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Plasma electrolytic treatments for advanced surface finishing technologies

Электролитно-плазменные технологии для перспективной финишной обработки материалов

E. V. Parfenov [*], V. R. Mukaeva, R. G. Farrakhov
Е. В. Парфенов [*], В. Р. Мукаева, Р. Г. Фаррахов

Ufa State Aviation Technical University, Marx str. 12, Ufa, 450008, Russia
[*] evparfenov@mail.ru

Уфимский государственный авиационный технический университет, Россия, 450008, Уфа, ул. К. Маркса, 12
[*] evparfenov@mail.ru

ABSTRACT

Classification of plasma electrolytic treatments (PET) is summarized with indication of main factors affecting the process mechanism. Two distinct features defining two major PET trends are identified as formation of oxide layer on valve metals and appearance of vapor gaseous envelope on non-valve metals. These elements appear to have the highest electrical resistance in the circuit; therefore, their properties define the process flow. Brief description of the majority of known plasma electrolytic treatments is given. Current challenges and advances in the process development include using advantages of the both trends, e.g. polishing of valve metals and oxidation of non-valve metals. The process of electrolytic plasma polishing for medical implants and surgical instruments is closely analyzed. Insights on further technology development are proposed. These challenges include polishing of valve metals (Ti implants), inner surfaces (canulated screws) and afterpolishing for additive manufacturing. The challenges can be resolved by using fluoride electrolytes, polishing head tool and in combination with grinding, respectively.

KEYWORDS

Plasma electrolytic treatments; plasma electrolytic oxidation; electrolytic plasma polishing; titanium; stainless steel.

АННОТАЦИЯ

Обобщена классификация электролитно-плазменных технологий (ЭПТ) с указанием основных факторов, влияющих на механизм процесса. Выявлены две ключевые особенности процесса, определяющие два основных тренда в ЭПТ: формирование оксидного слоя на вентильных металлах и появление парогазовой оболочки на невентильных. Указанные элементы имеют наибольшее электрическое сопротивление в цепи, следовательно, их свойства определяют ход процесса. Приведено краткое описание известных электролитно-плазменных технологий. Выявлены направления и вызовы для создания новых электролитно-плазменных технологий, которые включают объединение достоинств обоих трендов, например, полирование вентильных и оксидирование невентильных металлов. Детально рассмотрен процесс электролитно-плазменного полирования для медицинских имплантатов и хирургических инструментов. Рассмотрены тенденции дальнейшего развития технологии, которые включают полирование изделий из вентильных металлов (титановые имплантаты), внутренних поверхностей (канюлированные изделия), а также полирование как постобработку для аддитивных технологий. Указанные проблемы могут быть решены с применением фторидсодержащих электролитов, полировального электрода-инструмента и при комбинировании с механическим шлифованием соответственно.

КЛЮЧЕВЫЕ СЛОВА

Электролитно-плазменные технологии; плазменно-электролитическое оксидирование; электролитно-плазменное полирование; титан; нержавеющая сталь.

Introduction

Plasma electrolytic treatments (PET) gained significant attention in past two decades as environmentally friendly alternatives to conventional galvanic treatments [1, 2]. Being more efficient in terms of surface modification capabilities, this technology suffers from high energy demand [3], owing to the method main feature – use of relatively high voltages from 200 to 700 V. This high voltage applied within the electrochemical process promotes local electrolyte boiling and microdischarge evolution within thin boiling film or surface oxide layer. Currently, two main trends in the plasma electrolytic processes are under research and development – plasma electrolytic oxidation (PEO) and non-oxidizing treatments with vapor-gaseous envelope (VGE). This paper summarizes the recent advances within the both trends with respect to medical implant surface engineering.

