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ABSTRACT
Due to the increased surface to volume ratios, topological nanomaterials can enhance contributions from the topological surface states in transport measurements, which is critical for device applications that exploit the topological properties. It is particularly important for topological semimetals in which bulk carriers are unavoidable to make them into nanostructures to reveal the nature of the topological surface states, such as the Fermi arcs or nodal lines. Here, we report the nanostructure synthesis of the recently discovered triple-point topological metal MoP by direct conversion of MoO$_3$ nanostructures and study their transport properties. We observe that the initial size of the MoO$_3$ templates critically determines the crystalline quality of the resulting MoP nanostructures: large MoO$_3$ flakes lead to porous MoP flakes, while narrow MoO$_3$ nanowires lead to MoP nanowires without pores. The size-dependent porosity observed in MoP nanostructures is attributed to the volume change during the conversion reaction and nanoscale confinement effects. For MoO$_3$ nanowires with diameters less than 10 nm, the resulting MoP nanowires are single crystalline. The resistivity values of MoP nanostructures are higher than the reported values of MoP bulk crystals owing to the porous nature. However, despite the high porosity present in MoP flakes, the residual resistance ratio is $\sim 2$ and the temperature-dependent resistivity curves do not show any strong surface or grain-boundary scattering. Demonstration of the facile synthesis of MoP nanostructures provides opportunities for careful investigations of the surface states in transport measurements and exploration of future electronic devices, including nanoscale interconnects.

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INTRODUCTION
Roughly, a quarter of all electronic materials are suspected to be topological in nature in which their band structures belong to a different topology than those of ordinary materials. 1–3 The most recent additions to the family of topological materials are topological metals such as Dirac semimetals 4–8 and type-I and type-II Weyl semimetals, 9–11 which possess interesting Fermi arcs or nodal lines on the surface of the material. Unlike topological insulators, which are largely void of residual bulk carriers and have a Fermi level placed within the bulk bandgap, 12–16 topological metals retain their bulk carriers because of their metallic nature. One way to greatly suppress the bulk carriers is to fabricate the nanostructures of topological metals with increased surface to volume ratios. Several recent studies on Cd$_3$As$_2$ nanoslabs and nanoribbons, 17–19 WTe$_2$ nanoribbons and monolayers, 20,21 and MoTe$_2$ flakes 22 have demonstrated the clear advantage of using nanostructures to study surface states through transport measurements. Interestingly, the transport properties are closely tied to the overall dimensions and morphology of the nanostructures, providing a
powerful knob for controlling the surface states of these topological metals.

Among recently reported topological semimetals, the triple point topological metal molybdenum phosphide (MoP) has shown extremely low residual resistivity, high carrier density, high mobility, and long mean free path.\(^2\) The reported transport properties are intriguing not only for fundamental studies but may also have transformative impact in the semiconductor industry as a potential solution to current challenges with Cu interconnects. The topological protection predicted for MoP may suppress the surface and grain-boundary electron scattering that plagues the current Cu interconnects as they continue to shrink in size.\(^{25,26}\) MoP has also been studied as a promising catalyst for the hydrogen evolution reaction (HER). To enhance the catalytic activity, various MoP nanostructures including interconnected nanoparticles,\(^{27,28}\) thin films,\(^9\) and amorphous nanoparticles\(^7\) have been fabricated.

To advance our understanding of the nature of triple point topological metals and explore the feasibility of MoP as potential interconnects and HER catalysts, synthesis and transport property studies of MoP nanostructures of various sizes and shapes are essential. Here, we demonstrate the synthesis of nanostructured MoP by the direct conversion of MoO\(_3\) nanostructures in a tube furnace in the presence of PH\(_3\) gas, which was generated by the evaporation of P-containing precursors in H\(_2\) carrier gas. The resistances of MoP nanostructures were measured as a function of temperature and suggest a robust topological protection.

RESULTS

In this work, we synthesized MoP nanostructures by converting MoO\(_3\) nanostructures using a horizontal tube furnace, as shown schematically in Fig. 1(a). MoO\(_3\) nanostructures were first grown on SiO\(_2\)/Si substrates by the chemical vapor deposition (CVD) of MoO\(_3\) precursors\(^17\) or the oxidation of Mo powders. The MoO\(_3\) containing substrates were placed at the center of the tube furnace, and P-containing precursors were placed upstream, 15 cm away from the center. The tube furnace was heated to 700 °C with a constant flow of H\(_2\) gas, leading to the generation of PH\(_3\) gas. The reaction between the metal oxide nanostructures and PH\(_3\) converts the oxide into phosphide nanostructures.\(^{32-35}\) To obtain MoP nanostructures with different morphologies and configurations, conversion conditions such as the temperature, reaction time, H\(_2\) gas flow rate, and P-containing precursors were varied (details of the conversion conditions can be found in the section titled Experimental).

