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# Ni thin films vacuum-evaporated on polyethylene naphthalate substrates with and without the application of magnetic field

Hideo Kaiju<sup>a,\*</sup>, Akito Ono<sup>a</sup>, Nobuyoshi Kawaguchi<sup>a</sup>, Kenji Kondo<sup>a</sup>, Akira Ishibashi<sup>a</sup>, Jonghan Won<sup>b</sup>, Akihiko Hirata<sup>b</sup>, Manabu Ishimaru<sup>b</sup>, Yoshihiko Hirotsu<sup>b</sup>

<sup>a</sup> Research Institute for Electronic Science, Hokkaido University, Sapporo, Hokkaido 060-0812, Japan
<sup>b</sup> The Institute of Scientific and Industrial Research, Osaka University, Ibaraki, Osaka 567-0047, Japan

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## 1. Introduction

Fabrication of nanoscale patterns with the sub-10-nm feature size has been an important research target for potential applications in the next-generation memories, microprocessors, logic devices, and other novel functional devices. Typically, the International Technology Roadmap for Semiconductor (ITRS) in 2007 notes that an 11-nm node device is targeted for the year 2022 [1]. To achieve this milestone, optical lithography, such as ultraviolet (UV) optical lithography with immersion and extreme ultraviolet (EUV) optical lithography, is expected as the most commonly used technique for the fabrication of nanopatterns [2,3]. UV optical lithography using short wavelengths of 193 nm coupled with immersion to reduce the effective wavelength has demonstrated that 32-nm features can be patterned [2]. EUV optical lithography using a wavelength of 13.5 nm enabled the printing of nearly 27-nm half-pitch lines [3]. On the other hand,

#### ABSTRACT

We study the structural properties of the surface roughness, the surface mound size and the interfacial structure in Ni thin films vacuum-deposited on polyethylene naphthalate (PEN) organic substrates with and without the application of magnetic field and discuss its feasibility of fabricating quantum cross (QC) devices. For Ni/PEN evaporated without the magnetic field, the surface roughness decreases from 1.3 nm to 0.69 nm and the surface mound size increases from 32 nm to 80 nm with the thickness increased to 41 nm. In contrast, for Ni/PEN evaporated in the magnetic field of 360 Oe, the surface roughness tends to slightly decrease from 1.3 nm to 1.1 nm and the surface mound size shows the almost constant value of 28–30 nm with the thickness increased to 35 nm. It can be also confirmed for each sample that there is no diffusion of Ni into the PEN layer, resulting in clear Ni/PEN interface and smooth Ni surface. Therefore, these experimental results indicate that Ni/PEN films can be expected as metal/insulator hybrid materials in QC devices, leading to novel high-density memory devices.

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attractive patterning techniques, such as multi-step nanoimprint lithography (NIL) and mold-to-mold cross imprint (MTMCI) process, are currently proposed and pursued actively as an alternative patterning method [4–6]. A multi-step NIL using a mold produced by the spatial frequency doubling method has achieved the production of 17-nm line width patterning [4,5]. MTMCI process using silicon nanowires formed by spacer lithography, in which nanoscale line features are defined by the residual of a conformal film on the edges of a sacrificial support structure with the line width controlled by the film thickness, has produced a large array of 30 nm wide silicon nanopillars [6].

Recently, we have proposed a double nano-baumkuchen (DNB) structure, composed of two thin slices of alternating metal/ insulator nano-baumkuchen as a lithography-free nanostructure fabrication technology [7,8]. The schematic illustration of the fabrication procedure is shown in Fig. 1. First, the metal/insulator (organic films) spiral heterostructure is fabricated using a vacuum evaporator including a film-rolled-up system. Then, two thin slices of the metal/insulator nano-baumkuchen are cut out from the metal/insulator spiral heterostructure. Finally, the two thin slices are attached together face to face so that each stripe is crossing in a





<sup>\*</sup> Corresponding author. Tel.: +81 11 706 2878; fax: +81 11 706 2883. *E-mail address:* kaiju@es.hokudai.ac.jp (H. Kaiju).

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Fig. 1. Fabrication procedure of a double nano-baumkuchen (DNB) structure and a quantum cross (QC) device.

highly clean environment [9,10]. The DNB has potential application in high-density memory devices, the cross-point of which can be scaled down to ultimate feature sizes of a few nanometers thanks to the film thickness determined by the metal-deposition rate, ranging from 0.01 nm/s to 1 nm/s. One element of the DNB structure is called a quantum cross (QC) device that consists of two metal nanoribbons having the edge-to-edge configuration shown in Fig. 1 [8,11–13]. When molecular-based self-assembled monolayers (SAMs), such as rotaxanes [14], catenanes [15], and pseudorotaxanes [16], are sandwiched between the two metal ribbons, it can serve as a novel electronically switchable highdensity memory device [17,18]. When ferromagnetic materials with the induced uniaxial magnetic anisotropy are used for the two metal ribbons, a nanoscale spin injector can be also produced. Its device leads to novel multi-value memory devices.

