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Direct Observation of Microstructure Changes Arising from Electromigration

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1. Introduction

Responding to the needs of the times, various nanofabrication methods other than presently used lithography have been proposed, *e.g.* scanning probe microscopy based lithography [1] and shadow evaporation [2]. The break junction process of metallic narrow patterns by electromigration [2-4] is one of such methods. Though well controlled current flow has a possibility to realize reproducible formation of nanostructures [5-8], the method reported so far is more or less empirical without dynamical structural observations. For further development of this method as a fabrication method in nanometer scale, details on microstructure during the electromigration process should be investigated.

In this work, we selected Au as a test material, and observed and analyzed the rupture process of nanowires by transmission electron microscopy (TEM). For dynamic observation during the electromigration process, *in-situ* TEM was used where the current measurements are simultaneously performed together with the observation of the structural change of the wire.

2. Experimental details

Device patterns were made by using photo lithography and the lift-off process on Si-N (25 nm) /Si (100) substrates. First, electrode pads (Au/Cr) were prepared. Afterwards, wires of 35 nm thick Au was deposited. Typical size was 5 x 10 μm^2 (width x length). Finally, windows for TEM observations (100 μm square) were formed from the backside by using a KOH (25 wt.%) aqueous solution at 80 ^{o}C, and a self-standing Si-N membrane with Au patterns was obtained. The sample was set in a custom-made TEM holder with which the electric property can be measured during the TEM observation [9]. The TEM instrument used was a JEM-2010 microscope with a CCD video camera. Electric measurements (constant voltage mode) were performed by using a Yokogawa GS610 source measure unit.

3. Results and discussion

The initial resistance was about 60 Ω. By applying voltage, the current increased to the maximum value (1st stage), and grains merged together reducing the number of grain boundary while mazy gaps between grains expanded [10]. Afterwards, it gradually decreased (2nd stage) due to growth of mazy gaps to voids giving complicated current paths. Voids grew by the current flow, and the paths became narrower. The narrow paths ruptured one by one, and the current dropped to zero when all paths were destructed (3rd stage). In the followings, change of narrow wires between the 2nd and the 3rd stages were investigated by means of *in-situ* TEM.

A typical example of the narrowing process is presented in Fig. 1. The corresponding point is marked by triangles. At the start of the observation (Fig. 1a), the narrowest wire width was more than 50 nm. By continuing the current flow, the wire width was reduced and rearrangement and/or recrystallization of grains occurred (Figs. 1b-c). In Fig. 1d, the narrowest width of about 10 nm was formed at a grain boundary. To analyze the narrowing rate, the wire region in TEM images was divided

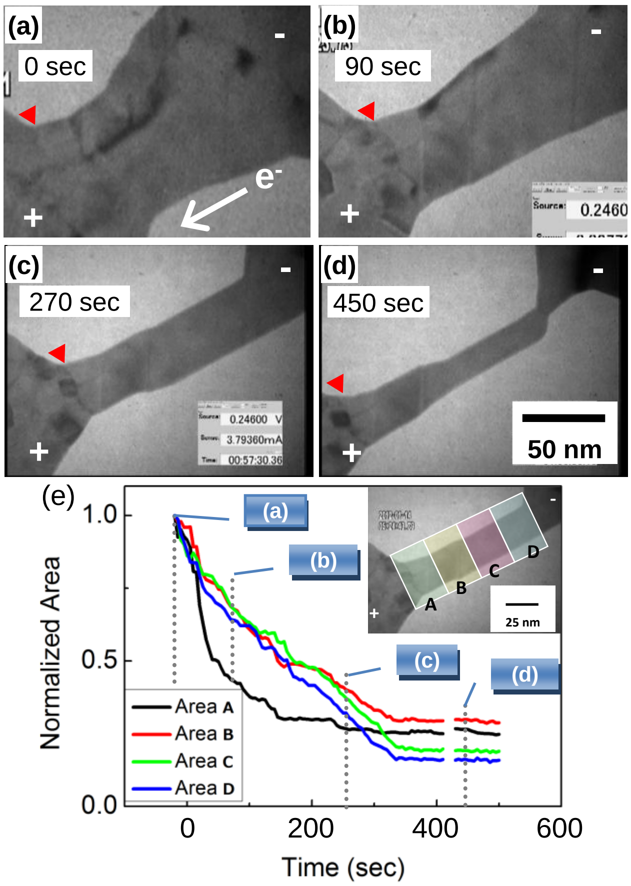

Fig. 1 (a)-(d): a series of TEM images of Au nanowire (black contrast area) during the continuous current flow (voltage: 246 mV). Red triangles on each images pointed the same grain boundary. (e): normalized area as a function of time.

into four parts (Fig. 1e), and the area of each part was measured as a function of time. The area values normalized by those at t = 0 s are summarized in Fig. 1e. The rate was not constant but fluctuating. Especially, at the time larger than 300 s, the wire narrowing was almost stopped. In this time range, the wire width was less than 20 nm which is smaller than the grain size at the nanowire. Electromigration is known to occur by several diffusion mechanisms at the surface, in the grain and along the grain boundary. In Fig. 1a, many grains constitute the nanowire forming a "stone wall" type grain boundary structure [11]. In this case, gold atoms are thought to move along grain boundaries parallel to the current flow. In Fig. 1c-d, nanowire was composed of almost single grain connecting side by side (so-called the "bamboo" type structure) [12]. In this case, the atom movement along grain boundaries is believed to be suppressed, and thus the narrowing rate becomes low [12]. In this work, this expectation was clearly confirmed by *in-situ* TEM that electromigration is much repressed when the wire region was composed of a single crystal.

