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Aging effect of rolled-up InGaAs/GaAs/Cr helical nanobelts

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ABSTRACT

We report an aging effect on as-fabricated InGaAs/GaAs/Cr helical nanobelts. It has been observed that over time the nanobelt diameter first decreases and then increases until a constant value is reached. The gradual change of the diameter of the helical nanobelts from their original value is due to the competition of stress relaxation along the transverse and longitudinal axes of the nanobelts. Finite element modeling (FEM) has been applied to validate the influence of the biaxial stress relaxation on the curvature change of these rolled-up helical nanobelts. In addition, the dependence of the pitch of the helix over time is investigated as well.

1. Introduction

A technique for fabricating three dimensional (3D) structures by a combination of "top down" and "self-organization" approaches, known as the self-scrolling technique, was introduced by Prinz et al. in 2000 [1]. This method is based on the coiling up of strained 2D thin films to generate 3D structures, an element of self-organization, after detachment from a substrate by selective etching. Using the self-scrolling technique, 3D micro-/nanostructures such as tubes and helices are fabricated in a controllable fashion [1–4]. Previously, it has been found that the diameter of the rolled-up micro-/nanotube can be precisely estimated theoretically, because the initial biaxial stress in the thin films are partially relaxed along the rolled-up direction [5], i.e. a uniaxial stress relaxation condition. In contrast to rolled-up tubes, the stress relaxation condition becomes more complicated for a rolled-up helical nanobelt in which the diameter, chirality, and pitch are also related to the width of the nanobelt due to the biaxial stress relaxation in the film plane [6,7]. Previous results indicated that the diameter of the helical nanobelts tends to increase as the stress relaxation condition changes from uniaxial to biaxial [6–8]. The robust fabrication process and the ultra-flexibility demonstrated for helical nanobelts have resulted in their application as nanosprings and linear-to-rotary motion converters [9–11]. Since diameter and pitch are two key geometrical parameters which determine the spring constant of the nanospring [12] and the conversion ratio of the linear-to-rotary motion converter [11], the stability of geometrical parameters of the helical nanobelts over time is important in determining the reliability of the devices. In this paper, the aging effect of InGaAs/GaAs/Cr helical nanobelts is investigated. Finite element analyses are also applied to analyze curvature dependence on the biaxial stress relaxation of the rolled-up helical structures.

2. Experimental methods

Freestanding InGaAs/GaAs/Cr helical nanobelts have been fabricated using the following procedure. First, AlGaAs/InGaAs/GaAs layers with thicknesses of 400 nm/16 nm/11 nm are epitaxially grown on a GaAs (001) substrate by molecular beam epitaxy (MBE) in which the AlGaAs layer acts as a sacrificial layer for releasing the InGaAs/GaAs bilayer from the substrate. The In concentration is 14% measured by X-ray diffraction (XRD), thus the misfit strain is 1.0% in the bilayer. Then a Cr layer is deposited on the top GaAs layer by e-beam evaporation with a thickness of 15 nm or 30 nm. For photolithographic patterning, photoresist S1818 (Shipley) is used for the generation of initial ribbon patterns, and then a mixture of Cl_2 and H_2 gases are employed for reactive ion etching (RIE) to transfer the patterns from the photoresist to the underlying layers. The main advantage of Cl_2 gas is its high etch rate on Cr and GaAs based materials [13]. The gap between the helical structure and the substrate should be large enough to avoid the helical structures sticking onto the substrate. Unlike the fabrication of freestanding Si-based helical nanobelts in which deep trenches are formed by wet etching of Si substrate [7], for fabricating freestanding GaAs based 3D structures on GaAs substrate, the trenches are created by RIE. Experiment results show that if the trench depth is larger than 1 μm, the as-fabricated 1–4 turn helices are freestanding. After oxygen plasma cleaning of the residue photoresist, a 1% HF aqueous solution is used to etch the AlGaAs

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sacrificial layer, and the patterned InGaAs/GaAs/Cr trilayer simultaneously detaches from the substrate and rolls up due to the internal stress. Finally, the samples are dried with a supercritical CO_2 dryer to eliminate the impact from surface tension of the liquid. To investigate the aging effect, the as-fabricated samples are kept at room temperature in air. All inspections were performed by field emission scanning electron microscope (FESEM) Zeiss Ultra 55 within the initial 108 days with the same viewing conditions.

