

REFERENCES

1. Kolobov Yu.R., Valiev R.Z., Grabovetskaya G.P. et al. Grain boundary diffusion and properties of nanostructured materials. – Novosibirsk: Nauka, 2001. – 232 p.
2. Mazurovskii M.I., Murzinova M.A., Salischev G.A., Afonichev D.D. Use of hydrogen alloying for formation of submicrocrystalline structure in two-phase titanium alloys // *Metals*. – 1995. – № 6. – P. 83–88.
3. Yoshimura H., Nakahigashi J. Ultra-fine grain refinement, superplasticity and its application of titanium alloys obtained through proplum treatment // *Mat. Sci. Forum*. – 2003. – V. 426–432. – P. 673–680.
4. Grabovetskaya G.P., Melnikova E.N., Kolobov Yu.R. et al. Evolution of structure-phase state of alloy Ti-6Al-4V at formation of submicrocrystal structure using reversible hydrogen alloying // *Izvestiya Vuzov. Fizika*. – 2006. – V. 49. – № 4. – P. 86–91.
5. Skvortsov S.V., Ilyin A.A., Gurtovaya G.V. et al. Phase and structure transformations occurring in titanium alloy VT20L under hydrogen // *Metals*. – 2005. – № 2. – P. 45–53.
6. Nosov V.K., Kolachev B.A. Hydrogen plasticization at hot plastic deformation of titanium alloys. – Moscow: Metallurgiya, 1986. – 118 p.
7. Kolachev B.A., Nosov V.K. Hydrogen plasticization and superplasticity of titanium alloys // *Fizika Metallov i Metallovedenie*. – 1984. – V. 57, Issue 2. – P. 288–297.
8. Kolachev B.A., Livanov V.A., Bukhanov A.A. Mechanical properties of titanium and its alloys. – Moscow: Metallurgiya, 1974. – 554 p.
9. Ilyin A.A., Mamonov A.M. Temperature-concentration diagrams of phase composition of hydrogen containing multicomponent alloys on the basis of titanium // *Metals*. – 1994. – № 5. – P. 71–78.
10. Presnyakov A.A. Localization of plastic deformation. – Moscow: Mashinostroenie. – 1983. – 56 p.

Received on 21.12.2006

UDC 536.46

FORMATION OF PHASE STRUCTURE IN THE SYSTEM Ti-3Al AT THE STAGE OF SECONDARY STRUCTURIZATION AT SYNTHESIS IN THE MODE OF THERMAL EXPLOSION

V.Yu. Filimonov, M.V. Loginova

I.I. Polzunov Altai state technical university, Barnaul

E-mail: VYFilimonov@rambler.ru

Features of phase formation processes in the system Ti-3Al at realization of self-extending synthesis in the mode of thermal explosion have been established with use of technological reactor enabling instant switching-off of the heating source. The analysis of synthesis finished products allows to draw a conclusion that phase structure of charge is abnormally depends on sizes of titanium particles. The single-phase product corresponding to initial stoichiometry is synthesized on fine and large fractions for the induction period. On intermediate fraction the product of synthesis is multiphase.

Introduction

Recently, interest of researchers in the field of fundamental and applied material science to compounds on the basis of titanium aluminide increased considerably. Use of intermetallic compounds on the basis of titanium and aluminum in various branches of engineering has wide perspectives owing to combination of a number of mechanical and physical-chemical properties. Titanium aluminides are characterized by low specific weight, high heat resistance, thermal stability, resistance to corrosive media action at high temperatures that makes it possible to apply them in aircraft construction, shipbuilding [1].

At the same time, rather small amount of publications are devoted to studying self-propagating high-temperature (SH) synthesis in this system in spite of the fact that the main field of application of specified compounds is detonation-gas or plasma spraying for obtaining protective coatings. From this point of view the main task of experimenter is to obtain monophasic powder materials of certain composition. It should be kept in mind here that the process of phase formation may be nonequilibrium.

