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Congruent melting of tungsten phosphide at 5 GPa and 3200 °C for growing its large single crystals*

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As one of important members of refractory materials, tungsten phosphide (WP) holds great potential for fundamental study and industrial applications in many fields of science and technology, due to its excellent properties such as superconductivity and as-predicted topological band structure. However, synthesis of high-quality WP crystals is still a challenge by using tradition synthetic methods, because the synthesis temperature for growing its large crystals is very stringently required to be as high as 3000 °C, which is far beyond the temperature capability of most laboratory-based devices for crystal growth. In addition, high temperature often induces the decomposition of metal phosphides, leading to off-stoichiometric samples based on which the materials' intrinsic properties cannot be explored. In this work, we report a high-pressure synthesis of single-crystal WP through a direct crystallization from cooling the congruent W–P melts at 5 GPa and ∼ 3200 °C. In combination of x-ray diffraction, electron microscope, and thermal analysis, the crystal structure, morphology, and stability of recovered sample are well investigated. The final product is phase-pure and nearly stoichiometric WP in a single-crystal form with a large grain size, in excess of one millimeter, thus making it feasible to implement most experimental measurements, especially, for the case where a large crystal is required. Success in synthesis of high-quality WP crystals at high pressure can offer great opportunities for determining their intrinsic properties and also making more efforts to study the family of transition-metal phosphides.

Keywords: congruent melting, tungsten phosphide, single crystals, high pressure and high temperature

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1. Introduction

Transition-metal (TM) phosphides have been widely studied in the fields of industrial catalysis,[1–11] magnetic refrigeration systems,[12–14] superconductivity,[15–19] optoelectronic devices, and semiconductors[20,21] due to their exceptional structural, electronic, magnetic, and catalytic properties. For most of binary TM phosphides, there often exist a large number of compounds with different compositions, mainly because the transition metals possess many d electrons, thus making it allowable that their oxidation states can be largely varied. In the W–P binary system, a series of materials has been reported including tetragonal W_3P[17] and WP_4,[22] orthorhombic WP,[2] and WP_2,[23] *etc*. Over the past decades, they have been intensively studied mainly due to their intriguing electronic properties for the study of diverse fundamental sciences, such as superconductivity (*e.g.*, W_3P),[17] Weyl semimetals (*e.g.*, WP_2).[24,25] Among them, WP is a shining member of phosphides, exhibiting superconductivity of a weak-coupling BCS type.[18] Very recently, based on the first-principles calculations, it has been predicted that WP possesses a unique topological band structure.[26] But such a theoretical prediction has not yet been validated experimentally, because the preparation of high-quality single-crystal WP samples is still a challenge by traditional reaction routes at

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atmospheric pressure. As a matter of fact, a similar issue also occurs for most of TM phosphides, which severely restricts the scientific study on this whole class of materials.

Traditional methods for synthesizing WP include the element reaction by ampoule techniques and arc melting,[2] the reduction of phosphate precursors in hydrogen,[6] the hydrogen plasma reduction,[7] and the decomposition of organometallics.[2] However, most of the obtained samples are poorly-crystallized and in nanocrystalline forms, which have been employed as catalysts for hydrogenating process.[2–5] Obviously, those methods cannot be used for preparing the high-quality WP crystals, because of the limited thermal stability of this compound at ambient pressure with a low decomposition temperature of $\sim$ 1000 ℃, even lower than its synthesis temperatures. As a result, the composition of final products often deviates from its ideal stoichiometry. The thus-degassed phosphor gas for traditional methods is chemically active and toxic, which may contaminate and damage the expensive furnace devices. Growing large single crystals of materials from their high-temperature melts is one of mostly exploited approaches, but it is almost impossible for refractory materials with very high melting points. Taking WC for example, its ambient-pressure melting point is close to $\sim$ 3000 ℃,[27] which is far beyond the temperature capability of most laboratory-housed furnace devices. As a sister material, the WP should also have an extremely high melting point. As such, disadvantages of traditional synthetic methods have posted a great challenge to the preparation of high-quality WP single crystals, although chemical vapor transport (CVT) technique has been used for growing single-crystal WP[18] or WP_2,[24] it still has some problems to be solved, such as high experimental cost, slow synthesis rate, low quality, and small-scale production.

