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In-situ reaction synthesis and decomposition of Ta_2AlC

Dense bulk Ta_2AlC ceramic was fabricated by in-situ reaction/hot pressing of Ta, Al and C powders. The reaction path and effects of initial composition on the purity were investigated. It was found that Ta_2AlC formed through the reactions between $AlTa_2$ and graphite, or between Ta_5Al_3C, TaC and graphite at 1500–1550 °C. By modifying the molar ratio of the initial Ta, Al, and C powders, single-phase Ta_2AlC was prepared at 1550 °C under an Ar atmosphere with an optimized composition of Ta : Al : C = 2 : 1.2 : 0.9. The lattice parameter and a new set of X-ray diffraction data of Ta_2AlC were obtained. In addition, Ta_2AlC was reported unstable above 1600 °C and decomposed to Ta_4AlC_3, and then to TaC_x.

Keywords: Ta_2AlC; Synthesis; Decomposition

1. Introduction

Due to the unique combination of properties, such as easy machinability [1–3], high elastic stiffness [4, 5], good electrical and thermal conductivity [2, 6], excellent thermal shock resistance and damage tolerance [2, 7, 8], $M_{n+1}AX_n$ (M, early transition metal; A, A group element; X, C or N; $n = 1-3$), also called MAX phases, have attracted attention for many years. Since Barsoum and his co-workers first fabricated bulk Ti_3SiC_2 [2], a number of layered MAX ceramics including Ti_3AlC_2 [9], Ti_2AlC [10–13], Nb_2AlC [14, 15], V_2AlC [16], Cr_2AlC [17], have been synthesized and their physical and mechanical properties reported. In the MAX family, Ta_2AlC has been determined to crystallize with $P6_3/mmc$ symmetry using X-ray diffraction (XRD) analysis in 1960s [18]. Recently, Sun et al. [19] calculated the elastic properties of Ta_2AlC by means of ab initio total energy calculations using the projector augmented-wave method. Manoun et al. [20] determined that the compressibilities of Ta_2AlC in *a* and *c* directions were almost identical. Lin et al. [21] confirmed the crystal structure of Ta_2AlC through high resolution transmission electron microscope (HRTEM) and *Z*-contrast imaging. Gupta et al. [22] reported that Ta_2AlC was not resistant to oxidation.

For understanding the intrinsic physical and mechanical properties of Ta_2AlC, single-phase Ta_2AlC needs to be prepared. However, in previous reports, Ta_4AlC_3 was always a coexisting phase [21, 22]. Seldom have works focused on synthesizing single-phase Ta_2AlC through investigating the reaction process. In the present work, the reaction path and the effects of initial element percent on the purity of Ta_2AlC were investigated using Ta, Al and C powders as initial materials by an in-situ reaction/hot pressing method [23]. Single-phase Ta_2AlC could be obtained at 1550 °C with an optimized molar composition of Ta : Al : C = 2 : 1.2 : 0.9. In addition, it is known that MAX phases are unstable and may decompose at high temperatures [24, 25]. Racault et al. [25] reported that Ti_3SiC_2 was unstable above 1300 °C (above its synthesis temperature of 1100 °C), and decomposed with the formation of nonstoichiometric titanium carbide and gaseous silicon. In our work, we found Ta_2AlC was unstable above 1600 °C and transformed into Ta_4AlC_3, and then to TaC_x with out-diffusing of Al. The decomposition mechanism of Ta_2AlC was investigated.

2. Experimental procedure

Commercially available powders of Ta (99.9 %, −200 mesh), Al (99 %, −300 mesh) and graphite (99 %, −200 mesh) were used as starting powders for fabricating Ta_2AlC. The Ta, Al and C powders with molar ratio of 2 : 1 : 1 were weighed initially in order to investigate the synthesis path. The powders were dry mixed in a resin jar, ball milled for 12 h, and then sieved. For the preparation of Ta_2AlC, the mixed powders were uniaxially pressed at 5 MPa to the green compacts in a BN-coated graphite die, and then heated in a graphite furnace at different temperatures under an Ar atmosphere. The heating rate was $15\ K \cdot min^{-1}$ and the holding duration was 20 min. Finally, the samples were cooled down to room temperature. A column of 25 mm in diameter and 3 mm in height was obtained after removing the surface contamination. The synthesized samples were examined by XRD (Rigaku D/max-2400, Tokyo, Japan) with Cu-K_α radiation ($\lambda = 1.54178$ Å) at a scanning speed of $0.04^\circ \cdot step^{-1}$. The ground surfaces of samples were analyzed by a scanning electron microscope (SEM) (LEO Supra35, Ammerbuch, Germany) equipped with an energy dispersive spectroscopic (EDS) system.