In the work [2] the criteria determining two ultimate mechanisms of occurring SH-synthesis processes depending on the ratio of characteristic burning time t_c and structure formation t_s were stated. In the case $t_s/t_c \ll 1$ equilibrium mechanism of structure formation takes place. In the process of burning all phases known at state diagram (Merzhanov mechanism) are formed. In opposite case $t_s/t_c \gg 1$ during burning reaction products being in metastable state are formed. At termination of chemical reaction phase and chemical transformations which are determined by diffusion processes (Borovinskaya mechanism) occur in these products. At this very stage the processes of structure formation may depend on environment, first of all, on heat dissipation condition and size of refractory component particles. Change of ratio of indicated times may give an opportunity to control the end product composition in the mode of layerwise burning or thermal explosion. In this case it should be kept in mind that times of structure formation are determined by diffusion coefficients at phase formation in the processes of reactionary diffusion or dissolution. If typical time of heat extraction is rather lower than structure formation time the metastable phases may be obtained [3].

In this paper the structure formation process in powder system Ti-Al at component synthesis in the mode of static thermal explosion, at different duration of synthesis processes and different sizes of refractory component particles were studied.

Experimental technique

Reactor represented a metal cylinder – 1 on lateral surface of which nichrome spiral – 2 was coiled through the insulating layer – 3. In order that system warming resulted in volume thermal explosion end surfaces were warmed up by special heating elements – 4 representing layers of asbestos cardboard «broached» with nichrome wire which was serially connected with spiral coiled on lateral surface. Temperature was measured using two chromel-alumel thermocouples – 5 one of which was in batch volume – 6 another one was calked in reactor side (7 – compensation junctions). Between the batch and end heater a layer of asbestos – 8 was laid. Spiral supply was switched off by a switch – 9. Signal from thermocouples was delivered to computer with built in multichannel card 10 LA 1,5 PCI through the normalizer – 10.

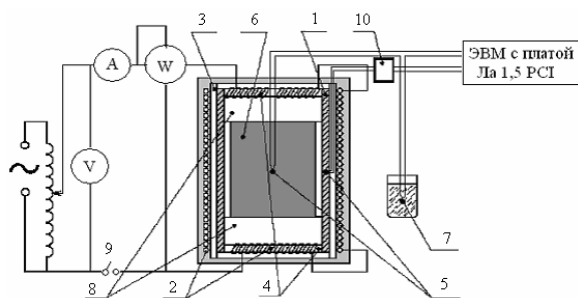


Fig. 1. Block diagram of technological reactor for carrying out SH-synthesis in thermal explosion mode

Inner diameter of reactor cavity amounted to 3,8 cm, cavity height was 5 cm.

Characteristic feature of reactor construction is the fact that lateral surface and ends of cylinder were heated by one and the same spiral that allowed approximating the system as much as possible to the mode of static thermal explosion as in this case reactionary volume is uniformly warmed up. In order to control temperature homogeneous distribution at synthesizing a junction of control thermocouple was placed at a distance of 1 mm from end surface and the difference in indices with thermocouple placed in the center of the reactor did not exceed 3 % at thermal explosion development. Presence of switch – 9 in the diagram allows switching off the spiral supply at any time and in this case batch is rapidly cooled.

The experiment results

Titanium powders of three marks PTH of three fractions: 55 ± 7 mkm, 130 ± 23 mkm, 242 ± 42 mkm and aluminum powders ASD-1 with average size 12 mkm were used in the experiment. At the first stage of the experiment the thermal explosion dynamics and phase composition of end product for an average fraction of titanium particle 1300 ± 23 mkm was studied. In Fig. 2 the ap-

propriate thermogram is given. Spiral power supply was switched off at the moment when batch had reached maximal temperature.

