Journal of Engineering Physics and Thermophysics, Vol. 65, No. 4, 1993

RELATIONSHIP OF THE STRUCTURAL AND PHYSICOMECHANICAL CHARACTERISTICS IN POROUS SHS MATERIALS BASED ON TITANIUM CARBIDE

V. N. Bloshenko, V. A. Bokii, V. I. Boyarchenko, A. A. Kashtanova, I. I. Fedorova, and O. Yu. Efimov

UDC 536.46

Structural and physicomechanical characteristics of the porous SHS materials TiC_{stoich}, $TiC_{0.9}$, and $TiC_{0.8}$ based on titanium carbide with a porosity of 55-58% are investigated. It is shown that as the combustion temperature increases, the compressive and breaking strength decreases monotonically and a smooth coarsening of the porous stucture occurs. Introduction of nickel into the carbide improves the strength and ductility of the porous SHS material. There is an optimum initial temperature (~ 500-600°C) ensuring maximum strength when nickel is introduced.

Along with nonporous materials the method of self-propagating high-temperature synthesis (SHS) enables one to produce porous materials [1]. Porous SHS materials produced by this method from various high-melting compounds, namely, carbides, borides, silicides, and compositions based on them, can be directly used as porous products: filters, catalysts, catalyst carriers, and structure elements. Each of the fields of application imposes specific requirements on the porous materials in terms of structural and physicomechanical properties: strength, porosity, and pore size. Therefore the aim of the present work is to investigate these characteristics for porous SHS materials based on titanium carbide. The latter was chosen since in SHS processes this is a model system, as it were, and also due to its practical suitability.

The investigations were carried out in the following manner. Using the procedure described in [1, 2], cylindrical porous specimens (with a pororsity of 53-58 vol.%) of diameter d = 10 mm and height h equal to the diameter of different composition were prepared. On an Instron 1195 instrument the specimens underwent axial or diametral compression to the point of destruction. In the first case, from the value of the breaking load P we determined the compressive strength of the material σ_c, and in the second case - the breaking strength σ_{br}.

We note that determination of σ_{br} is based on common assumptions of the theory of elasticity [3] and specific properties of brittle bodies consisting in a substantial difference between σ_c and σ_{br} ($\sigma_c >> \sigma_{br}$). In diametral compression between two planes of a cylindrical specimen the maximum tensile stresses develop in the plane of action of the external load and are directed perpendicularly to it. When a critical value of loading is attained in the diameteral plane of the specimen, coinciding with the load plane, a main crack develops which breaks the specimen into two parts.

In determining strength we carried out a series of 6-10 tests at each point. The values obtained were statistically processed. The pore and material structure was studied by the methods of optical metallography and electron microscopy. To prepare reaction mixtures, we used titanium, carbon, and nickel powders with degree of dispersion less than 40 μm.

In strength tests titanium carbide of stoichiometric composition behaved as an absolutely brittle material. Figure 1 (curve 1) gives a typical diagram in determining its compressive strength. It can be seen that the diagram terminates abruptly when the ultimate strength is attained. Just like a brittle body, nonstoichiometric titanium carbides of the compositions $TiC_{0.9}$ and $TiC_{0.8}$ were also destroyed.

Institute of Structural Macrokinetics, Russian Academy of Sciences, Chernogolovka. Institute for Problems of Superductility of Metals, Russian Academy of Sciences, Ufa. Translated from Inzhenerno-Fizicheskii Zhurnal, Vol. 65, No. 4, pp. 466-470, October, 1993.

 1062-0125/93/6504-1004$12.50

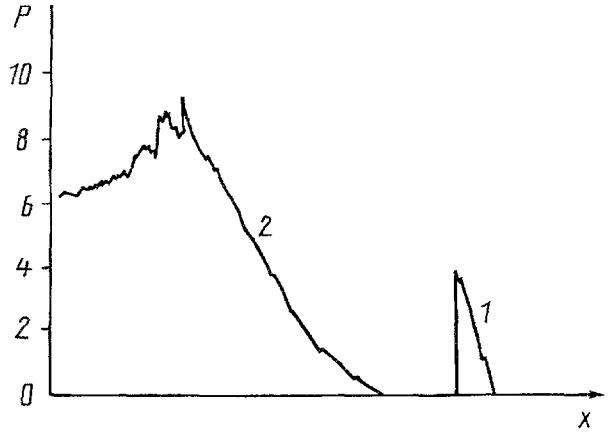

Fig. 1. Diagram of loading in compression: 1) stoichiometric TiC (П = 58%); 2) 95% TiC + 5% Ni (П = 54%). P, kH.