In our previous work, metal thin films on organic substrates have been studied toward the fabrication of QC devices and Ni thin films on PEN substrates have potential application in metal/ insulator hybrid materials for use in QC devices from the viewpoint of its surface smoothness [13]. However, the mechanism of surface smoothness, and other detailed structural properties, such as surface mound size and Ni/PEN interfacial structures including the interdiffusion or not of Ni into the PEN substrates, have not been clarified. Furthermore, although it is required that Ni thin films are evaporated in the magnetic field for QC devices working as spin injectors, Ni thin films evaporated in the magnetic field have not been investigated. In this paper, we have focused on the detailed structural properties of surface smoothness, surface mound size, and Ni/PEN interfacial structures in Ni thin films evaporated on PEN substrates with and without the application of magnetic field and discussed its feasibility of fabricating QC devices.

## 2. Experiments

Ni thin films were thermally evaporated on PEN substrates (2 mm width and 100  $\mu$ m thickness) without and with the inplane magnetic field of 360 Oe, respectively, in a high vacuum chamber at the base pressure of  $\sim 10^{-8}$  Torr. PEN films TEONEX Q65 were supplied by Teijin DuPont Japan, and cut down from 5 mm to 2 mm width by using the slitter with the film-rolling-up system in a clean environment. A boron nitride crucible N-1 made by DENKA and a tungsten filament made by CRAFT were used for the thermal evaporation of Ni thin films. The pressure during the evaporation was  $10^{-5}$  Torr and the temperature near PEN substrates was less than 62 °C, which was lower than the glass transition temperature  $T_g$  of 120 °C for PEN substrates. The growth rate was 0.93 nm/min at the evaporation power of 280 W. The Ni

thickness was measured by a mechanical method using the stylus surface profiler DEKTAK and an optical method using the diode pumped solid state (DPSS) green laser and the photo diode detector. The surface morphologies of samples were analyzed by atomic force microscope (AFM) Nanoscope IIIa. The analysis of the surface roughness and surface mound size, based on AFM images, were performed by the image analysis software Gwyddion. The microstructures as well as the Ni/PEN interfacial structures were examined using a JEOL JEM-3000F transmission electron microscope (TEM) operating at 300 kV. The cross-sectional TEM samples were prepared by a combination of mechanical polishing and Ar ion thinning. To reduce the beam-heating effects during ion thinning, the sample stage was cooled to -160 °C by liquid nitrogen conduction cooling.

## 3. Results and discussion

Fig. 2 shows the typical one-dimensional (1D) height profiles and three-dimensional (3D) surface images obtained from AFM observation for PEN and Ni/PEN evaporated without the application of magnetic field  $H_a$ . From 3D images, which are 1  $\mu$ m  $\times$  1  $\mu$ m in area, mound-like surfaces are observed in all the images and characterized by the surface roughness and the surface mound size. Here, the surface roughness R<sub>a</sub> is defined by  $R_{\rm a} = (1/L_x L_y) \int_0^{L_x} \int_0^{L_y} |h(x,y)| \, dx \, dy$ , where h(x,y) is the height profile as a function of x and y and  $L_{x(y)}$  is the scanning lateral size in the x(y)-direction. The surface roughnesses  $R_a$ 's are 1.13 nm, 1.08 nm, and 0.69 nm for the Ni thickness t of 6.3 nm, 17 nm and 41 nm, respectively. The surface mound sizes  $l_{\rm m}$ 's are 31.1 nm, 28.8 nm, and 79.0 nm, respectively. As plotted in Fig. 3, it is found that the surface roughness  $R_a$  decreases and the surface mound size  $l_m$ increases with increasing the Ni thickness t. In order to investigate the reason of the surface smoothness and the mound-size enlargement, we performed the electron diffraction analyses based on the cross-sectional TEM observation. As shown in Fig. 4, a ring pattern due to Ni with an fcc structure has been obtained. The electron diffraction pattern of Fig. 4 was obtained with a beam size of  $\sim$ 50 nm, and the ring patterns exhibit the formation of the fine-grained deposit with smaller than 5-nm size. This ring pattern has not been seen in the samples evaporated with the application of magnetic field, which is shown later in this paper. In the case of fine-grained deposit, the Ni grains are packed with little space and they can improve the surface smoothness. Under this condition, the smoothness and mound-size enlargement of Ni film surface have been enhanced due to a high mobility of adatoms and a low rate of deposition growth. The vapor Ni atoms parallel to the surface can be expected to move easily and

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