

INVESTIGATION OF SHS PROCESSES USING LASER HARDENING

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Using the titanium-boron system as an example, a procedure for ultrafast hardening (10^6 - 10^7 K/sec) of the SHS reaction using pulse laser radiation is developed. The dynamic scheme of the component interaction is obtained on the basis of metallographical and micro X-ray spectrum analysis. It is shown that under conditions of high-speed heating mass transfer on the interfaces has a pronounced convective character, ensuring an increased interaction rate.

To study the processes of SH synthesis in detail, it is essential to trace the entire complex of physicochemical transformations of components under conditions of temperature fields (10^3 - 10^4 K), scales (10^{-3} - 10^{-6} m), and time intervals (10^{-1} - 10^{-5} sec) of the real combustion wave. Pulse laser heating of the surface, which is used by us for revealing the character of the processes of chemical interaction of a powder system, is among the most effective methods for modeling these conditions.

Procedure. In the present work we performed investigations using the titanium-boron system as an example. Cylindrical specimens were prepared from a mixture of titanium and crystalline boron (titanium and boron particle size is $130 \mu\text{m}$) pressed to a relative density of 0.8 or from compact titanium with particles of crystalline boron pressed into the end. The specimen end was heated by a pulse of laser radiation with a wavelength of $1.06 \mu\text{m}$ for $t_i \sim 10^{-3}$ sec. The average density of the laser radiation flux was controlled in the interval $P = (0.3-3.0) \times 10^9$ W/m². The value of P was limited in the experiments by the appearance of noticeable fusion of the specimen end. To prevent oxidation, the specimen was blown with argon. After laser processing the specimen end was ground and polished, the thickness of the removed layer of material being 50-70 μm . The specimen was investigated using optical and electron microscopes and a CAMEBAX microanalyzer.

We perform an upper estimate of the heated layer thickness h_T and of the specimen surface temperature T_S at the moment the radiation pulse terminates, making use of the relations [1]

$$T_* = T_S \sqrt{\pi} \left[\text{erfc} \left(\frac{h_T}{2 \sqrt{at_i}} \right) \right], \quad T_S = \frac{2P}{\lambda} \sqrt{\left(\frac{at_i}{\pi} \right)},$$

where λ , a are the thermal conductivity and thermal diffusivity of the material; T_* is the minimum temperature of the heat treatment.

For $T_* = 1000$ K and the thermophysical parameters of titanium and boron the calculated values are in the ranges of $h_T = 10$ - $150 \mu\text{m}$, $T_S = 10^3$ - 10^4 . It is pertinent to note that the estimate does not take into account radiation reflection and phase transitions of the material, and hence the real values of h_T and T_S may be somewhat smaller.

The total time of the heat treatment, consisting of the sum of the pulse and heated-layer thermal relaxation times (of the order h_T^2/a), does not exceed 10^{-2} sec.

Results of the Investigations. As the investigations have shown, after laser heat treatment a pronounced chemical interaction occurs on the titanium and boron interfaces. The completeness of the interaction decreases with decreasing radiation intensity and with distance from the surface into the depth of the specimen, which is

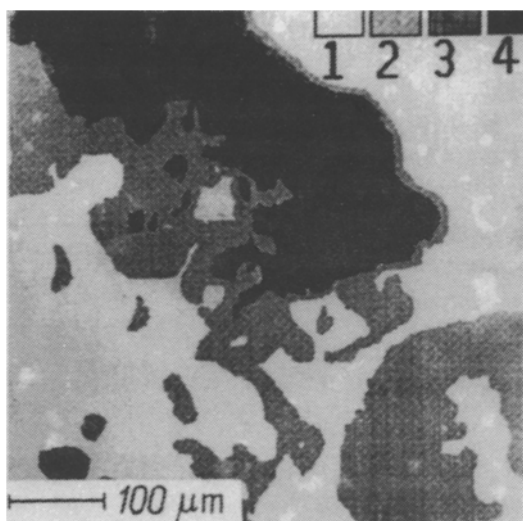


Fig. 1. Metallographic photographs of the process of interaction of titanium and boron in the 1st (a) and the 2nd (b) steps of the interaction: 1) initial titanium; 2) titanium melt; 3) initial boron; 4) boron melt; 5) zone of contact melting; 6) DChT zones. $\times 1000$ (a); $\times 600$ (b).

connected with a decrease in the heating temperature of the material. At a depth of more than $200\ \mu\text{m}$ there are no traces of chemical transformations.

Analyzing the interaction zones in different planes of the sections and with different laser radiation intensities makes it possible to formulate the following step scheme of the interaction under conditions of high-speed heating.

The 1st step. Mutual dissolution of the components and contact peripheral melting of boron particles. This is recorded if there is a halo contour around the boron particles (Fig. 1) with its characteristic structure and color. The amount of titanium in the halo contour is determined at a level of 81-96%, which includes the coordinate of the eutectic Ti-TiB (87 at.% Ti). The melt formed in contact melting fills microcracks and intercrystallite boundaries of the boron particles, which is evident from decoration and titanium saturation of the internal boundaries to concentrations of 96 at.%.

