

## Synthesis and formation mechanism of titanium lead carbide

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**Abstract:**  $Ti_2PbC$  was synthesized for the first time by pressureless reaction synthesis using  $Ti/Pb/TiC$  as starting materials at a heating rate of 2 °C/min and holding at 1370 °C for 2 h in a tube furnace protected by Ar atmosphere. The effects of starting powders, heating rates, and holding temperatures on the formation of  $Ti_2PbC$  were investigated. It was found that elementary mixture of  $Ti/Pb/C$  or higher heating rates fail to form  $Ti_2PbC$ . The decreased lattice parameters in the synthesized  $Ti_2PbC$  indicated the existence of Pb vacancies in the compound. A reaction mechanism was proposed to explain the formation of  $Ti_2PbC$ .

**Keywords:**  $Ti_2PbC$ ; MAX phase; differential scanning calorimetry (DSC); thermal stability



### 1 Introduction

MAX phases are a group of layered ternary compounds with the general formula of  $M_{n+1}AX_n$  ( $M$ : early transition metal;  $A$ : IIIA- or IVA-group element;  $X$ : C and/or N;  $n = 1\text{--}3$ ), which combine properties of ceramics and metals, such as low density, high elastic modulus, excellent self-lubricating property, and high electrical and thermal conductivities [1–3]. Less than a hundred of MAX phases (exclusive of solid solutions) have been obtained experimentally so far, although the number of the potential MAX phases is considerably larger. The research for further control over the properties of MAX phases with new functionalities has

resulted in an increased interest in MAX phase synthesis [1,4].  $Ti_2PbC$  is expected to be similar to other MAX phases, and may show unique functionality as the buffer layer in lead acid batteries [5–7]. In addition, the calculation by the first-principle suggests that the bonding between Ti and Pb is relatively weak and the diffusion coefficient of Pb element is high [8], indicating that  $Ti_2PbC$  could be an ideal raw material to prepare MXene, a newly emerging group of two-dimensional materials [9]. It should be pointed out that Pb is an element detrimental to human's health but also of great value in industrial application irreplaceably.

The fabrication of  $Ti_2PbC$  was reported only once by Jeitschko *et al.* [10] in 1964 by heating a  $TiC/Ti/Pb$  mixture to 850 °C for up to 600 h in vacuum. In this original paper, Jeitschko *et al.* measured the lattice constant and density of  $Ti_2PbC$ . The authors also described the structure of the  $Ti_2PbC$  that contains A-site vacancies intrinsically.

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Some researchers tried to synthesize  $Ti_2PbC$  at 850 °C as Jeitschko *et al.* did [5], but the synthesized  $Ti_2PbC$  powders contained a mass of impurities, such as  $TiC$ ,  $Ti-Pb$  compounds, which were resulted from the low synthesizing temperature. Except for this, there was no related literature on the synthesis of  $Ti_2PbC$  further according to the authors' knowledge.

Various processes were used to fabricate other MAX phase powders, including solid–liquid reaction [11], microwave hybrid heating [12], fluctuation synthesis [13], self-propagating high-temperature synthesis [14], etc. However, purity of the synthesized powders via these methods is still not satisfied. Previously, almost single-phase  $Ti_2SnC$  powders were synthesized through a simple pressureless reaction synthesis process of  $Ti/Sn/TiC$  mixed powders at 1200 °C in vacuum for 15 min [15]. Using the similar method, we have prepared high-purity MAX powders, including  $Ti_3AlC_2$ ,  $Ti_3SiC_2$ ,  $Ti_2AlC$  [16,17], etc. The objectives of the present work, therefore, are to explore the possibility of synthesizing high-purity  $Ti_2PbC$  by a pressureless reaction synthesis method, to optimize the processing parameters, and to understand the mechanism of the  $Ti_2PbC$  formation path.

