Synthesis of Ti$_2$AlN MAX-phase by sintering in vacuum

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Abstract. The dependence of MAX-phase synthesis on phase composition of the initial mixture and heat treatment terms in the electric furnace was investigated. Using Ti and AlN powders with the molecular ratio 2:1 as an example, the sequence of phase composition changes with increasing exposure temperature was tracked. It was established that the maximum amount of Ti$_2$AlN MAX-phase in the end products is obtained at 1400°C. At this temperature a single phase product (100% wt Ti$_2$AlN) was obtained for the initial mixture Ti:Al:TiN = 1:1:1. The principal possibility of scaling for obtaining a single-phase product of Ti$_2$AlN by vacuum sintering was experimentally shown on the example of the mixture of this composition weighing 500 g at a certain mode of thermal vacuum treatment.

1. Introduction

Ti$_2$AlN ternary compound belongs to MAX-phase systems with the general formula M$_{n+1}$AX$_n$ (usually $n = 1\div3$), where M is a transition metal, A is an element from IIIA and IVA groups of Mendeleev periodic system, X is carbon or nitrogen [1]. MAX phase compounds are the point of the scientific interest for different research groups since they possess properties of metal and ceramic simultaneously [2, 3]. Ti$_2$AlN-based materials are characterized by high electric and heat conductivity, high elastic modulus, low density, high thermal stability and ability to mechanical treatment that makes them attractive for various high-temperature applications [4].

The aim of this work was to obtain a single phase product (Ti$_2$AlN) by sintering different mixtures in the vacuum furnace. The additional aim was to define an opportunity of the process scaling to obtain a considerable amount of the single phase product of Ti$_2$AlN (using the example of 500 g specimen). Literature data analysis did not reveal any application of this method for obtaining Ti$_2$AlN.

2. Experimental

The powders of commercial Ti (PTS-1 brand), Al (ASD-4 brand), TiAl (PT65Yu35 brand) and home-made SHS-produced AlN, TiN were used as raw materials. The following powder blends: 2Ti + AlN (blend 1), TiAl + TiN (blend 2), and Ti + Al + TiN (blend 3) were intermixed in a ball mill. The prepared blends in quartz tubes were placed in a vacuum furnace (SNVE-16/16) and held at $T$ = 1100, 1200, 1300, 1400, and 1500°C (pressure $5.8 \times 10^{-6}$ mm Hg) for 60 min. The sintering products were characterized by XRD (DRON-3M) and SEM/EDS (Zeiss Ultra Plus, Carl Zeiss equipped with an INCA Energy 350 XT accessory, Oxford Instruments).
3. Results and discussion

Diffraction patterns of the products obtained by sintering of blend 1 are presented in figure 1. It is evident that at $T = 1100$–$1200°C$ the obtained product is multiphase. It contains $\text{Ti}_2\text{AlN}$, $\text{TiN}$, $\text{AlN}$, $\text{Ti}_3\text{AlN}$, and $\text{Ti}_3\text{Al}$. With increasing temperature, the $\text{Ti}_2\text{AlN}$ content is seen to grow from 20 to 52% (figure 2).

![Figure 1. Diffraction patterns of products obtained by sintering of blend 1 at various temperatures.](image)

With an increase in the temperature to $1300°C$ the obtained product contains 83% $\text{Ti}_2\text{AlN}$, 12% $\text{TiN}$ and 5% $\text{AlN}$. The product formed at $1400°C$ contains 94% $\text{Ti}_2\text{AlN}$ and 6% $\text{TiN}$. But in the product formed at $1500°C$ the amount of $\text{Ti}_2\text{AlN}$ decreases to 91% and the amount of $\text{TiN}$ increases to 9%.

The decrease in $\text{Ti}_2\text{AlN}$ amount coincides with the data in [5], which demonstrated that at $T > 1550°C$ in dynamic vacuum, aluminum evaporated from the MAX-phase to form non-stoichiometric titanium nitride:

$$\text{Ti}_2\text{AlN}(s) \rightarrow 2\text{TiN}_{0.5} + \text{Al}(g)$$

That implies that the maximum amount of $\text{Ti}_2\text{AlN}$ MAX phase (94%) can be obtained at $T = 1400°C$. That is why blends 2 and 3 were sintered at the same temperature.

After sintering of blend 2 the product formed at $1400°C$ contains 94% $\text{Ti}_2\text{AlN}$, 4% $\text{TiN}$, 1% $\text{Ti}_3\text{AlN}$ and 1% $\text{Ti}_3\text{Al}$. The diffraction pattern is shown in figure 3. After sintering of blend 3 the single phase $\text{Ti}_2\text{AlN}$ product was obtained (figure 4a). The material exhibits a nanolaminate structure typical of MAX compounds (figure 4b).
The scale factor investigation was carried out with blend 3 since the maximum product yield of Ti$_2$AlN MAX-phase was obtained with it. The experimental results are presented in figure 5.

Figure 6 shows the fracture surface of the product. The EDS results for the area indicated in figure 6 gave the following elemental composition: N 23.56 at %, Al 25.68 at %, and Ti 50.76 at %; it is in good agreement with that of Ti$_2$AlN.
4. Conclusions

Thus, as a result of the studies the exposure temperature of 1400°C was determined, at which the maximum content of Ti$_2$AlN MAX phase was observed in the end product. The maximum amount of Ti$_2$AlN MAX-phase (100%) was obtained for the mixture Ti:Al:TiN=1:1:1. Using the example of sintering of 500 g of mix 3, the principal possibility of scaling for the sintering process in vacuum and perspectives of this method for industrial application were demonstrated.

References

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