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#### ORIGINAL ARTICLE



# Phase transformation of molybdenum trioxide to molybdenum dioxide: An in-situ transmission electron microscopy investigation

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#### Abstract

We use in-situ environmental transmission electron microscopy (ETEM) to investigate the phase transformation of  $\alpha$ -MoO<sub>3</sub> in vacuum. In-situ selected area electron diffraction (SAED) indicates that  $\alpha$ -MoO<sub>3</sub> directly transfers to monoclinic MoO<sub>2</sub> without intermediate phase; however, high resolution transmission electron microscopy (HRTEM) study reveals that Mo<sub>4</sub>O<sub>11</sub> precipitates on the surface of MoO<sub>2</sub> matrix. Judging from the size of Mo<sub>4</sub>O<sub>11</sub> and its orientation relationship with MoO<sub>2</sub>, we believe that the formation of Mo<sub>4</sub>O<sub>11</sub> is due to the reaction between MoO<sub>2</sub> and the untransformed MoO<sub>3</sub>.

#### KEYWORDS

crystallographic shear plane, molybdenum dioxide, molybdenum trioxide

## **1** | INTRODUCTION

Molybdenum oxide has a lot of attractive properties due to its multiple valence states. For example,  $\alpha$ -MoO<sub>3</sub> finds its application as semiconductor,<sup>1</sup> photochromic materials<sup>2</sup> and a gas sensor.<sup>3</sup> Monoclinic MoO<sub>2</sub>, on the other hand, exhibits metal-like electronic conductivity due to the existence of delocalized electron in its valence band.<sup>4</sup>

 $\alpha$ -MoO<sub>3</sub> has an orthorhombic crystal structure (space group Pbnm. For simplicity, we use MoO<sub>3</sub> to represent  $\alpha$ -MoO<sub>3</sub> in the paper) with lattice constants a=0.3962 nm, b=1.3855 nm, and c=0.3699 nm. The structure of MoO<sub>3</sub> consists of bilayers of linked distorted MoO<sub>6</sub> octahedra parallel to (010) plane. The bilayer is composed of two corner-sharing rows of MoO<sub>6</sub> octahedra along [100] direction that links by sharing edges along [001] direction. The adjacent double-octahedral layers are then linked along [010] direction by weak van der Waals forces.<sup>5</sup> Generally  $MoO_2$  possesses a monoclinic structure (space group  $P2_1/c$ ) with lattice constants of a=0.56109 nm, b=0.48562 nm and c=0.56285 nm and a monoclinic angle of 120.95.6 The structure of MoO<sub>2</sub> is closely related to that of rutile (RO<sub>3</sub>) in which the MoO<sub>6</sub> octahedra are connected by sharing opposite edges along [001] to form strings. In MoO<sub>2</sub>,

however, the Mo atoms within the strings are pairwise drawn nearer to each other to form doublets. This results in the distortion of the  $MoO_6$  octahedra.<sup>7</sup>

Reduction in MoO<sub>3</sub> produces oxygen vacancies which are accommodated by partial screw dislocation at low temperature.<sup>8</sup> At higher reducing temperature, crystallographic shear (CS) planes are introduced to relax the chemical stress due to super-concentration of oxygen vacancies.<sup>8,9</sup> Different concentration level for oxygen vacancy leads to the formation of a number of suboxides (e.g. Mo<sub>9</sub>O<sub>26</sub>, Mo<sub>8</sub>O<sub>2</sub>, Mo<sub>5</sub>O<sub>14</sub> and Mo<sub>4</sub>O<sub>11</sub> for Mo<sub>n</sub>O<sub>3n-1</sub> family) between MoO<sub>3</sub> and MoO<sub>2</sub>. Two mechanisms are proposed to explain the reducibility of MoO<sub>3</sub> to MoO<sub>2</sub>. The first one is the one-step mechanism in which MoO<sub>3</sub> is directly reduced to MoO2.<sup>10</sup> The other mechanism, as suggested by Burch, involves MoO<sub>3</sub> transferring to Mo<sub>4</sub>O<sub>11</sub> first and then the latter continues transferring to MoO2.11 However, the in-situ X-ray Absorption Spectroscopy (XAS) and Xray Diffraction (XRD) by Ressler et al.<sup>12</sup> indicates that the actual process may be more complicated; in fact, they indicate that the governing mechanism depends on the experimental temperature range. When temperature is below 698 K, the reduction of  $MoO_3$  to  $MoO_2$  is a one-step process. When temperature is above 773 K, Mo<sub>4</sub>O<sub>11</sub> is formed from the reaction between MoO<sub>3</sub> and MoO<sub>2</sub>. This suggests that  $Mo_4O_{11}$  is not an intermediate in the reduction of MoO<sub>3</sub> but instead, is being formed in a parallel reaction from MoO<sub>3</sub> and MoO<sub>2</sub>. This mechanism was questioned by Sloczynski that an autocatalytic effect of MoO<sub>2</sub> should be taken into account.<sup>13</sup> Recently, Lalik proposed an autocatalytic comproportionation to explain the aforementioned discrepancy.<sup>14</sup> In his proposed mechanism, Lalik suggests that MoO<sub>2</sub> is first formed topotactically from MoO<sub>3</sub>. The formed MoO<sub>2</sub> then reacts with MoO<sub>3</sub> to produce Mo<sub>4</sub>O<sub>11</sub>. When MoO<sub>3</sub> is consumed, Mo<sub>4</sub>O<sub>11</sub> is reduced to MoO<sub>2</sub>.

