Growth and electrical properties of multidimensional tungsten nanobuliding blocks

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(Received 2 August 2004; accepted 5 October 2004)

We report the demonstration of tungsten nanoblocks such as nanorods, nanoblocks, and nanocylinders through a simple thermal process of sputtered W films. The formation of various types of tungsten nanostructures was based on the difference in the magnitude of the residual film stress and in its spatial distribution, which depends on the deposition pressure during the sputter deposition of self-catalytic W thin film. The resulting shapes displayed well-controlled structures that have characteristic dimensionalities such as zero-dimensional dot, one-dimensional wire, and two-dimensional nanosheet with the deposition pressure. The results demonstrate the possibility of construction of *in situ* multidimensional achitectures through the one-step thermal process which uses a self-catalytic function of each thin film. © 2004 American Institute of Physics. [DOI: 10.1063/1.1829156]

Metallic tungsten (W) nanowires have useful electrical properties like efficient field electron emission at a moderate electric field.¹ Conventionally, tungsten is a well-known material for interconnection lines, gate electrodes for integrated circuits, electron sources, protective coatings resistant to corrosion or atomic diffusion at high temperatures, and diffusion barrier in integrated circuits with blanket or selective deposited film.^{2–6} Many works have shown that the mechanical, physi-chemical properties of magnetron sputtered W thin films are strongly related to film structure and high residual stress.^{7–9}

Recent findings have proven that one-dimensional metallic nanowires to be attractive for tera-bit level integrated nanoelectronics and with nanomachine applications, showing very unique low dimensional quantum physics and thus has been intensively studied in various synthesis methods. A straight metallic nanowire can be offered the possibility of both the first level (nano gate) and second level (1-D nanocontact electrodes as well nano-interconnections) for all nano-circuits or active channels for nanodevices.^{10–19}

Previously, we demonstrated the fabrication of W nano wires by the transforming W thin film into straight form nano wires through simple thermal treatment under specific conditions. This displayed good field electron emission properties and their promising application as building blocks for functional devices.^{1,2} The objective here is to obtain nano-structured pure materials in various structures and discover general method that does not requiring separate investigation of the process for each desired shape. The tungsten films as auto-catalytic layer were deposited on (100)-oriented Si substrates by rf magnetron sputter using a pure tungsten target (purity 99.99%) and pure argon (purity 99.99%) as a sputter-

ing gas. The base pressure was about 4×10^{-6} Torr and the pressure of the Ar sputtering gas was varied from 5 to 20 mTorr without any intentional heating of the substrate. Tungsten has relatively low sputtering yield and consequently longer sputtering time to obtain the desired thickness. This resulted in oxygen contamination either in the form of residual oxygen in the working chamber or from the tungsten target.^{20,21} We applied a sputtering power density of 2.5 (W/cm^2) . After the deposition of W thin film as a selfcatalytic layer, the prepared substrate was inserted into the rapid thermal low pressure chemical vapor deposition (CVD) system. The CVD chamber temperature was rapidly raised to 750 °C within 30 s to 120 min by halogen lamps after evacuation down to 10⁻² Torr. The W layer coated Si substrates were treated under a mixture of Ar and hydrogen gas for a few minutes. During each run, the total pressure of the chamber was maintained at 300 mTorr and after the treatment, the chamber was purged continuously with a mixture of H₂ and Ar until the chamber temperature reached room temperature within 20 min.

Our study focused on the finding of a parameter that controls shapes of nanostructures. It showed that the different deposition pressure of the catalytic tungsten thin film resulted in widespread stress within the films after the deposition. In order to examine the effect of the stress of the catalytic W film on the formation of W nanostructure we measured residual stress on the as-deposited W films. Figure 1 shows the catalytic W thin film stress measured by Tencor FLX-2320 using two solid state lasers. Before studying the stress of the 190-nm-thick W thin films, we performed the measurements on substrate Si and W film on Si substrate as a standard value. The results reveal how the sputtering gas pressure influences the formation of residual stresses in the films. The films deposited in the pressure range of 5-12 mTorr show a decreasing tensional stress with decreasing sputtering pressure and then finally, transition from tensile to

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FIG. 1. (Color online) (a) Internal stress of self-catalytic W films sputtered in argon ambient vs sputtering pressure. (b) Spatial distribution of internal stress of self-catalytic W films on Si sustrate.

compressive stress occurred below 5 mTorr. The decrease in tensile stresses with decreasing sputtering pressure results in an easy wrinkling of the film, thus forming nanorods in W film deposited at 5 mTorr.