In order to investigate effects of the initial compositions on purity of Ta_2AlC, the starting powders with modified molar ratios of Ta : Al : C were subjected to hot pressing runs at 1550 °C under 25 MPa in an Ar atmosphere. XRD results showed that single-phase polycrystalline Ta_2AlC could be obtained using the initial powders with molar ratio of 2 : 1.2 : 0.9. For quantitative XRD analysis, powders drilled from bulk specimen were examined with a step size of 0.02° and a count time of $2\ s \cdot step^{-1}$ between 5° and 75°. Lattice parameters, *hkl*, $2\theta'$ and intensities were calculated by the Rietveld refinement method using a DBWS code in $Cerius^2$ computational program for material research (Molecular Simulation Inc., San Diego, USA).

The decomposition mechanism of Ta_2AlC was investigated by treating Ta_2AlC samples with a dimension of $10 \times 10 \times 1\ mm^3$ at 1600 – 1700 °C in a graphite crucible for 120 min under an Ar atmosphere (static pressure of 100 kPa). A yellow layer was observed forming on the surface of heated samples. Thereafter, XRD analysis was conducted on the surface layer and the sublayer after removing the as-formed surface layer. The surface scale and cross-section of treated samples were investigated by SEM and EDS.

3. Results and discussion

3.1. Effect of temperature on the synthesis of Ta_2AlC

Figure 1 shows X-ray diffraction patterns of samples synthesized at different temperatures. The detected phases are listed in Table 1 in sequence of temperature. No reaction occurred below 700 °C. When the temperature increased to 900 °C, Al_3Ta and TaC appeared, as described by reaction Eqs. (1) and (2):

$$Ta + 3\,Al \rightarrow Al_3Ta \qquad (1)$$

$$Ta + C \rightarrow TaC \qquad (2)$$

SEM analysis revealed that the phases showed different morphologies. EDS analysis indicates the equiaxial particles are Al_3Ta and the irregular particles are unreacted Ta, as shown in Fig. 2a. At 1100 °C, $AlTa_2$ appeared with the consumption of Al_3Ta and Ta. The reaction equation is:

$$5\,Ta + Al_3Ta \rightarrow 3\,AlTa_2 \qquad (3)$$

As temperature increased to 1300 – 1400 °C, the diffraction peaks showed new phases of Ta_5Al_3C, Ta_2AlC, Ta_4AlC_3 [26 – 28] and Ta_2C. The amounts of $AlTa_2$, TaC, Ta_2C and Ta_5Al_3C increase with the consumption of Ta, C and Al_3Ta (Fig. 1d and e). As can be seen in Fig. 2b and c, most of the gray particles are $AlTa_2$ at 1300 °C, and thereafter agglomeration and growth of $AlTa_2$ particles are observed at 1400 °C. The possible reactions for forming Ta_2C and

