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SELF-PROPAGATING HIGH-TEMPERATURE SYNTHESIS OF COMPOSITE MATERIALS. THE HIGH-MELTING COMPOUNDS (TiC, TiB_2)-INTERMETALLIDES, THEIR STRUCTURE AND PROPERTIES

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Regularities of the SHS of four-component powder mixtures of quasibinary sections (titanium carbide or diboride - intermetallide Ni_3Al or NiTi) are studied. Combustion rates and temperatures are determined. Specific features of the combustion as a function of the composition are revealed. The character of structure formation and the properties of the synthesized materials, including those produced in SHS compaction, are considered.

The most important problem in developing ceramic-metal compositions using the SHS method is the production of materials with a specified level of service characteristics. Yet, only a detailed study in combination with information of generalizing character on the regularities of SH processes and on the structure and the properties permits optimization of their synthesis modes.

The present study, using the group of quasibinary systems composed of a high-melting compound (Ti-C, Ti-2B) and an intermetallide (3Ni-Al, Ni-Ti), considered specific features of the combustion of four-component powder mixtures for initial temperatures of T_0 = 20-300°C. A fairly high thermicity of the mixtures, among them those complying with stoichiometry of the intermetallides, allows synthesis of materials in a wide range of compositions and their simultaneous pressure treatment at minimal energy expenditure.

Combustion regularities were studied in a constant-pressure bomb in a helium atmosphere (0.2 MPa) with preliminary pumping to a vacuum of 13.3 Pa. The combustion rate was determined by an FR-14 photorecorder. The temperature was recorded by an VR-5/VR-20 thermocouple (with a 100-μm diameter of the thermoelectrodes) with the aid of an Shch-1513 digital voltmeter and an N-307 graph plotter.

Figures 1 and 2 give data regarding the combustion regularities in the systems considered. Clearly, when significantly diluted (90% (3Ni-Al) and 70% (Ni-Ti)), the compositions do not ignite at room temperature T_{in}. Their ignition is possible only on preheating. A nonstandard combustion mode is generally observed close to the concentrations indicated. In the systems of Ti-C and Ti-2B with 3Ni-Al, temperature oscillations observable in the heating zone correspond, in frequency, to oscillations of the combustion temperature and the propagation rate. A rise in the initial temperature of synthesis resulted in a steady mode of the process. Increasing the fraction of the composition corresponding to an intermetallide in the original mixture leads to a lowering of the combustion temperature, which is higher the smaller the enthalpy of formation of the intermetallide (Ni_3Al, Ni-Ti). This is also the case with the combustion rate, except the (Ti-2B)-(Ni-Ti) system, in which u_c rises as the Ni-Ti content increases up to 15%. To elucidate the causes of the anomalous increase in the combustion rate, additional investigations were carried out. Attention was given to the fact that the Ti-2B composition and mixtures based on it are characterized by an appreciable release of admixed gases. This is indicated by visually observable gas flows issuing from the reaction zone of burning samples as well as by characteristic disruptions of their surface and a significant lengthening. In this connection, it was presumed that the combustion rates, measured by the usual procedure, may be undervalued, which is connected with the loosening power of the adsorbed gases, i.e., in the combustion wave, the actual porosity is in excess of the specified average over the volume of a raw sample. Here,

