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Picosecond Ultrasonic Spectroscopy for Cu Nanowires and Their Softening During Electromigration

ピコ秒超音波法によるCuナノワイヤの共振計測とエレクトロマイグレーション軟化の観測

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1. Introduction

Nanowires are used in various electromechanical devices as electric lines. Because of advances in fabrication technologies for nanostructures, wire thickness and width are significantly made smaller, and such a miniaturization has produced the problem of the difficulty of evaluating their. Typical failure mode is caused by electromigration (EM), which is attributed to a diffusion phenomenon of constitution atoms due to passing high-density current, by which vacancy flow is accompanied, resulting in the failure. Deterioration of the nanowires progresses with increases of current density and temperature[1].

To evaluate the reliability of nanowires, accelerated current-loading tests were conducted under high temperature and high current density. From these tests, the activation energy for vacancy migration was estimated, which predicts the possible limitation of the current density and temperature, below which the electric devices should be designed. These current tests however cannot provide the microstructure change, especially in earlier stages. In this study, we reveal that the stiffness of nanowires can be significantly lowered even for a low current density, with which the EM failure would not occur.

We take notice of mechanical resonance frequencies of nanowires, and monitor them along with the current loading tests using picosecond ultrasound spectroscopy[2, 3]. Resonance frequencies are sensitive to shapes, mass density, elasticity and boundary condition. Vacancies are diffused and accumulated on the grain boundaries to form thin defects, leading to the decrease in elastic constant. Therefore, we can evaluate evolution of defects in nanowires through resonance frequencies. Thus, we propose a novel characterization methodology of nanowires through the stiffness monitoring.

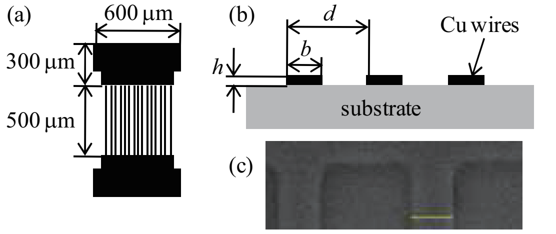

Fig. 1.(a) Schematic of the nanowire specimen. (b) Cross-section image of the specimen. (c) SEM image of the specimen. The bar indicates 300 nm.

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2. Experiments

We fabricated Cu nanowire specimen on (100) Si and glass substrate using the electronbeam lithography method. **Fig. 1(a)** shows the schematic of the nanowire specimen, and **Figs. 1(b)** and **(c)** show the cross-section and a scanning-electron-micrograph image of the specimen, respectively. The specimen consists of two flat regions on which aluminum wires are attached for the current loading tests, and 499 nanowires with 500 μm long.

We use the pump-probe picosecond ultrasound technique for performing the mechanical spectroscopic measurement for the nanowires. Irradiation of the specimen with the ultrafast light pulse (800 nm) excites coherent acoustic sources and generates vibrations related with the nanowires, including the thickness resonance inside nanowires, Rayleigh-wave resonance on the substrate surface, and Brillouin oscillations[4, 5] as shown later. They are detected by the delayed probe light pulse (400 nm) through the change in its reflectivity[2, 3, 6].

In addition, to cause EM in the nanowires, we apply the current pulses to the nanowire specimen with the duration of 1 μs and the current density of about 1 MA/cm^2, and monitored the changes of the resonance frequencies.

3. Results and Discussion

Fig. 2(a) shows an example of the time-resolved reflectivity change observed for a specimen on Si substrate with h=30 nm, b=300 nm and d=1000 nm. **Figs. 2(b)** and **(c)** are corresponding to FFT spectra, showing three kinds of peak frequencies near 2-10, 70, and 235 GHz. The highest frequency peak is identified to be Brillouin oscillation, which is caused by interference between the surface reflected probe light and that refracted by the longitudinal wave

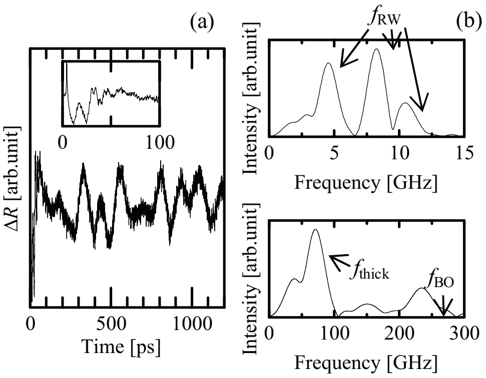

Fig. 2.(a) Time resolved reflectivity change observed for nanowires on Si substrate, and (b) their FFT spectra.

