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2020 7th International Congress on Energy Fluxes and Radiation Effects (EFRE)

Tomsk, Russia, September 14 – 26, 2020

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Study of the Microstructure of Synthesized Laminates

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***Abstract*—The studies show the possibility of obtaining titanium - titanium aluminide laminates by the interaction of titanium with aluminum. An important role in the study and obtaining of such materials is assigned to the study of diffusion processes between titanium and aluminum layers, and the formation of intermetallic layers and their microstructure. In the experiments the desired laminated microstructure is obtained by all four methods. At the same time, the disadvantages inherent in each of these methods are found. Ti-Al_3Ti metal - intermetallic laminates synthesized by thermal explosion, reaction sintering with and without pressure, explosion welding with further heat treatment for powder mixtures and foils are studied using a microstructural analysis. The structure and composition of the samples are studied by X-ray diffraction, X-ray microanalysis, and metallography. The best microstructural parameters are obtained by explosion welding of a multilayer package of titanium and aluminum plates, then by sintering this package in a muffle furnace with selection of time-temperature parameters. The conducted studies can be used to obtain laminates with a desired thickness of titanium and intermetallic layers.**

Keywords—metal-intermetallic laminate, synthesis, microstructure, foil, powder mixture.

I. Introduction

A new promising class of structural and multifunctional materials is metal - intermetallic laminates (MIL) [1–3]. Such materials have many useful properties and characteristics, such as high temperature strength, high oxidation stability, high creep resistance and others, which make them attractive for use in many fields of engineering [1, 4–7] and also for ballistic applications [1, 3, 8–13].

The studies show the possibility of obtaining titanium – titanium aluminide laminates by the interaction of titanium with aluminum. An important role in the study and obtaining of such materials is assigned to the study of diffusion processes between titanium and aluminum layers, and the formation of intermetallic layers and their microstructure [14–17].

In this paper, the microstructure formation of the samples synthesized by different methods with the use of a powder mixture and metal foils is studied.

II. Experimental Setup

Foils of titanium (VT1-0) and aluminum (8011) 0.3 and 0.15 mm in thickness, the plates of titanium (0.5 and 0.6 mm) and aluminum (1 mm) of the same grades, and the powders of titanium (PTS) and aluminum (ASD-4) were used in the experiments.

A stoichiometric powder mixture of titanium and aluminum and titanium foil 0.3 mm in thickness was used to synthesize titanium trialuminide Al_3Ti (37.2 wt.% Ti + 62.8 wt.% Al) in the thermal explosion mode (synthesis occurring simultaneously in the entire volume of the mixture). The powder mixture was pressed into tablets with a diameter of 20 mm and a porosity of 10–15% (pore volume in % of the total volume). A specially designed setup was used to conduct synthesis in the thermal explosion mode [18, 19]. To determine the temperature, a 200 μm tungsten-rhenium thermocouple was used, which was placed in the powder layer of the sample.

Ti and Al plates and a PM-12M muffle furnace were used for reaction sintering. External load was applied to reaction sintering to improve contact between the foils. The plates were polished and degreased. The package was made up of alternating titanium plate and three aluminum plates. The ratio of the number of titanium and aluminum plates was selected based on the layers of the intermetallide Al_3Ti and pure titanium formed during sintering. The package was sintered at a temperature of 700 and 900°C for 2, 4 and 6 hours.

The method of reaction pressing (reaction sintering under pressure) was applied at a special facility at the M.N. Mikheev Institute of Metal Physics of the Ural Branch of the Russian

The reported study was funded by the Russian Science Foundation, project No. 16-19-10264.

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Academy of Sciences, Yekaterinburg, Russia [4, 16]. Ti and Al plates were used, and temperature, pressure, and time of the process varied.