1. Mechanism-based classification of plasma electrolytic treatments

Analysis of current research in plasma electrolysis shows wide variety of technologies realized. However, the fundamental separation into two trends can be clearly seen (fig. 1). This separation originates in the semiconductor properties of the oxides that are formed on the surface of the substrate. Following the theory of metal/oxide/electrolyte interface conductivity, n-type oxides under anodic polarization of the workpiece make this junction reversely biased; therefore, the major voltage drop in the system occurs over this region [4, 5]. As a result, the electric properties of this interface define the behavior of the whole system. Therefore, if the valve metal oxides appear on the surface (Al_2O_3, TiO_2, MgO, ZrO_2, ZnO), its evolution follows the oxidizing trend. The mechanism of the oxide layer formation is predominantly electrochemical; this includes oxygen evolution due to the water electrolysis, and further interaction of the oxygen with the metal surface and metal ions coming from the substrate anodic dissolution, resulting in the oxide deposits on the surface [1]. On the other hand, if p-type oxides appear in the surface layer, the corresponding metal/oxide/electrolyte junction is open, and the other PET feature dominates in the system; this is the vapor gaseous envelope [2]. The VGE formation is typical for PETs of non-valve metals – steels, nickel, chromium and copper alloys. Since the surface is conductive, the current density comes to very high values; this results in high Joule heat release that creates a boiling envelope around the workpiece. Under anodic polarization non-valve metals can gain oxide layer on the surface, but it does not qualitatively affect the process mechanism; moreover, for the materials mentioned, it is desired to have polished, not oxidized surface in most applications. Usually, if the process is properly optimized, the VGE featuring PET treatment results in the non-oxidized surface; in this case, the anodic dissolution dominates over the oxide formation [6].

Like other electrochemical processes, oxidizing plasma electrolytic treatments operate within the temperature range defined by the electrolyte being liquid, i.e. generally from –10 to 110 °C. The workpiece temperature also stays in this range, and no heat treatment and phase transformations occur within the substrate bulk volume. Porous oxide layer is formed on the substrate [7]. This can be beneficial for producing coatings on nanostructured substrates [8]. However, when dealing with the VGE, various types of boiling must be taken into account [9, 10].

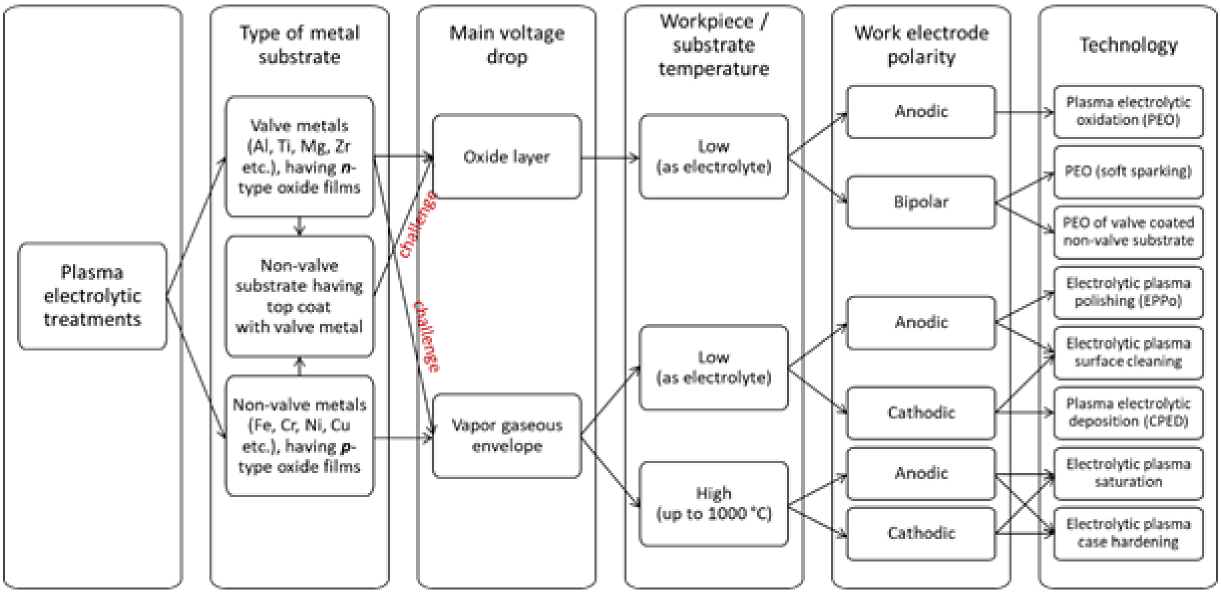


Fig. 1. Classification of plasma electrolytic treatments with indication of major factors affecting the process mechanism

Рис. 1. Классификация электролитно-плазменных технологий с указанием основных факторов, определяющих механизм процесса

If the VGE is not continuous, or it has bubble boiling, the workpiece temperature does not exceed significantly the electrolyte temperature, because the heat transfer through the liquid bridges is good. This can result in the surface cleaning and polishing for anodic processes, and coating deposition for cathodic processes [11, 12]. But if the VGE is continuous, and it has film type of boiling, this vapor-gaseous media has excellent heat resistance; as a result, the energy coming from Joule heat becomes shielded within the workpiece which can be heated up to 1000 °C within 15–30 s. Both cathodic and anodic processes can be used for case hardening as a result of quenching the heated workpiece, and for surface layer saturation with alloying elements. It should be pointed out that the VGE can exist over the valve oxide layer, and both mechanisms must be taken into account.