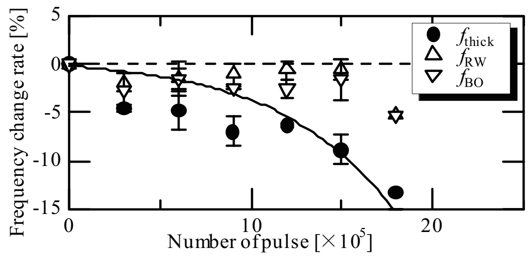

Fig. 3. Relationship between resonance frequencies and the number of pulse.

propagating in the Si substrate. Its theoretical frequency is given by $f_{BO}=2nv/\lambda$, where n, v, and λ denote the refractive index of the probe light in Si, longitudinal-wave velocity in Si, and the wavelength of the probe light, respectively. Handbook values for them predicts f_{BO}=235 GHz, showing good agreement with the experiment. We attribute the lower frequency peaks denoted by f_{RW} to the Rayleigh-wave resonances. Nanowires will vibrate coherently with the excitation by the light pump pulse, and they can be sources of Rayleigh waves. When the wavelength of the Rayleigh wave is close to the spacing d of the nanowires, significant amplitude enhancement occurs. Thus, the frequency of the Rayleigh-wave resonance is roughly estimated by $f_{RW}=n_R v_{RW}/d$, where n_R is the resonance order number and v_{RW} is the Rayleigh-wave velocity of Si. Using v_{RW}=5000 m/s, we have f_{RW}=5 GHz for n_R=1. In the case where nanostructures are periodically deposited on substrate, surface wave propagating on the substrate has a dispersion relation and observed surface wave velocity is lower than theoretical value because of the periodic additional mass. Thus, it is considered that three low-frequency peaks correspond to the Rayleigh–wave resonances for n_R=1, 2, and 3. The peak near 70 GHz is considered to be the through-thickness resonance of nanowires. Because of the large aspect ratio of the wire cross-section, the deformation in the thickness direction is governed by the longitudinal-wave modulus, $C_\perp$. Higher acoustic impedance of copper than that of the substrate allows assumption of the free boundary reflection at the substrate interface. Thus, the fundamental thickness resonance frequency is given by $f_{thick}=v/2h$, where $v=(C_\perp/\rho)^{1/2}$ and ρ denotes the mass density of the nanowires. Using $C_\perp$=198 GPa for polycrystalline copper, we have f_{thick}=78 GPa. The stiffness of deposited copper is usually lower than of the bulk by 10-20%[7], and the observed frequency near 70 GHz can be explained by the thickness resonance inside the nanowire.

Fig. 3 shows relationships between frequency changes and the number of applied current pulse. The pulse period was 100 Hz and pulse duration was 1 μs. As the number of pulse increases, f_{thick} significantly decreases by 15% from the initial value, while f_{BO} and f_{RW} remain nearly unchanged. These observations indicate two important factors. First, the nanowires do not show detachment from the substrate, because f_{BO} and f_{RW} can be observed when nanowires are tightly attached on the substrate. Second, the stiffness of the nanowire is significantly lowered by the current loading test. Because the elastic constant is proportional to the square of the resonance frequency, the decrease of f_{thick} in Fig. 3 suggests the decrease of $C_\perp$ by 30%. We attribute such a drastic stiffness decrease to the vacancy diffusion and formation of thin defects.

Generally, EM tests were conducted under the current density of the order of 10 MA/cm^2 at 200-300°C[8]. However, in the present study, we used the current density of ~3 MA/cm^2 at 20°C, which would not cause the EM failure. However, the resonance frequency decreased significantly, indicating softening of nanowires. We then consider that the macroscopic softening is caused by large aspect-ratio defects grown by vacancy diffusion and their accumulation at grain boundaries. There are many vacancies in the as-deposited nanostructures, and they are diffused predominantly along boundaries due to low diffusivity there by the applied electric field. At the crystal grain boundary, vacancies gather together and combine to form a thin defect. The macroscopic stiffness is highly lowered with the presence of the thin defects, even when their volume fraction is significantly smaller[9].

Thus, we discovered significant softening of nanowires in the early stage of EM, and the stiffness monitoring can be a powerful tool for evaluating reliability of nanostructures.

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