SPECIAL CHARACTERISTICS OF THE FILTRATION
COMBUSTION OF THE SYSTEM Ti—C—N IN AN
AUDIO-FREQUENCY SOUND FIELD

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The influence of sound fields on the filtration combustion of mixtures of titanium and carbon in nitrogen on the kinetics of self-propagating high-temperature synthesis and on the chemical and phase composition of the synthesis products is investigated. It is shown that filtration hindrances in a wide layer of the sample vanish if the charge material has an initial relative density of 0.2. The carbonitride component is formed most completely when the atomic fraction of carbon in the initial charge has values of 0.3 and 0.5. The optimum acoustic frequency stimulating the filtration of nitrogen deep within the briquette and yielding the most complete nitriding process is the resonance frequency of the acoustic system. Fast-framing photography exhibits the transient nature of the filtration combustion of titanium—carbon mixtures in nitrogen.

The application of combustion processes to the synthesis of high-melting inorganic compounds makes it possible, in the self-propagating high-temperature (SPHT) regime, to obtain not only simple carbides, nitrides, and borides, but also complex multicomponent products consisting of several metals and nonmetals.

The need to impart a high pressure (above 15 MPa) to the gaseous reagent [1] is one of the characteristic features of heterogeneous systems reacting by the filtration combustion mechanism. At a lower gas pressure combustion products with a high nitrogen content can be obtained by artificially removing filtration hindrances, for example, by means of acoustic vibrations of various intensities with a wide frequency range [2]. It is important to maintain the open porosity of the briquette and a high filterability on the part of the reaction products, thereby facilitating the diffusion processes in the afterburning zone and significantly influencing the depth of conversion in the synthesis reaction [3].

The carbide and nitride of titanium form a continuous series of solid solutions. The period of the crystal lattice varies with the composition essentially according to the Vegard law. A study of phase equilibrium in TiC—TiN—Ti alloys has disclosed a broad range of carbonitride solid solutions whose composition is described by the formula TiC_xN_y[ ]_z, where [ ] denotes a vacancy in the nonmetallic sublattice.

Variations of the carbon content in the alloys and of the density of nonmetallic vacancies, along with the substitution of nitrogen for carbon, influences both the lattice parameter and all the chemical and physical properties of the material; this is a major consideration in regard to its practical utilization [5].

The twofold objective of the present study is: to investigate the influence of the conditions attending the process and the carbon content in the initial charge on the kinetics and the chemical and phase composition of the combustion products in the system Ti—C—N at a constant nitrogen pressure; to determine the optimal conditions for the formation of the carbonitride composition in the case of filtration combustion in a sound field.

EXPERIMENTAL PROCEDURE

The influence of the vibration frequency on the kinetics of the process and the properties of the synthesis products have been investigated on the experimental stand described in [2]. The combustion process is recorded through a transparent window...
Fig. 1. Linear rate of propagation of the combustion front (a) and mass fraction of nitrogen \([N]\) in the synthesis products (b) vs frequency of the sound field, \(x_C = 0.2\), for rods of various densities. 1) \(\Theta_0 = 0.2\); 2) 0.3; 3) 0.4.

Fig. 2. Fractions of the carbonitride (a), nitride (b), and carbide (c) phases in the synthesis products according to the results of quantitative analysis vs frequency of the sound field, \(x_C = 0.2\). 1) \(\Theta_0 = 0.2\); 2) 0.3; 3) 0.4.

of the reaction chamber with a fast-framing camera, and the linear propagation rate of the combustion front \(v_c\) is determined in subsequent processing of the film records.

Titanium powders of the type PTS (with a dispersity of 125-160 \(\mu m\)) and P804T ash are used in the study. Rods are prepared by a wet pressing procedure (the binder is a 0.1% solution of polyvinyl butyral in ethyl alcohol); the relative density of the initial briquettes is \(\Theta_0 = 0.2-0.4\). In pressing, to exclude density fluctuation effects, pressure is applied in the direction perpendicular to the motion of the combustion front. The composition of the initial charge is calculated so as to obtain TiC\(_x\)N\(_y\) as the final product with an atomic fraction of carbon in the initial charge \(x_C = 0-0.6\). The mass fraction of nitrogen in the synthesis products is determined on a TC-136 (LECO) gas analyzer within 0.05% error limits. Fractographic analysis is performed by means of a JSM-35 (JEOL) scanning electron microscope. X-ray phase and quantitative analysis is performed on a DRON-4 diffractometer in CoK\(_\alpha\) emission (17.8892 nm) with automatic control of the monochromator in the step-scan mode\(^*\) over the angular range \(2\theta = 48-52^\circ\) [the (200) line is primary for titanium carbide, titanium nitride, and titanium carbonitride] in 0.05\(^\circ\) increments; the point exposure times range from 10 s to 30 s. An approximation method is used to process the diffraction spectra, the diffraction pattern being represented by a superposition of several model distributions, whose optimum parameters are determined after multiple iterations and a comparison of the initial and complete modeled spectrum. The model distributions used for processing are the squares of Lorentz functions, shifted by the amount

\(^*\)The multiplet separation software was developed by the Department of Radiography and Physics of Metals at the Moscow Institute of Steel and Alloys.