Two different MoO\(_3\) nanostructures—nanoflakes and nanowires—were used as templates for the MoP nanostructure synthesis. The MoO\(_3\) nanoflakes were synthesized by heating Mo powder to 900 °C for 1 h in air, and MoO\(_3\) nanowires were grown by CVD as detailed in our previous work.\(^1\) The synthesized nanoflakes give the substrate a black or dark bluish coloration, which is strongest at the leading edge (Fig. S1) where the nanowire yield is the highest. Figures 1(b) and 1(d) show scanning electron microscopy (SEM) images of the MoO\(_3\) nanoflakes and nanowires. X-ray diffraction (XRD) patterns confirmed the synthesis of MoO\(_3\) nanoflakes, which matched the standard XRD pattern of MoO\(_3\) (JCPDS card No. 47–1320) (Fig. S1). The microstructure of the MoO\(_3\) nanostructures was characterized by transmission electron microscopy (TEM), which showed that they were single crystalline (Fig. S1). To convert them into MoP nanostructures, the MoO\(_3\) nanostructures were placed at the center of a tube furnace with the Na\(_2\)H\(_2\)PO\(_2\) powder placed upstream, with H\(_2\) flow at atmospheric pressure and were heated to 700 °C for 1 h [Fig. 1(a), details of the conditions are provided in the section titled Experimental]. The Na\(_2\)H\(_2\)PO\(_2\) powder decomposes and reacts with H\(_2\) to generate PH\(_3\) gas. The high reactivity of PH\(_3\) and H\(_2\) induces the conversion of MoO\(_3\) to MoP.\(^{33,35}\) The nanoflake morphology changes after the conversion [Figs. 1(b) and 1(e)]; notable porous structures are observed in the converted MoP (scanning TEM (STEM) images are shown in Fig. S2).

The change in morphology of the nanostructures after conversion is attributed to the different crystal structure of MoO\(_3\) and MoP.

![FIG. 1. Synthesis, characterization, and classification of MoP nanostructures. (a) Schematic of the furnace setup used in the synthesis. (b)–(e) SEM images of the two different morphologies discussed in the main text: nanoflakes and nanowires of MoO\(_3\) [(b) and (d)] and MoP [(c) and (e)], respectively (scale bar: 200 nm). (f) Schematic of the morphology change during the conversion reaction. (g) Schematic of the crystal structure of MoO\(_3\); the Mo and O atoms are shown as light blue and red spheres, respectively. (h) Schematic of the hexagonal crystal structure of MoP where the Mo and P atoms are shown as light blue and orange spheres, respectively.](https://example.com/figure1)
Thermodynamically stable α-MoO₃ has an orthorhombic crystal structure with planar double layers, as shown in Fig. 1(g). MoP has a WC-type hexagonal crystal structure [Fig. 1(h)] with lattice parameters a = b = 3.22 Å and c = 3.19 Å, and Mo and P share the same coordination environment and coordination number of six in a trigonal prism. The change in the crystal structure and subsequent volume change during conversion can explain the observed porosity in the converted MoP nanoflakes. Unlike large flakes, however, MoP nanowires converted from MoO₃ nanowires do not show any pores and have relatively smooth surfaces [Figs. 1(d) and 1(e)]. This is attributed to the fact that nanowires can withstand much larger volume changes and strain, just as the well-known example of the lithiation of Si nanowires, which can undergo a volume change of 400% without creating pores or cracks.

The crystal structure and chemical composition of the synthesized MoP nanostructures were further confirmed by XRD. Figure 2(a) shows the XRD pattern of the MoO₃ and MoP nanostructures before and after the conversion reaction. The XRD pattern from the MoO₃ nanostructures matches the standard XRD pattern of MoO₃ (JCPDS card No. 47–1320) with the relative intensities of the (400) and (600) diffraction peaks being more significant, suggesting a preferred orientation during growth. After conversion, the XRD patterns of the final products show the characteristic diffraction peaks of MoP (JCPDS card No. 65–6024), demonstrating the successful synthesis of MoP nanostructures. The chemical composition of the MoP was analyzed using TEM energy-dispersive X-ray spectroscopy (EDS). Mapping of the Mo, P, and O elements was carried out to show the homogeneity of the composition (Fig. S3).