The work electrode polarity is another key factor separating plasma electrolytic treatments. Coming from the treatment name, plasma and electrochemical processes must be taken into account, and these processes significantly differ on anode and cathode [13, 14]. Cathode is the source of electrons, which come into gas via thermionic and other types of emission; the electrons get accelerated with the applied electric field, and the electron avalanche ionizes the gas. In the case of cathodic treatments, metal workpiece acts as a classical source of electrons, and the spark discharge appears in the VGE. As a result, the cathode workpiece surface always receives spark/arc discharge impact resulting in the increase of roughness [15]. However, in the case of anodic treatments, no obvious sources of electrons exist in the electrolytic cathode. According to [13], hydrated electrons can serve this task, and the discharge is considered as glow discharge with electrolytic cathode [16]. Since the number of such hydrated electrons is much less than the number of free electrons in metal, arc discharge is never obtained with anodic treatments.

From the electrochemical point of view, anode is the place where electrons come to the outer circuit. Consequently, metal ions come to the electrolyte; these and other cations can travel through the electrolyte to cathode and, as a result of reduction, gain the electrons back and deposit as a surface layer. Moreover, the electrolyte anions can travel to anode to form surface deposits. Depending on the ion reactivity (electrode potential), either water or dissolved electrolyte components participate in electrode half reactions; for electrolytes usually used in plasma electrolytic treatments, it is water which produces hydrogen on cathode and oxygen on anode [1]. Therefore, for oxidizing and polishing technologies, anodic polarization is critically important; for deposition technologies, cathodic polarization is essential.

From the variety of the PET technologies, each one uses specific features of the process mechanism. Plasma electrolytic oxidation provides protective oxide ceramic coatings on Al, Ti, Zr, Mg alloys [17–21]. Complex electrolyte compositions and introduction of nanoparticles widens the application of PEO coatings from wear and corrosion resistant to colored decorative and biocompatible [22–29]. Other advances in PEO concern electrical regime – progressing from DC, to AC (50 Hz), then to pulsed bipolar and currently to «soft sparking» regime providing compact coatings [30, 31]. Electrolytic plasma polishing (EPPo) is a technology which regains attention from the scientific community [32]. Being introduced into Russian industry for stainless steels and copper alloys [33, 34], now it is adapted for titanium [35]. Electrolytic plasma surface cleaning takes advantage of anodic dissolution, hydrodynamic action of VGE and electric discharge machining effect for cathodic process [11, 36–38]. Cathodic plasma electrolytic deposition combines electrochemical and microdischarge action resulting in coatings with interesting properties [39–41]. Anodic and cathodic plasma electrolytic case hardening and saturation provide quenching of workpieces and formation of nitride, carburized, boronized and other layers increasing the surface hardness [42–46]. Anodic processes usually form oxide layers which should be removed prior the workpiece use. The two trends create challenges that are now being addressed by researchers, i.e. PEO of non-valve substrates and polishing of valve metals. For non-valve substrates, aluminate and titanate electrolytes are typically used, or Al (or Ti) top pre-coating is sputtered or electrodeposited and then subjected to PEO [47, 48]. For polishing of Ti or Al, electrolytes and electrical regimes which suppress oxygen evolution, e.g. NH_4F and KF for Ti are used [49].

With respect to medical implants and surgical instruments, the process of electrolytic plasma polishing is of significant interest. This process can act as a final step for polishing titanium and stainless steel implants and surgical tools. Let us consider further discussion of this method capabilities.

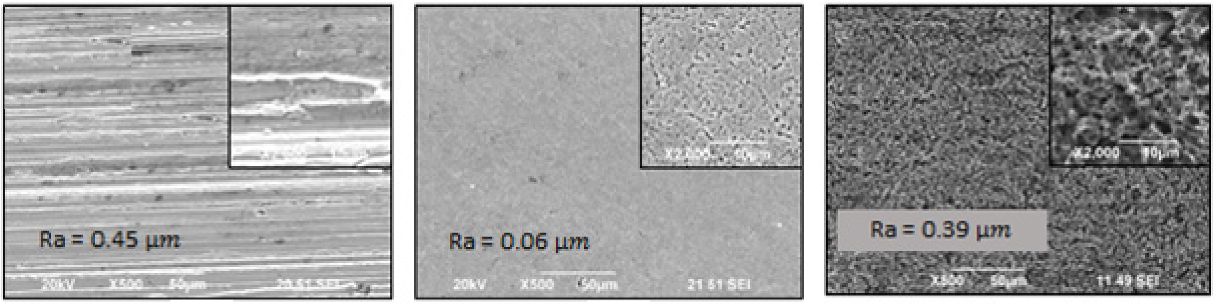
2. Electrolytic plasma polishing of medical implants and surgical instruments