FIG. 2. (Color online) Influence of the internal stress of catalytic W films on the dimensionality of resulting nanostructures. The shape of nanostructures made by thermal treatment strongly depended on the strength of the stress and distribution of the catalytic films. The scale bar represents 100 nm.



FIG. 3. (Color online) (a) A typical two terminal Nb–W nanorod–Nb junction and (b) two terminal electrical resistance vs temperature characteristics.

Highly magnified SEM images for the nanostructured materials fabricated from W films with the different stresses are shown in Fig. 2. The figure represents typical nanostructures obtained after simple thermal treatment under 300 mTorr of mixture gas. As we changed deposition pressure from 5 to 10 mTorr, while maintaining the same treatment time, we observed the transformation of nanorods to nanoblocks, which later took on an oval shape. Films deposited at higher sputter pressure beyond 12 mTorr resulted in reduced nanoballs after thermal treatment. One of the noticeable points in Fig. 2 is the fact that the nanostructure obtained by each catalytic W film is very uniform and even directionally aligned. The nanowires in Fig. 2(a), having filled an inner shell, consist of very sharp rods with the flattened top of a nearly perfect straight body. Majority of the materials have 5–10 µm length at maximum and below 100 nm in diameter with neat surface appearances. Also, the wavelength of the modulation in the HRTEM image is about 0.33 nm, the wavelength that corresponds to the interplanar distance of (110) lattice planes of bcc W.^{1,2} Another interesting fact is observed that when growth time increased, the areal density dramatically changed, and so, the extended growth time beyond about 30 min resulted in a dominant formation of a specific nanostructure. This was in competition with the extinction of trace of another nanostructure. As an extreme case, subsequent extrusions of tungsten at high pressure deposited W film occurred after an hour of thermal treatment and finally formed nanocylinders (see Fig. 2) are formed.

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The above observations indicate that the growth time, i.e., heat treatment time in furnace, could be another control parameter for the synthesis of a specific nanostructure from W films.

In order to examine the basic electrical properties of the nanorods, we fabricated metal electrode–W nanorod–metal electrode junction with backgate as shown in Fig. 3(a). Representative of two terminal resistance as a function of temperature shows very low resistance of a few ohm at room temperature. This then rapidly increases to about $10^3 \Omega$ at 70 K, showing typical behavior of the low dimensional system at low temperature. Their current levels reach to mA scale in this junction. Two terminal resistance (*R*) as a function of measuring temperature was depicted in Fig. 3(b), which reveals typical behavior of low dimensional material system, and showing an increase of resistance with the decrease of temperature.

In summary, there is a possibility of nanostructured tungstens by simply transforming of a continuous W thin film into discrete nanoblocks. Our sputter deposited films have an intrinsic residual stress and this provides one of the control factors for producing various types of nanostructures and even their conjunction such as multiscale junctions. Results show that W thin films with a lower tensile stress, i.e., higher compressive, tend to transform into high dense onedimensional nanorods/wire and film with a higher tensile stress. Additionally, a nonhomogeneous stress distribution may result in various kinds of zero-dimensional nanostructures. One of the exciting achievements for the multiscale structure is the mass production of a promising nanoprobe for scanning tunneling microscope and atomic force microscope applications and nanoblocks for nanorobot or any kind of nanostructure without the intentional use of any heterogeneous catalysts.

This work was supported by the Nano Core Technology(III) of the MOST in Korea, IMT 2000 Program, and partially by ITRC Project.

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