Study of the evolution of the metal surfaces topography under ion beam impact using statistic methods

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Abstract. Evolution of the state of cladding tubes outer surface from zirconium E110 alloy during ion-beam treatment was investigated using statistic methods. The samples have been irradiated on specialized installation ILUR-03 by radial Ar\(^+\) beam with wide energetic spectrum 0.5-5.0 keV up to doses (5-10)\times10^{18} \text{ ion cm}^{-2}. It is shown, that profile of the ion-modified surface may be described by means of autocorrelation function. Average relief roughnesses size increase after ion beam treatment is found. It is revealed that there are regular as well as random roughnesses at the surface investigated. It is established, that random component of relief, caused by mechanical abrasion, decreases during ion-treatment under given conditions.

1. Introduction
One of the most effective methods to improve quality of the different products surfaces is ion beam treatment. It is well-known, that surface of material and its near surface layer may contain a lot of technological impurities, which desorbed and sprayed during ion bombardment. Under certain parameters of ion beam, the ion polishing effect occurs. The surface roughness decreases, protrusions smooths and scratches heal [1].
It is well-known, that a lot of materials properties, such as friction, corrosion- and wear-resistance, are greatly affected by topography of its surfaces. In terms of that effect, parametric description of profile roughnesses is not universe because of strong dependence of physical processes, taking place at the surface, on relief features at equal values of \(R_a, R_q, R_z\) etc. Furthermore, using standardized criteria in precise surfaces analysis is not informative because of metrological causes [2-3].
In this way using statistic methods in analysis of ion-modified surfaces seems to be promising and actual goal.

2. Materials and tools
The experiments were performed on fragments of the cladding tubes from E110 alloy (Zr-1%Nb, length \(\sim 500 \text{ mm}, \text{ outer diameter} \sim 9.15 \text{ mm}\)). Outer surface of the specimens was previously polished mechanically and then treated on installation ILUR-03 [4] by radial beam of Ar\(^+\) ions with wide energetic spectrum 0.5-5.0 keV up to (5-10)\times10^{18} \text{ ion cm}^{-2}. Dose value has been chosen to provide sputtering of 1-2 \(\mu\text{m}\) layer of near-surface material. Sputtered layer thickness \(\Delta r\) has been determined from the results of microweighting on the basis of calculation 1 \(\text{mg} = 0.17 \mu\text{m}\). Microweighting was carried out on analytic weighing-machine GH-252 (A&D, Japan). Samples temperature during irradiation controlled by means of IR-pyrometers array and did not exceed 150-200 °C.
Profilograms of the samples outer surface $h(x)$ were filmed along a generator line on mechanical profilograph-profilometer TR-200 (Time Group Inc.) followed by automatic data transfer to PC. Traversing length was 1 mm, the measure step – 0.5 μm.

Summary data of the samples state are presented in table 1.

| #  | Surface state                      | $\Delta r$ (μm) | $R_s$ (μm) |
|----|-----------------------------------|-----------------|------------|
| 1  | Staff (mechanical abrasion)        | -               | 0.23       |
| 2  | Ion treatment, $D=5\times10^{18}$ ion cm$^{-2}$ | 1               | 0.19       |
| 3  | Ion treatment, $D=10\times10^{18}$ ion cm$^{-2}$ | 2               | 0.17       |

### 3. Profilograms processing technique

Profilograms of outer surface of the samples were processed in Microsoft Excel 2010. The straight line was used as a middle line of profile, coefficients of which were determined by the least squares method. For convenience of the surface state analysis all the features of the profile could be divided into systematic and random. Systematic component is a periodically arranged roughness of certain size that can occur as a result of regular technological factors operating. Random component is a result of the combination of a large number of irregular effects on the surface of the material during the formation of the relief. In first approximation, the random component of roughnesses can be regarded as a random stationary process. Thus, the actual surface profile can be represented as superposition of two selected components [3]:

\[
h(x) = h_{\text{sys}}(x) + h_{\text{rand}}(x),
\]

where $h_{\text{sys}}(x)$ – is certain periodic function, $h_{\text{rand}}(x)$ – is stationary random function with zero mean value.

For detailed analysis of the surface state autocorrelation function $K(\tau)$ for each of profilograms has been built. Autocorrelation function is known to be one of the most informative methods to describe stationary random processes and characterizes the dependence of process values at given point $x$ on the value of the same process at a different point $x + \tau$ with shift $\tau$. For a limited number of discrete values, the normalized autocorrelation function can be estimated by the formula [3]:

\[
K(\tau) = \frac{\sum_{i=1}^{n-1} (h(x_i) - <h>)(h(x_i + \tau) - <h>)}{\sqrt{\sum_{i=1}^{n-1} (h(x_i) - <h>)^2 \sum_{i=1}^{n-1} (h(x_i + \tau) - <h>)^2}},
\]

where $n$ – is number of measurements, $x_i$ – is coordinate of the $i$- measured point, $<h>$ - is arithmetic mean of the values $h(x_i)$ measured. The $K(\tau)$ error is calculated as follows:

\[
\Delta K = \sqrt{\frac{1-K^2}{n-2}}.
\]

