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Wire electrical discharge machining of E110 zirconium alloy

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Abstract. The paper deals with the results of experimental research carried out by scanning electron microscopy (SEM) and X–ray diffraction analysis (XRD) to define, how the modes of wire electrical discharge machining (WEDM) influence on the elemental and the phase composition of E110 zirconium alloy's surface layer.Investigation of the phase composition allowed us to determine the main α and δ phase's distribution through the depth of zirconium surface layer, in common with phases of oxygen, copper, zirconium, and niobium specific compounds. It was also established the maximum depth of the defect level containing amorphous phase for all of WEDM modes, and proposed the grinding and polishing as potential mechanical methods of its removal.

1. Introduction

Zirconium alloys are widely used in nuclear power engineering, zirconium is also used to create multifunctional coatings, including biocompatible ones; this makes it really important to solve the problem of efficient treatment and waste minimizing in the course of manufacturing the heavy–duty products using expensive zirconium alloys. Zirconium as other rare metals is demanded in engineering technology besides traditional alloys (steel, cast iron, heat–resistant, aluminum and brass alloy). Zirconium targets are used to create multifunctional coatings by magnetron sputtering. Zirconium alloys are also applied in manufacture of fuel elements, assemblies and spacer grids and nuclear reactors. The production cost of machine engineering products with its output rate, shaping and quality has always been one of the main criteria of their production efficiency. That is why we need to pay special attention to efficient waste minimizing in the process of rare metal parts manufacture, which is very cost–intensive. For example, we can lessen the cutting width, thus reducing the chip amount. Applying of wire EDM method helped to achieve significant results in waste minimizing, because the cutting depth in case of WEDM is only one and a half times bigger than the wire electrode diameter.

It is possible to process any electrically–conductive metal by using EDM wire cutting. The machinery manufacturers provide machinery units having a set of modes for treatment of most commonly used materials, such as steel, copper, aluminum, hard alloy, and graphite (ISO 63). In case, when there is a need to treat the metal, which is not included into the unit's data base, e.g. zirconium, we have to define the most efficient treatment mode experimentally.Despite the fact that EDM treatment of metals, compared to other treatment methods, has a lot of advantages, diffusion of the electrode material and dielectric medium into the surface layer of the product under treatment negatively affects characteristics and properties of the treated surface. The authors [1] particularly mention a high hydrogen absorption degree of the surface of Zr–2,5Nb zirconium alloy during its EDM treatment in a hydrocarbon dielectric. The requirements to the zirconium target surface are especially high in case of forming biocompatible coatings using the method of magnetron sputtering [2, 3]. In the course of

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treatment, particles of the electrode material and decomposition products are carried to the surface layer of the product and form a defect layer. Therefore, the aim of the current research is evaluation of depth and phase composition of a defect layer, which becomes one of the main quality criteria for wire electrical discharge machining [4].

2. Materials and methods

In the purpose of zirconium surface layer investigation after EDM treatmentit was prepared E110 zirconium alloy samples in the form of disks with diameter of 30 mm and width of 3 mm. The surface was treated using 1, 2, 3, and 4 cuts to control phase changes due to different number of cuts. The parameters of the WEDM modes of zirconium samples were experimentallydefinedand presentedin table 1.The treatment was performed using EDM machine unit Sodick VZ300L with brass wire electrode (weight content of copper and zinc was 65% and 35% respectively) and distilled water as a dielectric fluid. The basic elements content in the E110 alloy is as follows: Zr=99%, Nb=1% (by weight).

Microsections were made as follows: mounting into acrylic compound, grinding using paper SiC P320, P600, P800, P1500, P2500, polishing with diamond suspensions 6, 3 and 1 μm, final polishing with polishing suspension with the grain size of 0.05 μm.

Table 1. Parameters of the WEDM modes of E110 zirconium alloy.

Serial cut number	Average voltage, V	Average amperage, A	Offset (H), mm	Pulse duration (On)	Pause duration (Off)
1	60	2.3	0.106	008	014
2	55	2.1	0.031	002	011
3	45	1.3	0.011	001	001
4	40	1.0	0.005	001	001

Experimentally defined parameters of the modes provide stable treatment without wire breakages. Electron microscope investigations were carried out by using *JEOL JCM–5700* microscope with an energy dispersive X–ray spectrometer *JED–2300* in a high–vacuum mode. The signal type was secondary electrons (SEI). The SpotSize parameter was chosen 60, the value of accelerating voltage – 20 kV, magnification upto 5000×.