We systematically varied the reaction conditions of the MoP conversion and then used XRD to map out the phase diagram, as shown in Fig. 2(b). At a lower reaction temperature (650 °C) and 1 h reaction time, the product shows typical peaks of MoO₂ (JCPDS card No. 32–0671), but no distinct characteristic peaks of MoP (Fig. S4). At 700 °C for a reaction time of 0.5 h, the product shows typical peaks of MoO₂ and MoP, and when the reaction time is increased to 1 h, MoP peaks appear, while the peaks of MoO₂ disappear (Fig. S4). At higher reaction temperatures (750–800 °C), XRD patterns indicate MoP nanostructures for all reaction times (0.5, 1, and 2 h), as shown in Fig. S5. In other words, higher reaction temperatures of 700 °C and above result in a more efficient conversion of MoO₃ to MoP, while reaction temperatures less than 700 °C do not promote this transition well. Moreover, we note that the degrees of reactivity depend on the choice of P-containing precursors, which can be modulated by the flow rate of H₂ gas and which leads to the copresence of MoO₂ and MoP in the synthesis. For example, the flow rate of H₂ influenced the yield ratio of MoP and MoO₂ products for red phosphorus precursors, but it did not influence the yield ratio for NaH₂PO₂·H₂O precursors, as shown in Fig. S6.

The microstructure and crystal structure of MoP nanostructures were also characterized by TEM. Figure 3 shows TEM images of MoP nanostructures of various sizes and shapes. In the case of a nanoflake [Figs. 3(a) and 3(d)], the porous nature of the MoP flake is clearly visible with large crystalline grains reflected in the selected area electron diffraction (SAED) pattern [inset of Fig. 3(a)]. The spacing of the lattice fringes observed in a high-resolution TEM image was measured to be 0.21 nm, corresponding to the (101) plane of MoP [Fig. 3(d)]. Unlike the nanoflakes, MoP nanowires do not have pores and contain several grains along the length of the nanowires [Fig. 3(b)]. The lattice spacing along the axis of the nanowire was found to be 0.32 nm, corresponding to the (001) plane of MoP [Fig. 3(e)]. The crossover from porous to nonporous MoP nanostructures occurs at the critical width of ~100 nm; MoP nanowires with diameters <100 nm do not contain pores. Interestingly, MoP nanowires with diameters less than 10 nm were single crystalline [Figs. 3(c) and 3(f)]. This can be explained by the nanoscale confinement effect, where the lack of nuclei and the prohibitively high energy cost of grain boundaries at the nanoscale result in single-crystalline rather than polycrystalline grains.

We analyzed the grain size as a function of nanowire diameter, as shown in Fig. 3(g). Nanowires were observed to be single crystalline when their diameters were below 10 nm. Nanowires were polycrystalline and nonporous when their diameters ranged between 10 and 100 nm. For these nanowires, the average grain size increased with increasing nanowire diameter until it reached an asymptotic value of ~20 nm. MoP nanostructures with widths >100 nm were observed to be porous and showed an average grain size of ~23 nm. The grain size was also studied as a function of growth conditions by analyzing the XRD (101) peak using the Scherrer equation. The grains became larger at higher reaction temperatures, which can be expected due to coarsening [Fig. 3(h)]. Meanwhile, the
FIG. 3. Characterizations of the different morphologies of MoP converted from MoO$_3$ at 700 °C for 1 h. TEM images of nanoflakes [(a) and (d)] and nanowires [(b), (c), (e), and (f)]. (a) TEM image of a MoP nanoflake (scale bar: 200 nm). The inset in (a) shows the SAED pattern image (scale bar: 50 nm). (b) TEM image of a MoP nanowire (scale bar: 50 nm). (c) TEM image of a narrow (diameter < 10 nm) MoP nanowire (scale bar: 50 nm). [(d), (e), and (f)] High magnification TEM images of the red dotted box in (a), (b), and (c), respectively (scale bars: 5 nm). The MoP lattice fringes are indicated in white in (d)–(f). (g) Average grain size as a function of nanowire diameter obtained from TEM image analysis. For nanowires with diameters > 100 nm, pores were observed. (h) The average grain size and porosity of MoP nanostructures as a function of reaction temperature. The average grain size was obtained by analyzing the XRD (101) peak obtained from the growth substrate, which includes large MoP structures.

Porosity decreased at higher reaction temperatures (see Fig. S7 in the supplementary material for representative SEM images used in the analysis).