3. Results

Fig. 1a–d presents a series of FESEM images of a 3-turn left-handed InGaAs/GaAs/Cr helix exposed to air for the initial 21 days after fabrication, in which the helix rotates along the winding direction. The ribbon width of the helical nanobelt is 1.5 µm and the thickness of the Cr layer is 15 nm. When the Cr layer thickness is increased to 30 nm, an InGaAs/GaAs/Cr ring structure (non-closed), i.e. helical nanobelt with 0° helicity angle and zero pitch, is formed as shown in the inset of Fig. 2. It is worth noting that the orientation of the ribbons for fabrication of helices and ring structures are the same, thus the formation of the ring structure is due to the anomalous coiling of the ribbon [6,7]. The diameter of the as-fabricated helix and the ring structure is 3.2 µm and 14.1 µm, respectively. The measured results show that InGaAs/GaAs/Cr helical nanobelts decrease their diameter during an initial 14–28 day period, after which the diameter gradually increases. Eventually the diameter becomes almost identical to the original one after 42 days, as shown in Fig. 2. The change of the diameter of the helical nanobelts from its original value is attributed to stress relaxation along the transverse to longitudinal axes of the nanobelts. In the initial stage, the deformation of the cross sectional area of the nanobelts dominates the diameter reduction due to stress relaxation along the transversal axis of the nanobelts. Since the curvature radius along the transversal axis of the helix is increasing, the stiffness of the ribbon is reduced which leads to a reduction in the diameter of the helical nanobelts [14]. Then the stress relaxation along the longitudinal axis of the nanobelt becomes a major factor leading to an increase in diameter until it reaches a constant value. Based on experiments, the deviation of the diameter of the stress relaxed helical nanobelt is less than 10% of its original value. The results indicate that the diameter of the InGaAs/GaAs/Cr helical nanobelts becomes stable after approximately 6–7 weeks, and biaxial stress relaxation eventually ceases. The gradual stress relaxation is mainly attributed to the micro-

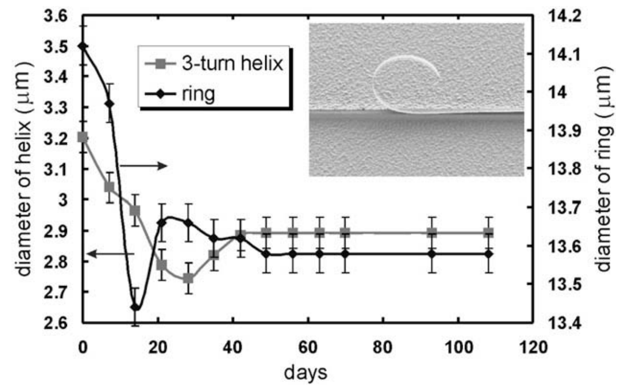

Fig. 2. Dependence of diameter of helical nanobelts on time. Both helical and ring structures have InGaAs/GaAs bilayers with thickness of (11/16 nm). Helical and ring structures are coated with 15 nm and 30 nm thick Cr, respectively.

structure evolution of the amorphous or polycrystalline evaporated Cr layer [15]. We also investigate the time dependence of the helix pitch as shown in Fig. 3. The results show the pitch value decreases for the initial 56 days, but afterwards becomes stable. We attribute the decrease of the pitch to a reduction of the anisotropy of the ribbon, e.g. the oxidation of the InGaAs/GaAs bilayer. It is notable that during the SEM inspection two effects may occur, one is the charging effect on the freestanding structure and the other is hydrocarbon deposition due to the contamination from the SEM chamber; however, results confirm that these effects are negligible. Assuming the freestanding structure is strongly charged by electrons on the surface, the pitch value and the diameter should increase during the inspection due to the repulsive force between the electrons. However, this is not the case. On the other hand, a thin hydrocarbon layer may be deposited on some area of the freestanding structure, but if the layer is significant, the shape of the helical nanobelts will be deformed locally.

