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Focused Ion Beam-induced Ripple and Nanoparticle Formation in $Cd_2Nb_2O_7$

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Ion beam techniques have been widely used for the fabrication of novel nanostructures by implantation, phase-decomposition and order-disorder phase transition processes. The formation of periodic ripple or a wavelike structure with a spatial wavelengths varying from nm to μm scale has been observed in many ion beam-bombarded solid surfaces and is believed to be caused by the competition between a roughening process as a result of ion beam sputtering (erosion) of surface and a smoothening process induced by thermal or ion-induced surface diffusion. Recently, ion sputtering-induced ripple structures have been of particular interest for the fabrication of nanoscale-textured materials via self-organization processes or as templates for the growth of nanowires, nanorods and nanodots. Here, we report the simultaneous formation of a ripple structure with the characteristic wavelength varying from nm to sub-μm range induced by ion sputtering and the formation of uniformly-distributed metallic nanoparticles with the size of 3~10 nm caused by ion irradiation-induced phase decomposition. Ion sputtering experiments were performed on a $Cd_2Nb_2O_7$ single crystal using FSEM/FIB dual beam system (FEI Nova 200 NanoLab). A focused Ga^+ beam with an energy of 30 keV was used for ion sputtering at beam currents from 10 pA to 5 nA at different incident angles. The surface morphology and microstructural evolution upon ion sputtering were characterized by in-situ SEM, ex-situ AFM and TEM techniques.

A ripple structure perpendicular to ion projection directions can be induced on $Cd_2Nb_2O_7$ substrate by Ga^+ ion sputtering at an incident angle varying from 30^o to 50^o. The characteristic wavelength of the ripple structure formed at a high dose of 4.01×10^{18} ions/cm^2 measured from the in-situ SEM image (Fig. 1A) is ~350 nm, and the surface roughness of the ripple structure was determined to be ~35 nm from the AFM image (Fig. 1B). By varying the ion flux and the incident angles, the characteristic wavelength of the ripple structure can be controlled from ~130 nm to 620 nm. Ripples were patterned at different areas of a $Cd_2Nb_2O_7$ thin foil for TEM observation in order to characterize the ripple microstructure. Fig. 2A shows a high angle annular dark field STEM image of the ripple structure patterned with 30 keV Ga^+ at an ion dose of 6.27×10^{16} ions/cm^2. The wavy-like contrast resulting from the variation of the sample thickness clearly demonstrated the formation of a ripple structure. The EDS line profile across the ridge of a ripple, as marked by a white line of Fig. 2A, indicates that the ridge of the ripple contains more Nb and O. This result suggests that surface diffusion of Nb and O might play an important role in the formation of ripples. No significant variation has been observed in the average signals of Cd (EDS profile); in contrast, uniformly-distributed nanoparticles, 3~10 nm, were observed in the Ga^+ sputtered $Cb_2Nb_2O_7$, as evidenced in the Z-contrast image (Fig. 2B). EDS maps (inset in Fig. 2B) show that the nanoparticles are Cd-rich, but depleted in Nb and O. A bright-field TEM image (Fig. 3A) shows the morphology of nanoparticles formed in Ga^+ sputtered $Cd_2Nb_2O_7$. The Nb-depleted feature of nanoparticles is further evidenced by the Nb map formed by energy loss electrons (Fig. 3B). The nanoparticles were identified as pure Cd metal nanoparticles by indexing the ring pattern (inset in Fig. 3A) and the lattice spacing of the nanoparticles (Fig. 3C). The formation of Cd metal nanoparticles was induced by the phase decomposition process of $Cd_2Nb_2O_7$ single crystals upon Ga^+ ion irradiation, concurrent with the formation of the self-organized ripple structure. This work was supported by the DOE Office of Basic Energy Sciences (DE FG02 97 ER45656) and an NSF NIRT grant (EAR-0403732).

