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O.A. Shkoda*

Tomsk Scientific Center, Siberian Branch, Russian Academy of Sciences, Tomsk, Russia
*(*E-mail: O.Shkoda@dsm.tsc.ru)*

Clarification of the state diagram of "Metal 1-Metal 2" systems by analyzing the "cloud" of data of combustion parameters of "Metal 1-Metal 2-Nitrogen" SHS systems

The combustion of the "Metal 1 — Metal 2 — Nitrogen" systems in the layer-by-layer mode of self-propagating high-temperature synthesis was studied. A large amount of experimental data was obtained by changing the initial synthesis parameters. As a result, a "cloud" of data was obtained, and not a single curve. Since self-consistency of many factors that ensure the propagation of the combustion wave occurs in this system, the structure of the "cloud" of data obeys strict laws characteristic of dissipative structures of nonequilibrium nonlinear systems. Variations in the initial data made it possible to obtain changes in the combustion parameters in a wide range. The combustion parameters reflect the properties of the solid-liquid medium that is formed in the chemical reaction zone in the combustion wave. The combustion temperature is between the "liquidus" and "solidus" temperatures of the corresponding "Metal 1-Metal 2" binary phase diagrams, where titanium, molybdenum, tungsten, and aluminum were used as the Metal. The resulting data "clouds" were compared with the corresponding phase diagrams of state.

It has been established that the shape and outline of the "cloud" of date for combustion parameters coincide with the shape and outline of the region bounded by the liquidus and solidus lines of the binary phase diagrams of the state "Metal 1 — Metal 2". Clarification in the phase diagrams of state has been carried out. The other arrangement of the liquidus and solidus lines on the melting diagrams of aluminum-tungsten and aluminum-titanium is proposed.

Keywords: self-propagating high-temperature synthesis, degree of conversion, phase diagram, nitrides, data "cloud", nonequilibrium system.

Introduction

The development of modern technology is associated with the development and production of materials that are able to operate in various extreme conditions. One of such materials are compounds of metals with nitrogen. Of important interest are metal nitrides, which have many useful properties, such as dielectric, semiconductor, chemical stability in various aggressive environments and other specific properties.

These properties of materials allow their use for products in microelectronics, laser technology, to work in contact with metal melts at high temperatures. Ternary systems "Metal 1-Metal 2-Nitrogen" exhibit unique properties characteristic of metals and ceramics [1–4]. Some compounds based on ternary systems belong to MAX-phases with a special layered nanolaminate structure, which allows them to exhibit both metallic and ceramic properties [5–7].

Self-propagating high-temperature synthesis (SHS) is a method for the production of metal nitrides, which makes it possible to obtain materials with the necessary different properties [2, 3]. However, the mechanism of formation of these compounds in the combustion wave is not fully understood, as well as the state diagrams of the systems used, which makes this work relevant.

The peculiarity of filtration synthesis is in the strong dependence of the composition of products and combustion parameters on pressure, composition of the gas medium, on the diameter of the briquette, its permeability. The way of gas reagent entering the combustion zone is important: spontaneous filtration (in which the combustion zone plays the role of a pump that sucks gas). The afterburning process also plays an important role in filtration synthesis. Due to the high temperatures that develop in the combustion zone, it can happen that the combustion temperature exceeds the melting point of the fusion products. The intermediates melt and the permeability of the sample is reduced. The gaseous reactant saturates only the layers of the sample on the surface by reaction diffusion, while the inner part of the sample often remains underreacted [8–10]. The nature of the temperature interval between the solidus temperature and the liquidus temperature (Liquidus-Solidus "L-S") affects the mode of combustion, the completeness of conversion, the maximum

temperature and the rate of combustion in this case. LS-melt means a two-phase suspension that occurs in the Liquidus-Solidus temperature interval (“L-S”).

