



## Microstructure and mechanical properties of V–Me(Cr,W)–Zr alloys as a function of their chemical–thermal treatment modes



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### ABSTRACT

Formation of regularities of the nanometric oxide precipitates and defect microstructure in vanadium-based low activation alloys V–Cr–Zr–(C,N,O) and V–Cr–W–Zr–(C,N,O) as a function of the regimes of their thermochemical treatment was investigated. Several methods of internal oxidation which provide formation of the nanosized ZrO<sub>2</sub> particles of controllable dispersion, ensure the nanometric size of the heterophase structure to be maintained up to the temperatures as high as 1300–1400 °C, and allow the recrystallization temperature to be increased up to  $\geq 1400$  °C were proposed. Formation of such microstructure contributes to dispersion- and substructural hardening and results in more than twofold increase in the yield stress of these alloys both at room and elevated (800 °C) temperatures, compared to the conventional thermo-mechanical treatment.

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### 1. Introduction

Low-activated vanadium alloys of V–Ti–Cr system (V–4Ti–4Cr is the best reference alloy) as the structural materials for cores of fusion and fission (fast) nuclear reactors are considered [1–4]. New compositions of vanadium low-activated alloys of V–Me(Cr, W)–Zr–C system demonstrate good prospects [5]. This prospect is related to the possibility of modifying these alloys' heterophase structure (a significant increase in the dispersity and the uniformity of the spatial distribution of the zirconium carbide particles–ZrC) by methods of thermomechanical treatment (TMT). According to [5], the significant increase of short-term high temperature ( $T = 800$  °C) strengthening (by 50%) and the recrystallization temperature (approximately by 100–200 °C) can be achieved using TMT methods in V–Me(Cr, W)–Zr–C alloys compared to V–4Ti–4Cr alloys due to higher volume fraction and thermal stability of the superfine non-metallic (ZrC) phase

particles. Significantly higher increase of the thermal stability of the nanostructured heterophase structure, the recrystallization temperature and characteristics of the long-term high-temperature strength of the vanadium alloys can be achieved with the use of chemical–thermal treatment (CTT) [6,7].

High solubility and diffusion mobility of the oxygen are typical for vanadium alloys at comparatively low temperatures ( $T \leq 0.4 T_{\text{melt}}$ ,  $T_{\text{melt}}$  is the melting temperature), which allow one to realize the non-equilibrium internal oxidation (IO) in Zr-bearing vanadium alloys, which gives rise to formation of ZrO<sub>2</sub> particles simultaneously in the entire volume of the specimens under the conditions of highly oversaturated non-equilibrium V–Zr–O solid solutions. It should be noted that as a result of slight changes in the conditions of nucleation and growth of a new phase in case of a deeper IO, it becomes possible to form the uniform distribution of ZrO<sub>2</sub> particles with an approximately unlimited degree of size fineness (particle's sizes < 10 nm).

High negative value of the thermodynamic potential of ZrO<sub>2</sub> phase formation ensures a high thermal stability of the fine-dispersed heterophase state and the possibility of an effective control over the coagulation rate of these particles by varying the oxygen concentration in V–Me(Cr,W)–Zr alloys. The possibility of forming of the

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fine-dispersed heterophase states (e.g., V–ZrO<sub>2</sub>) remaining stable at up to  $T \approx 0.8 T_{\text{melt}}$  was shown in [6,7]. These states suppress the recrystallization processes in specimens up to the temperature as high as  $0.8 T_{\text{melt}}$  under conditions of the IO of the specimens with a high defect density.

In this work we present the results of an effective dispersion hardening of V–Me(Cr,W)–Zr alloys with nanosized ZrO<sub>2</sub> particles using the TCT by the IO method.

## 2. Materials and experimental procedure

The investigation was carried out with the use of the vanadium low-activated alloys V–8.75Cr–0.14W–1.17Zr–0.01C–0.02O–0.01N (wt. %) and V–4.23Cr–7.56W–1.69Zr–0.02C–0.02O–0.01N (Alloy 1 and Alloy 2, respectively) manufactured in A.A. Bochvar Institute. Chromium and tungsten alloying provides the opportunity of values variation of the solid solution strengthening.

The initial specimens of the studied alloys represented the sheets of the 1 mm thick made using traditional thermomechanical treatment (TMT-I), similar to the treatment presented in [8], which involves the following stages:

1. Homogenizing vacuum annealing of the ingot at the temperature of 1300 °C for 8 h.
2. Extrusion (pressing) at the elevated temperature.
3. Conclusive rolling and upsetting cycles at room temperature with intermediate vacuum annealing at  $T = (950\text{--}1000)$  °C.
4. Conclusive stabilizing vacuum annealing at the temperature of 1000 °C for 1 h.

The specimens were heat-treated in the vacuum furnaces at  $\approx 3 \times 10^{-3}$  Pa. The structural studies were performed by the methods of optical metallography at Olympus GX-71 and NEOPHOT-21 microscopes and by transmission electron microscopy using Philips CM-30 and CM-12 microscopes at the accelerating voltages of 300 and 120 kV, respectively. The mechanical tests by the method of dynamic tensile loading were performed in vacuum at  $\approx 3 \times 10^{-3}$  Pa at the strain rate  $\dot{\epsilon} \approx 2 \times 10^{-3} \text{ s}^{-1}$  using flat specimens shaped as dumbbells with the gage section of 13 mm  $\times$  2 mm  $\times$  0.8 mm (at least 3 specimens for each temperature and condition).