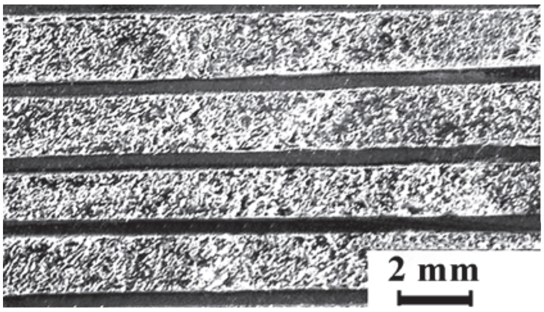
Explosion welding was conducted in the explosive chamber (Lavrentyev Institute of Hydrodynamics of the Siberian Branch of the Russian Academy of Sciences, Novosibirsk, Russia). The packages of 11 and 13 alternating titanium (0.5 and 0.6 mm thick) and aluminum (1 mm) plates with the size of 50x100 mm, the package of 21 titanium (0.6 mm) and aluminum (1 mm) plates with the size of 120 mm × 300 mm were obtained by explosion welding. The samples obtained by explosion welding were subjected to reaction sintering in a muffle furnace at temperatures of 700 and 900°C for 2, 4, 6 and 8 hours.

The synthesized samples were studied by X-ray diffraction (XRD), X-ray microanalysis (CAMECA), and metallography (Axiovert 200M).

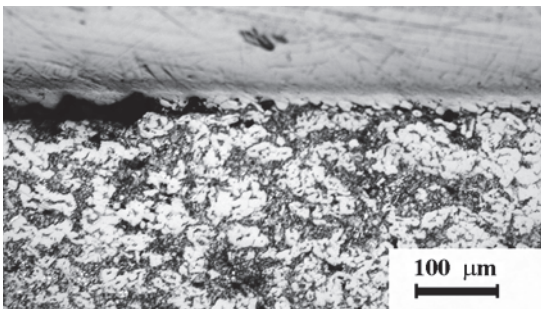
III. Results

A. Investigation of the Interaction of Titanium Foil and a Powder Mixture in Thermal Explosion Mode

The structure of the laminate synthesized from titanium foil and stoichiometric powder mixture Al_3Ti (37.2 wt.% Ti + 62.8 wt.% Al) is shown in Fig. 1a. At higher magnification, thin layers are observed at the interface between titanium foil and intermetallide synthesized from the powder mixture (Fig. 1b). X-ray diffraction data showed that the stoichiometric powder mixture reacted completely. X-ray diffraction patterns contain Al_3Ti peaks; peaks belonging to titanium oxide are infrequent.



(a)



(b)

Fig. 1. Microstructures of the laminate of titanium foils and a powder mixture (a) and the contact zone between titanium foil and the powder mixture (b).

B. Investigation of the Interaction of Titanium and Aluminum Foils in Thermal Explosion Mode

Synthesis with titanium and aluminum foils in the thermal explosion mode showed the porosity at the contacts between the layers. The layers of aluminum have unequal thickness along the sample, although initially they were of the same thickness. The reason for this may be, on the one hand, the non-uniformity of diffusion processes occurring within the one sample, on the other hand, aluminum could leak out from some layers. Separate inclusions and interlayers are visible on aluminum layers, which indicates the formation of new phases inside the aluminum layer.

Fig. 2 shows a fragment of a laminate sample after a thermal explosion. Zone 1 is the boundary between aluminum and titanium foils. The contact area between foils and the small areas of titanium and aluminum foils are in this zone. Microanalysis of the contact area showed titanium content in the range of 78 to 86 wt %. This ratio is close to the titanium content in the $AlTi_3$ phase on the phase diagram of the titanium – aluminum system.