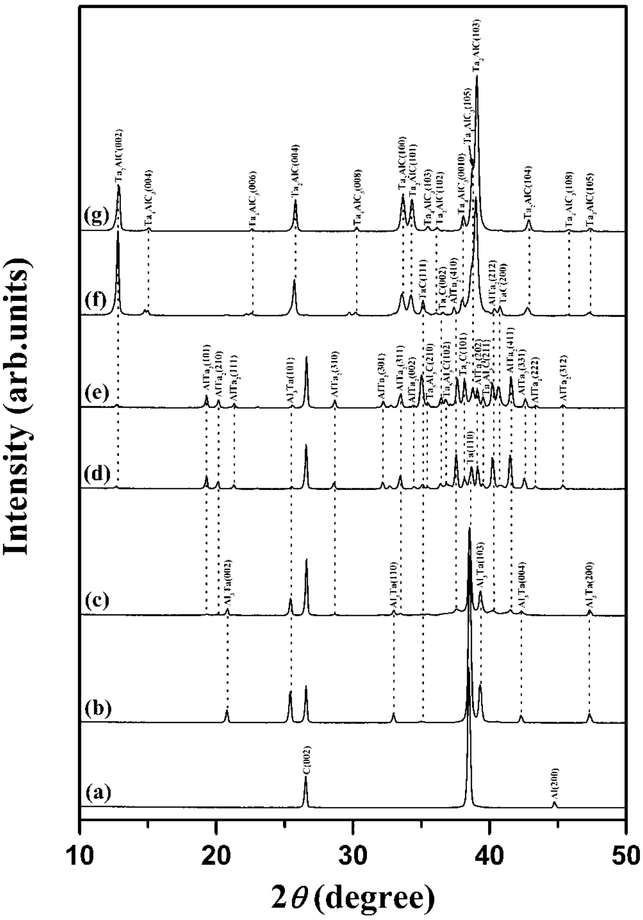

Fig. 1. X-ray diffraction patterns of 2Ta/Al/C mixed powders hot-pressed at (a) 700 °C, (b) 900 °C, (c) 1100 °C, (d) 1300 °C, (e) 1400 °C, (f) 1500 °C, and (g) 1550 °C.

Table 1. Phase compositions of the samples sintered in the temperature range from 700 to 1550 °C.

Temperature	Phase compositions in the samples
700 °C	Ta, Al, C
900 °C	Al_3Ta, TaC, Ta, C
1100 °C	$AlTa_2$, Al_3Ta, TaC, Ta, C
1300 °C	Ta_2AlC, Ta_5Al_3C, $AlTa_2$, Al_3Ta, Ta_2C, TaC, Ta, C
1400 °C	Ta_2AlC, Ta_4AlC_3, Ta_5Al_3C, $AlTa_2$, Al_3Ta, Ta_2C, TaC, C
1500 °C	Ta_2AlC, Ta_4AlC_3, $AlTa_2$, Ta_2C, TaC, C
1550 °C	Ta_2AlC, Ta_4AlC_3

