# Direct Observation of Strain Segregation in ZnO Nanorings Using X-ray Diffraction*

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## Introduction

A zinc oxide nanoring is transformed through coaxial, uniradius, and epitaxial coiling of a single-crystalline nanoribbon [1, 2]. The polar charge on ZnO nanoribbon's ±(001) surfaces, resulting from the termination by zinc and oxygen atomic layers, respectively, tends to fold itself if the surface charges are uncompensated during growth. It is believed that the mechanical configuration of such a nanoring reflects a balance among the polar charge, surface area, and elastic deformation. Although the research into functional oxide and one-dimensional nanostructures has rapidly expanded because of their unique and novel applications in optics, optoelectronics, catalysis, and piezoelectricity [3], the lattice deforms in response to the coiling stress remains largely unknown. We report here x-ray diffraction measurements of individual ZnO nanorings that clearly show the segregation of lattice strains under coiling stresses.

## Methods and Materials

Our studies were carried out at the 2-ID-D beamline, a hard x-ray microprobe facility, of the Advanced Photon Source of Argonne National Laboratory. Using advanced zone-plate optics, we focused 10 keV synchrotron x-rays, selected with a double-crystal Si (111) monochromator, into a 150 nm x 150 nm spot with a flux density of $4.5\times10^4$ photons/sec/nm$^2$/0.01%BW, allowing the structure of an inhomogeneous nanomaterial to be analyzed [4,5]. Single-crystal nanorings of ZnO were grown by a solid-vapor process from a mixture of ZnO, indium oxide, and lithium carbonate powders at the temperature of 1400°C [2]. The as-synthesized nanorings were transferred onto a silicon nitride membrane coated with a gold mesh of 120 μm/per grid and 1 μm thick. Scanning electron microscopy was used to characterize the lateral sizes of the rings and coordinate them against the mesh. To ensure the measurement on an individual nanoring, a ring is selected to be a specimen only if it is several tens of microns separated from others. The x-ray absorption image of the gold mesh was employed to establish coordinates of the rings with the x-ray beam. Zinc's Kα line fluorescence was used to finally locate a ring in the x-ray beam.

## Results

The diffraction patterns obtained from the (220) reflection of a ZnO nanoring with a diameter of 2.6 μm and a cross section of about 20 nm x 40 nm are displayed in figure 1. The (220) atomic plane lies parallel to the radius and normal to the tangent of the ring. The x-ray illumination along the ring circumference causes a 27 degree span of the diffraction along the normal (the $\chi$ direction or the vertical axis of figure 1) to the Bragg reflection in reciprocal space due to the continuous bending of the (220) plane. The horizontal axis of Fig. 1 is the angle between the incident beam and the diffracted beam ($2\theta$). The variation of the diffraction intensity along the $2\theta$ direction is a sign of the variation of the lattice strain. Lattice strain can vary along the arc of the ring, as suggested by the bottom parts of the diffraction patterns, by as much as 2.4%, although small domains of different strain also exist, as suggested by the small branching of the diffraction intensity. The two main branches of the diffracted intensity, as shown in the top parts of the diffraction patterns, indicate the existence of the two strain domains across the cross section of the ring with as much as 1.8% difference in (220) plane spacing.

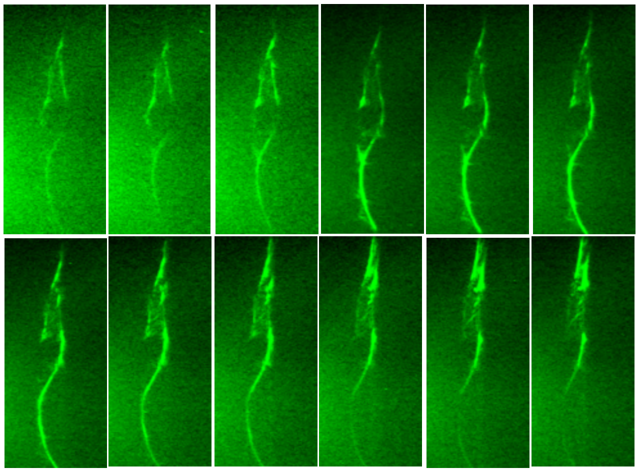

Fig. 1 Diffraction patterns captured by a CCD from the (220) reflection of a ZnO nanoring with incident angle ($\theta$) from 49.38° to 49.68° (from left to right and top to bottom). For each image the vertical axis represents the $\chi$ direction, which covers 27.5°, and the horizontal axis represents the $2\theta$ direction, which covers 6.6° and centered at 99.27°.

## Discussion

It is reasonable to expect that the coiling stress that forms a ring would cause a continuous distribution of (220) strains from compressive to tensile along the radius of the ring. Our studies show that the combined compressive and tensile strains can either collapse into domains of distinct strains across the cross section of the ring or segregate to have uniform strain across the cross section of the ring but vary continuously along the ring. We believe that the lattice response of the ZnO nanomaterials to the external stress observed in this study has important implications in mechanical characteristics of nanomaterials.

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