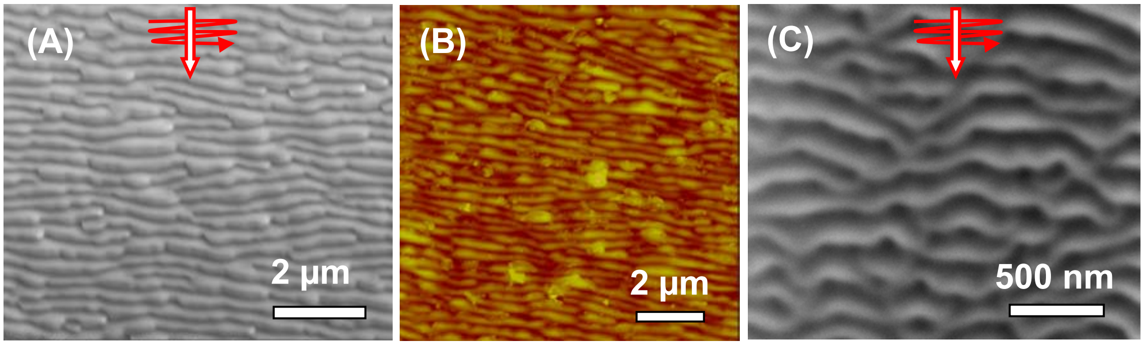

Fig. 1 (A) SEM and (B) AFM images of coarse ripples formed on 30 keV Ga^+ sputtered $Cd_2Nb_2O_7$ (at an incidence angle of 50^o and a high dose of 4.01×10^{18} ions/cm^2). (C) SEM image of finer ripples formed at a lower dose of 6.27×10^{16} ions/cm^2. The straight arrow indicates the projected ion beam direction and curved arrow shows the fast-scan direction.

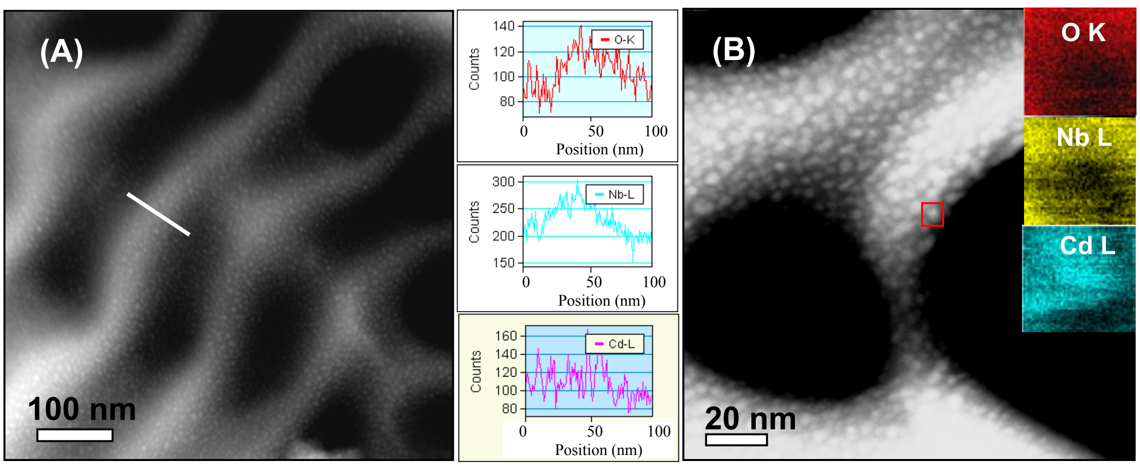

Fig. 2 HAADF-STEM images show the ion sputtering-induced ripples (A) and ion irradiation-induced nanoparticle formation (B) in $Cd_2Nb_2O_7$ at a dose of 6.27×10^{16} ions/cm^2. Insets of Figs. 2A and 2B are EDS profile and elemental mapping, respectively.

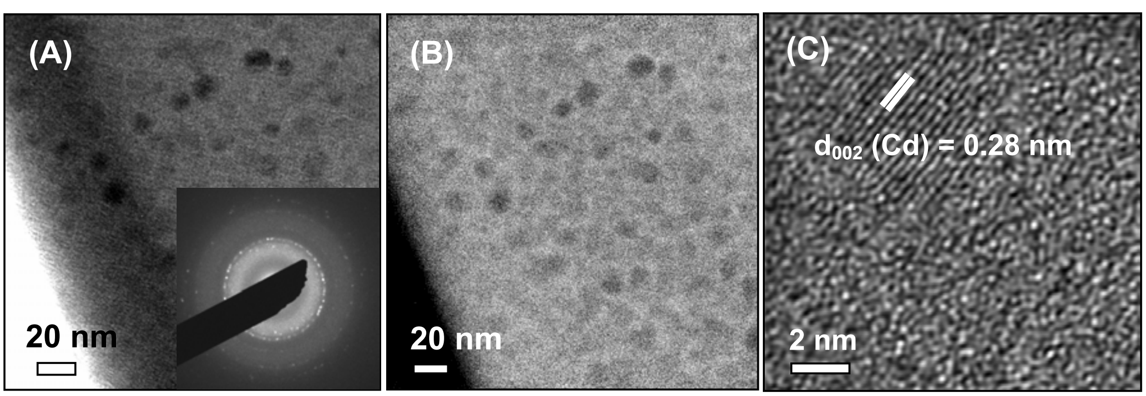

Fig. 3. (A) A bright-field TEM image and the corresponding Nb elemental map formed by energy-loss electrons. Inset in Fig. 3A is the electron diffraction pattern of nanoparticles. (C) High-resolution TEM image of a Cd nanoparticle.

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