In view of the equation (1), the autocorrelation function of the profile can be represented by a superposition of individually calculated autocorrelation functions for the systematic and random components of roughnesses:

\[
K(\tau) = K_{\text{sys}}(\tau) + K_{\text{rand}}(\tau),
\]
where $K_{sys}(\tau), K_{rand}(\tau)$ – are autocorrelation functions of systematic and random profile components $h_{sys}(x), h_{rand}(x)$ respectively. In first approximation profile systematic component can be represented by limited set of harmonic function in the form:

$$h_{sys}(x) = A\cos(2\pi\frac{x}{T} + \varphi),$$  \hspace{1cm} (5)$$

where $A$ – is roughness height, $T$ – period (wavelength), $\varphi$ – is random value on the interval $[0, 2\pi]$, connected with the starting point of the profilogram selection. For the random component of roughnesses description autocorrelation function corresponding to homogeneous isotropic field can be used:

$$K_{rand}(\tau) = B^2 \exp(-\tau/a),$$  \hspace{1cm} (6)$$

where $B^2$ – is certain constant, $a$ – is coefficient characterizing frequency content of the random roughnesses.

Thus autocorrelation functions may be approximated by the formula:

$$K(\tau) = \sum_{j=1}^{m} A_j^2 \cos(2\pi\frac{\tau}{T_j}) + B^2 \exp(-\tau/a),$$  \hspace{1cm} (7)$$

where $m$ – is a number of harmonic components with a wavelength $T_j$ and amplitude $A_j$.

Approximation parameters were determined using the Curve Fitting Tool package of MatLab R2012a program based on analysis of spectral density function of profile roughnesses, which estimated by the formula:

$$I(T) = \frac{1}{2\pi L} \int_{-L}^{L} \left| \exp(i\frac{2\pi}{T} \tau) h(\tau) \right|^2 d\tau,$$  \hspace{1cm} (8)$$

where $L$ – is profilogram length, $i$ – is imaginary unit.

Fraction of the profiles random component was estimated by the formula: [3]:

$$\nu = \frac{B^2}{(B^2 + \sum_{j=1}^{m} A_j^2)} \times 100\%.$$

4. Results and discussion

There are typical profilograms from outer surface of the samples in different states and corresponding normalized autocorrelation functions in fig.1. As can be seen, the ion treatment leads to visually noticeable increase in the average size of roughnesses with a slight decrease of peaks height and dents depth (fig. 1. a, c, e). It is observed, that regular component of the autocorrelation function allocates, and profile of the surface smooths while irradiation dose increases (fig. 1. b, d, f).
Functions obtained have been approximated by the form (7) for different number of harmonic components $m$ and taking into account error in assessment of the autocorrelation functions calculated by formula (3). It was observed, the accuracy of the approximation increases from $R^2=0.86-0.94$ for $m=0$ and $R^2=0.97-0.99$ for $m=3$. Parameters of the approximation functions for selected number of harmonic components are shown in table 2. As can be seen, with increasing irradiation dose, the proportion of random component of the profile roughnesses $\nu$ decreases from 85% to 55%, and its characteristic size grows indicating increase of uniformity of the surface relief.

Table 2. Autocorrelation functions approximation parameters.

| Surface state   | #1     | #2      | #3      |
|-----------------|--------|---------|---------|
| Staff           | 0.94   | 0.70    | 0.57    |
| $B^2$ (rel.un.) |        |         |         |
| $A_1^2$ (rel.un.) | 0.08   | 0.08    | 0.19    |
| $A_2^2$ (rel.un.) | 0.06   | 0.16    | 0.16    |
| $A_3^2$ (rel.un.) | 0.03   | 0.12    | 0.12    |
| $\nu$ (%)      | 85     | 66      | 55      |
| $a$ ($\mu$m)   | 6      | 10      | 28      |
| $T_1$ ($\mu$m) | 110    | 49      | 102     |
| $T_2$ ($\mu$m) | 278    | 197     | 174     |
| $T_3$ ($\mu$m) | 726    | 361     | 377     |
| $R^2$ (rel.un.) | 0.97   | 0.97    | 0.99    |

It is well-known, that mechanical polishing process causes a great number of irregular impacts because of great difference in sizes of abrasive particles, their location and pressure on the surface treated. That is why there is a big part of random roughnesses of surface of the samples in initial state. Mechanical polishing trails smooth with increase of ion beam treatment duration, because their characteristic size does not exceed the boundaries of the ion polishing maximum efficiency for the specified ion beam parameters.
Formation of the relief regular component may be caused by some features of the tubes production technological process, such parameters as shape, size, speed, and vibration of the tool.

5. Conclusion
Evolution of the state of cladding tubes outer surface from zirconium E110 alloy during ion treatment by radial Ar\textsuperscript{+} beam up to (5-10)×10\textsuperscript{18} ion cm\textsuperscript{-2} has been investigated using statistic methods. It is shown, that profile of the ion-modified surface may be described by means of autocorrelation function. Regular as well as random roughnesses at the surface investigated are found. It is revealed, that ion treatment at given beam parameters leads to smoothing of the surface relief while average relief roughnesses size grows and random component of the profile, concerning with mechanical abrasion, decreases.

References
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