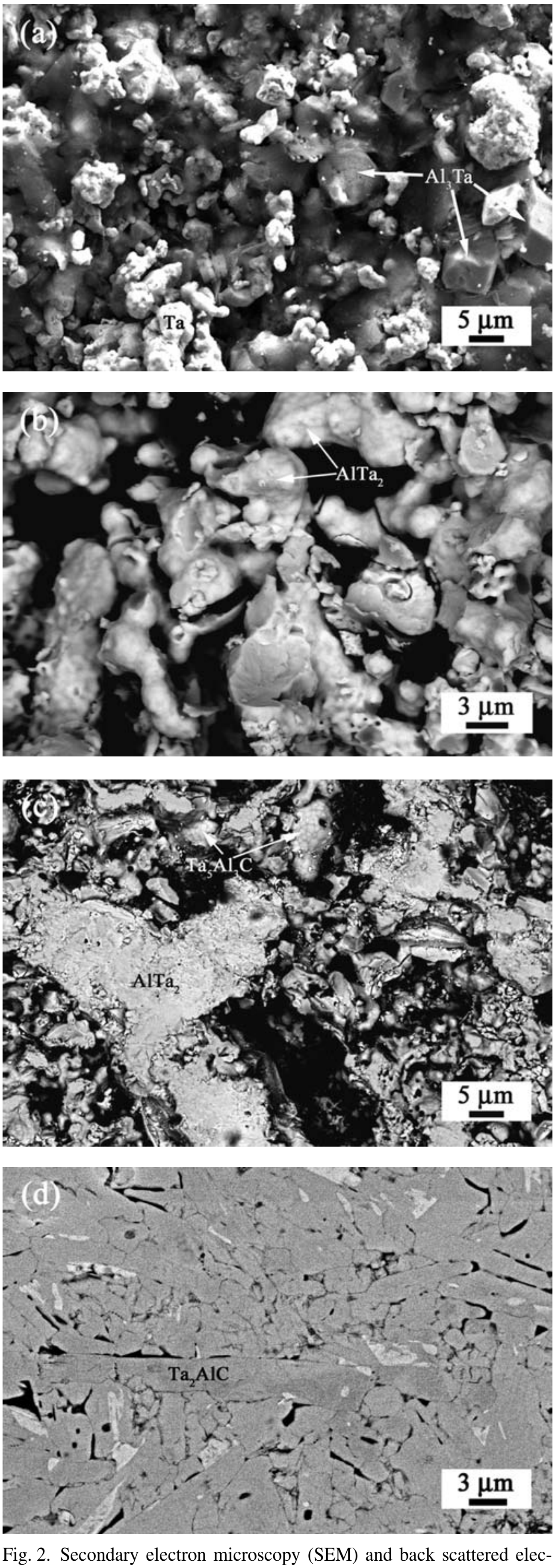

Fig. 2. Secondary electron microscopy (SEM) and back scattered electron (BSE) images of 2Ta/Al/C mixed powders hot-pressed at (a) 900 °C (SEM), (b) 1300 °C (BSE), (c) 1400 °C (BSE), and (d) 1550 °C (BSE).

Ta_5Al_3C are:

$$Ta + TaC \rightarrow Ta_2C \tag{4}$$

$$3\,AlTa_2 + 2\,C \rightarrow Ta_5Al_3C + TaC \tag{5}$$

Schuster et al. [29] reported that the new-formed phases at a lower temperature of 700 and 1000 °C were $AlTa_2$ and Ta_2C. The difference may be due to the kinetics of phase formation. When the temperature increased up to 1500 °C, the main crystalline phase was Ta_2AlC, together with small quantities of Ta_4AlC_3, TaC, Ta_2C, $AlTa_2$ and C. Lin et al. [30] confirmed that the Ti–Al twin boundaries provided the rapid diffusion paths for C, and contributed to the formation of Ti_2AlC. Similarly, the production of Ta_2AlC could be associated with the consuming $AlTa_2$ and graphite:

$$AlTa_2 + C \rightarrow Ta_2AlC \tag{6}$$

El-Raghy et al. [31] proposed that Ti_5Si_3C and TiC were the intermediate phases intimately involved in the formation of Ti_3SiC_2. Probably, Ta_2AlC could also be produced from Ta_5Al_3C, TaC and graphite following the reaction:

$$Ta_5Al_3C + TaC + C \rightarrow 3\,Ta_2AlC \tag{7}$$

When the temperature was raised to 1550 °C, only Ta_2AlC and Ta_4AlC_3 were detected. $AlTa_2$, TaC, Ta_2C and C were possibly consumed to produce Ta_4AlC_3 as follows:

$$AlTa_2 + 2\,TaC + C \rightarrow Ta_4AlC_3 \tag{8}$$

and

$$AlTa_2 + Ta_2C + 2\,C \rightarrow Ta_4AlC_3 \tag{9}$$

The polished surface of the sample sintered at 1550 °C revealed that both Ta_2AlC and Ta_4AlC_3 grains were gray and were hard to distinguish (Fig. 2d). Some white contrasting particles were identified as nonstoichiometric Ta–Al phase by EDS, distributing in the intergranular positions.

