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Development of Innovative SHS Technology Coupled with Thermal Explosion for Production of Single-Phase Nanocrystalline Materials of Ti-Al System

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ABSTRACT. Self-Propagated High-Temperature Synthesis (SHS), coupled with thermal explosion, is suggested for environmentally safe, energy-efficient production of nanostructured, single-phase intermetallics. Two new technologies, based on combustion and explosion, are developed for fabrication of compacted, pore-free, single-phase components. To solve this problem a device for conduction of SHS in the mode of explosion is designed and constructed. The developed technology allows production of single-phase, nanostructured intermetallics. *©2008 Bull. Georg. Natl. Acad. Sci.*

Key words: *Intermetallics, nanostructured, compacting, synthesis, thermal explosion, plastic deformation.*

Intermetallics represent a unique type of materials that retain ordered atomic structure up to melting point. The intermetallics of Ti-Al system are lighter and more inexpensive than nickel- and titanium superalloys, do not need protection against corrosion at operating temperatures, and possess sufficiently high strength (breaking point at 1473K amounts to 100MPa). These alloys may turn into effective substitutes for nickel superalloys used not only for aircraft- and surface engines, but also in other branches of industry [1].

Wide application of alloys based on titanium aluminides is restricted by the lack of effective and inexpensive production technologies.

Development of relatively simple and reasonably priced technology for production of Ti-Al intermetallics, is an important and still unsolved task. The most suitable technological process, possessing considerable potential for production of composites with peculiar properties is Self-Propagating High-Temperature Synthesis (SHS). The method is simple and characterized by low energy consumption, low cost, reliability of the equipment used, and purity of the synthesized product. SHS may be conducted in two modes: combustion and explosion. SHS in the mode of thermal explosion is usually performed in a muffle furnace, in which drastic alteration of temperature or heat sink is not possible. This limits the possibility of regulation of external thermophysical conditions of synthesis, variation of which might control the processes of microstructure formation during the synthesis of heterogeneous powder systems in the mode of thermal explosion [2].

The existing technologies of SHS-thermal explosion may produce compounds in the form of powders in the Ti-Al system (here, no single-phase compounds are available). These powders are used in powder metallurgy as a starting material, and as a coating for the surfaces of different components.

We are first proposing two technologies of fabrication of single-phase (by means of X-ray diffractometry) components made from Ti-Al compounds, which are compacted and pore-free. These technologies are performed in two modes: combustion and thermal explosion, with the subsequent compacting of the synthesized product. The material has fine microstructure, and in particular cases even nano-structure, which makes them very attractive because of expected increase in strength and plastic characteristics of the material.

In order to solve the above tasks we developed an original and universal equipment for performance of SHS in the mode of thermal explosion. The equipment is capable of fabricating materials (Ti-Al) and components of simple shape in one technological cycle.

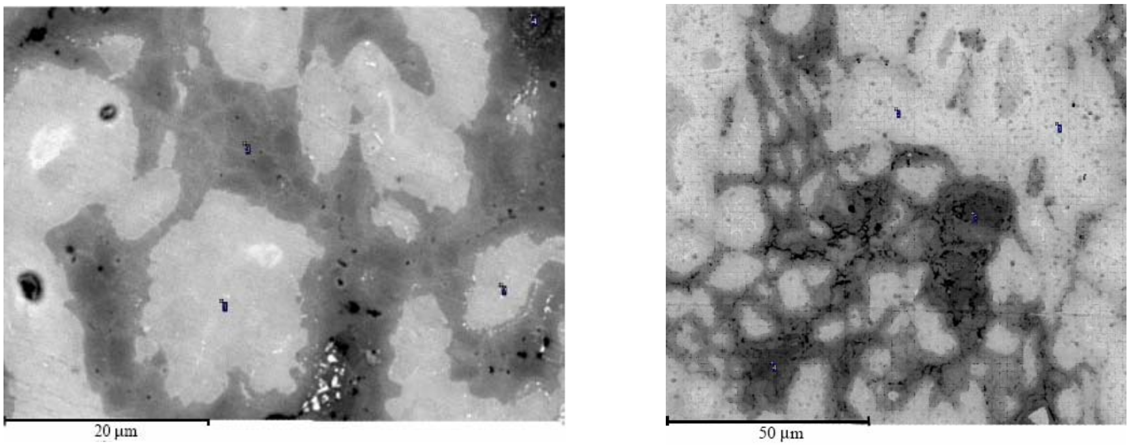
At this stage there is no adequate explanation of the mechanisms of microstructure formation in this system. However, study of grain size influence on mechanical properties of the intermetallics at room temperature revealed an unexpectedly strong dependence of plasticity on the above microstructure parameter. Thus, reduction of grain size to submicron level ($d<1\mu$) leads to a drastic shift of brittle-viscous transition temperature range to low temperatures, as well as an increase of plasticity at room temperature. The increase in plasticity, at room as well as at elevated temperatures, after grain sizes in Ti_3Al and TiAl reach a definite value, is related to the improvement of relaxation ability of grain boundaries [3].

It is shown that application of shear at pressure allows to deform the TiAl intermetallics (superstructure $L1_0$) up to extremely high rate, $\varepsilon=6.5$. This resulted in the formation of nanocrystalline structure with the sizes of the crystals, ~10nm.