## 2 Experimental procedures

Ti (300 mesh, ≥ 99.9% purity), Pb (300 mesh, ≥ 99.95% purity), C (625 mesh, ≥ 99.0% purity), and  $TiC$  (1340–2000 mesh, ≥ 99.95% purity) powders were used as starting materials. Table 1 shows the compositions and synthesis conditions, where  $Ti/Pb/C$  (molar ratio: 2:1.2:1) and  $Ti/Pb/TiC$  (molar ratio: 1:1.2:1, 1:1.0:1, 1:0.8:1) were used as starting powders and mixed in plastic jar for 24 h.

**Table 1 Compositions of raw materials and synthesis parameters for  $Ti_2PbC$  fabrication**

| Code | Raw material   | Synthesis temperature | Heating rate (°C/min) |
|------|----------------|-----------------------|-----------------------|
| A    | 2Ti/1.2Pb/1C   |                       | 10                    |
| B    |                | 1370 °C               | 2                     |
| C    |                |                       | 10                    |
| D    |                |                       |                       |
| E    | 1Ti/1.2Pb/1TiC | 1200 °C               |                       |
| F    |                | 1000 °C               |                       |
| G    |                | 850 °C                | 2                     |
| H    | 1Ti/1.0Pb/1TiC | 1370 °C               |                       |
| I    | 1Ti/0.8Pb/1TiC |                       |                       |

Powder mixtures were put into alumina crucibles that were placed in a graphite crucible, and then pressureless sintered at different temperatures for 2 h in Ar atmosphere to prepare  $Ti_2PbC$ . Two kinds of heating rates were applied: a high rate of 10 °C/min, and a low heating rate of 2 °C/min, from room temperature to the target temperature.

The constituent phase information and microstructure of prepared  $Ti_2PbC$  samples were analyzed by X-ray diffraction (XRD, D8-Discover, Bruker) and by scanning electron microscope (SEM, XL 30, FEI-Philips) equipped with energy dispersive spectrometer (EDS). To clarify the possible reactions, the specific heat was determined by differential scanning calorimetry (DSC, 04 F3, Pegasus) in the temperature range of room temperature to 1000 °C.

## 3 Results and discussion

### 3.1 Optimization of processing parameters for $Ti_2PbC$ synthesis

Samples A and B were synthesized from 2Ti/1.2Pb/1C powders at 1370 °C for 2 h at different heating rates of 10 and 2 °C/min, respectively. Their XRD patterns are respectively shown in Figs. 1(a) and 1(b), where no  $Ti_2PbC$  phase is identified in both samples A and B. It suggests that  $Ti_2PbC$  is difficult to form from the starting powders of 2Ti/1.2Pb/1C, at either of the heating rates applied.

Using 1Ti/1.2Pb/1TiC instead of 2Ti/1.2Pb/1C as starting materials, some obvious different results could be found. Although similar results are reached when heated at high rate of 10 °C/min (sample C, Fig. 1(c)),  $Ti_2PbC$  phase becomes the predominant one when heated at a low rate of 2 °C/min and held at 1370 °C for 2 h, as shown in Fig. 1(d) (sample D). It is well known that, during the synthesis of  $Ti_3SiC_2$  and  $Ti_3AlC_2$  from elemental starting powders, there exists always a combustion reaction between Ti and C [18,19]. This reaction releases a large amount of heat, which results in a sharp temperature pike in the synthesizing process. Similarly, such temperature pike would occur in the preparation of  $Ti_2PbC$ , leading to its decomposition. Using the  $TiC$  powders instead of Ti and C powder mixture prevents the Ti–C combustion reaction, lowering the system temperature and favoring the formation of  $Ti_2PbC$ .