It was noted in [10] that it is possible to construct a hypothetical diagram of state of the “Ti-Me-B” ternary system through the analysis of combustion parameters of the “Ti-Me-B” ternary system. Both in [10] and in this paper the idea that combustion parameters reflect the properties of the solid-liquid medium formed in the chemical reaction zone of the combustion wave, with the combustion temperature lying between the temperatures “L” and “S” of the corresponding double phase diagrams “Metal 1 — Metal 2”. By changing the initial synthesis parameters (composition, density, dispersity of powders, etc.), the properties of the medium (viscosity, the size of solid particles in suspension, the rate of nitrogen supply, etc.) also change.

For filtration combustion of three-component systems, such variation of initial parameters results in a “cloud” of data rather than a separate curve of dependence. The self-consistency of many factors in the system that provide propagation of the combustion wave occurs, and the structure of the data “cloud” obeys strict laws that are characteristic for dissipative structures of nonequilibrium linear systems. Different properties of the environment cause different types of formation of intermediate, unstable nitrides. Nanoscale suspension particles are the centers of chemical reactions of nitriding in a solid-liquid medium in which nitrogen is continuously supplied [9–14].

In [7, 13], devoted to the filtration combustion of a mixture of two metals in nitrogen, it was noted that all combustion parameters at appropriate scales are located within the “L-S” region of the melting diagram “Metal 1 — Metal 2”. The task arose to check to what extent the envelopes of the “cloud” lines of these combustion parameters of the ternary system “Metal 1 — Metal — Nitrogen” repeat the course of the liquidus and solidus lines of the double system “Metal 1 — Metal 2”. The systems “Ti-W” and “Ti-Mo”, having accurately constructed phase diagrams in coordinates “temperature-composition”, were chosen for verification. “Ti-Al” and “W-Al” were chosen as the diagrams that need to clarify the positions of the liquidus and solidus lines.

Experimental

Metal powders were used as initial components for synthesis: titanium of PTM grade; aluminum of ASD-4 grade of technical purity; finely dispersed molybdenum and tungsten (less than 10 μm). Gaseous nitrogen of high purity with oxygen content less than 0,001 % was used. The mixture of powders in the amount of 16 g was poured into cylindrical cups with a diameter of 20 mm and a height of more than 30 mm. The cups were made of well-permeable filter paper. Combustion was initiated by a tungsten spiral through an igniting tablet. The composition of the tablet was selected so that a combustion wave with a maximum temperature between the “L” and “S” temperature of the systems was formed. The cups were placed on a special stand, which was transferred to the reactor, which was a constant pressure unit with a volume of 3 liters. A constant nitrogen pressure of 20 atm was maintained in the reactor.

The amount of nitrogen assimilated during combustion was determined by weighing the sample before and after combustion with an accuracy of ±0.005 g. The maximum combustion temperature was measured by the thermocouple method using tungsten-rhenium thermocouples. The temperature data were recorded using a computer in the form of thermograms. Diffractograms were taken from the final synthesized combustion products on a “Drone-2” apparatus.

The completeness of conversion was determined as follows. For the case where the leading component is titanium, the completeness of transformation was calculated as the ratio of the percentage-weight content of nitrogen in titanium mononitrides of the final product to the percentage-weight content of nitrogen in stoichiometric titanium mononitride $TiN_{1,00}$:

$$\eta'=\eta_{Ti}=[N_c\cdot 100\ \%/(N_c+Ti_c)]/22{,}63\ \%.$$

Similarly, for the case where the leading component in the combustion wave is aluminum:

$$\eta''=\eta_{Al}=[N_c\cdot 100\ \%/(N_c+Al_c)]/34{,}17\ \%.$$

Here N_c is the amount of assimilated nitrogen (in grams) during SHS synthesis (calculated by weighing the sample before and after combustion with an accuracy of ±0.005 g), Ti_c and Al_c are the amounts of titanium and aluminum in the initial mixture (in grams, respectively).