High pressure (P) and temperature (T) synthesis has proven to be an effective route for the synthesis of TM-bearing compounds or for the discovery of their novel materials including borides, nitrides, and phosphides, because the oxidation states of metals tend to vary with pressure. Accordingly, a number of novel nitrogen-rich TM nitrides have recently been discovered under pressures in the Zr–N,[28,29] Hf–N,[28] Ta–N,[30,31] Mo–N,[32] W–N,[33] and noble metal nitride systems,[34–37] suggesting that the pressure can effectively suppress the high-T degassing and promote the involvement of d-electrons in chemical bonding.[38] Successful syntheses have also been achieved in the TM phosphides of ZnP_4,[39] Ir_2P,[40] and Cd–P (*e.g.*, CdP_4, and Cd_7P_{10})[41,42] and borides of HfB_2[43] and TiB_2.[44] Based on large-volume high-P devices, the millimeter-sized GaN single crystals have recently been grown from its continently melted liquid at 6 GPa and 2200 °C;[38] conversely, the complete decomposition of GaN has also been observed below 6 GPa, further confirming the crucial role of pressure played in this process. However, for materials with very high melting points (*i.e.*, above 3000 °C), it is difficult to implement this method for growing their single crystals, because for most available large-volume apparatus their temperature conditions are limited to $\sim$ 2300 ℃. Very recently, we have re-designed a new high-P cell assembly of large-volume cubic press and significantly optimized its heating efficiency, leading to extraordinarily extended P–T conditions of 10 GPa and 3700 °C.[27] Using the optimized cell, large single-crystal refractory materials of Mo, Ta, and WC have been made,[27] indicating that a similar method can also be used for the growth of WP single crystals from its congruent melts.

With these aims, in this work we perform high P–T experiments for growing large single-crystal WP from congruently pre-melted W and P liquids at 5 GPa and temperature up to 3200 °C, leading the high-quality WP crystals with large crystallite size of a few millimeters to success in being prepared.

2. Experiment details

The purchased high-purity W powders and red phosphorus were used as starting materials for high P–T growth of crystals. Prior to the experiment, W and P powers were homogenously ground and mixed in a molar ratio of 1 : 1. The mixed powders were compacted into a cylinder with 8 mm in diameter and 4 mm in height, which was surrounded by hBN capsule. Note that the hBN could dissolve into the W–P melts at high temperature; during cooling, it precipitated on the surface of each crystal, which facilitated the separation of crystals to avoid the formation of polycrystalline bulk continuum. High-P experiment was carried out in a 6×10 MN large-volume cubic press installed at the high-P laboratory of SUSTech. At the target pressure, the sample was gradually heated to the desired temperature. For a safe operation of pressure, the heating process was carefully program-controlled. Depending on the required experiment temperature, the heating duration was strictly restricted, in avoidance of blowout. Accordingly, at temperatures of 1800 °C, 2400 °C, 2700 °C, 3000 °C, and 3200 °C, the holding time for heating were 30 min, 5 min, 2 min, 1 min, and 0.5 min, respectively, followed by cooling with a rate of 300 °C/min. More experimental details can be found elsewhere.[27,45,46] The recovered samples were characterized by x-ray diffraction (XRD), scanning electron microscope (SEM), and thermogravimetric-differential thermal analyzer (TG–DTA). Refinement of crystal structure was performed by using the Rietveld method and the FullProf program.[47]

In addition, single-crystal diffraction was also performed to check the quality of as-prepared crystals by using the single crystal x-ray diffractometer (BRUKER D8 VENTURE) and Laue diffractometers. Energy dispersive spectrometer

(EDS) and electron probe micro-analyzer (EPMA) measurements were conducted to analyze the chemical composition of samples.

3. Results and discussion

In order to understand the high P–T reaction process for the formation of WP from reaction between W and P, a number of synthesis experiments are performed at pressures of 1.0 GPa–6.0 GPa and temperatures of 400 °C–3200 °C. Figure 1(a) shows the typical XRD patterns of recovered samples synthesized under different P–T conditions. At a low temperature of 800 °C, only W and P elements are identified, suggesting that there is no reaction occurring. With temperature increasing, the target material WP starts to appear at a temperature exceeding 900 °C, but impurity phases of WP_2 and WP_4 also occur in this temperature range. Above 1600 °C, the obtained product is phase-pure WP, which can form in a wide range of temperatures of 1600 °C–3200 °C. This is in striking contrast to that occurring at ambient pressure and a low decomposition temperature of ∼ 1000 °C, suggesting that its thermal stability is profoundly promoted at pressure. Figure 1 shows a summary of the P–T diagram for forming WP. Apparently, two boundaries are well defined to separate the formation of WP in the P–T space, according to the XRD measurement of recovered samples in Fig. 1(a). Obviously, the thus-determined P–T diagram for forming WP is valuable for the future laboratory and its industrial-scale production.

