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SUPERPLASTIC DUCTILITY OF OXIDE AND NONOXIDE CERAMICS

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The superplastic ductility of oxide and nonoxide SHS ceramics is considered. It is established that with certain temperature-rate dependences these ceramic materials manifest features typical of superplastic flow. It is shown that the ceramics undergo some specific microstructural changes under strain conditions.

Ceramics possess a number of unique properties that make them a promising structural material. In practice, different articles are manufactured from ceramics by the traditional, almost the only, method of powder metallurgy since the high melting point (~2000-3000°C) of ceramics and the lack of ductility under strain conditions do not allow the use of casting technologies and pressure treatment. Creation of ductile ceramics could overcome, to a significant extent, these difficulties and appreciably increase the survival capability of ceramic items. Some published data [1-5] point to the possibility of increasing the ductility of a ceramic by converting it to a superplastic ductility state. For this, a necessary condition is the presence of a stable submicrocrystalline microstructure with a grain size of less than 1 μm. However, the manufacture of such disperse powders is rather limited, and therefore investigation of the superplastic ductility of relatively coarse-grain ceramics is of particular interest.

For our investigations we have chosen a bismuth ceramic as one of the group of single-phase oxide ceramics. Analysis of the DTA curves has shown that the bismuth ceramic has the phase transitions $\alpha \to \delta$ at $T = 710^{\circ}$C and $\delta \to \beta \to \alpha$ on slow cooling. Experiments were conducted in the single-phase α-region with a grain size of approximately 10 μm (Fig. 1). At 550°C the Bi_2O_3 ceramic possessed slight ductility, which increased with increase in temperature. At 650°C the ceramic showed rather high rate sensitivity of flow stress (Fig. 2). At a strain rate of $\varepsilon = 10^{-3}$ sec^{-1} the coefficient of the rate sensitivity of flow stress m attained 0.42, and in the microstructure of the deformed samples the grains were displaced relative to each other. Hence, even a coarse-grained ceramic may display the features of superplasticity flow under certain temperature-rate conditions.

Investigations on increasing the ductility of nonoxide ceramics are few in number, though these materials represent an important class of wear-resistant and high-temperature ceramics. Among them, ceramic materials based on carbides of transition metals occupy a very important place. Such ceramics may be deformed only at temperatures exceeding that of brittle-to-ductile transition (BDT), which, as a rule, is 1400-1500°C and up.

A typical representive of nonoxide ceramics is a titanium carbide ceramic. This compound is advantageous since the BDT temperature of interstitial phases (one of which is TiC) is strongly dependent on the chemical composition. Titanium carbide with nonstoichiometric composition was produced by SHS followed by forced compacting. In the initial state the samples had a two-phase structure: carbide grains are surrounded by micropatches of a titanium phase whose volume share amounts to 12% (Fig. 3). The mean size of the carbide grains is 10-12 μm. The lattice parameter of the carbide phase, determined by an x-ray diffraction method, is 4.29 Å, which corresponds to the compound $TiC_{0.47}$.

We now consider the high-temperature behavior of the material in the lower homogeneous region. In the case of compression strain at temperatures up to 700°C the carbide ceramic behaved like a typical ceramic material with inherent brittleness. At 700°C and a strain rate of $\varepsilon = 10^{-4}$ sec^{-1} a brittle-to-ductile state transition occurs. With increasing strain temperature, ductility increases while the flow stress decreases monotonically (Fig. 4). For the given composition of the material at $\varepsilon = 10^{-4}$ sec^{-1} the BDT temperature was 700°C. Increasing the strain rate

