Influence of thin oxide layers on tribological properties of E110 alloy tubular specimens under dry friction conditions

B A Kalin, N V Volkov, R A Valikov, A S Yashin and T V Yakutkina

National Research Nuclear University MEPhI (Moscow Engineering Physics Institute) Kashirskoe highway 31, Moscow, 115409, Russia

E-mail: NVVolkov@mail.ru

Abstract. Experiments to simulate wear process of fuel cladding in case its contact with spacing grid. System «sphere-plane» selected as friction pair. Tubular parts of fuel claddings from E110 alloy diameter of 9.15 mm and length of 50 mm (wall thickness 1 mm) were used for investigations. Some claddings were subjected to ion cleaning and polishing under the influence of Ar^+ ion beam with average energy of 3 keV. Samples were oxidized in steam-water conditions (T=300° C, p=17 MPa, time up to 100 h) to create thin oxide layers with a thickness of 1 mkm on the tubes surface. It is found that wear of the metallic samples takes place in elastically plastic deformation conditions at initial stage (2-5 min). Presence of thin oxide layer (of thickness up to 200 nm) on the samples surface contributes to reduce wear due to the uniform redistribution its fragments on the friction track, and wear also samples takes place in elastically plastic deformation conditions. Presence of oxide layer with thickness of 700 nm on the samples surface increases wear in conditions of abrasion friction.

1. Introduction
Progress of nuclear energy and nuclear technology has put forward a completely new tasks in the area of tribology. For example, one of the most pressing tasks is to increase the wear resistance (endurance) and service life of tribo-contact pairs, such as fuel cladding - spacing grid.

2. Materials and tools
Tubular parts of fuel claddings from E110 alloy diameter of 9.15 mm and length of 50 mm (wall thickness 0.65 mm) were used for investigations.

Some claddings were subjected to ion cleaning and polishingunder the influence of Ar^+ ion beam with average energy of 3 keV, on the installation ILUR-03 [1] designed for ion beam processing the surface of long tubes with a diameter up to 20 mm (Figure 1). Samples of the tubes in the initial state and after ion treatment were oxidized in a steam environment (T = 300 °C, P = 17 MPa, a time of 10, 20 and 100 hours) for creating on the surface of the tubes of thin oxide layers. Before the experiments, in order to control the process of deterioration, we measured the surface roughness R_a on profilograph TR-200 and the surface microhardness of the PMT-3 (136° angle of a diamond pyramid, the load 90 and 120 g).

Analysis of the conditions of deterioration shells in the case of its contact with the spacer grid shows [2,3] that the closest approximation to real conditions corresponds to deterioration of a pair "sphere-plane" (Figure 2). The motion of a spherical indenter is advantageously carried out around the circumference of the shell, i.e., while rotating the tube around its axis. The indenter motion along tube geneatrix increases influence of local anisotropy of properties.
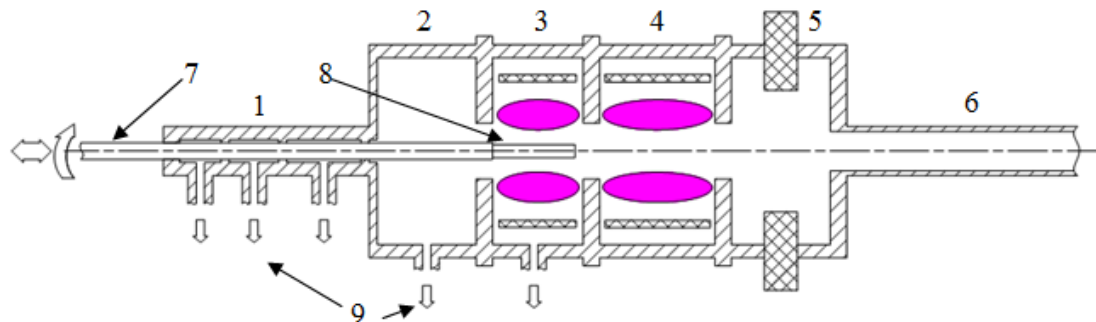
Figure 1. The scheme of the installation ILUR-03: 1 – gateway, 2 – transition chamber, 3 – ion cleaning chamber, 4 – ion polishing chamber, 5 – magnetron chamber, 6 – chamber to exit processing items, 7 – manipulator with a sample holder, 8 – processing item, 9 – vacuum pump connection.
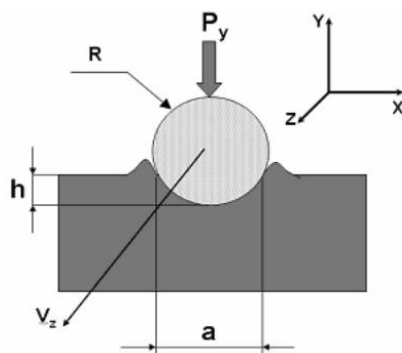

Figure 2. The scheme of deterioration of the friction pair "sphere-plane": 1 - cylindrical sample, 2 - ball indenter of radius R = 0,5 mm, a – width of the friction track, h - depth of the friction track, P_y - load, V_z - speed of movement.

