Research of the texture and residual stress in E110 after surface modification and hydrogen exposure

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Abstract. The effect of modifying the surface of components from the zirconium alloy E110 by pulsed carbon ions, irradiating electrons and titanium ions in the oxygen medium on the formation of a structural-phase and stress state in them and the patterns of interaction of surface modification with hydrogen were studied. Treatment by carbon and titanium ions does not change the basal texture of the initial E110 sheet. Irradiation by electrons leads to a significant weakening of the basal texture of the initial sheet. Holding in hydrogen leads to the formation of ZrH₁.₅ hydride in samples treated by carbon and titanium ions, but in samples, irradiated by electrons even traces of hydride were not detected. It is shown that surface modifications are accompanied by a transition from the compressive stresses of the initial product (100–120 MPa) to tensile stresses from 100 to 150 MPa. It was shown that after carbon and titanium ion modification the hydrogen exposure leads to a change in the stress state from tension to compression (20–70 MPa). In samples irradiated by electrons tensile stresses (70 MPa) were preserved.

1. Introduction

Toughening the working conditions of modern power plants increases the rate of corrosion processes in zirconium products and increases the level of their hydrogenation [1]. Crystallographic texture has a significant effect on the performance of zirconium components [2–7]. It was also shown [8–10] that during the formation of oxide layers on zirconium products at the interface with the metal (Me/O), a dense oxide layer is formed that contains, in addition to the monoclinic 8 % tetragonal oxide, the amount of which decreases as you move away from the interface and go to the loose outer oxide layer. In this case, the tetragonal oxide has a pronounced texture (100) [001], which is related to the monoclinic oxide (m) by the orientation relationship (OR): (103)ₘ//(100), or (103)ₘ//(101), and [100]//(010)ₘ [9].

In addition to the texture, the properties of the oxide layer depend on the level of the compressive stresses, which decrease as the sections are removed from the boundary Me/O. It is known that the morphology and texture of hydrides depends on the texture of the substrate. Most often between them is realized OR: (0001)ₐ-Zr//(111)ₕ-ZrH₁.₅ [11, 12]. However, under the influence of tangential tensile stresses, other ORs can arise, especially {1011}ₐ-Zr//(111)ₕ-ZrH₁.₅. It is important to note that the change in the OR is accompanied by a reorientation of the hydrides from the tangential to the radial direction of the tubular products. In the present work we investigated the effect of surface modification and the subsequent hydrogen saturation of the alloy E110 on the formation of texture and residual macro stresses therein.
2. Texture and hydride formation in E110

Three variants of modifying the surface of the alloy E110 (Zr – 1 % Nb) were realized: treatment with carbon ions with a pulse energy of 1 J/cm$^2$, $n = 3$ (iC); electron irradiation (EI) and irradiation with titanium ions in an oxygen medium (Ti). The texture and residual stresses were determined by X-ray diffraction on a DRON-4 diffractometer in filtered CuKα radiation. Residual stresses were determined by the "sin$^2 \psi" method for the reflex (1015) with "turn" angles $\psi = 0, -20, -40, -50^\circ$.

Figures 1–3 show the X-ray diffraction patterns of zirconium specimens after various variants of surface modification and hydrogen saturation. Treatment by carbon ions according to the iC regime (figure 1(a)) and irradiation with titanium ions in the oxygen medium according to the Ti regime (figure 2(a)) practically does not change the basal texture of the initial sheet from the E110 alloy. Irradiation with electrons leads to a significant weakening of the basal texture of the initial sheet (figure 3(a)).

Figure 1. The X-ray patterns for the E110 product after surface treatment by carbon ions (iC) (a) and hydrogenation (b): □ – $\alpha$-Zr; ▲ – $\delta$-ZrH$_{1.5}$.

Figure 2. The X-ray patterns for the E110 product after surface treatment by titanium ions in the oxygen medium (Ti) (a) and hydrogenation (b): □ – $\alpha$-Zr; ▲ – $\delta$-ZrH$_{1.5}$. 
Hydrogen exposure leads to the formation of ZrH$_{1.5}$ hydride in samples treated in iC and Ti modes, in which the texture of the original material has not changed (figures 1(b), 2(b)). At the same time, the intensive hydride reflex (111) it is observed (figures 1(b), 2(b)), which agrees with the basal texture of zirconium and the orientation relationship between the metal and hydride (0001)$_{\alpha-Zr}$/\{111\}$_{\delta-ZrH1.5}$. In samples irradiated by electrons in which weakening of the initial basal texture was observed, even traces of hydride were not detected (figure 3(b)).

![Figure 3. The X-ray patterns for the E110 product after electron irradiation (El) (a) and hydrogenation (b): □ – α-Zr.](image)

3. Residual stress formation after surface modification and hydrogen exposure
Figure 4 shows the results of measuring residual stresses in zirconium products. It is shown that all three methods of surface modification are accompanied by a transition from the compressive stresses of the initial product (100–120 MPa) to tensile stresses, the magnitude of which varies from 100 to 150 MPa (figure 4(a)).

![Figure 4. The magnitude of residual stresses for various variants of modifying zirconium products (a) and subsequent hydrogenation (b).](image)

It was found that in specimens treated in the iC and Ti regimes in which the original basal texture does not change, holding in hydrogen leads to the formation of hydride, which is accompanied by a change in the stress state from tension to compression (20–70 MPa), figure 4(b), which is a natural consequence of the volume increase as a result of hydride formation. At the same time, tensile stresses
(70 MPa) were preserved in samples irradiated by electrons (figure 4(b)) in which the basal texture was weakened and hydrogen exposure did not lead to the hydride formation.

It is likely that the formation of hydrides in samples modified by titanium and carbon ions indicates a lower permeability of their surface layers by hydrogen, possibly in connection with the formation of low-permeability oxide layers, which is facilitated by the basal texture of the metal substrate that survived after treatment. In addition, the presence of compressive stresses in the surface layers after these treatments prevents the penetration of hydrogen, figure 4(b).

4. Conclusions
It was experimentally discovered that treatment by carbon ions and titanium ions in an oxygen medium does not change the basal texture of the initial E110 sheet. Irradiation by electrons leads to a significant weakening of the basal texture of the initial sheet.

It was shown that a hydrogen exposure leads to the formation of ZrH$_{1.5}$ hydride in samples treated in iC and Ti ions, but in samples irradiated by electrons even traces of hydride were not detected.

It was shown that surface modification is accompanied by a transition from the compressive stresses of the initial product (100–120 MPa) to tensile stresses from 100 to 150 MPa. It was found that in specimens treated by the iC and Ti ions in which the original basal texture does not change, hydrogen exposure leads to the formation of hydride, which is accompanied by a change in the stress state from tension to compression (20–70 MPa). However, tensile stresses (70 MPa) were preserved in samples irradiated by electrons, in which the basal texture was weakened and hydrogen exposure did not lead to the hydride formation.

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