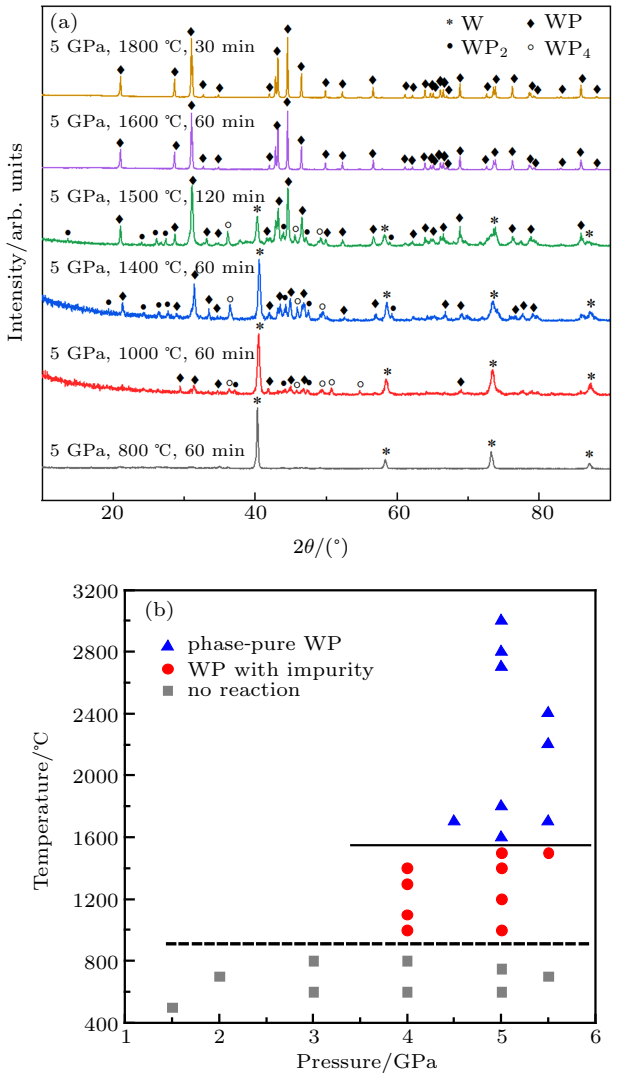


Fig. 1. (a) XRD patterns of recovered samples synthesized under different high P–T conditions. (b) High P–T forming region of WP. No reaction is observed below black dashed line. Phase-pure WP occurs above black line. Between the two lines formed is WP with by-products of WP_2 and WP_4.

Figure 2 shows the refined powder XRD pattern. It is evident that all the diffraction peaks of this material match with an orthorhombic structure with a space group of *Pnma* (No. 62). The thus-refined lattice parameters are $a = 5.72786(3)$ Å, $b = 3.24745(2)$ Å, and $c = 6.22046(4)$ Å, which are in good agreement with previous reports.[35–37]

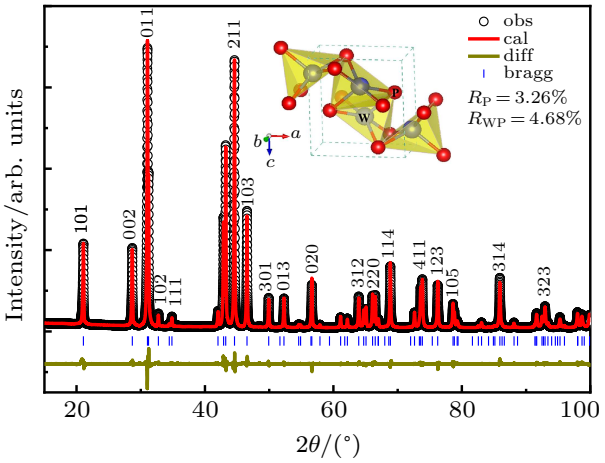


Fig. 2. Refined XRD pattern of polycrystalline WP synthesized at 5 GPa and 1600 °C taken at ambient conditions by using a copper radiation. Inset shows a polyhedral view of its crystal structure.