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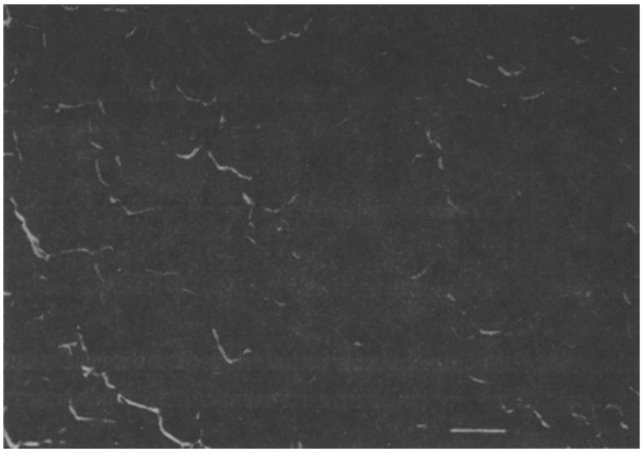

Fig. 1. Microstructure of a Bi_2O_3 ceramic (1000 magnification).

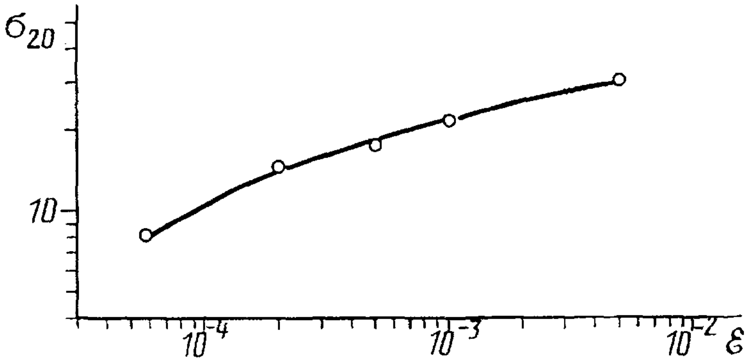

Fig. 2. Rate sensitivity of the flow stress to strain of a Bi_2O_3 ceramic. ε, sec^{-1}; σ_{20}, MPa.

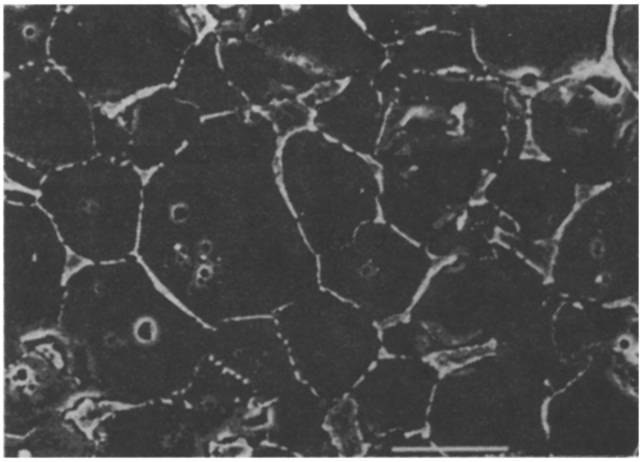

Fig. 3. Microstructure of a $TiC_{0.47}$ ceramic (2000 magnification).

to 10^{-3} sec^{-1} caused a BDT temperature rise to 750°C. The σ-ε curves are typical for the process of dynamic recrystallization. At temperatures above 900°C the flow achieves a steady state and the samples become deformed by 80-90% without failure.

The rate sensitivity of flow stress at $\varepsilon = 0.2$ (1), 25 (2), and 50% (3) to strain at 950°C is shown in Fig. 5. In the strain rate range from 10^{-3} to 10^{-1} sec^{-1} a linear dependence of σ on ε is observed, with the coefficient of the rate sensitivity of flow stress m being constant and equal to 0.2. As the strain rate decreases to 10^{-4} sec^{-1}, the coefficient m increases to 0.32, while at high strain rates it becomes 0.4.