Finite element modeling (FEM) was performed for validating the prediction of the biaxial stress relaxation of ring structure using the commercial software COMSOL. A triangular mesh is utilized as it provides a good approximation for curved areas and boundaries. To simplify the simulation, the InGaAs/GaAs/Cr trilayer is replaced by GaAs/Cr bilayer, in which the GaAs layer in the simulation has the same thickness as the InGaAs/GaAs bilayer of the real samples. In principle this simplification only changes the absolute value of the calculated diameter but not the tendency of the

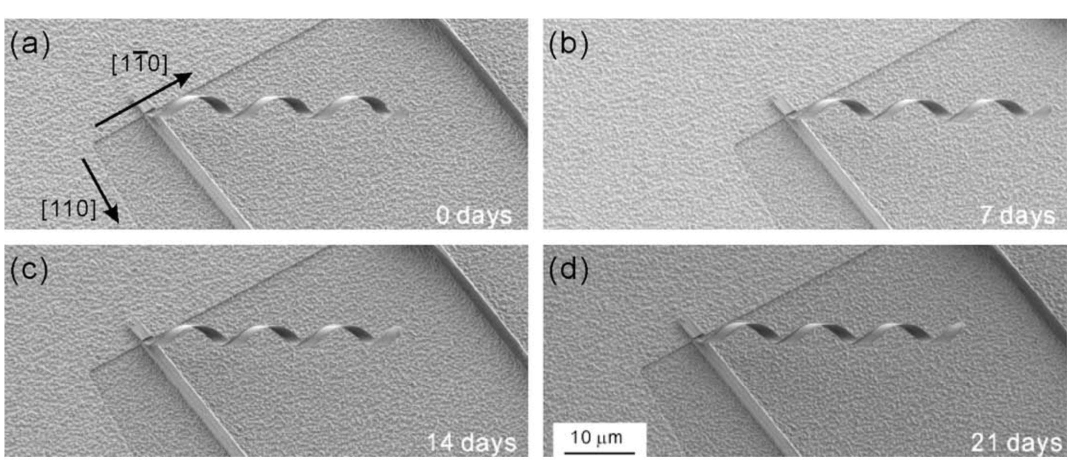

Fig. 1. The as-fabricated sample is exposed to air for several weeks. The sample is inspected by FESEM with the same viewing orientation. All four images have the same scale bar. The helix was prepared on a GaAs (001) wafer with a trilayer, i.e. InGaAs/GaAs/Cr (11/16/15 nm). The orientation of the ribbon-like patterned mesa has 10° deviation from the [110].

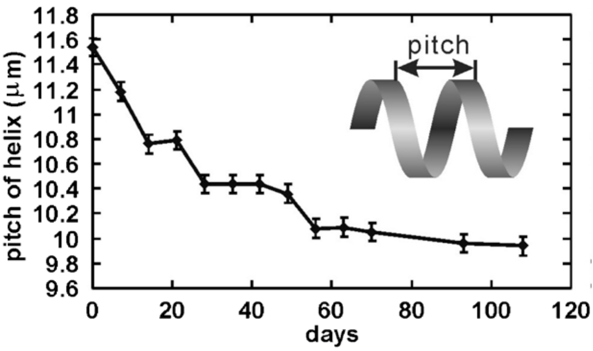

Fig. 3. Dependence of helix pitch with time, the helix has three turns and has the same stacked layers as the helix shown in Fig. 1. The inset shows the pitch of the helix, which means the translational distance along the helix axis with one complete helix turn.

diameter via the stress relaxation which is the key part of the FEM analysis. In the simulation, a 1.5 μm wide and 10 μm long GaAs/Cr bilayer ribbon is applied, where Cr is 30 nm and GaAs is 27 nm thick, respectively, and we assume that the Cr layer has a misfit strain of 1.5% [16]. Since the length of ribbon does not influence the scrolling curvature/radius, only a portion of the total length of the ring is used to observe the small deviation of deformation of the ribbon. Elastic properties were set according to the physical properties of the materials in real experiments, such as Young's moduli, Poisson ratios and shear moduli. In Fig. 4a and b, it is shown that the process in which the radius changes is due to the interplay of strain relaxation in the orthogonal directions of the X–Y plane. The radius in the longitudinal direction decreases due to stress relaxation between two layers, while the radius in the Y–Z plane increases. This allows us to assume the opposite trend in radius in two planes. Similar situations can be observed when the radius in X–Y plane increased. The reason for this interaction is that the change of curvature in one plane leads to the change of stiffness in the other plane, which causes simultaneous scrolling in two directions and, thus suppressing effects on each other. The final position is determined by the balance between bending moment and the initial stress, it is the result of self-adjusting the radius in two perpendicular planes due to biaxial stress competition. The schematic diagram of the simulation results shows that for the longitudinal axis of the nanobelt, the curvature radius is initially reduced due to the relaxation process and then increased (see Fig. 4c), which is consistent with experimental results.