As presented in Figs. 3(a)–3(c), at 5 GPa and a relatively low temperature of 1600 °C, the obtained samples are polycrystalline bulk continuum of WP. The crystallite size is very small (*i.e.*, ∼ 5 μm), despite a prolonged heating time of 60 min (Fig. 3(c)). We also study the influence of cooling rate on grain growth as seen in Figs. 3(b) and 3(c); despite a slow cooling rate, the grain size is retained. This suggests that the applied temperature is insufficient for diffusing atoms, which is a prerequisite for grain growth. As temperature increases to 2400 °C (Fig. 3(d)), the recovered sample is still polycrystalline WP and the grain boundaries are seemingly formed with a grain size of around 100 μm–200 μm.

To our surprise, large WP crystals occur as temperature exceeds 3000 °C, the results are shown in Figs. 3(e)–3(f). This strongly implies the fusion of WP above 3000 °C under a pressure of 5 GPa, which explains the reason why large crystals can grow, although the cooling process is short, within only a

few minutes. Clearly, at such extreme *P*–*T* conditions, both W and P can melt together to form uniform melting liquid. From this viewpoint, the WP nucleates and grows from congruent melts made of W and P atoms. Using a similar method, Utsumi *et al.* have also prepared large crystals of GaN at 6 GPa and 2200 °C.[38] Besides, this is also the first time for measuring melting point of WP at ∼ 3200 ℃ around 5 GPa, close to that of WC,[27] which makes it stand out as an excellent refractory material with very high melting point. Evidently, the thus-formulated high-*P* approach to growing large crystals of WP will also be applicable for other TM phosphides. Figure 3(g) shows an optical image of WP single crystal synthesized at 3200 °C and 5 GPa. It is worthwhile to mention that the crystals can readily be separated from each other, as a result of formation of hBN that is coated on the surface of each crystals. This is because capsule material hBN can be partially dissolved in the W–P melts at high temperature; during cooling, it precipitates on the surface of each crystals. Nevertheless, this offers an excellent solution to the separation of the obtained single crystals. Also noted is the morphology of obtained WP crystal (in Fig. 3(g)), having a uniform angular shape, remarkably differs from those of WP crystals synthesized by using CVT method with a shiny needle-like shape.[18]

To further check the quality of such-prepared WP crystals, we conduct single-crystal diffraction measurements, and the results are shown in Figs. 3(h)–3(i). All the Laue diffraction spots in Fig. 3(h) can be identified and correspond to the (100) crystallographic plane, which is a typical characteristic of single crystal. In addition, based on a 200-μm crystal, a number of small and clear diffraction spots are also recorded by using the single-crystal diffractometer as seen in Fig. 3(i), further confirming high quality of crystal. The crystal structure is also refined by using the single-crystal XRD data; the thus-refined lattice parameters are $a = 5.7248(4)$ Å, $b = 3.2474(3)$ Å, and $c = 6.2198(5)$ Å, which are in excellent agreement with those of polycrystalline sample and the reported values.[18,48–50] The obtained lattice parameters are tabulated in Table 1.