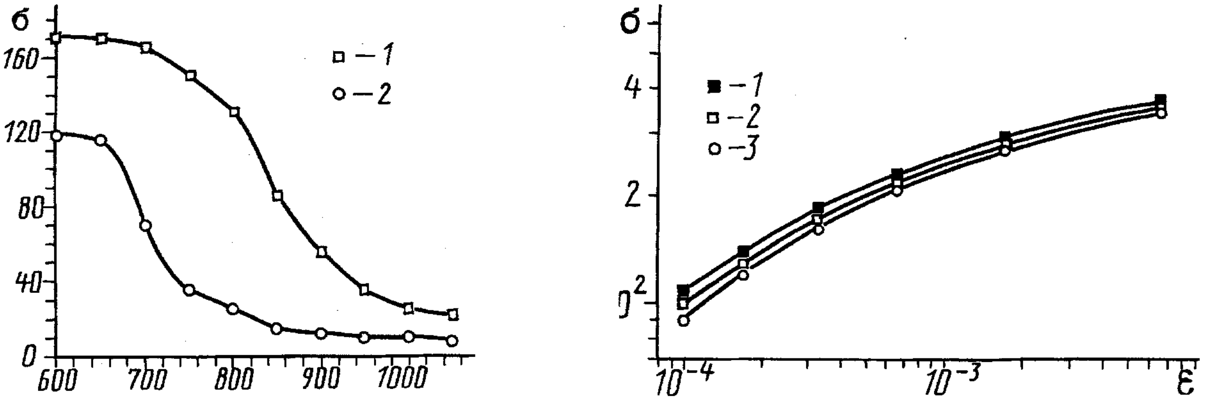

Fig. 4. Mechanical properties of a $TiC_{0.47}$ ceramic under compression: 1) 12 μm; 2) 2 μm. T, °C; σ, MPa.

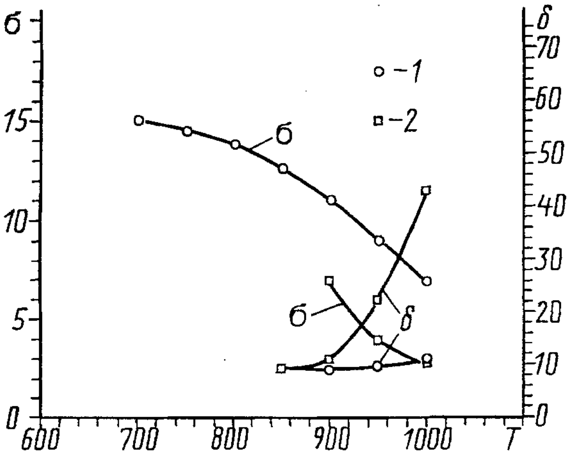

Fig. 6. Mechanical properties of a $TiC_{0.47}$ ceramic under tension.

In the case of tensile strain with $\varepsilon = 10^{-3}$ sec^{-1} (curve 1) the flow stress begins to decrease starting from 750-800°C; however, marked ductility is only manifested at a temperature above 800°C (Fig. 6). The maximum elongation to failure depends weakly on the temperature; it amounts to 7-12% in the temperature range from 850 to 1000°C. With decrease in the strain rate to 10^{-4} sec^{-1} (curve 2), the flow stress decreases markedly with increase in temperature, and the ductility increases abruptly. For instance, whereas at 900°C $\delta = 12\%$, at 1000°C it increases to 40%.

Thus, the results of mechanical tests show that under certain temperature-rate conditions of compression and tension strains the titanium carbide ceramic possesses relatively high ductility. In order to elucidate the reasons for such behavior of the ceramic, we will consider the microstructural changes caused by strain (Fig. 7).

Even at small degrees of strain some grains become deformed, and in them, micropatches of a metallic titanium phase with distinct crystallographic orientations within the boundaries of a grain appear. They form continuous chains which separate fine from coarse grains. The absence of such micropatches on annealing and their appearance in strain are indicative of their deformation origin and crystallographic nature. With increase of the degree of strain, the volume of fine recrystallized grains separated by interlayers of the titanium phase increases. The formation of fine grains under strain conditions decreases the flow stress, and when the volume portion of those grains becomes sufficiently large some features of superplastic ductility are observed: the coefficient m increases with the degree of strain. At high degrees of strain ($\varepsilon = 80\%$) the microstructure becomes more homogeneous; however, some coarse grains or fragments of grains with micropatches of the metallic titanium phase are preserved.