3.2. Effect of molar ratio of initial elemental powders on the purity of Ta_2AlC

It is obvious that pure Ta_2AlC cannot be synthesized using Ta, Al and C powders with a molar ratio of 2 : 1 : 1, as discussed in the above section. Ta_4AlC_3 always coexists with Ta_2AlC in the sample prepared above 1500 °C. The attempts to fabricate single-phase Ta_2AlC are focused on modifying the initial composition. At first, we compared the atomic percent of Al and C in Ta_2AlC and Ta_4AlC_3. The atomic percent of Al in Ta_2AlC is higher than that in Ta_4AlC_3, while the atomic percent of C is lower in Ta_2AlC. Therefore, in order to eliminate Ta_4AlC_3, this highlights a way to increase the content of Al and to decrease C in the starting mixed powders. The starting powders with molar ratios of Ta : Al : C = 2 : 1.1 : 1, 2 : 1.2 : 1, and 2 : 1.2 : 0.9 were selected and sintered at 1550 °C for 20 min. The diffraction patterns of samples prepared with different compositions are plotted in Fig. 3. It is seen from Fig. 3a and b that those samples synthesized with the molar ratios of Ta : Al : C = 2 : 1.1 : 1 and 2 : 1.2 : 1 still contain small quantities of Ta_4AlC_3. When the Ta : Al : C molar ratio changed to 2 : 1.2 : 0.9, single-phase Ta_2AlC is obtained, as shown in Fig. 3c. A similar strategy was successfully used to prepare single-phase Ti_3AlC_2 with the molar ratio of Ti : Al : C = 3 : 1.1 : 1.8 [32].

The density of single-phase polycrystalline Ta_2AlC was measured by the Archimedes method, and yields $11.46\ g \cdot cm^{-3}$, which is close to the theoretical value of $11.52\ g \cdot cm^{-3}$. The Rietveld refinement of the XRD pattern for drilled Ta_2AlC powders is conducted and plotted in Fig. 4. A comparison between the experimental powder diffraction data and the calculated pattern of Ta_2AlC is also shown in the figure. The separated short dashed lines represent the positions of diffraction peaks. The line plot denotes experimental diffraction spectra, and the cross is the simulated pattern. Deviation between them is plotted as the lowest curve. The reliability factors are $R-P = 8.48\,\%$ and $R-WP = 11.23\,\%$, respectively. The refined lattice parameters are $a = 3.081$ Å and $c = 13.89$ Å, agreeing well with previous results reported by Manoun et al. (a = 3.086 Å, c = 13.85 Å) [20], Lin et al. (a = 3.079 Å, c = 13.86 Å) [21], and ICDD card No. 29-0092 (a = 3.075 Å, c = 13.83 Å).

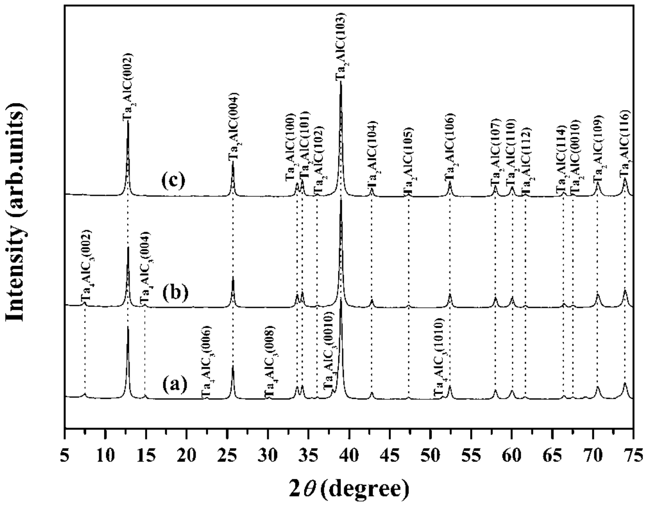

Fig. 3. X-ray diffraction patterns of Ta_2AlC samples prepared using the starting powders with molar ratios of Ta : Al : C = (a) 2 : 1.1 : 1, (b) 2 : 1.2 : 1, and (c) 2 : 1.2 : 0.9, showing the effect of initial element composition on purity.