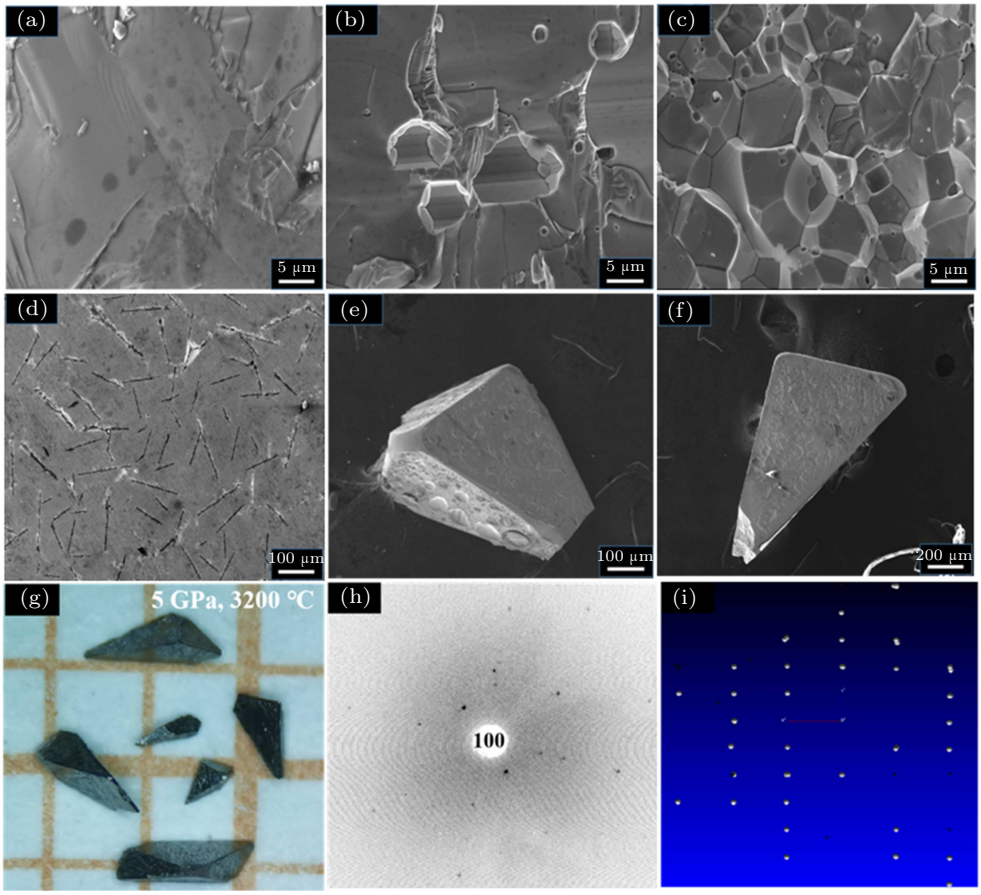


Fig. 3. SEM and optical observations and single-crystal diffraction measurements for WP, showing SEM images of WP prepared at 5 GPa and different temperatures with different holding durations at (a) 5 GPa and 1600 °C for 30 min, (b) 5 GPa and 1600 °C for 60 min and followed by quenching, (c) 5.0 GPa and 1600 °C for 60 min with a cooling rate of 40 °C/min, (d) 5.0 GPa and 2400 ℃ for 5 min, (e) 5.0 GPa and 3000 °C for 0.5 min with a cooling rate of 600 °C/min, (f) 5.0 GPa and 3200 °C for 0.5 min with a cooling rate of 800 °C/min, (g) optical image of WP synthesized at 3200 °C. Size of the background grid is 1 mm × 1 mm, (h) Laue diffraction pattern, and secondary symmetry of the (100) plane is identified, (i) single-crystal diffraction patterns. In panels (h) and (i), diffraction data are taken at room temperature.

To determine the chemical composition of WP crystals, we also perform EDS and EPMA measurements on high-*P* synthesized WP crystals, and the results are shown in Fig. 4. As expected, the elements are uniformly distributed over the whole crystal. The EDS analysis shows that the determined element ratio of WP is W : P = 51.82 : 48.18, slightly deviating from a unity because of the remarkable difference in sensitivity of the EDS technique to light element (*e.g.*, P) and heavy element (*e.g.*, W). In contrast, the EPMA probe is often more accurate and has extensively been used for characterizing the composition of TM phosphides; the obtained element ratio is W : P = 50.82 : 49.18, close to 1 : 1. We thus conclude that the as-prepared WP crystals have nearly a stoichiometric composition, in spite of the fact that the sample is synthesized at extremely high temperature of 3200 °C. On the other hand, this also infers that the pressure can profoundly improve the thermal stability of TM phosphides and suppress their decomposition.

Table 1. Refined crystal structure parameters for WP by using single-crystal diffraction data.

Empirical formula	WP
Formula weight/(g/mol)	214.81
Temperature/K	293
Wavelength/Å	0.71073
Crystal system	orthorhombic
Crystal color	black
Space group	*Pnma* (62)
a/Å	5.7248(4)
b/Å	3.2474(3)
c/Å	6.2198(5)
α/(°)	90
β/(°)	90
γ/(°)	90
Cell volume/Å^3	115.631(16)
Z	4
Calculated density/(g/cm^3)	12.339
R_{wp}/%	3.14