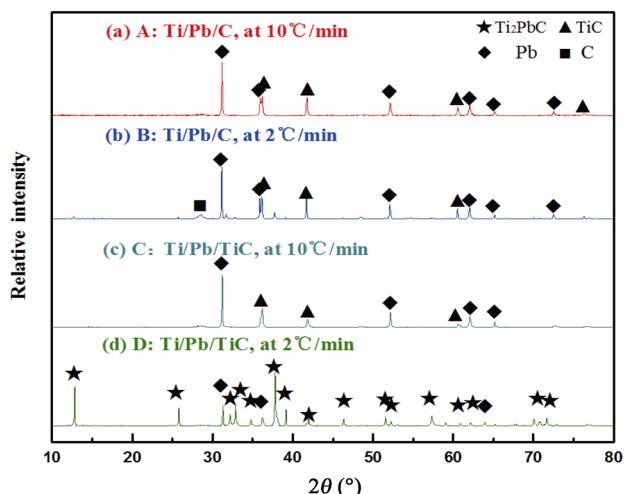
It is obvious that the same starting materials in

sample C and sample D do not lead to the same synthesized product due to the difference in heating rate. As shown in Fig. 1(c), with an increase in heating rate to 10 °C/min, sample C synthesized from Ti/Pb/TiC, the same with sample D, contains no  $\text{Ti}_2\text{PbC}$  phase. Low heating rate allows long reacting time between raw materials and favors a thermodynamic equilibrium, leading to the formation of  $\text{Ti}_2\text{PbC}$ . A high heating rate is likely to induce over-heating and the equilibrium is hard to reach.

Accordingly, all the following samples E–I were synthesized from Ti/Pb/TiC with a heating rate of 2 °C/min.

Samples D, E, F, and G were synthesized from 1Ti/1.2Pb/1TiC at different synthesis temperatures for 2 h at a heating rate of 2 °C/min. The corresponding XRD results are shown in Fig. 2. When synthesized at 850 °C,  $\text{Ti}_2\text{PbC}$  phase has already formed, but TiC and Pb impurities are the predominant phases. In addition,  $\text{Ti}_4\text{Pb}$  phase is formed, which may be a reaction product at low temperature, because the reaction between liquid Pb and Ti occurs at temperatures above 400 °C [20,21].

When the synthesis temperature increases to 1000 °C,  $\text{Ti}_2\text{PbC}$  becomes predominant, and its content increases with increasing synthesis temperature. However, TiC and Pb impurities always accompany  $\text{Ti}_2\text{PbC}$  even when the synthesis temperature increases to 1200 °C.  $\text{Ti}_2\text{PbC}$  becomes the dominant phase with small amount of Pb when sintered at 1370 °C. Apparently, for the starting materials of 1Ti/1.2Pb/1TiC, the over

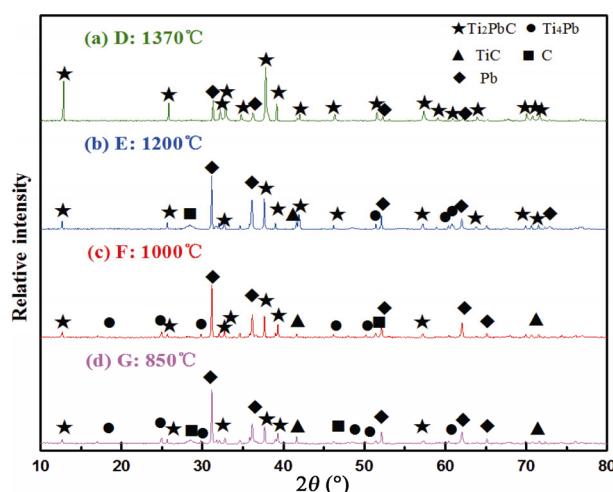


**Fig. 1** XRD patterns of (a) sample A from Ti/Pb/C at 10 °C/min; (b) sample B from Ti/Pb/C at 2 °C/min; (c) sample C from Ti/Pb/TiC at 10 °C/min; (d) sample D from Ti/Pb/TiC at 2 °C/min. All samples were synthesized at 1370 °C.