The difference between η_{Ti} and η_{Al} is determined by the different stoichiometric nitrogen content: in titanium mononitride — 22.63 % wt. % and in aluminum mononitride — 34.17 % wt. %, and by the different contents of titanium and aluminum in the initial mixture. If both aluminum and titanium are nitrided to form mononitrides simultaneously, the completeness of the transformation is calculated by the following formula:

$$\eta''' = [\alpha N_c\cdot 100\ \%/(\alpha N_c+Ti_c)]/22{,}63\ \% + [\beta N_c\cdot 100\ \%/(\beta N_c+Al_c)]/34{,}17\ \%\ \text{or}$$

$$\eta''' = [\alpha N_c \cdot 4{,}419/(\alpha N_c + Ti_c)] + [\beta N_c \cdot 2{,}926/(\beta N_c + Al_c)] \text{ or}$$

$$\eta'''_{TiN+AlN} = \alpha \cdot \eta' + \beta \cdot \eta''.$$

Here α and β are the fraction of titanium and aluminum in the mixture, respectively. In arriving at this formula, we were guided by the fact that titanium and aluminum have almost equal enthalpies and nitrogen is distributed between them according to the initial composition.

Results and Discussion

The limiting factor in filtration combustion is the problem of gas delivery to the nitriding reaction site. The porosity structure of the medium is of great importance for the kinetics. If a large amount of liquid appears in the reaction zone, combustion stops due to the impossibility of gas penetration to the reaction site. In such cases, inert additives are used to lower the combustion temperature.

The composition of the synthesized product depends on the initial ratio of components. Up to 40 weight % Ti the product consists of aluminum mononitride, Al3Ti phase and pure aluminum. The diffractograms record clear, well-formed AlN peaks. In the range of 40 to 60 weight % Ti, the following are found: aluminum mononitride, titanium mononitride, TiAl phase and pure aluminum. At the ratio from 60 weight%Ti and above in the synthesized product are found: titanium mononitride, Ti3Al phase, αTi, and also, here are found small peaks of MAH phases Ti2AlN, Ti4AlN3. The diffractogram shows high broadened peaks, from which peaks of titanium mononitride, Ti3Al, αTi intermetallide with aluminum dissolved in it, emerge. Any traces of aluminum mononitride are absent [7].

An equilibrium suspension consisting of particles of solid solutions or compounds is formed when two different metals are heated above the solidus temperature. A vortex motion that entrains nitrogen gas and delivers it to the reaction site is easily formed in such a heterogeneous medium at high temperatures. In this case, the melt in any quantity is not an obstacle to the penetration of gas to the reaction centers, which are solid particles in the "L-S"-melt.

Figure 1, a shows two combined diagrams: a part of the phase diagram "Mo-Ti" and a "cloud" of values of the degree of conversion η. It can be seen that the enveloping "cloud" completely repeats the solidus and liquidus lines. Inside the cloud, the η data are located along the lines that repeat the course of the liquidus curve. Figure 1, b shows the superposition of the state diagram for the "W-Ti" systems and the "cloud" of η data taken from the combustion analysis of the "W-Ti-N" system. The lower envelope coincided exactly with the course of the solidus line. The upper envelope partially coincided with the liquidus line, the rest of the data repeat its course. Complete filling of the "L-S" space did not occur, apparently, solid suspension particles of the required size were not formed at high combustion temperatures [13–16].

"Cloud" data of maximum combustion temperatures (Tmax) are plotted on the fusibility diagram of the "Mo-Ti" system (Fig. 2, a). The presence of two "clouds" of Tmax data can be seen. The lower cloud completely coincides with the "L-S" space. The second cloud, which is located above the "L" line, is the result of "flare afterburning", when the gaseous part of the product, consisting of suboxides, burns in the form of flares, outside the combustion wave [9]. The data inside the second "cloud" also lie on curves repeating the course of the liquidus line. Figure 2, b shows a "cloud" of combustion rate (Uc) data. If the combustion rate (Uc) data are plotted in a mirror-hyperbolic reflection, this "cloud" of data coincides completely with the "L-S" region, as does the transformation completeness (η) data. This suggests that the relationship between the combustion rate (Uc) and the degree of transformation (η) is proportional to ~1/1-η, in contrast to the cases of single metal combustion in nitrogen, for which this relationship is defined as ~1/η [12].