The electrolytic plasma polishing process is a balance between the anodic dissolution and the oxide formation; this balance is shifted towards the dissolution by the action of the vapor gaseous envelope which prevents the precipitation of the oxides on the treated surface [6]. Fig. 2 shows the surface topography for the samples treated at different voltages, with and without the VGE (350 and 9 V), and at the same current density of 0.3 A cm^{-2}. According the Faraday's law, the electrochemical processes are the same, but the effect is different; therefore, it is induced by the VGE. Moreover, no electric discharge craters appear on the surface, suggesting a volume type of the discharge – glow discharge with electrolytic cathode.

As shown in Ref. [50], the EPPo process is successfully applied for finishing of the medical implants and surgical instruments (fig. 3 and 4). This research group works on industrial applications of the EPPo; therefore, significant information regarding the electrolytes and treatment regimes is omitted, probably, as confidential information [32]. However, as other publications show, ammonia salts with concentration 4–7% are usually applied. The most widely used component is ammonium sulfate for EPPo of stainless steel, nickel, chrome and copper alloys [34, 51]. Ammonium fluoride is used for titanium alloys polishing [35].

Current challenges and advances in the EPPo technology are summarized in Fig. 5. This includes polishing of valve metals, e.g. Ti and Al. This is resolved by the appropriate electrolyte composition, usually by introducing chlorides and fluorides. Polishing of the inner surfaces can be achieved by using a head tool which is run through the inside of the tube [52, 53]. Also, it is possible to polish non-valve metal coatings on organic materials [54].

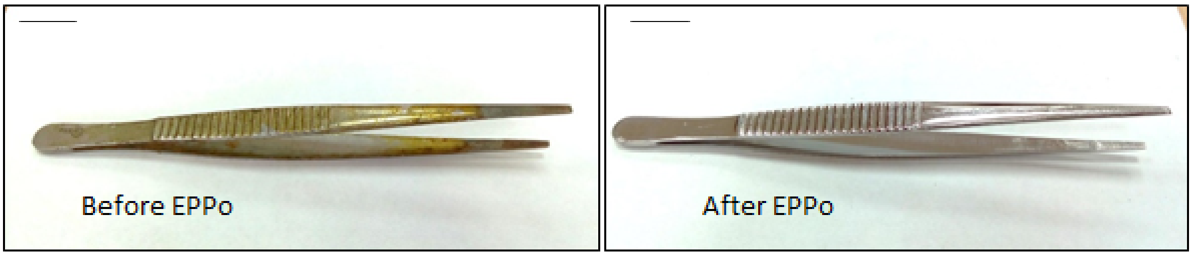
EPPo can act as a good option for the surface finishing of the workpieces produced by additive technologies, e.g. selective laser melting (SLM) [55, 56]. Typically, the roughness average Ra after this type of manufacturing is 5-6 µm. The capabilities of the EPPo reach Ra below 0.05 µm. However, this is only possible with the initial roughness of 0.2–0.4 µm. After SLM, the direct use of EPPo provides Ra from 1.2 to 2.0 µm; better polishing needs mechanical treatments, e.g. grinding, to decrease the roughness into the 0.2–0.4 µm range.



a *b* *c*

Fig. 2. Surface plane SEM images of the stainless steel samples before (*a*) and after EPPo treatments during 15 min. at different voltages U: *b* – 350 and *c* – 9 V [6]