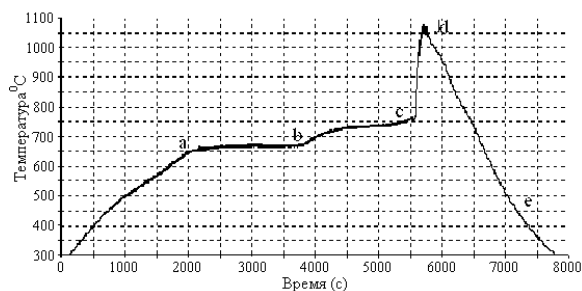


Fig. 2. Thermogram of the process of synthesis in the mode of thermal explosion at switching off heating source at the moment of reacting batch achieve of maximal temperature. Reactor wall temperature is 750°C , titanium particle size is 130 ± 23 mkm

Area *ab* corresponds to aluminum melting. Area *bc* corresponds to batch warming up to the temperature of the wall (aluminum in liquid phase). Area *cd* is the thermal explosion (primary structure formation), area *de* is the batch cooling down room temperature after switching off power source.

The appropriate diffractogram of synthesis end product is given in Fig. 3.

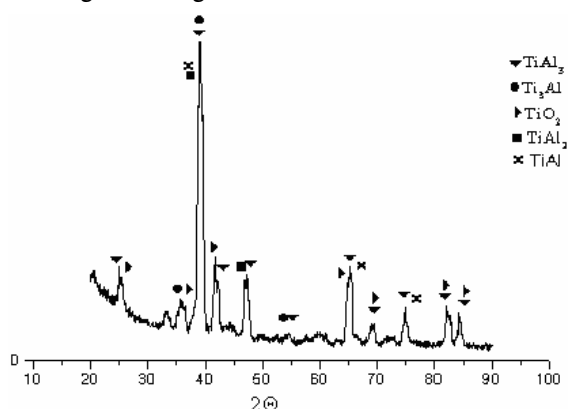


Fig. 3. Diffractogram of synthesis end product at switching off the power source at the moment of achieving maximal temperature by reacting batch. Titanium particle size is 130 ± 23 mkm

It follows from the view of diffractogram that the end product is multiphase with the content of all phases existing at state diagram [4] in this case aluminum reflections are not observed. The conclusion may be drawn that the formed phases are oversaturated with aluminum as the original composition corresponds to stoichiometry of the phase TiAl_3 .

Thermogram of synthesis for particles of the same size but with cure time 4300 s after achieving maximal temperature by the system at temperature of reactor wall 800°C is given in Fig. 4. There is a well-marked plateau which corresponds to heat release from phase formation at secondary structure formation at thermogram.

Possibility of secondary structure formation is conditioned by the fact that wall temperature is supported

rather high therefore, heat extraction rate decreases in comparison with the previous case. It follows from the view of proper diffraction pattern (Fig. 5) that only two phases remain in the end product.

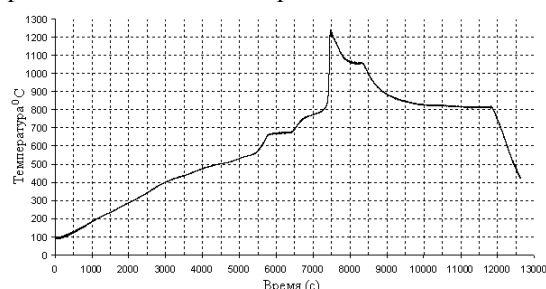


Fig. 4. Thermogram of thermal explosion process at temperature time lag of reactor wall (800 °C) during 4300 s. Titanium particle size is 130 ± 23 mkm

Then the system was kept 6600 s after achieving temperature peak and in this case thermogram differs qualitatively in no way from the given one in Fig. 4. The appropriate diffraction pattern is given in Fig. 6.

At annealing synthesis product obtained in the mode of source switching off in the point of thermogram maximum, diffraction pattern is completely identical, synthesis product is monophase. Thus, the conclusion may be drawn that metastable phases oversaturated with aluminum relax at long-term annealing to equilibrium state which is determined by original stoichiometry.