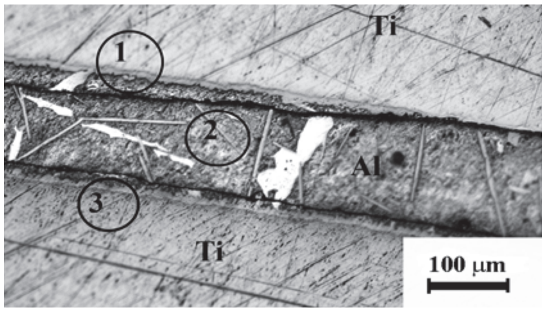


Fig. 2. Contact zone between titanium and aluminum foils after thermal explosion.

Zone 2 is the zone of aluminum foil. According to X-ray microanalysis, the titanium content decreases in this with increasing the distance from the titanium foil. The titanium content reduced to 2 to 3 wt% at certain points in the middle of aluminum foil. But points containing 100% aluminum were not found. The light inclusions inside the aluminum foil contain titanium, the weight of which is close to that in the Al_3Ti phase.

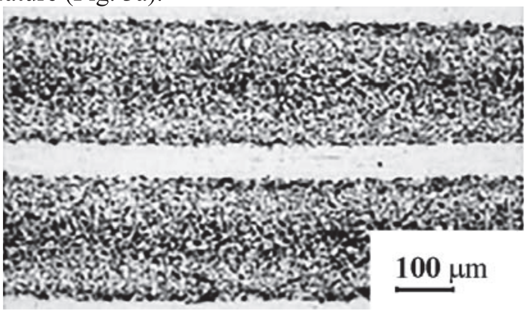
Zone 3 is the zone of titanium foil. Pure titanium was not found in this zone at a distance of 0.15 mm from the boundary with aluminum foil. The titanium content increases with the distance from the boundary with aluminum.

C. Investigation of the Interaction of Titanium and Aluminum Plates in the Reaction Sintering Mode

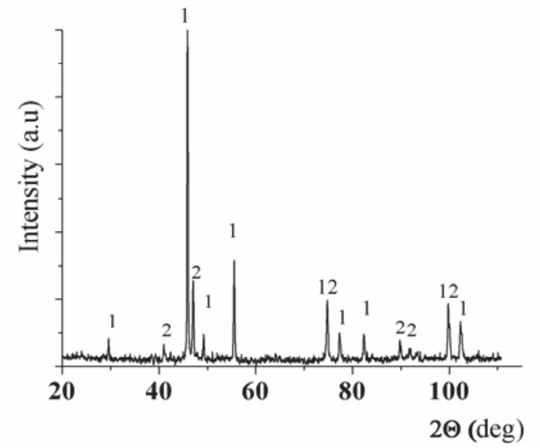
The reaction sintering method combines sintering with a chemical reaction of the intermetallide synthesis. The sintering of the samples at T = 700°C initiates diffusion processes that form layers between titanium and aluminum plates, which provides a relatively strong bond between the plates. Depending on the sintering time, aluminum may not be completely consumed.

At a sintering temperature of 900°C, a laminate sample was obtained, consisting of alternating layers of titanium and two-phase layers, which were formed by the interaction of liquid aluminum with solid titanium (Fig. 3a). At the boundary between the titanium interlayer and the two-phase area, a diffusion region providing a strong bond between the layers is observed. Two phases are identified in the X-ray spectrum of the sample: Ti and Al_3Ti (Fig. 3b).

The two-phase area consists of the Al_3Ti phase and pores. In some places between the layers there are gaps, the occurrence of which can be associated with a high sintering temperature (Fig. 3a).



(a)



(b)

Fig. 3. Microstructure (a) and X-ray diffraction pattern of a laminate (1-$TiAl_3$; 2-Ti) (b) after sintering at T = 900°C for 6 hours.

D. *Investigation of the Interaction of Titanium and Aluminum Plates after Explosion Welding and Sintering*

Fig. 4 shows the microstructure of a titanium-aluminum laminate after explosion welding. The wavy nature of the interface is visible, which enhances the contact and adhesion between the layers of titanium and aluminum.

