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In Situ Heating Study of 2H-$MoTe_2$ to Mo_6Te_6 Nanowire Phase Transition

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Transitional-metal dichalcogenides (TMDs) have attracted extensive research interest for nano-electronic and photovoltaic applications because of their novel structure and remarkable properties [1]. Except the commonly observed semiconducting 2H phase of TMD materials, the available metallic 1T or semi-metallic 1T' phase makes it a promising 2D contact material for applications in field-effect transistors (FETs). Among various TMDs, mono-layer 2H-$MoTe_2$, with a direct band gap of 1.1 eV, is a highly attractive material for FETs [2]. Apart from the 1T'-$MoTe_2$ phase, theoretical calculation shows that Mo_6Te_6 nanowires (NWs) phase (see Figure 1a) is also metallic and can be a potential candidate as a contact material for $MoTe_2$–based FETs [3]. Previously, Mo_6X_6 type NWs (X=S, Se, and Te) has been synthesized only via ternary intercalation method. Until recently, Lin *et al* show that Mo_6S_6 and Mo_6Se_6 NW can be fabricated using electron beam engineering in the transmission electron microscope (TEM) [4].

In this study, we show that $MoTe_2$ can change from 2H phase to Mo_6Te_6 NW phase under vacuum annealing [3]. $MoTe_2$ thin flakes (~15-20 layers thick, Figure 1b-c) have been exfoliated from the bulk crystal and transferred on to an *in situ* heating TEM grid via the mask assisted transfer method. *In situ* heating experiment has been performed on thin $MoTe_2$ flakes under the TEM (JEM ARM 200F with a probe Cs-corrector operated at 200 kV) so as to observe the 2H-NW phase transition. The phase transition starts when the flake is heated to 450 °C, which is consistent with the Te melting temperature of 449.5 °C. During the heating, Te atoms start to dissociate from 2H-$MoTe_2$ and evaporate into vacuum, thus creating a Te deficiency environment favorable for the formation of the relatively more stable Mo_6Te_6 NW phase. As shown in Figure 2(a), "streak line" features of Mo_6Te_6 NW bundles propagate across the flake along the three-fold 2H-$MoTe_2$ <11-20> directions, matching with the fast Te loss directions on the flake surface. Figure 2(b) shows the *in situ* observation of a Mo_6Te_6 NW bundle propagated on the flake. The phase transition is so fast that NW bundles can grow to about 50 nm in less than 27s. After 24 min annealing at 450 °C, most of the flake has been covered by Mo_6Te_6 NWs. To observe the 2H-NW interface after the heating experiment, a cross-sectional TEM specimen (Figure 2c) has been prepared by cutting across a Mo_6Te_6 NW bundle. The viewing direction is along the axial direction of NWs, which is also along 2H-$MoTe_2$ [11-20] zone axis. Atomically sharp interface between 2H-$MoTe_2$ and Mo_6Te_6 NWs is observed as shown in Figure 2(c). Due to the difference in layer spacing between the two materials, eight layers of $MoTe_2$ converted into seven layers of Mo_6Te_6 NWs. This is an evidence that the 2H-NW phase transition is not restricted in one layer. Our energy dispersive X-ray spectroscopy (EDS) analysis shows that the Te/Mo ratio at the NWs region is 1.07, which is consistent with the stoichiometry of Mo_6Te_6. Considering that Te loss into vacuum is permanent, the 2H-NW phase transition is irreversible and Te deficiency plays an important role during the phase transition. Our experimental findings have provided a potential approach for the application of FETs fabrication and more efforts are required for the precise controlled growth of Mo_6Te_6 NWs on 2H-$MoTe_2$ flake [6].