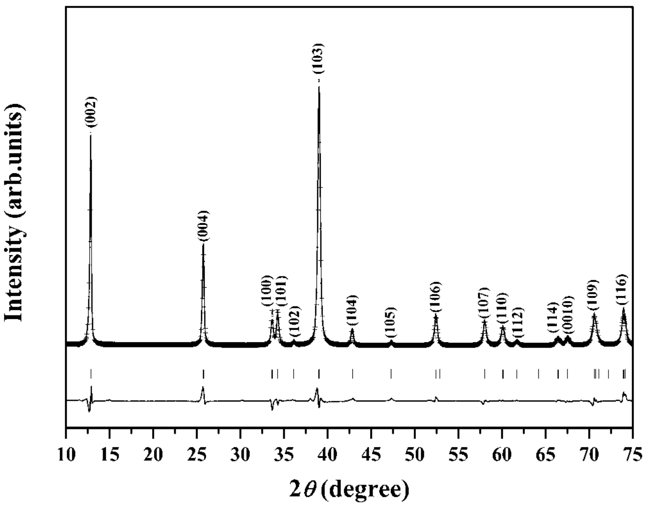

Fig. 4. Comparison of experimental powder diffraction data with the calculated pattern of Ta_2AlC.

Table 2. Calculated, experimental and ICDD card No. 29-0092 data of reflections, $2\theta'$ and intensities for Ta_2AlC.

Reflection (hkl)	$2\theta_{Cal.}$ (°)	$2\theta_{Obs.}$ (°)	$2\theta_{ICDD.}$ (°)	$I/I_{0Cal.}$ (%)	$I/I_{0Obs.}$ (%)	$I/I_{0ICDD.}$ (%)
002	12.773	12.780	12.791	80.12	78.71	70
004	25.709	25.701	25.749	39.82	41.94	12
100	33.618	33.664	33.626	17.65	15.36	20
101	34.253	34.300	34.263	18.10	17.67	22
102	36.099	36.149	36.115	1.79	1.97	4
006	38.988	38.948		58.14	58.20	
103	39.007	39.062	39.028	100.00	100.00	100
104	42.797	42.859	42.823	9.57	10.78	5
105	47.306	47.376	47.347	2.21	2.93	5
106	52.409	52.488	52.460	21.61	22.82	10
008	52.840	52.924		0.02	0.02	
107	58.022	58.112	58.085	20.09	19.04	5
110	60.116	60.204	60.132	15.81	15.77	12
112	61.738	61.829	61.761	3.33	3.46	3
108	64.101	64.203		0.17	0.18	
114	66.468	66.569	66.498	6.99	7.04	3
0010	67.585	67.509		7.36	6.72	
109	70.633	70.750	70.722	13.66	12.86	10
200	70.670	70.778		1.03	0.97	
201	71.046	71.154	71.065	2.72	2.51	6
202	72.166	72.278		0.34	0.33	
116	74.007	74.125		19.05	21.66	
203	74.019	74.135	74.056	12.75	14.50	16

Table 2 lists the calculated reflections and intensities of Ta_2AlC, together with those of experimental and in ICDD card No. 29-0092. All reflections are in good agreement with the data from different sources. The present study also presents some new reflections, which were not reported before. Therefore, we complement the XRD data of Ta_2AlC in the present work.

3.3. Decomposition mechanism of Ta_2AlC

In the present processing conditions, Ta_2AlC is typically observed to coexist with Ta_4AlC_3. Possibly, a transformation exists between Ta_2AlC and Ta_4AlC_3 at 1550 °C or higher temperatures. Zhou et al. [33] reported that Ti_2AlC lost half of its aluminum layers and then transformed to Ti_3AlC_2 when synthesizing Ti_3AlC_2 by hot pressing using starting materials of Ti, Al_4C_3 and activated carbon. In order to prove this supposition in the Ta–Al–C system, single-phase Ta_2AlC samples are heated at temperatures of 1600, 1650 and 1700 °C under an Ar atmosphere to determine possible transformation. A yellow-colored layer is formed on the surface of treated samples. The top scale is determined to be cubic TaC by XRD, as shown in Fig. 5. The separation of diffraction peaks above 57° is attributed to Cu-K_α and Cu-K_β. Generally, tantalum carbide TaC is nonstoichiometric and presented by TaC_x. From the 111, 200, 220, 311 and 222 peaks in the profile, the lattice parameter a_0 is determined as 0.444592, 0.444405 and 0.444505 nm. According to the Bowman's equation: $x = 63.98a_0 - 27.516$ [34], the x values are calculated as 0.929, 0.917 and 0.923, respectively. Figure 6 shows a cross-section of a Ta_2AlC sample after treating at 1700 °C for 120 min. The outer layer is a compact TaC_x layer. The inset is an enlargement of the rectangular region. EDS analysis reveals that the equaxial particles are TaC_x and the lamellar