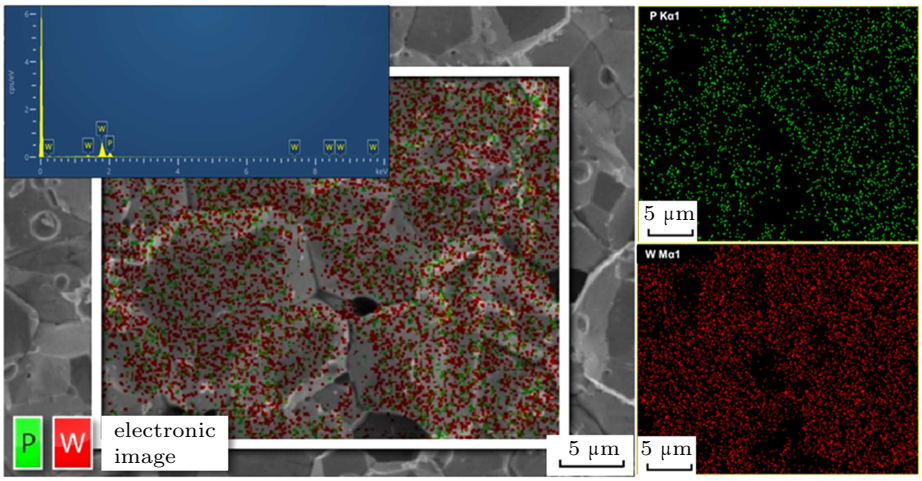


Fig. 4. Element mapping for WP crystals based on EDS measurement.

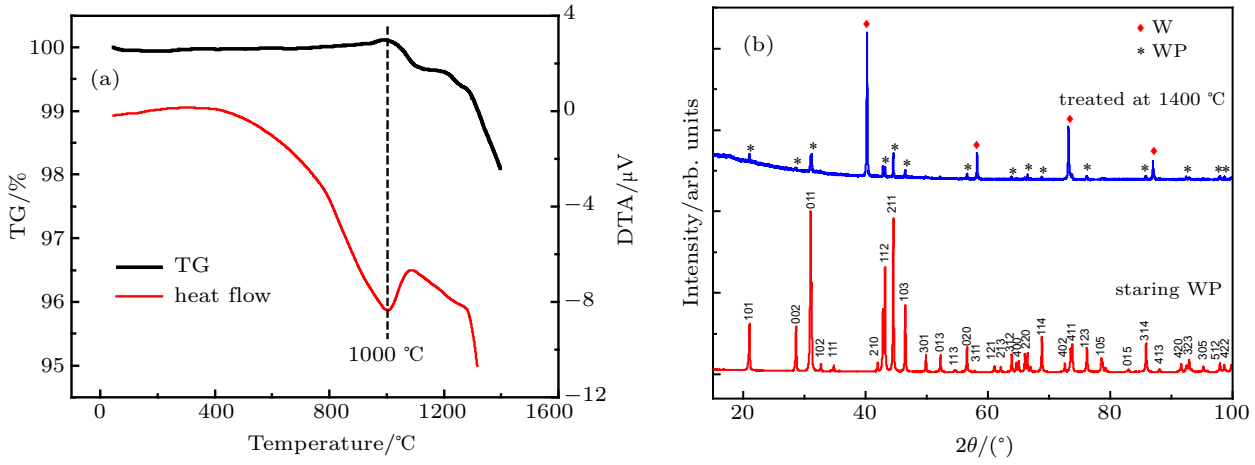


Fig. 5. (a) Determined TG-DTA data for polycrystalline WP. (b) XRD patterns of the sample treated at 1400 °C to look into decomposition process of panel (a).

Finally, the thermal stability of WP is evaluated by using a regular TG-DAT analysis. As seen in Fig. 5(a), as the temperature is elevated, a clear weight loss happens around 1000 °C and corresponds to a dramatic variation in heat flow, indicating the occurrence of decomposition or oxidation of WP. Therefore, in order to investigate the phase evolution at high temperature, XRD analysis is carried out on the high-T treated sample, specifically, at 1400 °C in air, and the results are plotted in Fig. 5(b); the final product is a mixture of W and WP, which clearly indicates a decomposition process at 1000 °C rather than oxidation.

4. Conclusions

In this work, using our recently designed high-P cell, we successfully prepare large single-crystal WP from cooling the congruent melts of W–P at 5 GPa and 3000 °C–3200 °C. The obtained crystal possesses high quality with a crystallite size over one millimeter, which will facilitate further experimental determination of its intrinsic properties for further exploring the predicted interesting physics in this material such as topological band structure. Besides, the formulated synthetic methodology can also be exploited for growing high-quality crystals for most TM phosphides, which would open a new avenue to the study of condensed-matter physics and material science.

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