Although transmission electron microscopy (TEM) was employed in investigation on the reduction of MoO<sub>3</sub> several decades ago, only several in-situ works have been reported.<sup>8,15</sup> In the published work, the in-situ work was conducted either at low temperature<sup>15</sup> or at relatively low magnifications,<sup>8</sup> phase transformation from MoO<sub>3</sub> to MoO<sub>2</sub> was not discussed in details. In a more recent work, the phase transformation sequence of MoO<sub>3</sub> to MoO<sub>2</sub> was studied, but the phase transformation was induced by the radiolysis effect of electron beam.<sup>16</sup> Therefore, a study on phase transformation of MoO<sub>3</sub> by in-situ heating, which is governed by other mechanisms, is necessary. As first part of this series work, herein we report the microstructure alteration of MoO<sub>3</sub> under heating in vacuum revealed by environmental TEM (ETEM).

### 2 | EXPERIMENTAL

The MoO<sub>3</sub> used in the current investigation is powder form and purchased from Sigma-Aldrich (ACS reagent, ≥99.5%). A dedicated H-9500 ETEM was employed in the current investigation. Part of column is modified by installing a set of apertures to restrict gas leakage into the upper column. A safety valve is used to further maintain a safe high vacuum in the gun area. A high speed turbo molecular pump (TMP) is added to specifically pump the specimen chamber. In order to keep the MoO<sub>3</sub> crystalline intrinsic during the in-situ investigation, a low accelerating voltage of 100 kV was chosen to reduce the atomic sputtering through knock-on displacement by high energy electrons.<sup>16</sup> A combination of suitable condense-aperture size and voltage/current of LaB<sub>6</sub> filament adjustment was optimized to lessen radiation damage. Beam was kept off during the heating process unless image was collected. The heating rate was 10°C/min. The electron energy loss spectrum (EELS) was acquired using an installed GIF Tridiem Filter. An energy dispersion of 0.20 eV/pixel was applied in order to record the Mo M2.3- and O K-edges on the same spectrum. The simulation of selected area electron diffraction (SAED) pattern was performed using commercial software named Desktop Microscopist.

#### **3** | **RESULT AND DISCUSSION**

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As stated above,  $MoO_3$  can easily be damaged by electron beam. Figure 1 shows a high resolution transmission electron microscopy (HRTEM) image of the specimen prior to heating. Domains with different sizes are seen in the image. These domains are obviously formed by the irradiation of electron beam. Due to weak Van der Waals force between the double layers parallel to (010) plane (Figure 2A),  $MoO_3$  can easy cleavage along (010) and hence a plate-like habit of  $MoO_3$  with [010] as normal direction is common. Figure 2B shows the SAED pattern orientated at [010]. The intensities of the reflections in the pattern are not perfectly symmetrical because of the maximum tilting angle limit for the specimen stage. The space-group-forbidden [100] and [001] reflections appear in the pattern. This could come from high order Laue zone.

When specimen was heated up to 400°C, new features emerged overlapping the original [010] zone axis reflections (Figure 3A). The most obvious feature is the streaks parallel to [100]. This stems from the short-ordering of oxygen vacancies formed by the partial removal of Van der Waals force connected oxygen atoms between the consecutive double layers parallel to (010) plane. According to Bursill,<sup>17</sup> the elimination of pairs of the oxygen vacancies can be realized by shearing along the edges of MoO<sub>6</sub> octahedra along vector of  $\mathbf{a}/2 + \mathbf{b}/7$ . In addition to the reflection of (100)<sub>MoO3</sub>, sharp reflections such as the yellow arrowed spot come out form the streaking reflections. The d-spacing (0.342 nm) of the indicated spot is very close to



**FIGURE 1** HRTEM image of  $MoO_3$ . The inset shows domains with different sizes in a region within the image. The image shown in the inset is processed by Fourier filter

