A research of microstresses and phase composition in alloys T30K4, T5K10 using diffraction of thermal neutrons

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Abstract. Samples T30K4, T5K10 are examined using diffraction of thermal neutrons in temperatures interval 20-600°C on reactor ИБР-2М (ОИЯИ). Temperature dependencies of a parameter of a hurdle of inner stresses of 3-rd type were calculated using Ritveld method taking into account diffraction specter. Experimental results of changes of crystal hurdles (TiW)C and WC are presented.

1. Introduction
Hard alloys based on tungsten carbide and titan with cobalt binding phase are basis for production of various tools. A coefficient of thermal expansion of carbids and cobalt differs thrice. There can appear residual stresses which can influence on work characteristics of a tool under cooling of these composites from high temperatures of synthesis [1, 2, 3] A level of microstresses appearing because of carbids defectiveness can influence on physical-mechanical features of these composites [4, 5].

The object of the research was to examine macrotensions in triple system W-Ti-Co with a high level (T30K4) and low level (T5K10) of titan.

Diffraction experiments were held using a neutron fourier-stress-difractometer (FSD) [6] with resolution 0.002 under d=0.2 nm.

2. Pilot studies of alloys T30K4 and T5K10
Measurements of diffraction specters of hard alloys have been held under temperatures of 20°C, 200°C, 400°C and 600°C for 6 hours in each spot. A heater was represented as a halogen spotlight with thin cylindrical clamps made of stainless steel. An analysis of neutron-diffraction photographs showed that alloys T30K4, T5K10 have three phases: TiC, WC, Co. (see table 1 and figure. 1, 2).

A nominal phase content of hard alloys T30K4 and T5K10 is enlisted in table 2.

Table 1. Phase content of alloys T30K4, T5K10.

| The structure description | TiC       | WC        | Co        |
|--------------------------|-----------|-----------|-----------|
| Monosymmetric system and | Cubic     | Hexagonal | Cubic     |
| space group              | (Fm3m, Nb225) | (P6m2, Nb 187) | (Fm3m, Nb225) |
| The hurdle parameter     | a=4.323   | a=2.909, c=2.834 | a=3.545   |
| Atoms coordinates        | Ti(0,0,0) C (1/2,0,0) | W(0,0,0), C(1/3,2/3,1/2) | Co(0,0,0) |
Table 2. The nominal content of hard alloys T30K4, T5K10.

| The alloy grade | WC  | TiC | Co |
|-----------------|-----|-----|----|
| T30K4           | 66  | 30  | 4  |
| T5K10           | 85  | 5   | 10 |

Figure 1. Alloy T30K4 under a room temperature. Experimental points calculated using method of Ritveld are shown. Positions of diffraction picks of WC, TiC and Co are shown by strokes.

Figure 2. Alloy T30K4 under the temperature of 600°C. Experimental points calculated using method of Ritveld are shown. Positions of diffraction picks of WC, TiC and Co are shown by strokes.

3. Phase content of hard alloy T30K4.
Processing of diffractional data of alloy T30K4 is held using method of Ritveld by means of software suite Vmria. Under study of neutron-diffraction photograph of TiC phase we can see that reflections of
200 and 220 type are almost redeemed (see figure 3) because of different marks of amplitudes of coherent scattering by cores of titan and carbonium. Intensions of these reflections of TiC phase increase on experimental neutron-diffraction photographs of hard alloy T30K4. It is clearly seen on the neutron-diffraction photograph captured under the temperature of 600°C (compare figure 2 and figure 3). It is obvious that in alloy T30K4 atoms of tungsten and cobalt with positive amplitude of scattering enforce in subarray of Ti and that influences on intensities of reflection of 200 and 220 type. Calculations for tungsten show that 13-15% of W atoms decompose in TiC under temperature of 600°C.

Figure 3. Model neutron-diffraction photograph of titanium carbide. Reflections with sharp indexes are almost scattered.

Increase of a parameter of TiC hurdle in alloy T30K4 with a temperature is shown on figure 4 and table 3.

Figure 4. A temperature dependence of the parameter of TiC hurdle in alloy T30K4.
Table 3. Value of the parameters of TiC hurdle in alloy T30K4

| Temperature of changes | a, Å   |
|------------------------|--------|
| 20°C                   | 4.323  |
| 200°C, 2 h             | 4.327  |
| 400°C, 1h              | 4.333  |
| 600°C, 6h              | 4.341  |

Study of microstresses of phase (TiW)C in alloy T30K4 was held according to a dependence of squares of half-width on squares of interplanar spacings in this phase for different temperatures. Data for two temperatures $T_{room}$ and 600°C is enlisted in table 4.