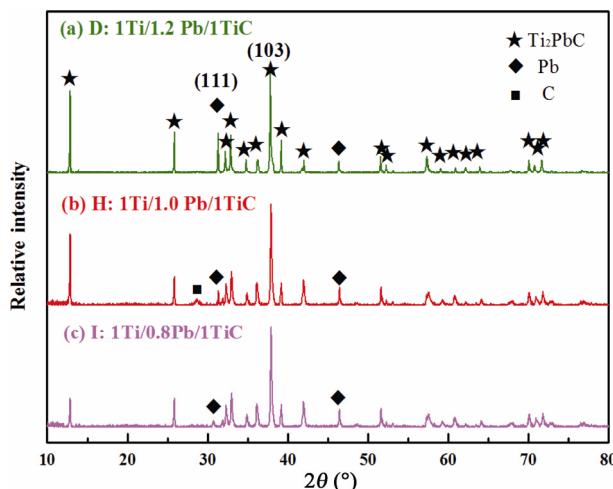
stoichiometric Pb concentration is likely the reason why Pb always remains in the synthesized materials. Consequently, we decreased the molar ratios of Pb in the starting mixtures to prepare high-purity  $\text{Ti}_2\text{PbC}$  powders.

Samples D, H, and I were synthesized from 1Ti/xPb/1TiC, and  $x$  value is ranged from 0.8 to 1.2, respectively, with their XRD patterns shown in Fig. 3.  $\text{Ti}_2\text{PbC}$  is the main phase in all samples, while Pb still remains in the final products. And the intensity of characteristic peaks of Pb decreases gradually with the decreasing  $x$  values.

Pb is still detected when using a stoichiometric composition of 1Ti/0.8Pb/1TiC to prepare  $\text{Ti}_2\text{PbC}$ ,



**Fig. 2** XRD patterns of samples D–G synthesized from Ti/Pb/TiC at a heating rate of 2 °C/min at (a) 1370 °C, (b) 1200 °C, (c) 1000 °C, and (d) 850 °C.



**Fig. 3** XRD patterns of samples D, H, and I synthesized at 1370 °C with a heating rate of 2 °C/min from 1Ti/xPb/1TiC, where  $x$  is (a) 1.2, (b) 1.0, and (c) 0.8.

suggesting the possible formation of Pb vacancies in the crystal structure of  $\text{Ti}_2\text{PbC}$ . This hypothesis could be verified from two aspects. First, the monovacancy formation energy for Pb in  $\text{Ti}_2\text{PbC}$  is reported to be 0.99 eV, which is much smaller than those of other A-site metals, such as Ga (2.59 eV), In (2.19 eV), Sn (2.08 eV), and Cd (1.62 eV) [8]. Second, the lattice parameters of  $\text{Ti}_2\text{PbC}$  measured by XRD also confirm the existence of Pb vacancies. Table 2 lists the previously reported and the presently measured lattice parameters of  $\text{Ti}_2\text{PbC}$ . We can find that  $a$  and  $c$  of  $\text{Ti}_2\text{PbC}$  in present study are smaller than those in the literature. Especially the decreased amount in  $c$  axis ( $-1.5\%$ ) is much larger than that in  $a$  axis ( $-0.7\%$ ), indicating the possible existence of Pb vacancies.

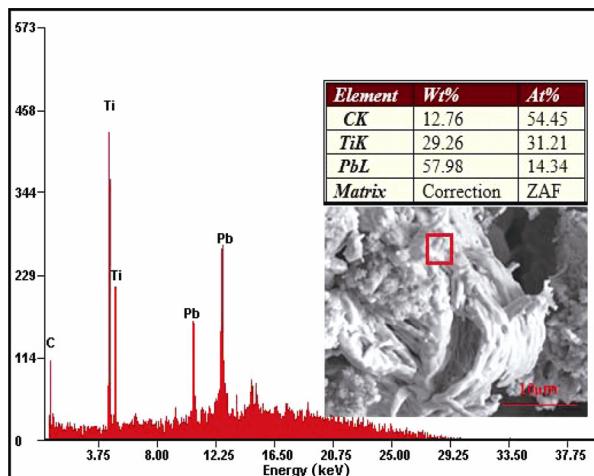
We used EDS to measure the Ti:Pb ratio of five random areas in sample D and obtained the average value. As shown in Fig. 4, the Ti:Pb ratio is 2.17:1, indicating the existence of Pb vacancies in  $\text{Ti}_2\text{PbC}$  that has also been mentioned by Jeitschko *et al.* [10].