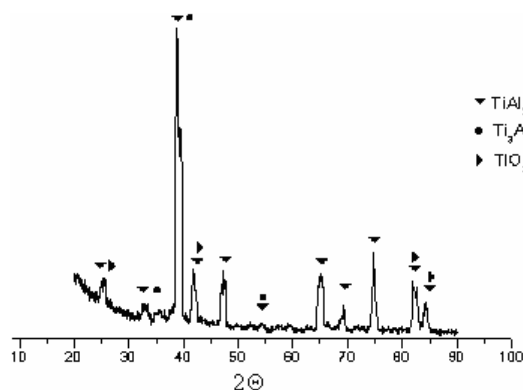


Fig. 5. Diffraction pattern of synthesis end product at temperature time lag of reactor wall (800 °C) during 6600 s. Titanium particle size is 130 ± 23 mkm

Synthesis for fine fraction 55 ± 7 mkm in the mode of source switching off was implemented at the next stage of the experiment. The result of synthesis was monophase product of composition $TiAl_3$. Diffraction pattern of synthesis product corresponded completely to that given in Fig. 6.

Then synthesis for the coarsest fraction of titanium particles 180 ± 32 mkm was carried out.

Thermogram obtained in the mode of source switching off is given in Fig. 7; the appropriate diffraction pattern is given in Fig. 8.

It follows from the diffraction pattern view that synthesis product is monophase. At first sight such result is seemed to be unexpected as at particle size growth the

wide range of synthesized phases should be expected by the reason of relatively long time of structure formation.

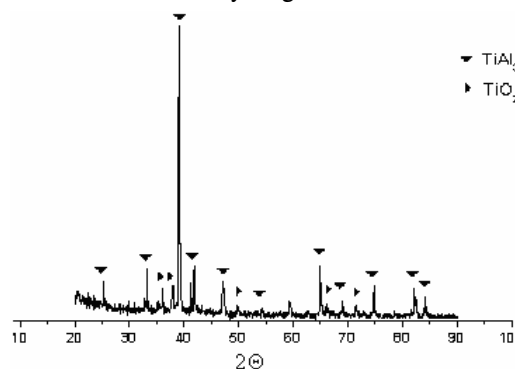


Fig. 6. Diffraction pattern of synthesis end product at temperature time lag of reactor wall (800 °C) during 6600 s. Titanium particle size is 130 ± 23 mkm

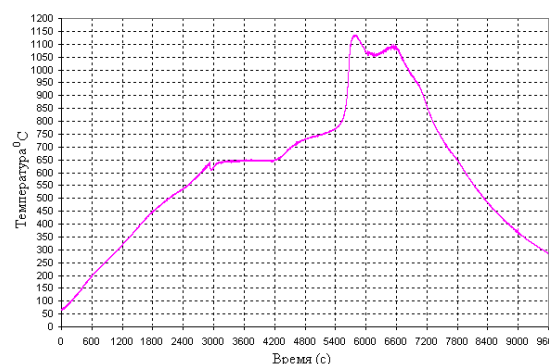


Fig. 7. Thermogram of synthesis process in the mode of thermal explosion at switching off heating source at the moment of achieving temperature peak by reacting batch. Temperature of reactor wall is 700 °C, titanium particle size is 242 ± 42 mkm

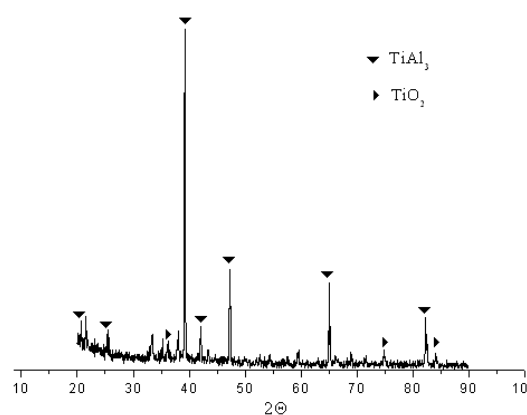


Fig. 8. Diffraction pattern of synthesis end product obtained in the mode of source switching off. Reactor wall temperature is 700 °C. Titanium particle size is 242 ± 42 mkm

However, large size of particles give an opportunity to implement the process of secondary structure formation and in minimum point of thermogram the rates of the processes of heat release and heat emission are compared. Then heat release rate begins to dominate that stimulates formation of the main phase according to stoichiometric composition. The latter determines the

presence of the second maximum at thermogram which gives an opportunity of system supplementary reaction. Then the product cools as an inert body.