Of particular interest is the change in properties of the material after extensive preliminary strain where a microduplex microstructure has already been formed. Strain of the ceramic at 950°C by $\varepsilon = 80\%$ is followed by

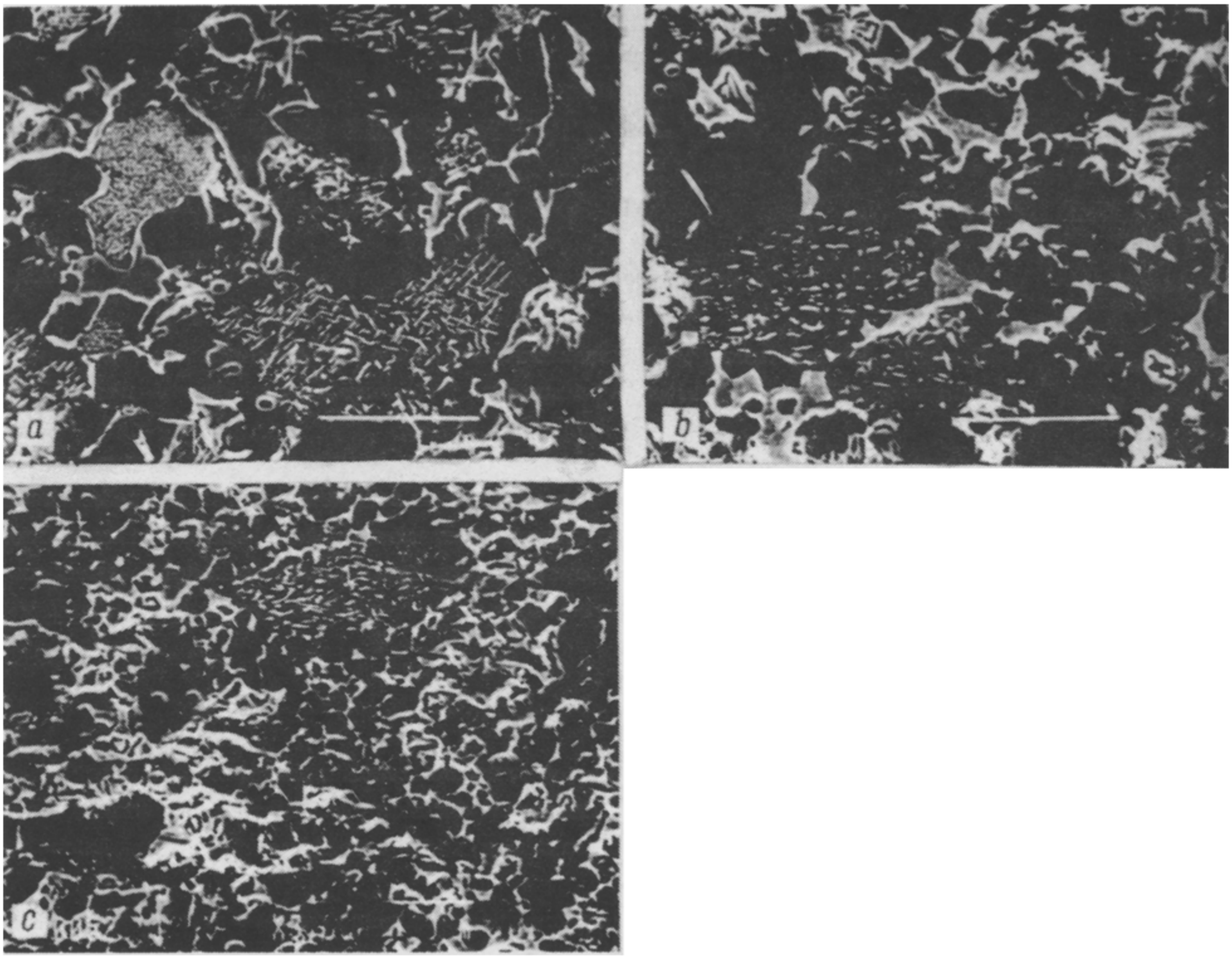

Fig. 7. Microstructure of a $TiC_{0.47}$ ceramic after strain. a) $\varepsilon = 10\%$; b) 50%; c) 80% (3000 magnification).