The 2nd step. Formation of local regions with deep chemical transformation (DChT), which expand in the form of "jets" from a boron particle, on the initial boundaries of titanium and boron (Fig. 2). The titanium content in the DChT regions is in the range 20-80 at.%, which includes the coordinates of the compositions of all basic compounds of the system TiB, Ti_3B_4 , TiB_2 , and Ti_2B_5 . Simultaneously from the internal and external boundaries of the initial particle, melting of the boron occurs, as a result of which crystalline fragments form together with the melt. The presence of up to 1 at.% Ti in the boron melt suggests a eutectic character of melting, which is apparently due to contact interaction of the crystalline boron and the titanium melt. Melting of the internal portions of the boron particle is ensured by titanium that previously penetrated into the internal boundaries in the 1st step.

The formation of the DChT zones is of a clearly nondiffusion character, which can be seen from the absence of symmetry of their growth with respect to the initial boron particle, the absence of lamination, and the monotonic change in the component concentration as we move from boron to titanium. Within the DChT zones there are fairly extended (up to $150\ \mu\text{m}$) portions of practically pure titanium (82-96 at.%), fragments of pure boron ($5-50\ \mu\text{m}$), and local portions enriched with one or another component (Fig. 2). In addition, diffusion transfer is unable to mix the components on the scales of the DChT regions, which attain $400\ \mu\text{m}$, since otherwise the presence of unreal diffusion coefficients of $10^{-4}\ \text{m}^2/\text{sec}$ is necessary.

The most probable mechanism of formation of the DChT regions depends on microconvective flows developing in the zone of contact peripheral melting of boron particles. The convective flows ensure mixing processes that are several orders faster than diffusion, can carry boron fragments away from the initial particle surface, and create nonuniform portions in the DChT regions. The spatial expansion of the DChT regions is

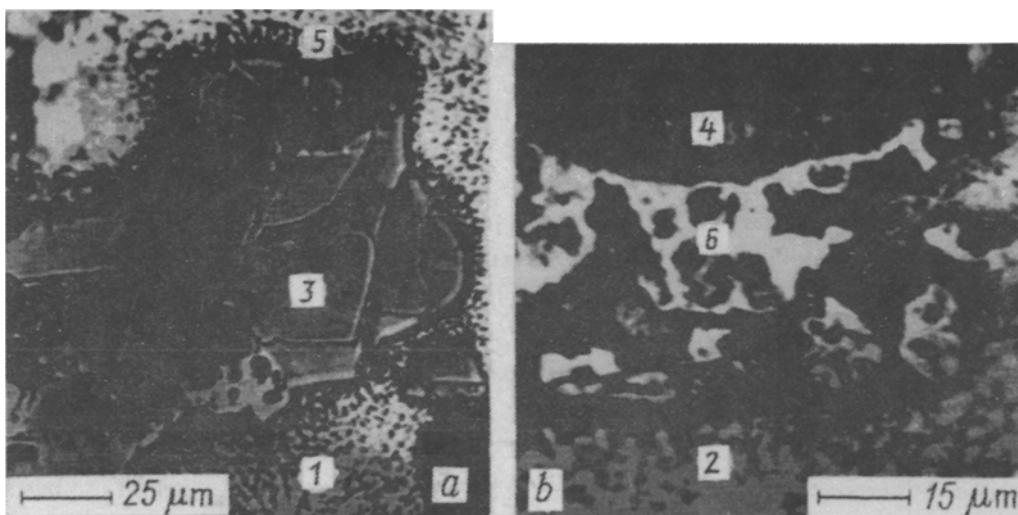


Fig. 2. Map of the distribution of the component concentration in the 2nd step of the interaction. Titanium, at. %: 80-100 (1), 60-80 (2), 20-60 (3), 0-20 (4); boron: the rest; 2, 3) DChT zones. $\times 150$.

accompanied by their mixing with the boron melt also by the mechanism of convection, which is seen from the presence in the boron melt of titanium-enriched (0.5-2.5 at. %) zones 10-20 μm in size adjacent to the DChT regions.

The presented step nature of the interactions averages observations over the set of reaction zones of a large number of particles. In individual cases the sequence of transformations may deviate from the above scheme, but on the whole all types are present in each particle.

Moving to the analysis of the nature of convection observed in titanium-boron interaction, it is pertinent to note that microconvection (the minimum flow scale is 10^{-7} m, the rate is up to 10^{-1} m/sec) was observed earlier on the boundaries of water solutions and organic materials [2] and in surface layers of metals in laser fusion [1]. The driving force of microconvection is the surface tension gradient on the interface, and the tendency of the system to minimize the surface energy creates conditions for development of liquid flows.

In pulse laser heating of the Ti-B system (just as in the combustion wave) the temperature drop $\Delta T \sim 10^3$ K of the component concentration $\Delta C \sim 10$ at. % on the scale $\Delta l \sim (10^4 - 10^5)$ is attained. For high-melting borides and titanium- and boron-based melts the surface tension gradient on the melt-gas and melt-boride boundaries under these conditions attains the value

$$\text{grad } \sigma \sim \frac{\Delta \sigma}{\Delta l} \sim 10^4 \div 10^5 \text{ N/m}^2,$$

which reflects the value of the driving force for melt flows per unit interphase surface. We note that the value obtained should be considered as a lower estimate of the driving force, since no account is taken here of the sharp decrease in the interphase energy during the intensive chemical interaction. We also add that the value obtained exceeds the driving force of the microconvection observed in water solutions and organic compounds by one-two orders.

Thus, it may be assumed that the presence of melt microflows in fast high-temperature interaction of titanium and boron is connected with decidedly surface instability of interfaces of the components of the system.

REFERENCES

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