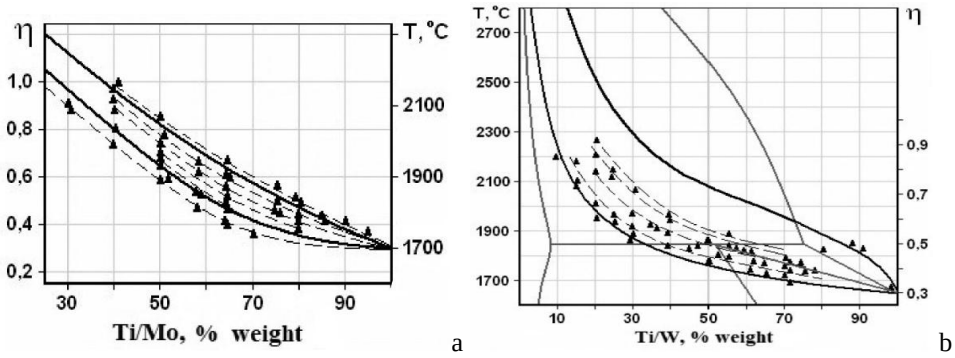
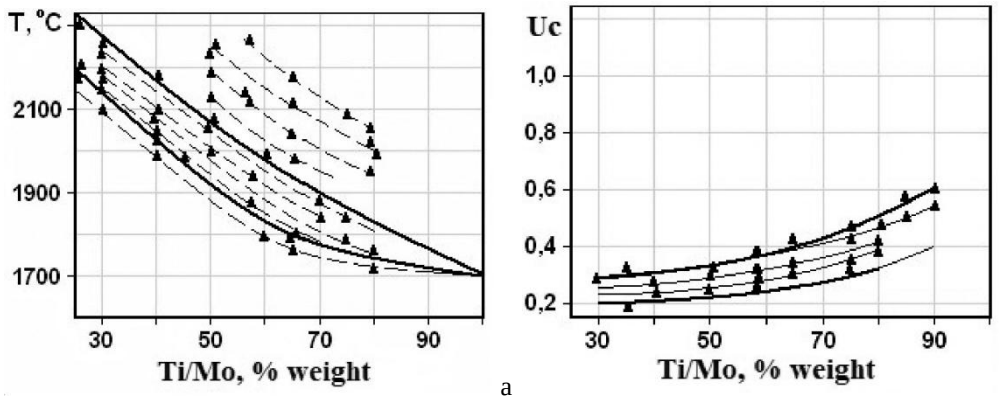


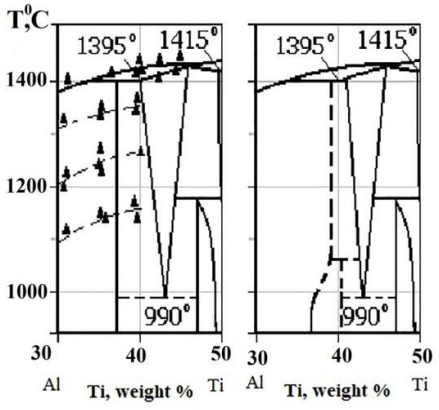
Fig. 1. a — Combined melting diagram of "Ti-Mo" with "cloud" data of the degree of conversion η,
b — Combined melting diagram of "Ti-W" with "cloud" data of the degree of conversion η.



a b

Fig. 2. a — Combined melting diagram of "Ti-Mo" with "clouds" of maximum temperature (Tmax) data, b — "Cloud" of combustion velocity (Uc) data for the "Ti-Mo-N" system.