## 3. The method of diffusion doping of vanadium alloys by oxygen

The stability of the oxidative medium on the stage of the alloys' saturation by the oxygen plays an important role in designing of the practical modes of controlled diffusion doping of vanadium alloys by oxygen. Conventionally, such saturation is performed in degrading oxides medium [6]. However, the existing practices of the IO of heat-resistant alloys demonstrate that application of this method is complicated by the necessity of removing of the extraneous impurities from these oxides, in particular, from volatile compounds containing oxygen (e.g., lower oxides).

One of the possible ways to stabilize the velocity of the diffusion-assisted saturation of the specimens with oxygen is to apply the diffusion doping via heat treatment in air followed by the transfer of the oxygen from the surface scale into the bulk of the specimens by high-temperature vacuum annealing. In this work, the above-described process via the modes of diffusion doping with oxygen is used. It was shown that these operating modes ensure a sufficiently high stability of the oxidation velocity. The technological process is simple and would not involve any additional costs associated with designing of the special equipment to perform the IO and to monitor the oxidation medium.

To achieve high thermal stability of the oxide phase, the concentration of oxygen in the internally oxidized specimens ( $C_O$ ) should be higher than the concentration required for complete binding of the

oxide-forming element during formation of the oxides of the corresponding stoichiometric compositions [6,7]. To achieve this goal in the studied alloys, the oxygen concentration should be  $C_O > 1.3$  at.% (Alloy 1) and  $C_O > 1.9$  at.% (Alloy 2) under corresponding zirconium concentrations and formation of ZrO<sub>2</sub> oxides. In this work, relying on these data and aiming at the use of different values of the volume fraction and thermal stability (coagulation rate) of the oxide phase, we performed two types of chemical–thermal treatment (CTT) procedures for each of alloys.

CTT-I—diffusion doping modes to achieve oxygen concentrations of  $C_O \approx 1.4$  and 2.1 at.% in Alloy 1 and Alloy 2, respectively, in order to ensure high thermal stability of nanosized ZrO<sub>2</sub> particles and, hence, a considerable increase in the alloy's recrystallization temperature;

CTT-II—diffusion doping modes corresponding to twice lower oxygen concentrations ( $C_O \approx 0.7$  and 1.2 at.% in Alloy 1 and Alloy 2, respectively) and volume fractions of the oxide particles, and much lower thermal stability of the heterophase nanostructured state, compared to CTT-I.

The concentration of the oxygen absorbed by the specimens was measured by weighing using the analytical laboratory scales. For the specimens used in this experiment, the oxygen-concentration measurement error was  $\pm 3 \times 10^{-3}$  at.%. The annealing after the IO of the specimens was performed at  $T = 1000$  °C for 1 h.

## 4. Experimental results

Before a CTT, the heterophase structure of both studied alloys was characterized by the presence of oxycarbonitride zirconium particles with a high content of carbon (ZrC particles) with the sizes from  $\approx 0.1$  to 0.3  $\mu\text{m}$  (Fig. 1a). These particles retard recrystallization of the alloys, so the fine-crystalline or polygonal structure becomes formed after TMT-I (grain and polygon sizes from  $\approx 1$  to  $\approx 3$   $\mu\text{m}$ ) characterized by high dislocation density.

Two types of ZrO<sub>2</sub> particles are observed in the electron microscopy replicas with the extracted second-phase precipitates (Fig. 1b and c) after the IO and the finishing annealing at 1000 °C for 1 h, irrespective of the CTT regime:

1. Comparatively large particles (Fig. 1b) with size (tenths of a micrometer) and shape similar to the initial carbide particles (Fig. 1a).
2. ZrO<sub>2</sub> particles with size of a few nanometers (Fig. 1c).

According to the mechanisms of low-temperature IO of two-phase bcc alloys (containing carbide particles) [6], formation of the oxides occurs via oxidation of the initial carbide particles due to a low rate of carbide dissolution in the reaction zone of IO (compared to oxygen saturation rate). Since the shape and features of the spatial distribution of comparatively large particles of the first type are similar to ZrC particles in the initial (before the IO) state, it is clear that these particles are produced during the IO via the above-mentioned mechanism. The finer particles of the second type are formed via precipitation from a solid solution, their fine size being determined by low mobility of zirconium atoms.

As follows from the electron diffraction patterns (Fig. 1b and c), the particles of different sizes have different types of the crystal lattice: monoclinic (the particle size are tenths of a micron, Fig. 1b) and face-centered cubic (fcc) or face-centered tetragonal (fct) lattices (for the particle sizes of a few nanometers, Fig. 1c). According to [6], these differences result from the changes in the type of the crystal lattice (monoclinic  $\rightarrow$  orthorhombic  $\rightarrow$  fct  $\rightarrow$  fcc) in the course of particle's size reduction.

Investigation of the thermal stability of microstructure indicates a considerable increase in the recrystallization temperature after the IO (Fig. 2). In both alloys, the temperature of the onset of collective recrystallization corresponds to that of the carbide phase dissolution and is found to be  $\approx 1300$  °C, the grain size after a one-hour

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