Realization of aligned three-dimensional single-crystal chromium nanostructures by thermal evaporation

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Abstract Aligned three-dimensional single-crystal chromium nanostructures are fabricated onto a silicon substrate by thermal evaporation in a conventional thermal evaporator, where the incident angle of Cr vapor flux with respect to the substrate surface normal is fixed at 88°. The effects of the deposition time and incident angle on the morphology of the resulting nanostructures are investigated. The achieved Cr nanostructures are characterized by scanning electron microscopy, energy dispersive X-ray analysis, X-ray diffraction, transmission electron microscopy, high-resolution transmission electron microscopy, and surface area measurement. This study provides a convenient way to fabricate three-dimensional single-crystal Cr nanostructures, which is suitable for batch fabrication and mass production. Finally, the same technique is employed to fabricate the nanostructures of other metals such as Ag, Au, Pd, and Ni.

1 Introduction

Nanoscale metals are being extensively investigated because of their promising applications and improved properties compared to their bulk or micro counterparts [1, 2]. Chromium, an interesting transition metal, has found various applications in many fields including metallurgy, catalysis, magnetic field, and semiconductors [3–10]. For instance, Cr is a commercially available transition metal that adheres well to silicon substrates, which makes Cr being widely used in silicon based semiconductors both in research and in industry. Cr thin films [5–7] and nanoparticles [8–10] have been intensively studied, and improved catalytic and magnetic performances have been found compared to those for bulk Cr. Although Cr thin films and nanoparticles have been extensively investigated, there are very few studies on two-dimensional (2D) and three-dimensional (3D) Cr nanostructures. It is widely accepted that 2D/3D nanostructures are important components for nanoscale devices with many promising applications, due to their large surface areas and other unique properties [11–15]. Consequently, it will be very interesting to synthesize 2D/3D Cr nanostructures.

In this work, a convenient way is presented to synthesize aligned 3D single-crystal Cr nanostructures onto a silicon substrate using thermal evaporation in a conventional thermal evaporator. The experimental processes are first described. The effects of the deposition time and incident angle on the morphology of the resulting nanostructures are studied by scanning electron microscopy (SEM). The synthesized 3D Cr nanostructures are also characterized by energy dispersive X-ray analysis (EDX), X-ray diffraction (XRD), transmission electron microscopy (TEM), high-resolution transmission electron microscopy (HRTEM), and surface area measurement. The same technique is employed to realize other metal nanostructures as well.

2 Experiment

The 3D nanostructures of Cr are synthesized by a VEECO 770 thermal evaporating system using 99.99% pure Cr pellets as the source material. An alumina crucible is used as the source holder, and a tungsten filament is employed to
thermally heat the source holder. Half of a 4-inch p-type single-crystal silicon wafer is used as the substrate that is cleaned using acetone, chromic sulfuric acid mixture, thoroughly rinsed by deionized water and blow dried by nitrogen. Then the silicon substrate is put into an oven for further drying at 200°C for 20 min. The silicon substrate is mounted into a specific movable holder inside the evaporation chamber. The incident angle of Cr vapor flux with respect to the silicon substrate surface normal is fixed at 88°. The distance between the top of the substrate and the source holder is around 18 cm. The Cr is evaporated with a constant supply current of 23 A and a vacuum level of $7 \times 10^{-6}$ mbar. The deposition rate is set to be 5 Å/s, and the temperature of the substrate is about 45°C during the thermal evaporation. The deposited Cr nanostructures on the substrate are directly characterized by SEM and EDX. For XRD characterization, the Cr nanostructures are scratched from the substrate to avoid the effect of the well-crystallized silicon substrate. XRD patterns are recorded on a Bruker D4-Endeavor diffractometer using Cu Kα radiation (40 kV, 40 mA) from 20 to 100° in 2-Theta with a 0.016° step scan, a real-time/step of 0.13 s on a linear Bruker LynxEye detector, and 30 rotation/min. For TEM and HRTEM characterizations, the Cr nanostructures are manually scratched from the silicon substrate, mixed with ethanol, and deposited onto carbon-coated copper grids. TEM images and electron diffraction patterns are obtained on a JEOL 2010 microscope running at 200 kV. HRTEM images are taken with a JEOL 2100F microscope operating at 200 kV.

3 Results

3.1 SEM characterization

Figure 1(a) is a top view SEM image of the Cr nanostructures, and the deposition time is set as 400 s. Most of the Cr nanostructures become bigger and bigger as they grow from the substrate surface. Figures 1(b) and (c) are cross-section view SEM images of the Cr nanostructures taken at different directions. The length of the larger Cr nanostructures is around 0.2 μm. However, besides the larger ones, smaller and shorter structures can be seen from Figs. 1(a)–(c). Figure 1(d) shows a 45° titled view SEM image. Roughly, the top surface of most of the Cr nanostructures is crescent-shaped. Figures 1(a)–(d) confirm the 3D structure of the realized nano Cr. Experiments show that relatively homogeneous Cr nanostructures can be formed on the entire surface of the half of a 4-inch silicon wafer. Therefore, the approach is suitable for batch fabrication and mass production.

It is possible to tailor the dimensions of the Cr nanostructures by controlling the deposition conditions. For example,