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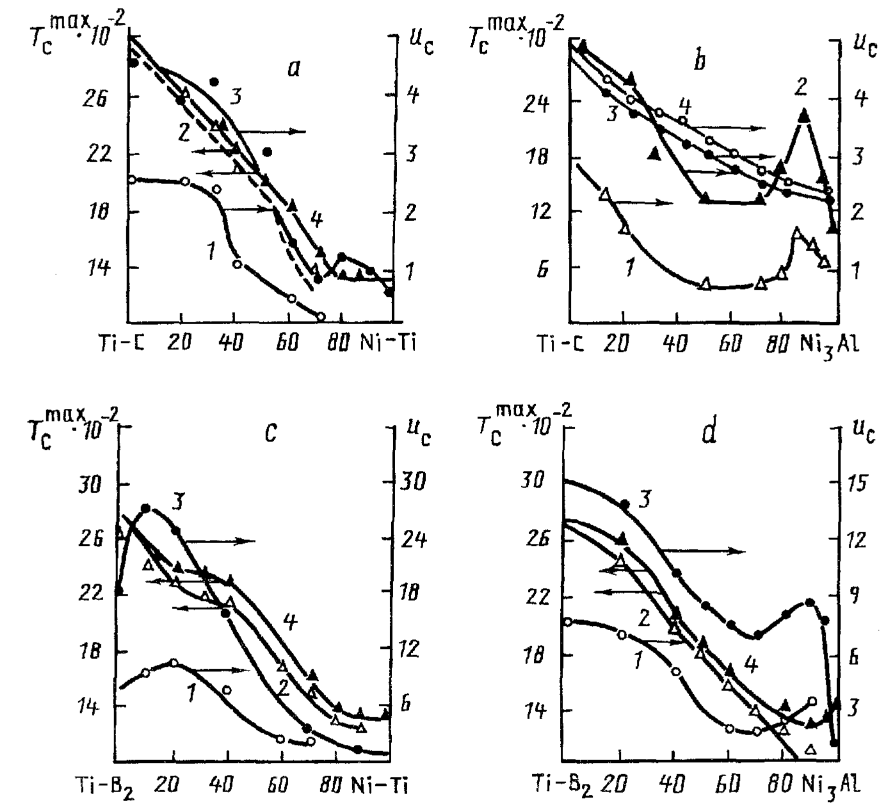
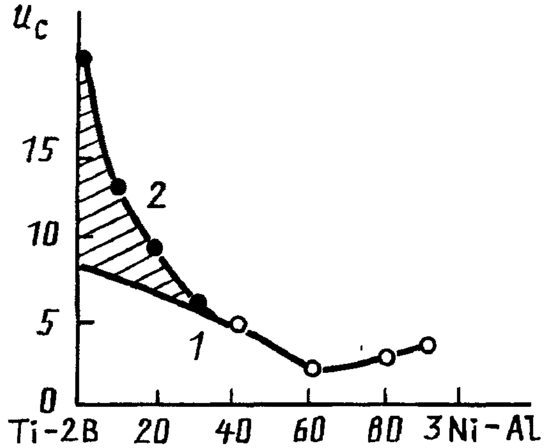


Fig. 1. Concentration dependence of the combustion rate and temperature: 1, 2) $T_0 = 20°C$; 3, 4) 300. T_c^{max}, °C; u_c, cm/sec; Ti-C, Ni-Ti, Ti-B_2, Ni_3-Al, at. %.

the combustion rate that is characteristic of true gasless combustion is not attained and should be higher. Obviously, the wave of gas admixtures, while accompanying the combustion wave, increases the initial porosity of the mixture in the heating and reaction zones due to the excess pressure of filtering gases. This entails a change in the initial thermophysical characteristics of the reacting mixture and, as a consequence, a change (a decrease) in the combustion rate. The fact of a decrease in the combustion rate with increasing porosity for the Ti-2B system is well known [1]. Gas releases in the SHS may be accompanied by significant deviations of the process from the true kinetics toward deceleration. In a first approximation, the degree of such a deviation can be assessed from the change in the initial porosity along the calibration curve, which is an ideal porosity dependence of the combustion rate, i.e., is plotted for true gasless combustion (Fig. 3). In a first approximation such a curve is the dependence of u_c on η plotted for preliminarily degassed samples and using a limitation of the growth of the sample height during the synthesis, when the initial and final porosities are fairly close to each other. A more careful check showed that a similar effect of the admixed gases is also revealed for other systems that we investigated. Thus, during "gasless" combustion in powder mixtures of (Ti-2B, Ti-C)-(3Ni-Al, Ni-Ti) systems, the combustion parameters are largely dependent on the amount of admixed gases and the experimental conditions.

Bends are also observed in the concentration dependences of the combustion rate and the maximum temperature of combustion near the composition corresponding to the intermetallic compound. Here, the combustion rates increase (see Figs. 1b and 2) or change slightly (Figs. 1a and 2). Particularly noticeable is the difference in the combustion rates when T_0 rises. The recorded behavior of u_c and T_c may result from the eutectic on the equilibrium phase diagram of the system of intermetallide-high-melting compound. The performed calculational and thermographic investigations showed that the eutectics lie in the region of compositions for which kinetic singularities of combustion are observable.

Analysis of the results obtained indicates not only a regular, on the whole, decrease in the maximum temperature and the rate of combustion as the fraction of the intermetallide component increases but also an



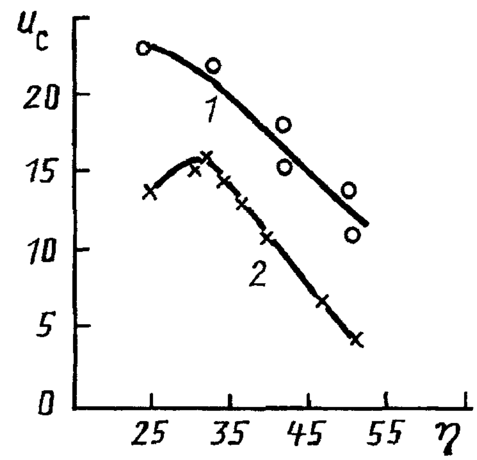
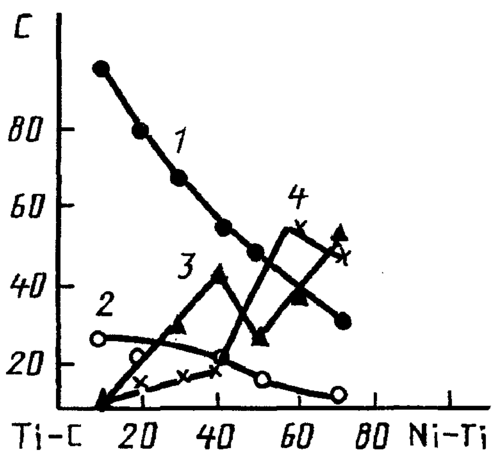