The evolution of the microstructure during plastic deformation was studied, using X-ray analysis and electron microscopy. It was revealed that fragmentation of the alloy during deformation passes through the stages of lath formation, mainly because of twinning and subsequent loss of stability of grains and formation of ultra-micro- and nano-crystalline structures [4].

Because of peculiarities of Ti-Al compound formation in the developed equipment of SHS in the mode of thermal explosion, at the first stage of the process the starting materials (Ti and Al) are pressed at 0.25 – 2 Kbar (depending on the end product Ti_3Al, $TiAl_3$, TiAl). After heating to definite temperature with definite rate of heating (different for different phases, Ti_3Al, $TiAl_3$ and TiAl), a synthesis of the material in the mode of combustion or thermal explosion is conducted. After the synthesis of the compounds in the mode of thermal explosion, a compacting at high temperature, 1300 – 1600^{o}C is performed (depending on the end product, Ti_3Al, $TiAl_3$, TiAl). This causes plastic deformation of dislocation structure and formation of new grain boundaries, resulting in grain size refinement and formation of very fine microstructure with the dimensions, ~80nm.

Metallographic and phase analyses were conducted with scanning electron microscope (SEM) and x-ray microanalyzer (EPMA). In order to determine phase and elemental compositions of the synthesized samples, SEM and EPMA studies were performed. The SEM/EPMA was conducted on a Quanta 200 (FEI, USA) scanning electron microscope equipped with INCA (Oxford Instrument, UK) energy dispersive spectrometer (EDS). SEM images were captured using secondary electron detector as well as backscattered electron detector operating in compositional contrast mode. EDS measurements were carried out using the accelerating voltage 10kV, the probe current, 1nA at working distance of 10nm. The take



a) b)

Fig. 1. Microstructure of TiAl(a) and Ti_3Al(b) samples.

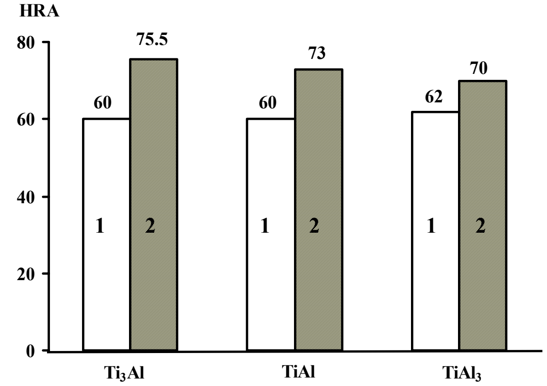


Fig. 2. Hardness alteration diagram for the materials $TiAl_3$, TiAl, Ti_3Al:
1 – samples before plastic deformation
2 – samples after plastic deformation

angle of x-ray radiation was 35°. Acquisition time was 50s per single measurement. Quantitative analysis was performed using the conventional correction procedure included in INCA software. The final results were normalized to 100%.

In Fig.1 the microstructures of TiAl(a) and Ti_3Al(b) samples are shown. It is obvious that because of multiple plastic deformation, breakage of dislocation structure takes place, leading to the formation of new boundaries of block (lath) structure and the refinement of TiAl and Ti_3Al grains to 70-80nm.

In Fig. 2, alteration of TiAl and Ti_3Al hardness is shown, depending on the compacting of the samples. The hardness of the samples, which were not compacted and plastically deformed after the synthesis, were compared with that of the compacted at 1300-1500°C and plastically deformed samples. It is shown that because of plastic deformation, the hardness of Ti_3Al varies from 60 HRA (220Hv) to 75.5 HRA (490Hv); for TiAl from 60 HRA (220Hv) to 73 HRA (445Hv); for $TiAl_3$ from 62 HRA (245Hv) to 71 HRA (400Hv). The strength of the material is also increased, for example for Ti_3Al it rises from $\sigma_b = 29\text{-}30\text{kg/mm}^2$ to $\sigma_b = 51\text{-}55\text{kg/mm}^2$.

The above described leads us to the conclusion that the developed technology makes it possible to produce nanostructured single-phase Ti-Al intermetallics.

მასალათმცოდნეობა

თმს თბური აფეთქების ინოვაციური ტექნოლოგიის შემუშავება Ti-Al სისტემაში ერთფაზიანი ნანოკრისტალური სტრუქტურის მქონე მასალების მისაღებად

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ერთფაზიანი ნანოკრისტალური სტრუქტურის მქონე ინტერმეტალიდების მისაღებად შემოთავაზებულია თმს-თბური აფეთქების ენერგოდამზოგავი, ეკოლოგიურად სუფთა ტექნოლოგია. შემუშავებულია ორი ახალი ტექნოლოგია (წვის და აფეთქების რეჟიმში) Ti-Al სისტემაში კომპაქტური, უფორო, რენტგენოერთფაზიანი ნაკეთობების მისაღებად. დასახული ამოცანის გადასაწყვეტად შექმნილია დანადგარი აფეთქების რეჟიმში

მაღალტემპერატურული სინთეზის ჩასატარებლად. შემუშავებული ტექნოლოგიური პროცესი საშუალებას გვაძლევს მივიღოთ ერთფაზიანი ნანოკრისტალური სტრუქტურის მქონე ინტერმეტალიდები.

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