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**FIGURE 2** (A) Crystal structure of MoO<sub>3</sub> orientated close to [100]. (B) The selected area electron diffraction pattern selected area electron diffraction of a MoO<sub>3</sub> plate orientated at [010] [Color figure can be viewed at wileyonlinelibrary.com]



FIGURE 3 The evolution sequence of selected area electron diffraction pattern at different temperature [Color figure can be viewed at wileyonlinelibrary.com]

the d-spacing of  $(01\bar{1})_{MoO2}$  plane. In contrast to  $(100)_{MoO3}$ , there is no sharp satellite appearing beside  $(001)_{MoO3}$ . Instead, a symmetrical diffuse reflection emerges. This relates to the rigid chains of edge-sharing octahedra within the double layers.<sup>18</sup> This diffuse reflection zone becomes bigger (Figure 3B) with temperature, indicating more oxygen vacancies generated in the double layers. At 950°C, the weak ring pattern belongs to MoO<sub>2</sub> forms, implying that the original MoO<sub>3</sub> transfers to small MoO<sub>2</sub>. Among the rings, reflections of  $(01\bar{1})_{MoO2}$  and  $(\bar{1}11)_{MoO2}$  are pronounced, suggesting the orientation of the large portion of MoO<sub>2</sub> particles is close to [211]. Figure 4 shows the HRTEM image of a MoO<sub>2</sub> particle with such an orientation. If all the Van der Waals force connected oxygen

atoms between the consecutive double layers are removed, we can obtain the MoO<sub>2</sub> in composition. According to Bursill,<sup>17</sup> the removal of oxygen in MoO<sub>3</sub> is realized by slipping of the two consecutive layers along  $\mathbf{a}/2$  followed by collapse along  $\mathbf{b}/7$ . The stacking of the (010) layers one upon the other brings stress to the formed MoO<sub>2</sub>, thus structure like twinning will be incorporated into the structure to relax the strain. This is what exactly happened. On closer inspection of the diffraction pattern in Figure 3C, we do find the existence of twinning structure orientated at [211] (see the two parallelograms in Figure 3C). Because there is no intermediate phase between MoO<sub>3</sub> and MoO<sub>2</sub> as revealed by SAED, and the formation of MoO<sub>2</sub> is associated with oxygen vacancies removal by crystallographic



**FIGURE 4** The HRTEM image of a monoclinic  $MoO_2$ . The inset is the power spectrum of  $MoO_2$ 



**FIGURE 5** Electron energy loss spectrum spectra collected at different temperature [Color figure can be viewed at wileyonlinelibrary.com]

shear planes, we believe that the transformation from  $MoO_3$  to  $MoO_2$  is a topotactic process.

Electron energy loss near edge structure (ELNES) revealed by core loss spectrum in EELS can be envisaged in a first-order approximation as an image of a momentum and atomic resolved projection of the unoccupied density of state, thus they are sensitive to the chemical bonding. Because oxygen K-edge is sensitive to its coordination



**FIGURE 6** The HRTEM image of MoO<sub>2</sub>. Precipitates with different sizes are visible on the surface of MoO<sub>2</sub>

variation, we focus on the O K-edge alteration in the current investigation. Figure 5 shows the EELS spectra obtained at different heating temperatures. In Figure 5, peak 1 of O K-edge including the arrowed shoulder is explained as transitions from the O 1s core state to a hybridized O 2p-Mo 4d states.19 The octahedral crystal field splits the Mo 4d band into  $t_{2g}$  and  $e_g$  sub bands, and furthermore, the distortion of  $\text{MoO}_6$  splits  $t_{\rm 2g}$  and  $e_{\rm g}$  into four peaks. Because of the energy resolution of the EELS spectrum (~1.5 eV), we can only resolve peak 1 and its shoulder. Compared to that in ambient temperature, the full width at half maximum (FWHM) of peak 1 increases at 450°C. When temperature reaches 916°C, the FWHM becomes larger. This is because that the intensity of the shoulder of peak 1 (highlighted by the arrow in EELS spectrum at 450°C) increases with temperature while the sharp part of peak 1 keeps decreases. In the end, a flat peak forms. In MoO<sub>3</sub>, the six  $t_{2g}$  and the four  $e_g$  orbitals are empty, thus O 1s electron can be excited and jump into the empty  $t_{2g}$  and  $e_g$  orbitals. In MoO<sub>2</sub>, the  $t_{2g}$  orbitals are filled with two electrons, which results in the increase the weight of  $e_g$  orbitals and decrease the weight of  $t_{2g}$  orbitals. Hence, difference weighing in  $t_{2g}$  and  $e_g$  for MoO<sub>3</sub> and  $MoO_2$  explains the shape changes of peak 1.