All analyses of the combined graphs allowed us to assume a more correct position of the liquidus line, peritectic horizontals and solidus lines for the phase diagrams of state of the systems "Al-Ti" and "Al-W". Experimental data of completeness of transformation (η) for these systems in certain concentration limits were located in the regions of known diagrams, where there is no solid-liquid melting zone, which according to the assumed mechanism of phase formation in the combustion wave cannot be. Therefore, we propose another variant of the melting diagram section, which is shown in Figure 3 (a, b) and Figure 4.



a b

Fig. 3. "Al-Ti" combined melting diagram with "cloud" of degree of transformation data (η): a) known; b) proposed.

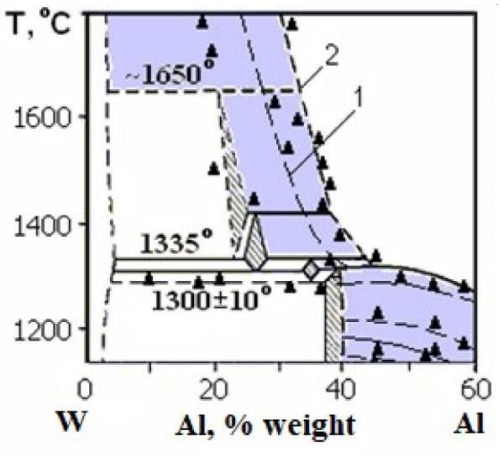


Fig. 4. "Al-W" fusibility diagram with "cloud" of conversion degree data (η): curve 1 — known, curve 2 — proposed.

The proposed changes result in a diagram with the presence of a high-temperature phase β-Al_3Ti, in addition to α-Al_3Ti, for the "Ti-Al" system. The modified melting diagram of "W-Al" differs from the previous one by the shift of the liquidus line towards increasing Al concentration starting from the temperature ~1310 ºC.

Conclusions

A different arrangement of liquidus and solidus lines in the melting diagrams of "W-Al" and "Ti-Al" is proposed. It is shown that the shape and outline of "clouds" of these combustion parameters of SHS system "Metal 1 — Metal — Nitrogen" repeat the area of the zone between "L" and "S" of double diagrams "Metal 1 — Metal 2".

It is revealed that the relationship between UG and η for "Metal 1 — Metal — Nitrogen" SHS systems is determined by the formula: Uc~1/1-η (or η~1/1/1-Uc).

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О.А. Шкода

«Металл 1–металл 2–азот» СВС жүйелерінің жану параметрлері туралы деректердің «бұлтын» талдау арқылы «металл 1-металл 2» жүйелерінің балқыту диаграммасын нақтылау мүмкіндігі туралы

«Металл 1–металл 2–азот» жүйелеріне өздігінен таралатын жоғары температуралық синтездің қабат-қабат режимінде жануы бойынша зерттеу жүргізілді. Бастапқы синтез параметрлерін өзгерту арқылы тәжірибелік мәліметтердің үлкен көлемі алынды. Құрамы, тығыздығы, ұнтақтардың дисперсиясы және басқалары сияқты синтездің бастапқы параметрлерін өзгерту арқылы ортаның қасиеттері де өзгереді, мысалы, тұтқырлық, суспензиядағы қатты бөлшектердің мөлшері, азотпен қамтамасыз ету жылдамдығы және т.б. Үш компонентті жүйелерді фильтрациялық жану үшін аталған бастапқы параметрлерді өзгерту нәтижесінде жеке қисық емес, деректердің «бұлты» алынды. Бұл жүйеде жану толқынының таралуын қамтамасыз ететін көптеген факторлардың өзіндік сәйкестігі болғандықтан, деректер «бұлтының» құрылымы тепе-теңдіксіз сызықты емес жүйелердің диссипативті құрылымдарына тән қатаң заңдарға бағынады. Бастапқы деректердің өзгеруі жану параметрлерінің кең ауқымдағы өзгерістерін алуға мүмкіндік берді. Жану параметрлері жану толқынындағы химиялық реакциялар аймағында түзілетін қатты-сұйық ортаның қасиеттерін көрсетеді. Бұл жағдайда жану температурасы титан, молибден, вольфрам және алюминий металдар ретінде пайдаланылған «металл 1-металл 2» сәйкес қос фазалы диаграммаларының «сұйықтық» және «қатты» температуралары арасында болады. Алынған деректер «бұлттары» сәйкес фазалық диаграммалармен салыстырылды. Бұл жану параметрлерінің «бұлтының» пішіні мен контуры «металл 1-металл 2» күйінің қос фазалық диаграммаларының ликвидус және солидус сызықтарымен шектелген аймақтың пішіні мен контурымен сәйкес келетіні анықталды. Фазалық диаграммалар нақтыланды. Алюминий-вольфрам және алюминий-титан балқитын диаграммаларындағы ликвидус пен солидус сызықтарының басқаша орналасуы ұсынылды. Өздігінен таралатын жоғары температуралық синтездің қабат-қабат режиміндегі «металл 1-металл 2-азот» жүйелері үшін жану жылдамдығы мен конверсия дәрежесі арасындағы байланыс формуласы анықталды.