Tribological experiments were conducted on the installation Friction-T (diagram is shown in Figure 3). As the friction pair selected "sphere-cylinder" diameter of the spherical indenter 1 mm, the type of friction - dry, load on the indenter P_y - constant. Selection of load caused by need to ensure the conditions close to the real, i.e., on the indenter at a load of 35 g corresponds to a pressure of 170 MPa, 50 g - 200 MPa.

Deterioration is implemented with a movement of the indenter along the surface with the velocity V_z = 1.4 cm/sec, which corresponds to the actual fuel rod conditions, the oscillation frequency of 0.5 Hz. Time Test - 2, 5, 10, 20, 30, 40, 60 minutes. As the main controlled parameter of deterioration was selected width of friction track – a, which was measured using an optical or scanning electron microscopy [4].

The approximation model of the "ideal indentation" of the ball in the cylindrical shell, friction track depth may be calculated according to the formula $h(t) = R - \{R^2 - [0.5a\,(t)]^2\}^{0.5}$ where: h - the depth of the friction track, R - the radius of the spherical indenter of 0.5 mm, a - the width of the friction track.
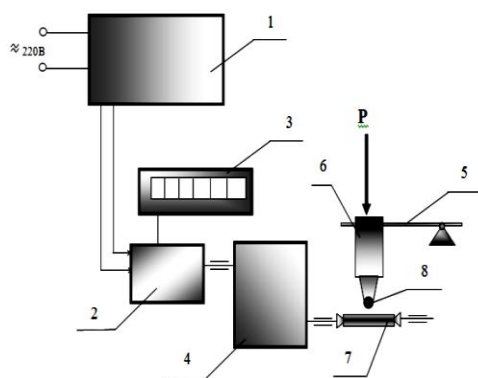

Figure 3. The Installation scheme Friction-T: 1 - power supply, 2-motor, 3 - speed control panel, 4 - precision reducers, 5 - balk of the loading indenter and its movement along the sample, 6 - the holder of a spherical indenter, 7 - cylindrical sample, 8 - ball indenter.

Volumetric wear I_v and linear wear rate I_h friction surface samples was calculated using the measure of h(t), which allows on the basis of tests carried out accelerated depreciation forecast to hold for long term use products [5,6].

3.Results and discussion

The measurements of the surface roughness shells samples indicate that in the normal state (after mechanical polishing), the value of surface roughness value is typically R_a = 0.9-1.5µm after ion treatment is reduced to R_a = 0.5-0.7 µm, and after oxidation in a steam environment is at level: the samples in the normal state - R_a = 1.5-1.0 µm, after ion cleaning - R_a = 0.7-0.9 µm.

Microhardness of samples in the normal state varies in the range H_μ = 127 ± 12 kg mm^{-2}, and after the ion cleaning there is a slight increase to H_μ = 140 ± 12 kg mm^{-2}.

In Figure 4 exemplary shows photographs friction track samples in normal condition, after 5 and 30 minutes of testing. From which it is seen that at the initial stage of deterioration (Figure 4a) are formed circular (longitudinal) strain bands (indicated by arrows), are also observed metal particles formed as a result of the effect of setting indenter with subsequent separation from the sample.With the increase in test time (figure 4b) the surfaceof friction track filled metal particles, some of them are covered with an oxide layer and pressed into the surface layer of the sample. At this stage, grinding the surface layer of the material and the stabilization of its restructured.When test time more than 30 minutes there is a further coloring material particles and their subsequent grinding, which contribute to increasing the abrasive component of deterioration, the track profile is formed completely of friction and it is further deepening.

In the case of cylindrical samples at the surface of thin oxide layer process of deterioration is slowing. As seen in Figure 4c width of friction track substantially less (test time of 30 minutes: the layer - 214 µm, in a normal state - 350 µm) compared to a sample placed in the normal state (Figure 4a), annular strips of metal deformation is practically not observed . At higher magnification in a metal layer revealed a large number of oxide particles (size of 50-100 nm), pressed into the surface layer of the sample.