Near the interface the island diffusion inclusions Ti(Al), the chemical composition of which is given below, are visible in aluminum and inside aluminum layers:

1. The aluminum layer inclusions located in the middle of the layer consist of Al – 11.831, Ti – 88.169 at%.

2. The aluminum layer inclusions located near the boundary with titanium have average values: Al – 5.822, Ti - 94.178 at%.

3. Narrow contact zone between foils: the formed phase with the atomic ratio corresponding to the Al3Ti phase is clearly traced. Also this zone contains the areas with the Al values from 9.314 to 75.545, Ti - 90.686 to 24.455 at%.

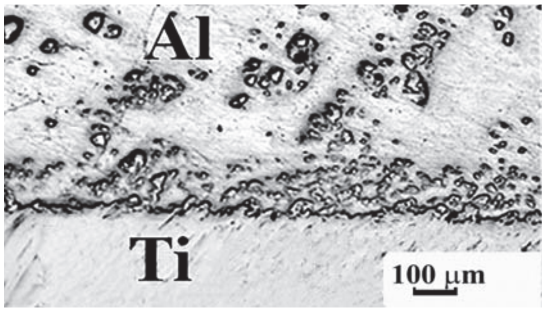


Fig. 4. Microstructure of a titanium-aluminum laminate after explosion welding.

To obtain metal-intermetallic composites, laminated packages were sintered after explosion welding. Fig. 5 shows the microstructure of the contact area of the composite after sintering at T = 700ºC and holding for 2 hours. The inclusions of the formed products are visible in the aluminum layer near the interface.

Chemical composition data were obtained using a scanning electron microscope and an X-ray dispersion energy spectrometer. This micrograph shows that the intermetallic layer formed by the diffusion interaction of titanium with liquid aluminum is not homogeneous. The growth of the diffusion interlayer begins with the formation of local sections, which, with an increase in the sintering time, increase in size and form the one homogeneous layer. In the micrograph, the numbers indicate the areas in which the chemical composition was determined. It was found that points 1, 3 are pure titanium, points 2, 4 are Al_3Ti, and point 5 is Al. The round particles inside the Al foil are Al_3Ti intermetallide grains surrounded by solidified Al(Ti) melt.

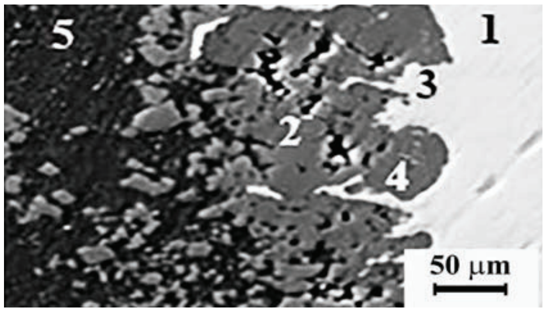


Fig. 5. Microstructure of the contact area of the laminated composite after sintering at T = 700ºC and 2- hour holding time.

Fig. 6 shows the fragments of the laminate microstructure after sintering at T = 700°C for 4 hours. Microstructural studies showed that an increase in the holding time to 4 hours led to the formation of a homogeneous layer of the Al_3Ti intermetallide at the interface with titanium and the occurrence of a two-phase structure in aluminum (Fig. 6a). In the zones indicated by numbers (Fig. 6b), microanalysis determined the content (at%) of the following elements:

1. Al – 0, Ti – 100.0;

2. Al – 0, Ti – 100.0;

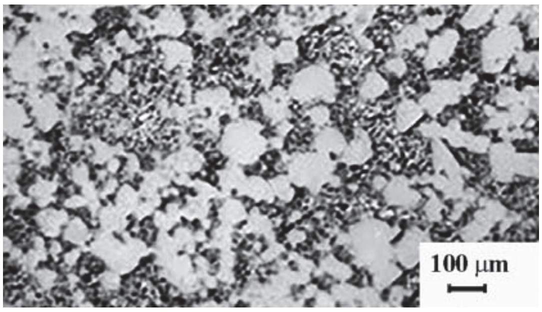
3. Al – 0, Ti – 100.0;