On figure 5 we can see that there are almost no microstresses in this phase and that they are left on a level of stain-free powder of iron, which is enlisted for comparison.

![Image of graph](image)

Figure 5. Alloy T30K4. Dependence of half-widths squares $W^2$ of diffractional picks on squares of interplanar spacings $d^2(Å)$ for $α$-Fe, $□$-(TiW)C-under the room temperature and $■$-(TiW)C- under the temperature of 600°C.

Table 4. The destination table of the microstresses of (TiW)C phase

| Temperature of changes | $ε\times10^{-3}$ |
|------------------------|-------------------|
| 20°C                   | 0.345             |
| 200°C                  | 0.346             |
| 400°C                  | 0.342             |
| 600°C                  | 0.324             |

Now let us study behavior of WC phase of the same alloy T30K4 in that interval of the temperature changes. The dependence of the squares of half-width on the squares of inter-planar spacings WC in alloy T30K4 for two temperatures $T_{room}$ and the temperature under 600°C is enlisted on figure 6.
Figure 6. Alloy T30K4. Dependence of half-widths squares $W^2$ of diffractional picks on squares of interplanar spacings $d^2$ for $\alpha$-Fe, (TiW)C-under the room temperature and (TiW)C-under the temperature of 600°C.

After the conducted tests for alloy T30K4 we suppose that tungsten carbide decomposes in titanium carbide under a sintering temperature with a formation of hard alloy. The analysis showed that microstress WC in alloy T30K4 during measurement from the room temperature up to 600°C has no change. We suppose that the process of metals redistribution is going on. It is found that 13-15% of tungsten decomposes in TiC that increases intensities of even reflections of this phase.

4. Phase content of hard alloy T5K10.

The same research was held with alloy T5K10. Alloy T5K10 also has three phases: TiC, WC, Co (see table 1). The nominal content of alloy T5K10 is enlisted in table 2 in percentage.

The parameters of WC hurdle in alloy T5K10 with increase of the temperature from the room one up to °C. The data is enlisted in table 5, and figure 7, 8, 9.

Figure 7. The temperature dependence of the parameters of “a” hurdle and with WC in alloy T5K10.
Table 5. Value of WC hurdle parameters in alloy T5K10.

| The temperature of changes, °C | a, Å  | c, Å |
|-------------------------------|-------|------|
| 20                            | a=2.909 | c=2.833 |
| 200, 2 h                      | a=2.912 | c=2.837 |
| 400, 1 h                      | a=2.915 | c=2.840 |
| 600, 6 h                      | a=2.918 | c=2.843 |

Figure 8. Neutron-diffraction photograph T5K10 under the room temperature. Experimental points calculated using method of Ritveld are shown. Positions of diffraction picks of WC, TiC and Co are shown by strokes.

Figure 9. Neutron-diffraction photograph T5K10 under the temperature of 600°C. Experimental points calculated using method of Ritveld are shown. Positions of diffraction picks of WC, TiC and Co are shown by strokes.
It is hard to receive statistically significant intensities of TiC and Co in alloy T5K10 with 6% of TiC and 9% Co content. Maybe that is the reason why no structure changes are indicated in these phases under different temperature measurements. The data of the microstresses is indicated only for WC phase as in the previous alloy. The data is enlisted on figure 10 and table 6.

Figure 10. Alloy T5K10. Alloy T30K4. Dependence of half-widths squares W^2 of diffractional picks on squares of interplanar spacings d^2 (Å) for -α-Fe, - (TiW)C-under the room temperature and - (TiW)C- under the temperature of 600°C.

Table 6. The destination table of the microstresses of WC phase.

| The temperature of changes °C | εX10^-3 |
|------------------------------|---------|
| 20                           | 2.451   |
| 200                          | 2.443   |
| 400                          | 2.445   |
| 600                          | 2.442   |

The analysis showed that under different temperatures from the room one up to 600°C with the interval of 200°C and 6 hours of withstanding at the specified the microstresses do not change, they exist but they are not significant.

The structure and microstresses of WC phase for this alloy is studied certainly. The transition of titanium into tungsten carbide or the reverse transition was not found maybe because of the low statistical validity of the intensities of the titanium carbide which is hard to be indicated. There are no structure changes and changes of the microstresses with low level of titanium in alloy T5K10. The analysis of the neutron-diffractive photograph showed that under different temperatures from the room one up to 600°C with the interval of 200°C and 6 hours of withstanding at the specified the microstress WC in alloy T5K10 does not change, they exist, but it is not significant. Residual stresses for separate phases were not evaluated in this research for lack of temperature dependencies of clear phases used for comparison with phases in alloys.

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