### 3.2 Morphology of the synthesized $\text{Ti}_2\text{PbC}$ powders

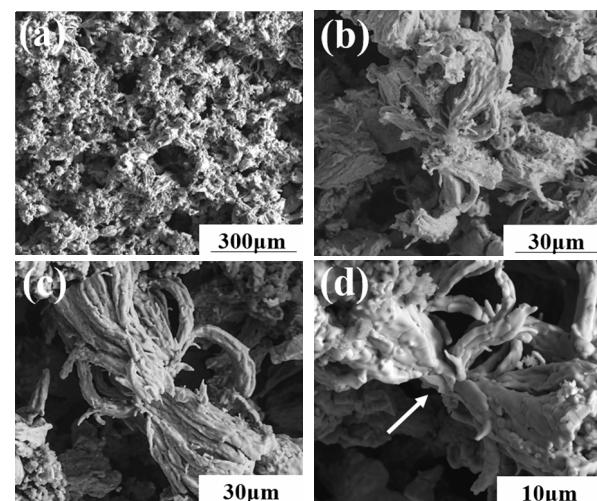
Figures 5(a)–5(d) show the representative morphologies of the as-prepared  $\text{Ti}_2\text{PbC}$  powders from Ti/Pb/TiC at 1370 °C at 2 °C/min for 2 h (sample D).  $\text{Ti}_2\text{PbC}$

**Table 2** Lattice parameters of  $\text{Ti}_2\text{PbC}$  reported in the literature and measured from XRD results in this study for samples synthesized from 1Ti/1.0Pb/1TiC

| Data resource   | Lattice parameter (Å) |            |
|-----------------|-----------------------|------------|
|                 | $a$                   | $c$        |
| Previous report | 3.222 [10]            | 13.99 [10] |
| Present study   | 3.201                 | 13.78      |



**Fig. 4** EDS spectra from the marked area of sample D.



**Fig. 5** SEM images of  $\text{Ti}_2\text{PbC}$  powders synthesized from Ti/Pb/TiC after synthesis at 1370 °C at 2 °C/min for 2 h (sample D): (a) the as-prepared  $\text{Ti}_2\text{PbC}$  powders, (b) the flower-like  $\text{Ti}_2\text{PbC}$ , (c) the bowknot-like  $\text{Ti}_2\text{PbC}$ , and (d) higher magnification images of the bowknot.

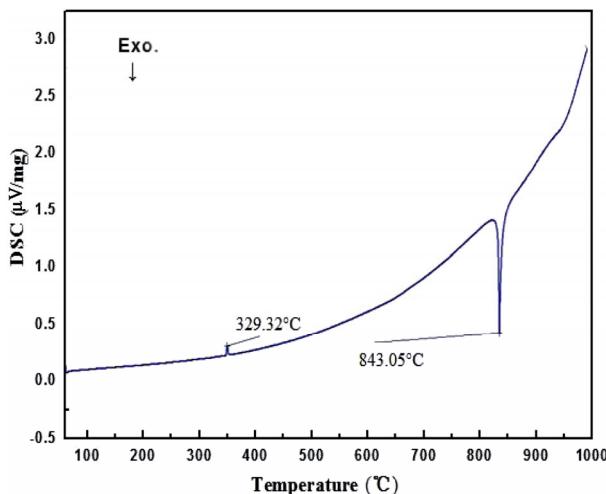
particles display mainly flower-like or bowknot-like shapes, as shown in Fig. 5(a) and enlarged in Fig. 5(b) and Fig. 5(c), respectively.

At a higher magnification, we can see that the rod-like  $\text{Ti}_2\text{PbC}$  is gathered to form the wings of bowknot in Fig. 5(d). The layered microstructure in the middle of the bowknot where the arrowhead points, is a typical feature of MAX phase, shown in Fig. 5(d). Such layered grains are identified to be  $\text{Ti}_2\text{PbC}$  with EDS results (not shown).