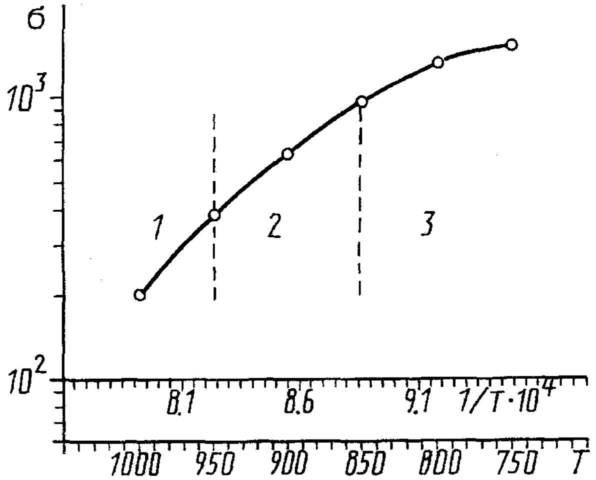

Fig. 8. Dependence log σ - $1/T$ of a $TiC_{0.47}$ ceramic. $1/T$, K.

dynamic recrystallization. As a result, the size of the carbide grains decreases to 1-2 μm, and the composition of the carbide phase changes from $TiC_{0.47}$ to $TiC_{0.75}$, while its volume portion increases simultaneously to 40%. We now consider the behavior of this fine-grain ceramic under high-temperature strain.

Formation of the microduplex microstructure causes a decrease in the BDT temperature to 850°C (see Fig. 4). The flow stress decreases monotonically with increase in the strain temperature. The fine-grain ceramic shows considerably less flow stress than the coarse-grain ceramic and this difference remains practically unchanged in the entire temperature range.

Analysis of the results of mechanical tests and microstructural changes reveals a decidedly dislocation character of the plastic strain in titanium carbide. The curve of flow stress vs inverse temperature (Fig. 8) allows us to qualitatively judge the action of one or another type of strain mechanism. The log σ - $1/T$ curves have two bendings, which is indicative of changes due to a strain mechanism. The first bending pertains, obviously, to the transition of the brittle-ductile temperature and the appearance of deformability. The second bending can be caused by a conservative-to-nonconservative transition of dislocation motion. As is seen, different temperature regions have different activation energies: $Q_1 > Q_2 > Q_3$.

Conclusions

1. Nonstoichiometric titanium carbide manifests high ductility at temperatures above the BDT temperature under compression and tensile strain conditions. The BDT temperature depends on the scheme and rate of strain as well as on the chemical composition of the compound.

2. Plastic strain of nonstoichiometric titanium carbide with an initial coarse-grain microstructure causes changes in the chemical composition of a compound due to micropatches of a metallic titanium phase over some crystallographic planes.

3. Hot plastic strain of titanium carbide leads to development of dynamic recrystallization which is accompanied by a reduction in the size of the microstructure. Creation of the microduplex microstructure under certain temperature-rate conditions of strain favors the transition of the ceramic to a superplastic ductility state.

4. The size of the grains is an important structural factor for a ceramic that determines its deformability. The reduction of grains in size results in a decrease in the BDT temperature and the level of flow stress, thus increasing the ductility of the material.

NOTATION

ε, strain rate; m, coefficient of rate sensitivity of flow stress; δ, ductility; Q, activation energy; σ, flow stress.

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