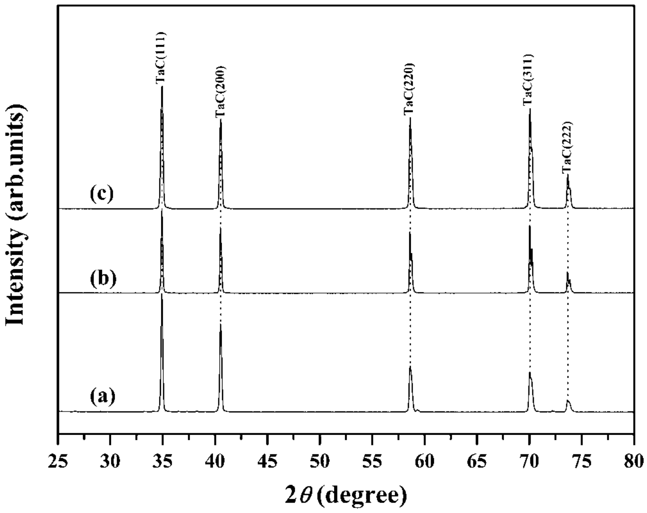

Fig. 5. X-ray diffraction patterns of Ta_2AlC samples treated at (a) 1600 °C, (b) 1650 °C, and (c) 1700 °C.

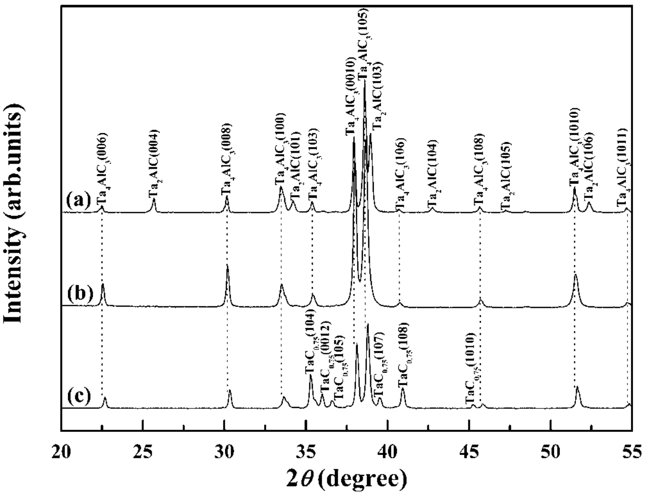

Fig. 7. X-ray diffraction patterns of Ta_2AlC samples after treating at (a) 1600 °C, (b) 1650 °C, and (c) 1700 °C and then removing the surface layer.

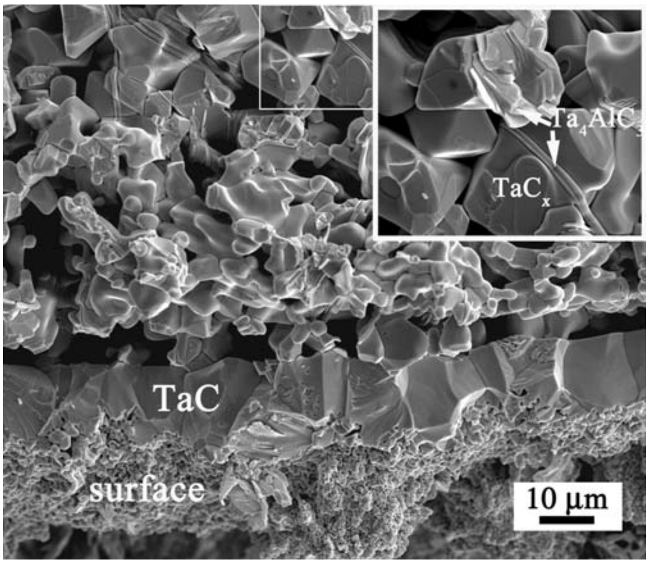

Fig. 6. SEM micrograph of a cross-section of the Ta_2AlC sample after treating at 1700 °C for 120 min. The inset is a high magnification view of the rectangular region.