Кілт сөздер: өздігінен таралатын жоғары температуралық синтез, түрлену дәрежесі, фазалық диаграмма, нитридтер, деректер бұлты, тепе-теңдік емес жүйе.

О.А. Шкода

О возможности уточнения диаграммы плавкости систем «металл 1–металл 2» через анализ «облака» данных параметров горения СВС систем «металл 1–метал 2–азот»

Проведено исследование горения систем «металл 1–металл 2–азот» в послойном режиме самораспространяющегося высокотемпературного синтеза. Меняя начальные параметры синтеза, получено большое количество экспериментальных данных. Варьируя начальные параметры синтеза, такие как состав, плотность, дисперсность порошков и другие, меняются и свойства среды, такие как вязкость, размер твердых частиц в суспензии, скорость поступления азота и т.д. Для фильтрационного горения трёхкомпонентных систем в результате варьирования перечисленных исходных параметров получено «облако» данных, а не отдельно взятая кривая. Поскольку в этой системе происходит самосогласование многих факторов, обеспечивающих распространение волны горения, то строение «облака» данных подчиняется строгим законам, характерным для диссипативних структур неравновесных нелинейных систем. Вариации исходных данных позволили получить изменения в параметрах горения в большом диапазоне. Параметры горения отражают свойства твердожидкой среды, которая образуется в зоне химических реакций в волне горения. Температура горения при этом находится между температурами «ликвидус» и «солидус» соответствующих двойных фазовых диаграмм «металл 1–металл 2», где в качестве металла использовались титан, молибден, вольфрам, алюминий. Полученные «облака» данных были сопоставлены с соответствующими фазовыми диаграммами состояния. Установлено, что форма и очертание «облака» данных параметров горения совпадают с формой и очертанием области, ограниченной линиями ликвидус и солидус двойных фазовых диаграмм состояния «металл 1–металл 2». Проведено уточнение в фазовых диаграммах состояния. Предложено иное расположение линий ликвидус и солидус в диаграммах плавкости «алюминий–вольфрам» и «алюминий–титан». Определена формула соотношения между скоростью горения и степенью превращения для систем «металл 1–металл 2–азот» при послойном режиме самораспространяющегося высокотемпературного синтеза.

Ключевые слова: самораспространяющийся высокотемпературный синтез, степень превращения, диаграмма состояния, нитриды, «облако» данных, неравновесная система.

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Information about the authors

Shkoda Olga Aleksandrovna — Candidate of technical sciences, senior research officer, Tomsk Scientific Center of the Siberian Branch of the Russian academy of Sciences, Tomsk, Russia; e-mail: *O.Shkoda@dsm.tsc.ru*. ORCID ID: https://orcid.org/0000-0002-6068-4817