The physical properties of titanium carbide depend to a large extent on its composition in its wide homogeneity range, between TiC_{0.47} and TiC_{0.988} [1, 2]. Because of this, it is very important to be able accurately to determine the amounts of combined carbon and titanium in this carbide. The existing chemical methods for the determination of carbide compositions involve calculating first the total amounts of carbon and titanium and then the amounts of free carbon and titanium [3, 4], the actual carbide compositions being then computed from the results of these measurements. Precise determination of free titanium by the two most widely used methods, chemical and x-ray phase analyses, is a matter of considerable difficulty.

The method described below enables the amounts of combined carbon and titanium in a carbide to be determined direct from a neutron diffraction photograph. It is based on the fact that the intensity of diffraction reflections for titanium carbides (structure type NaCl) is proportional to the square of the structural factor $F^2 = (b_{Ti} + 1_{BC})^2$, where $b_{Ti} = 0.34 \cdot 10^{-12}$ cm and $b_{C} = 0.665 \cdot 10^{-12}$ cm are, respectively, the amplitudes of coherent neutron scattering for titanium and carbon [5] and $x$ is the combined carbon content of a carbide of composition TiC$_x$. Here the sum corresponds to reflections from planes with even Miller indices and the difference to reflections from planes with odd indices. This makes it possible to find the carbon content $x$ of a carbide from the reflection intensity ratio for suitably chosen planes. The intensity of a diffraction reflection for a polycrystalline specimen of cylindrical shape [6] is given by the expression

$$I_{hkl} = k \rho V N_C \frac{I_{hkl}}{p \sin \theta \sin 2\theta} |E|^2 e^{-2\mu} A_{hkl},$$

where $k$ is the instrument constant, $V$ the volume of specimen material in the beam, $p'$ the specimen density determined experimentally, $p$ the theoretical value of the density, $N_C$ the number of elementary cells in a unit volume, $I_{hkl}$ the (111) the repeatability factor for a plane with indices $(hkl)$, $\theta$ the Bragg reflection angle, $e^{-2\mu}$ the Debye thermal coefficient, and $A_{hkl}$ the absorption factor.

In a neutron diffraction photograph of titanium carbide there are pairs of diffraction reflections with even and odd indices occupying close angular positions, for example $(111)$ and $(200)$ or $(220)$ and $(311)$, which have similar values of Debye thermal coefficient. Hence, the sought carbon content of a carbide can be obtained with the formula

$$x = -\frac{b_{Ti}}{b_{C}} \frac{1 + \beta}{1 - \beta},$$

where

$$\beta = \pm \sqrt{\frac{I_{odd} (\sin \theta \sin 2\theta)_{even} I_{even}}{I_{even} (\sin \theta \sin 2\theta)_{odd} I_{odd}}}.$$

Neutron diffraction photographs of titanium carbides of various compositions were taken at room temperature in a horizontal neutron diffractometer at the Institute of Nuclear Physics, Academy of Sciences of the UzbSSR [7]. The detector of the effect was a pair of SNM-16 helium filled counters placed one behind the other in the direction of the beam. This counter arrangement increased the effectiveness of neutron recording (k = 1.08 Å) one and a half times.

Test specimens in powder form were rammed into thin-walled (wall thickness 0.1 mm) cylindrical vanadium cassettes of 12-mm diameter and 50-mm height. Neutron diffraction photographs were taken, using the monitoring counter, by scanning at 10° intervals, the measurement time at each point being about 5 min. Under these conditions each analysis lasted about 3 h. The results of our determinations of the compositions of the titanium carbides investigated are summarized in Table 1. Included for purposes of comparison are data yielded by chemical analyses.*

Error in the calculation of x by the neutron diffraction method is determined mainly, as can be seen from Eq. (2), by error in the measurement of the intensities Ieven and Iodd, which in turn is determined by statistical error. In the present work error in the determination of the intensities did not exceed 2%, and error in the determination of x was thus less than 1%. It should be noted that statistical error in the determination of diffraction peak intensities can be reduced by increasing measurement time or the amount of powder being examined.

Any free carbon, in the form of graphite, and free titanium present in a specimen can be determined from the intensities of appropriate reflections. Calculation is then performed in the same way as in quantitative x-ray phase analysis [8]. Free carbon is usually found in carbides of compositions close to equiatomic, the combined carbon and titanium contents of which can conveniently be determined from the intensity ratio of even and odd reflections. Free titanium is generally found in carbides with compositions close to the lower limit of the homogeneity range, characterized by low-intensity reflections with even indices. Because of this, free titanium contents are best determined from the intensity ratio of titanium diffraction reflections in neutron diffraction photographs of a pure titanium reference specimen and the carbide being investigated. The chemical method used in this work for determining free titanium contents was similar to that described in [9].

Comparing the results given in the table, it will be seen that chemical analysis yields distinctly low values of free titanium content. The reason for this is probably that in chemical analysis free titanium only partially enters into chemical reaction.

The method proposed can be employed also for determining the compositions of carbides of other transition metals with the NaCl structure. Here it should be noted that, compared with the neutron diffraction method, the x-ray diffraction technique [10] suffers from a major drawback, for the amplitude of coherent scattering of x-rays strongly depends on the atomic number and the scattering angle; apart from this, in this technique it is necessary to allow for x-ray absorption.

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