For transport measurements, we fabricated four-point contact nanodevices on a porous MoP nanoflake using standard e-beam lithography and thermally evaporated Cr/Au contacts [Fig. 4(a)]. The four-point probe resistance of the MoP nanoflake was measured as a function of temperature from room temperature down to 2 K [Fig. 4(b)]. The average height profile of the flake, obtained by atomic force microscopy (AFM), was 51 nm with a root-mean-square (rms) roughness of 13 ± 5 nm (Fig. S8). Despite the highly porous nature and surface roughness, which would normally induce large surface scattering, the starting resistivity was 116 μΩ cm at room temperature and decreased to 54 μΩ cm at 2 K with a residual resistance ratio of ~2 [red curve in Fig. 4(b)]. We note that the resistivity was calculated using the geometric area without factoring in the porosity. Based on our previous studies of WTe$_2$ and SnTe nanostructures, which are quite resistive and suffer from surface
The measured resistivity of the porous flake was compared to sintered MoP bulk samples made by sintering commercially purchased MoP powders at 1000 °C for 12 and 24 h. Figure 4(b) shows the resistivity of the bulk samples as a function of temperature, which shows 114.6 μΩ cm and 88.1 μΩ cm at 300 K and reaches 15.5 μΩ cm and 1.7 μΩ cm at 2 K, respectively [black and blue curves in Fig. 4(b)]. Figures 4(c) and 4(d) show cross-sectional SEM and TEM images of the bulk sample, which reveals that the grains are 200–500 nm wide, much larger than the average grain size of ~23 nm for the porous nanoflake. Thus, the reduced resistivity of the polycrystalline bulk is attributed to the larger grain size. Indeed, the resistivity of MoP single crystals is 8 μΩ cm at 300 K. We note that the transport properties of single-crystalline and polycrystalline MoP nanowires could not be obtained due to challenges related to fabricating nanodevices on such small nanowires and their propensity to be damaged easily. Efforts to measure narrow MoP nanowires are ongoing.

**CONCLUSION**

In summary, we demonstrate a reliable synthesis strategy of triple-point topological metal MoP nanostructures and report resistivity measurements of the resulting porous nanoflake. A phase diagram for the conversion reaction from MoO$_3$ to MoP nanostructures was constructed as a function of reaction time and reaction temperature. The resulting MoP nanostructures tend to be porous to accommodate the volume change that occurs during conversion. Narrow MoP nanowires did not contain pores and could be single crystals due to the geometrical confinement effects. The transport properties of porous MoP nanostructures were measured using four-point probe geometry. The synthesis strategy demonstrated in this work opens opportunities to study the nature of topological surface states in this topological metal, which could be sensitive to the overall morphology and dimensions of the nanostructures, and explore their feasibility as low-dissipation interconnects as well as effective HER catalysts.

**EXPERIMENTAL**

**MoO$_3$ flake synthesis**

1 g of molybdenum powder (Sigma-Aldrich, 99.99%) was placed at the center of a furnace and heated up to 900 °C for 1 h in ambient air. Mo was converted to MoO$_3$ flakes with varying dimensions.

**MoO$_3$ nanowire synthesis**

As previously reported by our group, MoO$_3$ nanowires were grown by CVD. 0.15 g of the MoO$_3$ source powder (Sigma-Aldrich, 99.95%) was placed at the center of a 1 in. tube furnace with [100] Si substrates located 15 cm downstream. After purging with Ar, the system was pumped down to 100 mTorr, and then, H$_2$ was flowed at 20 sccm, bringing the furnace pressure to 5 Torr. The furnace was heated to 600 °C in 15 min and held at that temperature for 10 min to produce MoO$_3$ nanowires with high yield.
Fabrication of polycrystalline MoP bulk rod and transport measurements

Commercially purchased MoP powder (Alfa-Aesar, 99.95%) was pressed at 10 tons/cm² into a cylinder-shaped mold and subsequently sintered at 1000 °C for 12 and 24 h in a 5% H₂/Ar environment. The sintered pellet was sliced with a diamond saw into a rod shape. Four electrodes with 1–2 mm spacing between them were fabricated by thermal deposition of a 20/300 nm-thick Cr/Au layer (M Braun MB-EcoVap). Transport measurements at low temperature were performed using a Quantum Design Dynacool physical property measurement system equipped with a base temperature of 2 K. Resistance scans were taken as a function of temperature at an applied current value of 5 mA.

SUPPLEMENTARY MATERIAL

See the supplementary material for supporting Figs. S1–S8.

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The authors declare no competing financial interest.

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