Note that all micrographs were taken from the surface areas of the sample, where the  $\text{Ti}_2\text{PbC}$  grains may grow freely. Carefully observing the  $\text{Ti}_2\text{PbC}$  grains in Fig. 5(d), we found that they generally grow into elongated shapes, suggesting that  $\text{Ti}_2\text{PbC}$  shows an anisotropic growth behavior. This feature is attributed to the varied grain growth rate in different crystal planes, similar to other MAX phases [22]. Furthermore, the liquid phase formed from low-melting-point Pb during synthesis would benefit the anisotropic growth of  $\text{Ti}_2\text{PbC}$  grains. This supposition is further confirmed by the much coarser  $\text{Ti}_2\text{PbC}$  grains in the 1Ti/1.2Pb/1TiC reaction system (not shown), where excessive Pb provides more liquid amount during synthesizing process.

### 3.3 Reaction mechanism for formation of $\text{Ti}_2\text{PbC}$

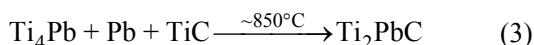
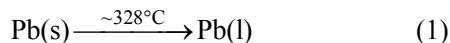
Figure 6 presents the DSC curve of a mixture of 1Ti/1.0Pb/1TiC powders in the temperature range from room temperature to 1000 °C at a heating rate of 2 °C/min.



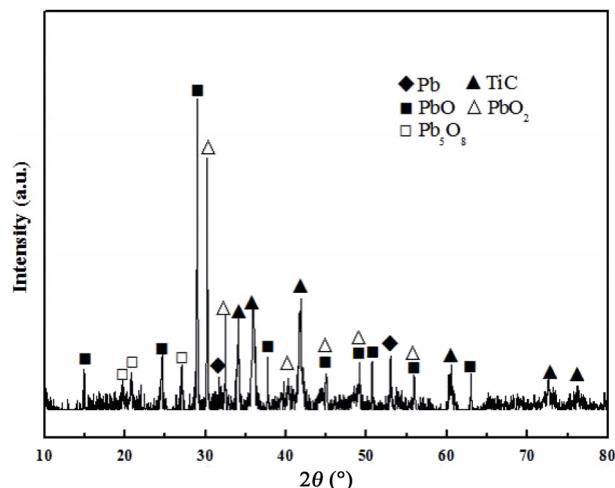
**Fig. 6** DSC curve of a mixture of Ti/Pb/TiC powders.

The endothermic peak at 329.32 °C corresponds to the melting of Pb. The exothermic peak at 843.05 °C may be associated with the reactions between the Ti–Pb compounds and TiC to form  $Ti_2PbC$ . According to the XRD result (Fig. 2(d)) and Ti–Pb binary diagram, intermetallic compound like  $Ti_4Pb$  is surely formed at temperatures between 400 and 850 °C. The possibly exothermic peaks related to the reactions between Ti and Pb are negligible in the DSC curve. This may be attributed to the mild reactions between Ti and Pb occurred in a relatively wide temperature range, dispersing the corresponding heat pike.

Based on the DSC analysis and XRD results, a reaction mechanism was put forward. For the homogeneous powder mixture of Ti/Pb/TiC, Pb first starts to melt as temperature increases to be above 328 °C. Then Ti reacts with Pb to form Ti–Pb compounds with increasing temperature. Once the temperature reaches 850 °C, reactions between the Ti–Pb compounds and TiC occur to form  $Ti_2PbC$  phase, which may proceed on TiC surfaces and be promoted by the liquid Pb environment. Thus, the reaction sequence during the synthesis of Ti, Pb, and TiC powders is as follows:



It is interesting to find that the prepared  $Ti_2PbC$  powders are easily decomposed during the following planetary ball milling.  $Ti_2PbC$  powders and corresponding zirconia balls (with the ball-to-powder mass ratio of