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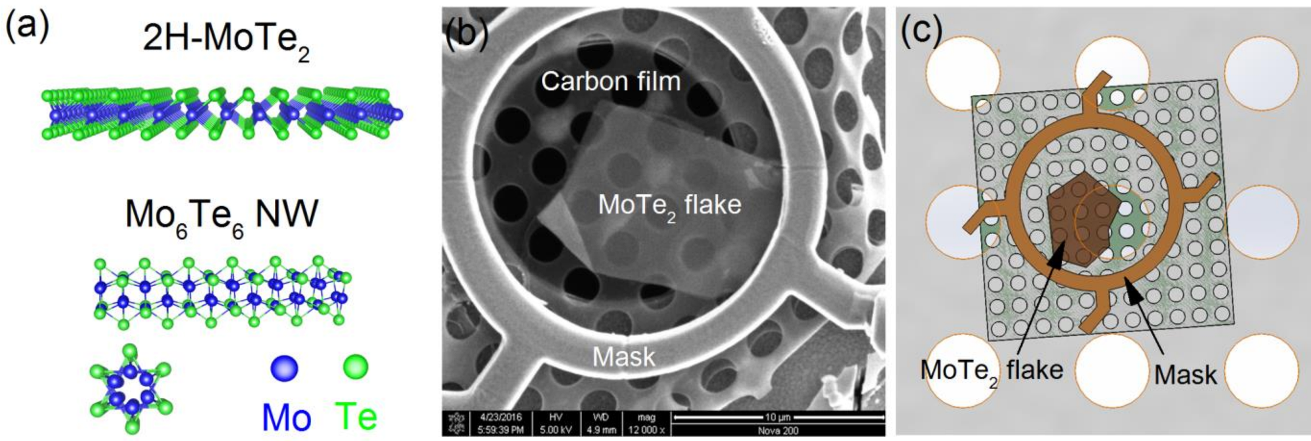

Figure 1: (a) The structure of 2H-$MoTe_2$ and Mo_6Te_6 NW. (b) SEM image of a 2H-$MoTe_2$ flake transferred on the heating E-chip. (c) Cartoon illustration of the flake on heating E-chip. Masks have been used during the flake transfer to prevent ion beam damage to the flake.

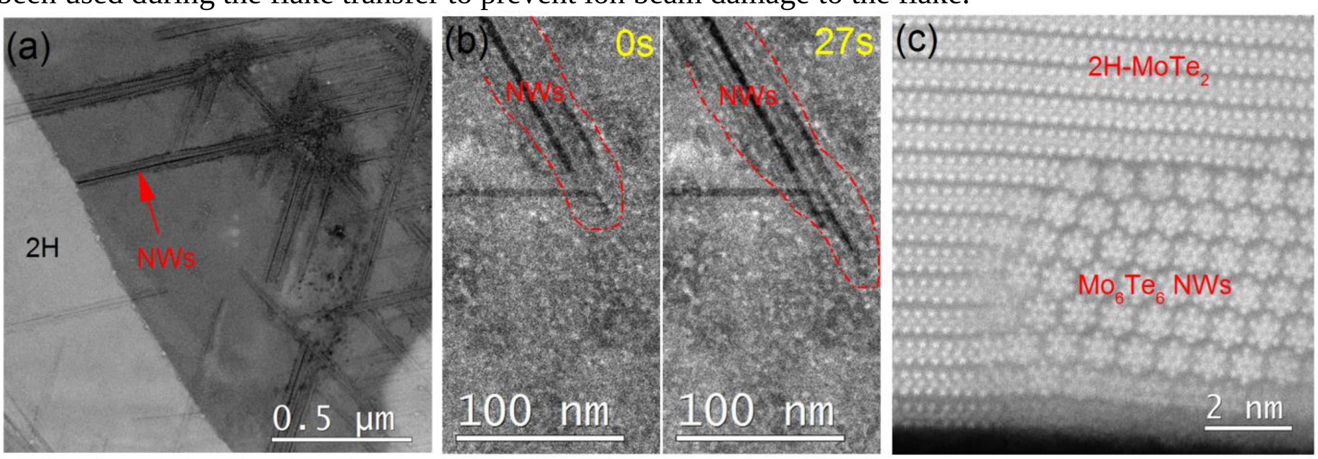

Figure 2: (a) Mo_6Te_6 NWs on 2H-$MoTe_2$ flake. (b) Propagation of Mo_6Te_6 NWs during *in situ* heating experiment. (c) High resolution STEM image shows the 2H-NW interface. All the images are acquired in HAADF mode.

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