To investigate whether the above mentioned phenomenon was caused by electromigration, the experiment with polarity alternation of electron flow was performed (Fig. 2). Electromigration may introduce alternation of structural change, while such phenomenon should not occur by thermal effect. Positive bias applied to the right-bottom side of the wire (Fig. 2a) resulted in wire narrowing as described above (Fig. 2b). By polarity alternation between Figs. 2c and 2d, reduction and gain in area occurred at negative and positive voltage side, respectively. This corresponds to upper and lower side of the electron stream. This is clearly recognized by comparing with dotted line indicating the contrast edge of Fig. 2b. Further polarity alternation between Figs. 2d and 2e showed the same tendency. The positions with area reduction and gain were inverted, and the wire became narrow (fig. 2f). The series of result in Fig. 2 can be a proof that the wire narrowing occurred in this work was caused by electromigration.

By continuing the voltage application (or current flow), the wire is ruptured and a nanogap is formed. An example is shown in Fig. 3. The nanowire with about 3 nm in width (Fig. 3a) was further constricted near the grain boundary indicated by dotted curves (Fig. 3b), and finally ruptured forming a nanogap (Fig. 3c). The nanogap had a tendency to be formed in the vicinity of a grain boundary.

4. Conclusion

Wire narrowing which appertain to electromigration was dynamically investigated by using *in-situ* TEM. All of the results pointed out that the crystal growth and grain boundary arrangement during the current flow must be important factor to obtain well qualified nanogaps.

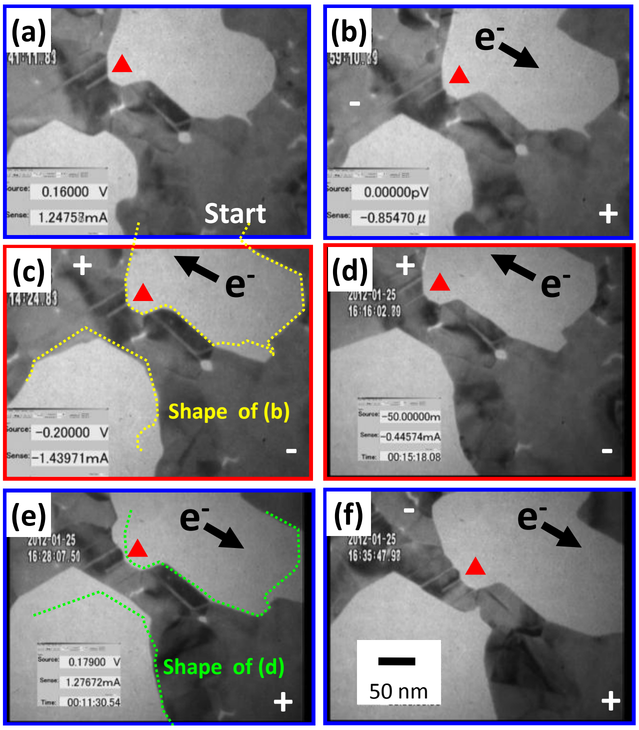

Fig. 2 (a)-(f): geometrical change during alternation of the current flow. The arrows indicate the flow of electron. Red triangles denote the corresponding position. Yellow dotted line in (c) indicated the shape of nanowire in (b), and Green dotted line in (e) indicated that in (d).

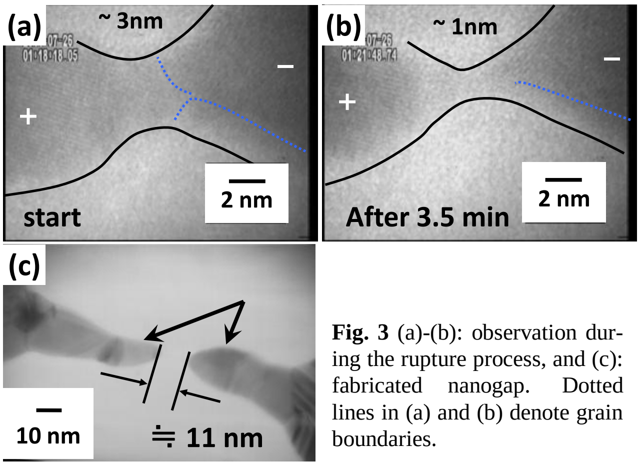

Fig. 3 (a)-(b): observation during the rupture process, and (c): fabricated nanogap. Dotted lines in (a) and (b) denote grain boundaries.

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