**Fig. 7** XRD pattern of sample D after ball milling.

10/1) were placed in stainless steel jars. The milling process was performed for 1 h in air, at 500 r/min by a ND7-1L planetary ball mill. The corresponding XRD pattern shows that no  $Ti_2PbC$  but TiC and Pb are found, with  $PbO$  and  $Pb_3O_8$  as minor phases (as shown in Fig. 7). The decomposition of  $Ti_2PbC$  phase is mainly attributed to the weak Ti–Pb bonding [23,24], which is consistent with the results of lattice parameter calculation (Table 2), namely easy formation of Pb vacancies. Decomposition of MAX phases, induced by the outward diffusion of A element from the layered structure under certain conditions, is well reported. When  $Ti_3SiC_2$  is placed in a harsh environment such as molten Al [25–27], Si atoms migrate outward, leaving the  $TiC_x$  layers behind. It is also confirmed that  $Ti_3AlC_2$  decomposes at 1450 °C in vacuum by the outward diffusion of Al atoms and can be corroded in molten KOH at 700 °C [28,29]. This work reveals another manner of decomposition of an MAX phase, by ball milling.

#### 4 Conclusions

$Ti_2PbC$  powders were successfully synthesized for the first time by pressureless synthesis.  $Ti_2PbC$  was obtained by synthesizing Ti/Pb/TiC at a low heating rate of 2 °C/min. Starting materials of Ti/Pb/C or higher heating rates resulted in the failure of  $Ti_2PbC$  synthesis. The decreased lattice parameters in the synthesized  $Ti_2PbC$  indicated the existence of Pb vacancies in the compound. A possible reaction mechanism was proposed to explain the formation of  $Ti_2PbC$ , namely Pb melts at 328 °C and Ti–Pb compounds

form by the reactions between Ti and liquid Pb, and subsequently  $Ti_2PbC$  forms through the reaction between Ti–Pb compounds and TiC. It is the first report on the decomposition of  $Ti_2PbC$  into TiC and Pb during ball milling.