X–ray phase analysis was performed using X–ray diffractometer Shimadzu XRD–7000 by the sliding beam method [5] using Kα–radiation of a copper tube with Θ angles from 1° to 10°. The X–rays patterns were interpreted by using PowderCell 2.4 software.

3. Results and discussion

Interpretation of X–rays patterns allowed us to receive the numeric data for phase depending on a tube angle Θ.For example the phase composition of a surface layer after 4th cut is shown in table 2. We can clearly see that there are two main phases of zirconium: hexagonal and cubical, compounds of copper and niobium ($CuNbO_3$), amorphous phase, in the second cut there is pure copper. Moreover, there are two phases of zirconium dioxide: tetragonal and monoclinic.

Table 2. The phase composition of a surface layer after 4th cutdepending on a tube angle Θ.

Θ	Zr hexagonal	Zr cubic	ZrO_2 tetragonal	ZrO_2 monoclinic	$CuNbO_3$	Amorphous phase
1	55.80	6.40	8.80	0.20	1.80	27.00
2	54.10	11.90	6.80	1.20	4.00	22.00
3	47.40	14.10	8.00	1.90	4.60	24.00

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4	45.00	16.20	10.60	1.50	6.70	20.00
5	46.90	19.40	10.20	3.40	5.10	15.00
6	55.30	17.30	9.60	1.90	5.90	10.00
7	54.00	16.40	11.10	5.20	3.30	10.00
8	59.20	15.50	5.50	2.10	7.70	10.00
9	68.50	16.50	6.10	0.80	8.10	0.00
10	71.50	16.70	5.00	1.50	5.30	0.00

The diffusion depth of wire electrode material was evaluated on cross–sections by energy–dispersive spectroscopy (EDS). For example dotted analysis of E110 zirconium alloy sample after 4th WEDM cut is presented on figure 1.Cross–sectioncontains the points where the energy dispersive analysis was performed.We moved from the surface deep into the material, until impurity matter disappeared, i.e. until zirconium concentration achieved 100%. Maximum depth of the copperdiffused layer amount13.0 µm for the 1st cut,6.7 µm for the 2nd cut,3.8 µm for the 3rd cut and 3.0 µm for the 4th cut.

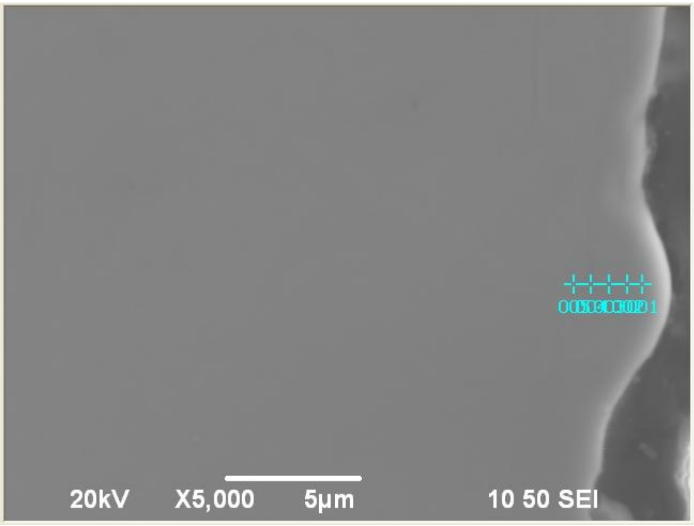

Figure 1. Cross–section of E110 zirconium alloy after 4th WEDM cut. SEM, x5000.

Technical requirements under cathodes manufacture for magnetron sputtering setup [6, 7] do not allow any foreign matter present in the surface layer, because in the course of sputtering first the foreign matter is sputtered, and only after that – zirconium; this results in different thickness of zirconium layer in different parts of the product [8]. It is obvious that during WEDM treatment the workpiece's surface and electrode are exposed to high temperatures and pressure. It causes formation of different phases complying with minimum potential energy principle. The receive data show that different phases form in a pre–surface layer, thus adversely affecting usage of different parts, applied for manufacture of fuel elements, cathodes and other products [9, 10].

4. Conclusion

Taking into account the above–said, we can argue that the WEDM treatment is an efficient method of precise zirconium machining. Maximum depth of the copperdiffused layer amount13.0 µm for the 1st cut, 6.7 µm for the 2nd cut, 3.8 µm for the 3rd cut and 3.0 µm for the 4th cut.

Though the received data, concerning depth of the electrode material penetration into the surface layer of zirconium target, we can make a conclusion that it is necessary to oversize for finishing grinding or polishing before using the products as intended. Further research of the mentioned zirconium samplesfor defining exact grinding or polishingmodesare necessary to be performed.

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