Fig. 2. Effect of thermovacuum treatment on the combustion rate: 1) without thermovacuum treatment; 2) with thermovacuum treatment. Ti-2B, 3Ni-Al, at. %.

Fig. 3. The effect of porosity on the combustion rate. Designations are the same as in Fig. 2.



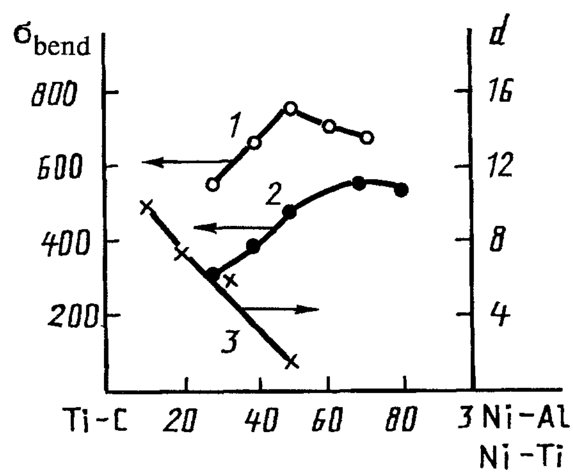


Fig. 4. Effect of the binder concentration on the phase composition: 1) TiC; 2) Ni_3Ti; 3) NiTi; 4) tetragonal lattice. c, wt. %.

Fig. 5. Effect of the composition on the strength of the composite material and on the grain size of TiC: 1) Ni-Ti; 2, 3) Ni-Al. σ, MPa; d, mm.

important role of the combined effect of the nature of the phase equilibrium, the presence of gas releases, the level of mechanical properties, and the temperature T_0 on the parameters of the SH process. Investigations of the synthesized materials demonstrated that, as the content of the intermetallide binder increases, the average particle size of the carbides and borides diminishes. The particles are mainly of rounded shape. (The characteristic change in the dimensions of the high-melting component is given in Fig. 5.)

The structure of the synthesized materials was studied using metallographic, x-ray phase, and local micro-x-ray spectrum analysis. It is found that the synthesis products along quasibinary sections are mainly two-phase and correspond to the specified stoichiometries. For all compositions up to 70 at.% of the intermetallide, there is steady formation of stoichiometric titanium carbide or diboride. In a number of systems the composition of the binder forming in SHS depends on the concentration of the intermetallide component. Specifically, in the TiC-NiTi system (Fig. 4) with small concentrations of the binder, it is composed mainly of the Ni_3Ti phase. With a subsequent increase in the concentration, the basic NiTi phase appears in the same amount. Furthermore, a third phase with a tetragonal lattice with the parameters a = 3.093 Å and c = 0.088 Å appears. The regions of concentrations close to the intermetallides are characterized by traces of incomplete reaction. In particular, in systems with carbon in the synthesis product we detected free carbon, nickel-based solid solutions, and traces of the NiAl phase.

Additional studies showed that it is possible to control the composition of the SHS product using the methods of directional alloying and heat treatment. In pressure treatment, no perceptible distinction is revealed between the mechanical properties of materials produced by the methods of extrusion and deformational compaction. The specific weight and the hardness of the composites considered change practically linearly with the fraction of the intermetallide component. Comparison of the physicomechanical properties of the compacted materials with the phase composition and the structure of the synthesis products permitted the conclusion that the maximum mechanical strength is attained in the cases where the titanium carbides and diborides have a fine-grain structure and the phases with the maximum level of mechanical properties dominate in the binder composition. This is especially clear for the TiC-NiTi system (Fig. 5).

The complex of the studies performed and the process designs indicate that a broad range of porous and compacted ceramic-metal materials (plates, tubes, rings, rods, etc.) with a prescribed level of physicomechanical properties and service characteristics can be obtained using the SHS method.

NOTATION

u_c, T_c, combustion rate and temperature; η, porosity; T_0, initial temperature; ρ, density; HRA, hardness; d, grain size; σ_{bend}, strength of the composite material; c, binder concentration.

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