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## References

- [1] Sun ZM. Progress in research and development on MAX phases: A family of layered ternary compounds. *Int Mater Rev* 2011, **56**: 143–166.
- [2] Barsoum MW. The  $M_{N+1}AX_N$  phases: A new class of solids: Thermodynamically stable nanolaminates. *Prog Solid State Ch* 2000, **28**: 201–281.
- [3] Xu L, Zhu D, Grasso S, et al. Effect of texture microstructure on tribological properties of tailored  $Ti_3AlC_2$  ceramic. *J Adv Ceram* 2017, **6**: 120–128.
- [4] Arróyave R, Talapatra A, Duong T, et al. Does aluminum play well with others? Intrinsic Al-A alloying behavior in 211/312 MAX phases. *Mater Res Lett* 2017, **5**: 170–178.
- [5] Lara-Curcio E, An K, Kiggans Jr. JO, et al. Lightweight, durable lead-acid batteries. U.S. Patent 8,017,273. 2011.
- [6] Sun ZM, Barsoum MW. Spontaneous room temperature extrusion of Pb nano-whiskers from leaded brass surfaces. *J Mater Res* 2005, **20**: 1087–1089.
- [7] Zhang P, Zhang Y, Sun Z. Spontaneous growth of metal whiskers on surfaces of solids: A review. *J Mater Sci Technol* 2015, **31**: 675–698.
- [8] Liu B, Wang JY, Zhang J, et al. Theoretical investigation of  $A$ -element atom diffusion in  $Ti_2AC$  ( $A$  = Sn, Ga, Cd, In, and Pb). *Appl Phys Lett* 2009, **94**: 181906.
- [9] Zheng W, Sun Z, Zhang P, et al. Research progress on MXene, two dimensional nano-materials. *Mater Rev* 2017, **31**: 1–14.
- [10] Jeitschko W, Nowotny H, Benesovsky F. Die H-Phasen  $Ti_2TiC$ ,  $Ti_2PbC$ ,  $Nb_2InC$ ,  $Nb_2SnC$  und  $Ta_2GaC$ . *Monatshefte für Chemie* 1964, **95**: 431–435.
- [11] Wang X, Zhou Y. Solid–liquid reaction synthesis of layered machinable  $Ti_3AlC_2$  ceramic. *J Mater Chem* 2002, **12**: 455–460.
- [12] Guan C, Sun N. Synthesis of high-purity  $Ti_2SC$  powder by microwave hybrid heating. *J Adv Ceram* 2016, **5**: 337–343.
- [13] Sun Z, Zhou Y. Fluctuation synthesis and characterization of  $Ti_3SiC_2$  powders. *Mat Res Innovat* 1999, **2**: 227–231.
- [14] Riley DP, Kisi EH, Wu E, et al. Self-propagating high-temperature synthesis of  $Ti_3SiC_2$  from  $3Ti+SiC+C$  reactants. *J Mater Sci Lett* 2003, **22**: 1101–1104.
- [15] Li S-B, Bei G-P, Zhai H-X, et al. Synthesis of  $Ti_2SnC$  from  $Ti/Sn/TiC$  powder mixtures by pressureless sintering technique. *Mater Lett* 2006, **60**: 3530–3532.
- [16] Li S, Xiang W, Zhai H, et al. Formation of a single-phase  $Ti_3AlC_2$  from a mixture of Ti, Al and TiC powders with Sn as an additive. *Mater Res Bull* 2008, **43**: 2092–2099.
- [17] Hashimoto S, Takeuchi M, Inoue K, et al. Pressureless sintering and mechanical properties of titanium aluminum carbide. *Mater Lett* 2008, **62**: 1480–1483.
- [18] Li J-F, Matsuki T, Watanabe R. Combustion reaction during mechanical alloying synthesis of  $Ti_3SiC_2$  ceramics from  $3Ti/Si/2C$  powder mixture. *J Am Ceram Soc* 2005, **88**: 1318–1320.
- [19] Ge Z, Chen K, Guo J, et al. Combustion synthesis of ternary carbide  $Ti_3AlC_2$  in Ti–Al–C system. *J Eur Ceram Soc* 2003, **23**: 567–574.
- [20] Ji B, Fang P. Study on the mechanism of strengthening titanium alloys with plumbum. *Vacuum* 2003, **1**: 40–41. (in Chinese)
- [21] Guo Q, Wang G, Guo G. *Common Non-Ferrous Metal Phase Diagram Atlas of F Binary Alloy*. Chem Ind Press, 2010. (in Chinese)
- [22] Hartman P, Perdok WG. On the relations between structure and morphology of crystals. II. *Acta Cryst* 1955, **8**: 521–524.
- [23] Liu Y, Zhang P, Ling C, et al. Spontaneous Sn whisker formation on  $Ti_2SnC$ . *J Mater Sci: Mater Electron* 2017, **28**: 5788–5795.
- [24] El-Raghy T, Chakraborty S, Barsoum MW. Synthesis and characterization of  $Hf_2PbC$ ,  $Zr_2PbC$  and  $M_2SnC$  ( $M$  = Ti, Hf, Nb or Zr). *J Eur Ceram Soc* 2000, **20**: 2619–2625.
- [25] Barsoum MW, El-Raghy T, Farber L, et al. The topotactic transformation of  $Ti_3SiC_2$  into a partially ordered cubic  $Ti(C_{0.67}Si_{0.06})$  phase by the diffusion of Si into molten cryolite. *J Electrochem Soc* 1999, **146**: 3919–3923.
- [26] El-Raghy T, Barsoum MW, Sika M. Reaction of Al with  $Ti_3SiC_2$  in the 800–1000 °C temperature range. *Mat Sci Eng A* 2001, **298**: 174–178.
- [27] Aldica G, Khodash V, Badika P, et al. Electrical conduction in initial field assisted sintering stages. *Journal of Optoelectronics and Advanced Materials* 2007, **9**: 3863–3870.
- [28] Li S-B, Zhai H-X, Bei G-P, et al. Synthesis and microstructure of  $Ti_3AlC_2$  by mechanically activated sintering of elemental powders. *Ceram Int* 2007, **33**: 169–173.
- [29] Sun D, Zhou A, Li Z, et al. Corrosion behavior of  $Ti_3AlC_2$  in molten KOH at 700 °C. *J Adv Ceram* 2013, **2**: 313–317.

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