As stated above, a consecutive mechanism is proposed to explain the transformation from  $MoO_3$  to  $MoO_2$ , in

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**FIGURE 7** (A) Another precipitate in MoO<sub>2</sub> matrix. Indexation of power spectrum and its simulation ((B) and (C)) suggests the precipitate is Mo<sub>4</sub>O<sub>11</sub>. HRTEM image reveals  $(311)_{Mo4O11}/(\bar{1}11)_{MoO2}$ 

which Mo<sub>4</sub>O<sub>11</sub> is treated as an intermediate phase.<sup>13</sup> However, our in-situ electron diffraction investigation gives supporting evidence to the other mechanism, i.e., MoO<sub>2</sub> topotactically formed from MoO<sub>3</sub> directly. HRTEM provides lattice image of secondary phase and matrix from which we can decide the orientation between them, thus we can postulate the actual formation mechanism. Figure 6 shows the HRTEM image of a region right after in-situ experiment is finished. Several secondary phases are seen in the image. The matrix is  $MoO_2$  (see power spectrum as inset) orientated at [100]. Judging from the sizes of the secondary phases, we postulate that they nucleate and grow up from the matrix MoO2. Figure 7 shows another region where the Moiré fringes of secondary phase and matrix are clearly visible. Indexation of the power spectrum of the secondary phase (Figure 7B,C) suggests the secondary phase is Mo<sub>4</sub>O<sub>11</sub> and there is orientation relationship between  $Mo_4O_{11}$  and  $MoO_2$  (e.g.,  $\left(311\right)_{Mo4O11}/(111)_{MoO2}$ (Figure 7A). Because the composition of  $Mo_4O_{11}$  is between  $MoO_3$  and  $MoO_2$  and there is no other phase formed between them as revealed by in-situ electron diffraction, we believe that the oxygen of the underneath unreacted MoO<sub>3</sub> diffuses and reacts with MoO<sub>2</sub> to form Mo<sub>4</sub>O<sub>11</sub>. The existence of OR between the precipitated  $Mo_4O_{11}$  and  $MoO_2$  matrix supports such postulation.

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In response to Sloczynski's comment on whether the existence of  $Mo_4O_{11}$  indicates an autocatalytic mechanism and two-step process for the reduction in  $MoO_3$  to  $MoO_2$  in hydrogen, Ressler argued that  $Mo_4O_{11}$  is not intermediate in the reduction from  $MoO_3$  to  $MoO_2$ .<sup>12,13,20</sup> Our work agrees to Ressler's result. In a subsequent study on

reduction in MoO<sub>3</sub> with propene,<sup>21</sup> Ressler, however, found there was an intermediate Mo18O52. The inconsistency in MoO<sub>3</sub> reduction with different reactant (e.g., hydrogen and propene) reveals the complexity of the reduction process for MoO<sub>3</sub>. There are at least seven different suboxides between  $MoO_3$  and  $MoO_2^{22}$   $Mo_{18}O_{52}$ (MoO<sub>2.89</sub>), according to Bursill,<sup>9</sup> can be viewed as a layout of  $3 \times 6$  MoO<sub>6</sub> octahedra shear structure on the (010) plane of MoO<sub>3</sub>, which indicates that the structure of  $Mo_{18}O_{52}$  is still quite close to MoO<sub>3</sub> although short ordering of oxygen vacancies happens. Bearing in mind that the streaking feature shown in the electron diffraction pattern in Figure 3A is associated with short ordering of oxygen vacancies, it would not be unexpected if Mo18O52 precipitates as long as the annealing time is longer enough. However, the existence of MoO<sub>3</sub>-structure related Mo<sub>18</sub>O<sub>52</sub> does not mean that the formation of a ReO<sub>3</sub> type of structure such as  $Mo_4O_{11}$  is unavoidable.

### 4 | CONCLUSIONS

The reduction in  $MoO_3$  is investigated by in-situ electron diffraction. As a result, we find that  $MoO_3$  is directly reduced to the monoclinic  $MoO_2$ . Although  $Mo_4O_{11}$  is found by HRTEM observation, it is postulated that  $Mo_4O_{11}$ is formed by the reaction between  $MoO_3$  and  $MoO_2$  considering the small size of  $Mo_4O_{11}$  and the orientation relationship between  $Mo_4O_{11}$  and the matrix  $MoO_2$ . The emerging of streak in the diffraction pattern indicates the short ordering of oxygen vacancies. The elimination of the oxygen vacancies can potentially lead to the formation of  $MoO_3$ -related structure such as  $Mo_{18}O_{52}$  or  $Mo_{13}O_{38}$ ; however, due to the rapid heating rate and relatively short duration time, we did not find any of these phases.

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