4. Al – 75.040, Ti – 25.364;

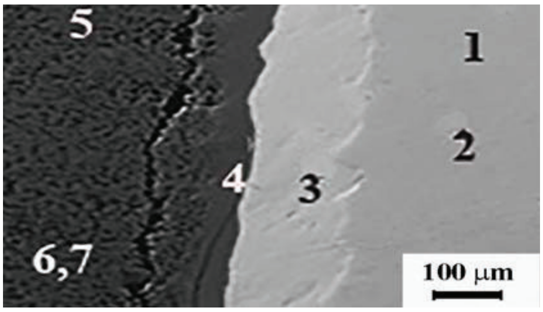
5. Al – 75.719, Ti – 24.281;

6. Al – 91.580, Ti – 8.420;

7. Al – 92.144; Ti – 7.856.



(a)



(b)

Fig. 6. Microstructure of the laminated composite after sintering at T = 700ºC and 4-hour holding time.

Fig. 7 shows the fragments of the microstructure of a laminate after sintering at *T* = 700°C for 6 hours. Titanium layers (narrow layer) and wide layers of Al_3Ti intermetallide can be seen. Dark narrow areas, which are two-phase regions consisting of Al_3Ti and solidified Al(Ti) melt are visible in all intermetallic interlayers in the central part. These areas are characterized by greater porosity.

In the zones indicated by numbers (Fig. 7b), microanalysis determined the content of the following elements (at%):

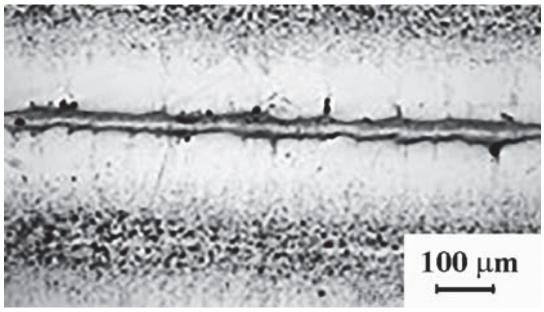
1. Al – 0, Ti – 100.0;

2. Al – 74.573, Ti – 25.427;

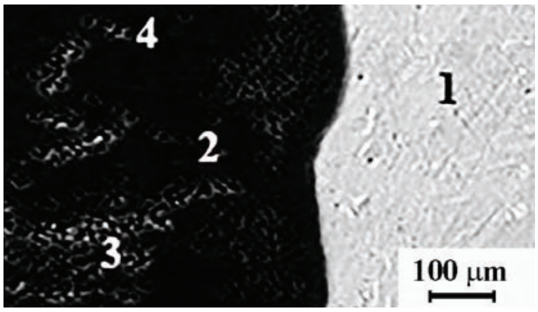
3. Al – 67.573, Ti – 32.427;

4. Al – 74.412, Ti – 25.585.

A homogeneous laminated sample consisting, according to the data of X-ray spectral and X-ray diffraction analyzes, of alternating layers of Ti and intermetallic Al_3Ti was obtained increasing the holding time to 8 hours.



(a)



(b)

Fig. 7. Microstructure of the laminate after sintering at *T* = 700ºC and 6-hour holding time.

E. Investigation of the Interaction of Laminates after Reaction Sintering under Pressure

The microstructural studies of laminates from titanium and aluminum plates obtained by the reaction sintering method combined with pressing are presented. Fig. 8 shows the microstructure of the sample, and Fig. 9 shows the contact zone between the layers at a higher magnification.