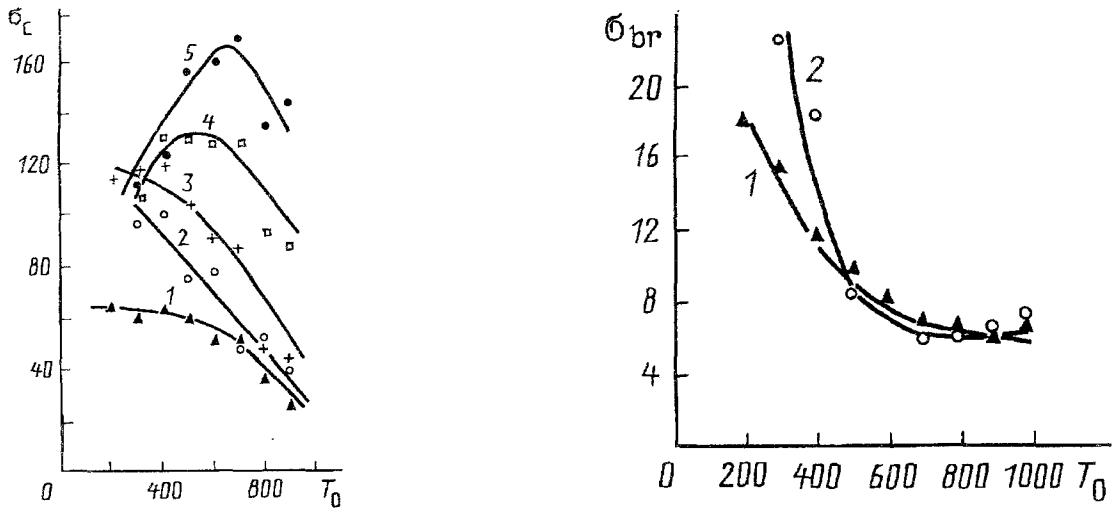

Fig. 2. Compressive strength vs initial temperature: 1) TiC_{stoich} (П = 58%); 2) $TiC_{0.9}$ (П = 56%); 3) $TiC_{0.8}$ (П = 55%); 4) 95% TiC + 5% Ni (П = 54%); 5) 85% TiC + 15% Ni (П= 53%). σ_c, MPa; T_0, °C.

Fig. 3. Breaking strength vs initial temperature: 1) TiC_{stoich}; 2) $TiC_{0.9}$.

Figure 2 gives the compressive strength as a function of the initial temperature T_0 at which the combustion process was initiated, and the combustion temperature T_c for the basic parameter on which the properties of the porous material depend was determined. Measuring the combustion temperature is a more difficult problem than measuring the initial temperature. Therefore this work investigates the mechanical characteristics as functions of T_0. According to combustion theory, the value of T_c is related to T_0 by the relation $T_c = T_0 + Q/C$ (Q and C are the thermal effect of the combustion reaction and the heat capacity of the mixture). Figure 2 shows a monotonic decrease in strength with increasing T_0 for both stoichiometric and nonstoichiometric titanium carbide as well as a higher strength for nonstoichiometric carbides.

The breaking strength as a function of the initial temperature is given in Fig. 3. Here a decrease in strength also occurs with increasing T_0; however there is practically no difference in the strength of stoichiometric and nonstoichiometric titanium carbide for initial temperatures higher than 400°C. The level of values of the breaking strength itself is 4-8 times lower than that of the compressive strength.

The monotonic decrease in strength as the initial temperature increases is explained by a change in the microstructure of these porous materials. An investigation of ground ends showed that with increasing T_0 (for a constant value of the overall porosity) there is a smooth coarsening of the structure: the average sizes of the carbide body and the pores increase (Fig. 4). For example, for titanium carbide of stoichiometric composition the average

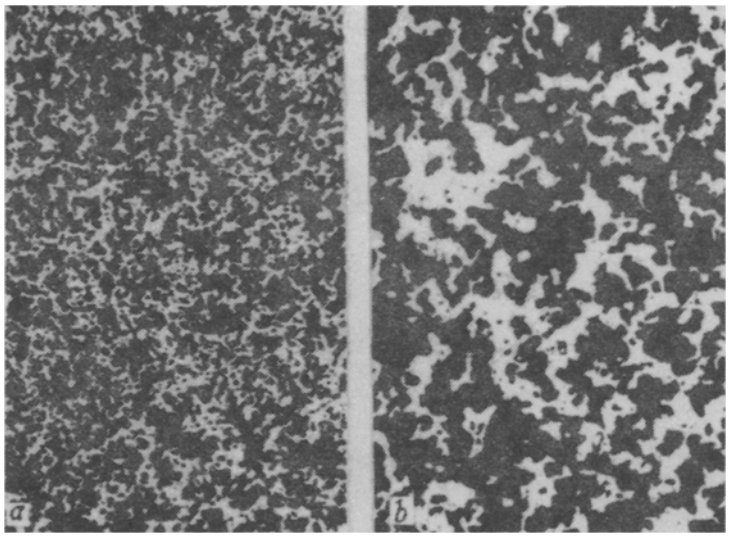

Fig. 4. Microstructure of TiC_{stoich} specimens for different initial temperatures: a) $T_0 = 200^{\circ}C$ ($d_{av} = 24\ \mu m$); b) $T_0 = 700^{\circ}C$ ($d_{av} = 55\ \mu m$). ×50.