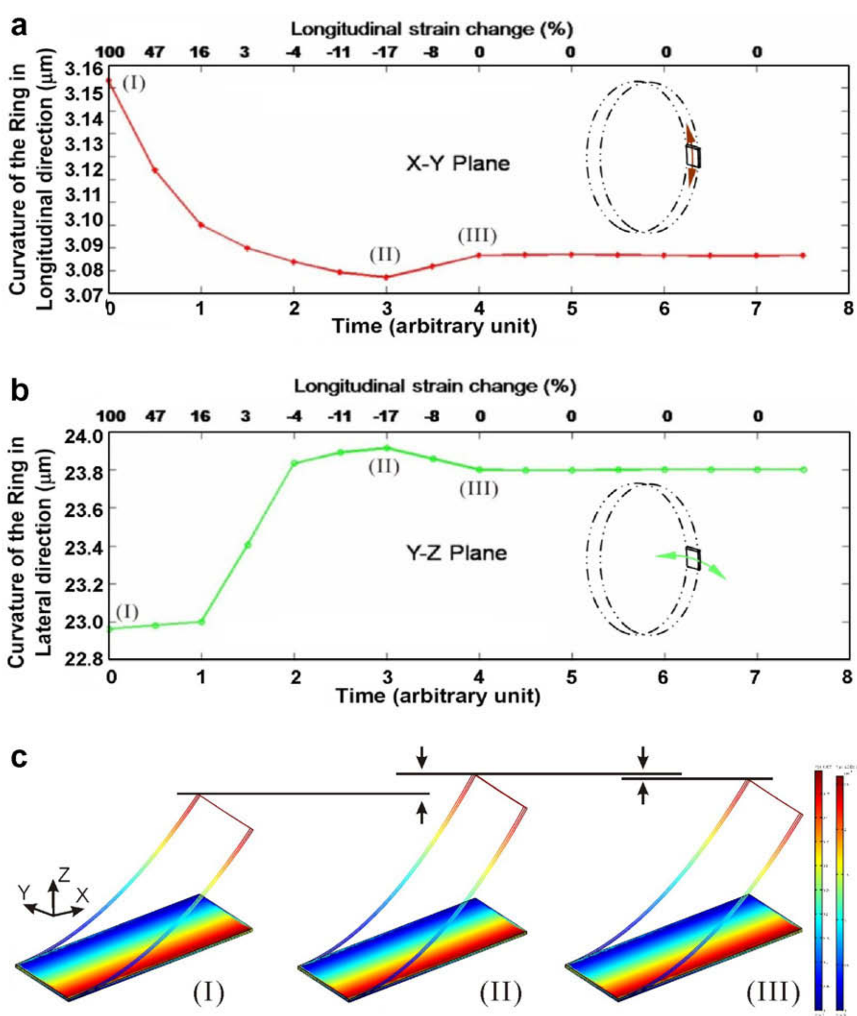

Fig. 4. FEM results of curvature evolution in biaxial directions for ring structure. (a) Curvature change in the longitudinal axis (X–Z plane) of the ribbon due to stress relaxation. (b) Curvature change in the transversal axis (Y–Z plane) of the ribbon due to stress relaxation. (c) Curvature radius to stress states of the ribbon for (I) (II) (III) in (a). The length and the width of the ribbon for simulation are 4 μm and 1.5 μm, respectively.

4. Conclusion

The aging effect of InGaAs/GaAs/Cr helical nanobelts has been investigated. The change in diameter of helical nanobelts occurs in three stages over time, i.e. decrease, increase and final balance, due to the biaxial stress competition. The pitch of the helix is reduced with time at the beginning but eventually becomes constant as well. The results indicate that the shape of rolled-up helical nanobelts is not stable in the initial 6–8 weeks after fabrication, but eventually returns to a shape similar to its original one.

Acknowledgments

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