grains are Ta_4AlC_3. It is difficult to accurately determine the x value because of the light C element [35]. We further studied the X-ray diffraction patterns of treated Ta_2AlC samples after removing the surface layer, as shown in Fig. 7. Obviously, the Ta_4AlC_3 phase appears in the sample treated at 1600 °C for 120 min (Fig. 7a). When the temperature is raised to 1650 °C, Ta_4AlC_3 is detected as the predominant phase (Fig. 7b). At 1700 °C, both Ta_4AlC_3 and TaC_x are detected (Fig. 7c). From 1600 to 1700 °C, it is seen that the diffraction peaks of Ta_4AlC_3 shift to higher angles, indicating the decrease in lattice parameters of Ta_4AlC_3 with Al out-diffusion [24, 33]. The lattice parameter c values of Ta_4AlC_3 are established as 2.383, 2.359 and 2.290 nm, respectively. The diffraction profile of TaC_x is similar to that of rhombohedral $TaC_{0.59}N_{0.15}$ (structure $R\bar{3}m$) which was formed by substituting carbon atoms for about 20 at.% nitrogen atoms in Ta_4C_3 [36]. Based on the XRD profile (Fig. 7c), the lattice parameters of TaC_x are calculated as $a = 0.3118$ nm and $c = 2.986$ nm, which is close to those of $TaC_{0.59}N_{0.15}$ ($a = 0.3107$ nm and $c = 3.009$ nm) and Ta_4C_3 ($a = 0.3120$ nm and $c = 3.005$ nm) [36]. So, TaC_x may have the same rhombohedral structure as $TaC_{0.59}N_{0.15}$ and Ta_4C_3. Additionally, Emmerlich et al. [24] reported the rapid decomposition of Ti_3SiC_2 by Si out-diffusion along the basal planes to form Ti_3C_2 slabs above 1100 °C, and then the Ti_3C_2 slabs detwinning into (111)-oriented $TiC_{0.67}$ layers. Therefore, the x value may be 0.75. The results suggest that Ta_2AlC is unstable above 1600 °C and decomposes into Ta_4AlC_3 or $TaC_{0.75}$ by Al out-diffusion. The decomposition sequence may be that Ta_2AlC firstly decomposes to Ta_4AlC_3, and then to rhombohedral $TaC_{0.75}$. In addition, Zhang et al. [37] reported that Ti_3AlC_2 preferred to form h-TiC_x and then C vacancies redistributed more uniformly to form c-TiC_x when studying the structure stability of Ti_3AlC_2 in Cu above 850 °C. Possibly, the transformation of rhombohedral $TaC_{0.75}$ into cubic $TaC_{0.75}$ happened during the heat treatment. The higher C content of cubic TaC_x in the surface scale is probably attributed to the C-supply from the graphite crucible [35]. Also, Al_4C_3 was found on the wall of graphite crucible. It may be formed according to reactions (10) and (11):

$$Ta_4AlC_3(s) \rightarrow 4\,TaC_{0.75}(s) + Al(g) \tag{10}$$

$$4\,Al(g) + 3C \rightarrow Al_4C_3(s) \tag{11}$$

4. Conclusions

Ta_2AlC ceramic was fabricated by an in-situ reaction/hot pressing method using Ta, Al and C powders as initial materials. Investigation of the reaction path showed that Ta_2AlC was produced by reaction between $AlTa_2$ and graphite, or between Ta_5Al_3C, TaC and graphite at 1500–1550 °C. Ta_4AlC_3 was the impurity phase in Ta_2AlC and was difficult to eliminate. Through modifying the element composition, single-phase Ta_2AlC could be obtained using an optimized composition with the molar ratio of Ta : Al : C = 2 : 1.2 : 0.9 and hot pressing at 1550 °C. Above 1600 °C, Ta_2AlC was unstable and decomposed into Ta_4AlC_3, and then to TaC_x.

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