Increasing the test period (Figure 4a) causes a broadening of the friction road which state is characterized by a homogeneous structure consisting of fine particles of the oxide layer (smaller than 50 nm). Areas related to the separation of the material are practically not observed.
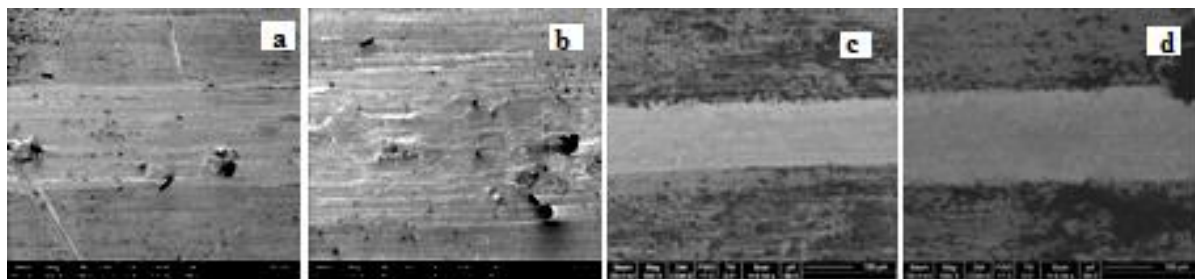

Figure4. Photos of the friction tracks of sample E110 alloy in a normal state and after oxidation in a steam medium: a - the test for 5 minutes; b - the test 30 minutes; c - 5 min test time, the thickness of the oxide layer 200 nm; d - the test 30 minutes, the thickness of the oxide layer 200 nm.

Figure 5 shows experimental data depth change friction tracks of test time h (t) samples in normal condition and the oxide layer (curves 1 and 3), after ion treatment (curve 2), and after ion cleaning with an oxide film (curve 4) that a good approximation to describe the kinetic curves logarithmic following form h (t) = A × Ln (t) + B, where A and B coefficients are selected for each state of the samples.
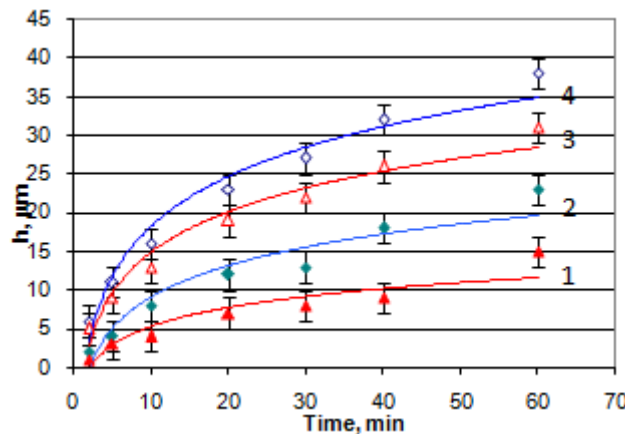
Figure 5. Changing the depth of the friction track of the test time h (t) samples: 1 - sample in normal state; 2 - sample after ion cleaning; 3 - the sample in the normal state with the oxide layer; 4 - sample after ion polishing with the oxide layer.

The presence of the oxide layer on the samples in the normal state and after ion treatment substantially reduces wear. Based on the kinetic dependences of deterioration from test time $h(t)$ were calculated loss material volume $V(t)$ and the intensity of the linear wear $I_h(t)$.
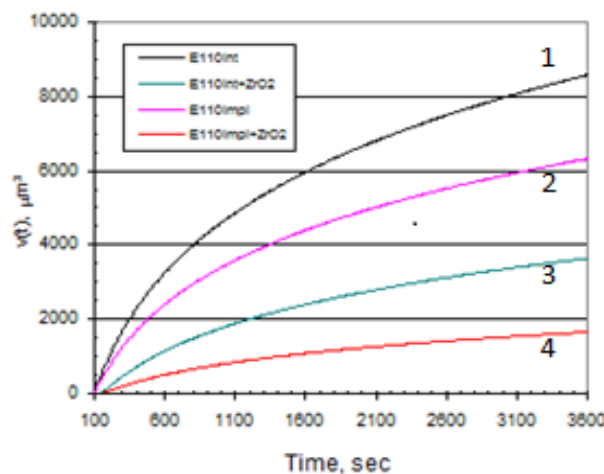

Figure 6. The dependence of volume loss in material from the test time V(t): 1 - the sample in normal state; 2 - sample after ion cleaning; 3 - the sample in the normal state with the oxide layer; 4 - sample after purification by ion oxide layer.

Figure 6 shows the dependence of V(t) the studied samples which show that the minimum loss of volume of the material observed in the samples after ion polishing with oxide layer (curve 4) $V(t) \approx 1500 \text{ m}\mu^3$.

4. Conclusion
Based on the tribological testing of cylindrical samples of zirconium alloys E110 in conditions of dry friction (with a load of 35 g, the rate of movement of the contacting surfaces 2.67 cm/sec) is set as follows:
• at the initial stage of deterioration metal samples (2-5 min) formed with track friction characteristic annular deformation zones, i.e. Wear is under elastic plastic deformation;
• ion polishing reduces abrasive component of wear;
• presence on the surface of samples of thin oxide layer (thickness 200 nm), thereby reducing of deterioration and wear depth approximation depending on the time with a good reliability is described by a logarithmic dependence which is characteristic of friction in terms of elastic-plastic deformation.

Acknowledgements

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