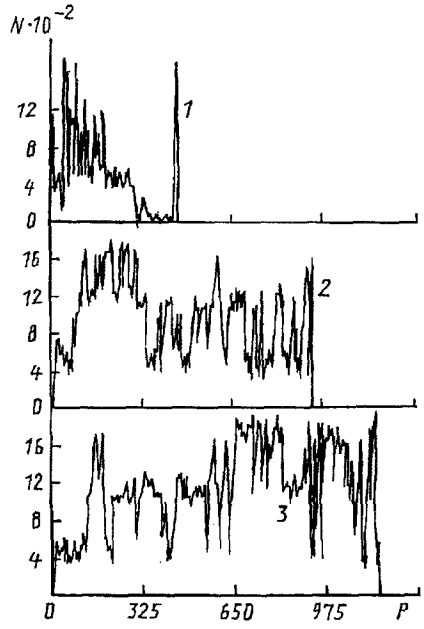

Fig. 5. Acoustograms obtained in diametral compression: 1) TiC_{stoich}; 2) 95% TiC + 5% Ni; 3) 85% TiC+ 15% Ni. N, sec^{-1}; P, kH.

size of pores increases from 24 μm at $T_0 = 200^{\circ}C$ to 55 μm at $T_0 = 700^{\circ}C$. And such a change in the structure must lead to a decrease in strength. The microstructure of nonstoichiometric titanium carbides also changes in a similar way. It should also be noted that in all cases porous SHS materials based on titanium carbide had a completely open character of the pores, i.e., the fraction of closed pores was less than 1 vol.%.

Introduction of nickel additives into titanium carbide of stoichiometric composition leads to a change in the properties of the porous skeletons. Whereas without a nickel additive the diagram of loading terminated abruptly, with introduction of nickel it has the form shown by curve 2, Fig. 1. Due to the appearance of ductility the diametral compression method for the specimens with nickel failed since in this case main cracks do not form in deformation of the specimens.

Investigation by the acoustic emission (AE) method gives additional information on the structure of porous SHS materials and their behavior under load. Figure 5 give characteristic acoustograms obtained in diametral compression of specimens to the point of destruction. The compressive force is plotted on the abscissa and the

acoustic emission intensity, i.e., the number of acoustic pulses per unit time, is plotted on the ordinate. The acoustogram for testing titanium carbide (curve 1) shows a brittle character of destruction. Early in the loading there is an intense release of acoustic signals due to microdestruction of weak links and crumbling under the bearings. Then their intensity gradually decreases (all weaknesses are taken up). Subsequently, for some time, as the load is increased, the material resists elastically (there are almost no sound pulses). And, finally, there is a sudden AE spike, i.e., formation of a main crack and specimen destruction. For the specimens with Ni (curve 2), a more uniform pattern of release of acoustic signals is observed in loading the specimens to the point of destruction, and the average AE intensity and the total value of AE (the area under the acoustogram), proportional to the released energy, increase. This points to the increased ductility of the material. When 15% Ni is introduced into TiC a transition from the brittle character of destruction to the ductile one already occurs (curve 3).

Curves 4, 5 in Fig. 2 give the compressive strength as a function of T_0. As compared to pure titanium carbide the strength increased noticeably and its dependence on T_0 acquired a nonmonotonic character. The position of the maximum on the curve $\sigma_c(T_0)$ shifts in the direction of high initial temperatures as the nickel concentration increases, i.e., in the direction of increased average pore size. The emergence of the maximum suggests the appearance of a new factor improving the strength and competing with the structure coarsening, i.e., the factor diminishing the strength. This new factor is the presence of nickel, which concentrates solely along grain boundaries, heals structure defects, and improves ductility and strength. The maximum on the strength curve is connected with the fact that with increasing T_0 a more uniform distribution of nickel in the volume of the carbide body occurs, which is confirmed by the data of metallography and electron microscopy.

REFERENCES

1. A. G. Merzhanov, I. P. Borovinskaya, V. N. Bloshenko, and V. A. Bokii, Porous Reractories and Their Manufacture, PCT Int. Appl., WO 9007, 013, June 28, 1990.
2. V. N. Bloshenko, V. A. Bokii, I. P. Borovinskaya, and A. G. Merzhanov, Fiz. Goreniya Vzryva, No. 6, 90-94 (1984).
3. M. M. Frokht, Photoelasticity [in Russian], Vol. 2, Moscow (1950).