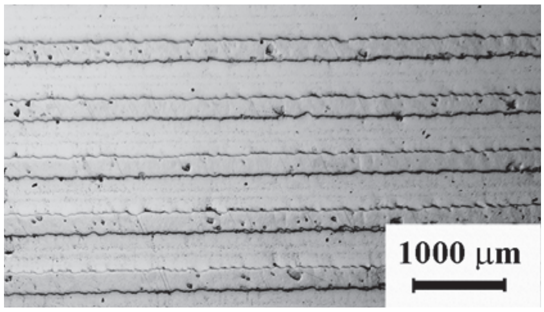


Fig. 8. Micrograph of the sample layers.

The microhardness measurement of some layers showed the following:

Dark narrow layer: ~ 141 kg/mm^2;

wide light layer: ~ 752 kg/mm^2.

Table values: titanium: 140 - 200 kg/mm^2,

TiAl: 180 - 280 kg/mm^2,

Al_3Ti: 595 - 620 kg/mm^2.

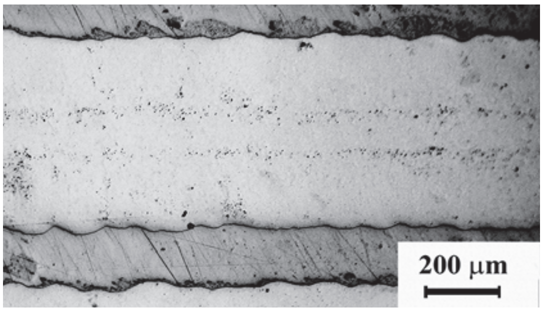


Fig. 9. Contact zone between the layers.

Based on these data, it can be concluded that the dark narrow layer is titanium, and the light wide layer is Al_3Ti intermetallide. The data of X-ray diffraction analysis (Fig. 10) show that the laminate consists of two phases: titanium and Al_3Ti, which is in good agreement with the microhardness measurements. Based on the experimental results obtained, it was shown that it is possible to obtain a laminate based on titanium aluminide Al_3Ti and Ti by reactive sintering under pressure.

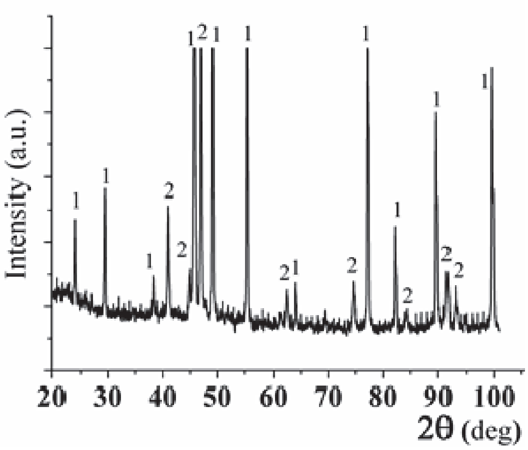


Fig. 10. X-ray diffraction pattern of a laminate obtained by reaction sintering under pressure.

IV. Conclusion

In the experiments the desired laminated microstructure was obtained by all four methods. At the same time, the disadvantages inherent in each of these methods were found.

After synthesis in the thermal explosion mode, the intermetallic layers of the samples have high porosity and low strength. There are also problems of mechanical and physico-chemical compatibility of dissimilar materials at the interface between the layers, which leads to the absence of a strong bond between the titanium foil and the synthesized intermetallide. After reaction sintering, an intermetallic layer has high porosity and low strength, while a region of increased porosity is formed in the middle of the intermetallic layer. The reaction pressing method partially solves the problem of high porosity in the intermetallic layer, but nevertheless, there are the pores that are unevenly located in the layer, which requires additional improvement of the synthesis modes for metal - intermetallic laminates. After combined processing by explosion welding and sintering, a central layer of increased porosity is also formed, which reduces the strength characteristics of the composite.

Acknowledgments

The authors appreciate the financial support from the Russian Science Foundation (RSF) (grant no. 16-19-10264). The authors thank N. G. Kasatsky, V. I. Mali and A. M. Patselov for help in conducting experiments.

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