of Zr NSWs during pulse-voltage impression was observed in situ by lattice imaging, and simultaneously, the variation in conductance was measured. The pulse-voltage impression caused the reversible phase transition between the crystalline and amorphous phases. The average ratio of the conductance per a unit area at the crystalline states to that at amorphized states was estimated to be 0.87. The amorphized region expanded gradually by every pulse-voltage impression, and the conductance decreased stepwise. It was demonstrated that the conductance of the NSWs can be controlled in a stepwise manner via pulse-voltage impression, leading to the application of Zr NSWs to nanometer-sized functional devices, e.g., non-volatile phase-change random access memories and switches, which enables the reduction of energy consumption and resultant higher integration. In particular, since Zr NSWs exhibited no precipitation during cycles of the fast phase transitions under certain operation conditions, the NSWs are expected to become degradation-free nanometer-sized functional devices.

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