Comparatively rapid batch cooling at particle size 130 ± 23 mkm gives no opportunity to implement the process of secondary structure formation; plateau appears instead of the second peak, the system has no time to transfer to equilibrium state according to the original stoichiometry.

Conclusion

It follows from the carried out series of experiments that phase composition of the product of SH-synthesis carried out in the mode of thermal explosion in the system Ti+3Al depends on particle size and monophase

product of stoichiometry $TiAl_3$ is obtained both on small (in the experiment conditions 55 ± 7 mkm) and large (180 ± 32 mkm) size of titanium particles at induction period termination. At intermediate fraction (130 ± 23 mkm) at induction period termination the product of synthesis is monophase. Similar anomalous effect is explained by the fact that at small particle size the all-burn time is compared with induction time and heat release has no time to influence considerably on phase formation process. Synthesis is finished at the stage of primary structure formation. At large particle size the long diffusion time develops conditions for intensive occurrence of the process of secondary structure formation that is displayed in appearance of the second peak at thermogram. Temperature growth connected with it stimulates formation of the phase corresponding to original stoichiometry.

REFERENCES

1. Antsiferov V.N., Smetkin A.A. Mechanically doped intermetallic alloys of titanium aluminides // *Perspektivnye Materialy*. – 2003. – № 6. – P. 12–15.
2. Merzhanov A.G. Combustion process of condensed system. New direction in research // *Bulletin of USSR AS*. – 1979. – № 8. – P. 10–18.

3. Evstigneev V.V., Volpe B.M., Milyukova I.V., Saygutin G.V. Integrated technologies of self-propagating high-temperature synthesis. – Moscow: Vysshaya Shkola, 1996. – 274 p.
4. Titanium alloys. Metallography of titanium alloys / E.A. Borisova, G.A. Bochvar, M.Ya. Brun et al. Ed. by S.G. Glazunov and B.A. Kolachev (editor-in-chief). – Moscow: Metallurgiya, 1980. – 464 p.

Received on 14.11.2006

UDC 538.9:621.785.6

CHANGE OF THE SPRING Cr-Ni ALLOY MICROSTRUCTURE AFTER AGEING

M.K. Skakov, E.B. Mukazhanov, D. Erbolatuly, Zh.M. Isaeva

S. Amanzholov's Eastern-Kazakhstan state university, Ust-Kamenogorsk, Kazakhstan

It has been stated that ageing of the tempered alloy 47XHM at temperature 500 °C during 5...10 h does not result in disintegration of oversaturated firm solution, at ageing temperature rise up to 600 °C attributes of disintegration in particles of α -phase of homogeneous type start to be shown. It was shown that after tempered samples ageing at 700 °C the faltering disintegration with allocation of not coherent α -phase on the basis of chrome develops intensively, and its volume fraction increases with increase in ageing time reaching the maximal values in 5...10 h of ageing.

As it is known [1] owing to high elastic properties the precipitation hardening alloys on Ni-Cr basis are applied at manufacturing elastosensitive elements of various devices and machines. Moreover, alloys Ni-47Cr-Mo are perspective materials for elements of construction of nuclear and thermonuclear reactors [2]. In work [3] it is shown that radiation resistance of alloy 47XHM may be considerably improved changing the initial structural-phase state.

Investigations [4] of hardened alloy 47XHM show that sample phase-structural state is influenced by hardening temperature, cure time for hardening and cooling rate. In work [5] peculiarities of change of mechanical properties of alloy 47XHM depending on hardening modes are detected and hardening optimal conditions are determined.

The aim of the given paper is to study the change of microstructure of spring alloy 47XHM at aging in temperature range 500...700 °C.

Material and investigation technique

Precipitation hardening alloy 47XHM of standard chemical composition (47 % – Cr, 5 % – Mo, res. – Ni) is selected as research material in this paper.

Carrying out heat treatment of alloy 47XHM, heating for hardening from 1250 °C during 1...2 min and aging in temperature range 500...700 °C were carried out in laboratory electric-tube furnace of resistance of the type SUOL-0,4.4/12-M2-U4.2 in vacuum with residual pressure not more than 1 Pa. Sample state was recorded by hardening in cold water.