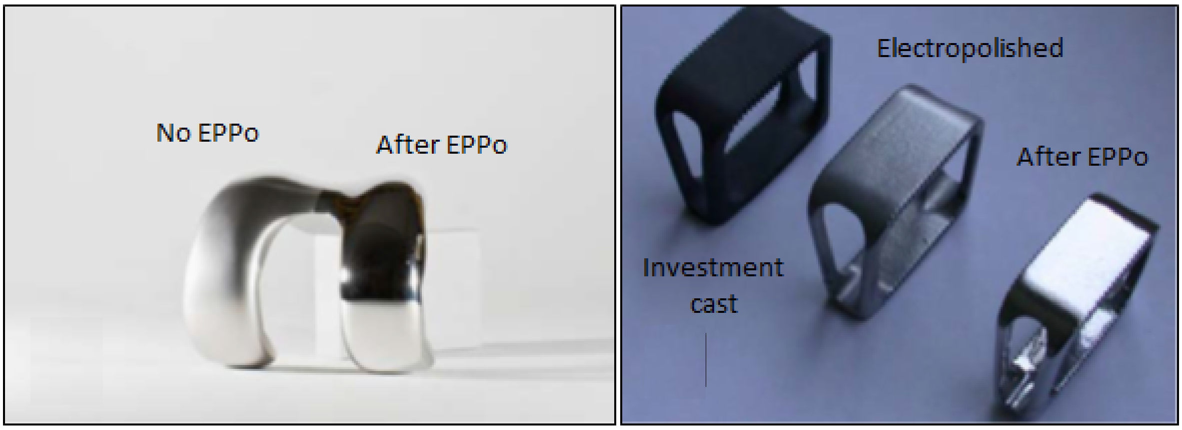
Рис. 2. РЭМ-фотографии поверхности нержавеющей стали перед (*a*) и после электролитно-плазменного полирования в течение 15 минут при различных напряжениях: *b* – 350 и *c* – 9 В [6]



a *b*

Fig. 3. Appearance of tweezers before (*a*) and after (*b*) EPPo applied for cleaning and polishing

Рис. 3. Внешний вид пинцета до (*a*) и после (*b*) электролитно-плазменного полирования



a *b*

Fig. 4. CoCr knee cap, right-hand side part after EPPo (*a*); Ti6Al4V, investment cast (left), electropolished (center), electrolytic plasma polished (right) (*b*) [50]

Рис. 4. CoCr деталь эндопротеза колена, правая часть после ЭПП (*a*); деталь из Ti6Al4V, после литья (слева), после электрохимического полирования (в центре), после ЭПП (справа) (*b*) [50]

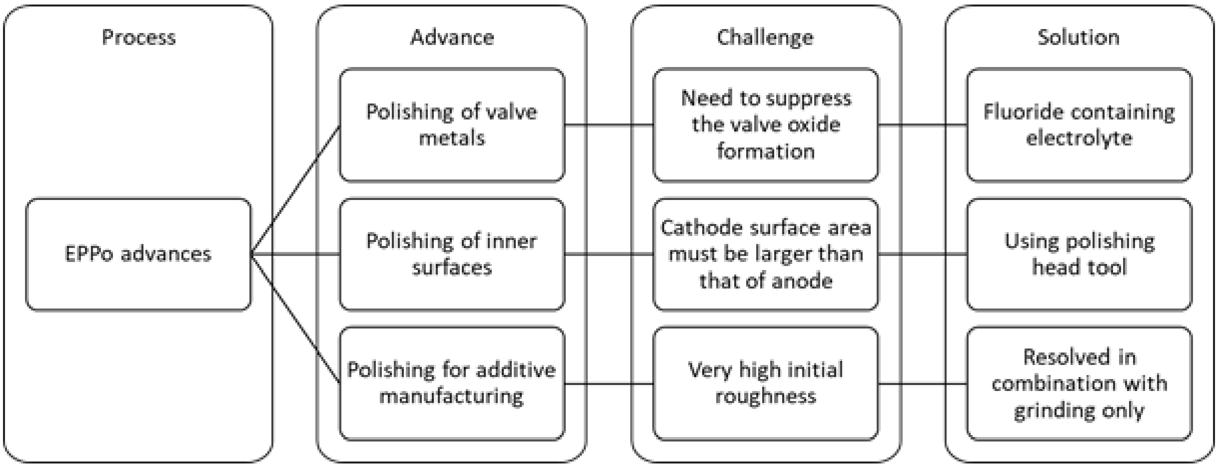


Fig. 5. Current advances and challenges in the EPPo technology

Рис. 5. Перспективы развития и вызовы в развитии технологии электролитно-плазменного полирования

Conclusion

In the past 20 years, plasma electrolytic treatments found their applications in various areas of engineering. Two distinct features of the PETs – formation of oxide layer on valve metals and appearance of vapor gaseous envelope on non-valve metals defined current and prospective applications. Joining of the benefits of the both features constitute some of the current challenges in the technology. Challenges in electrolytic plasma polishing for biomedical applications have been identified as: polishing of the valve metals, inner surfaces and workpieces after additive manufacturing comprise the advances in the technology. Finally, it is expected that plasma electrolytic technologies will find more industrial applications for surface finishing of medical implants and surgical instruments.

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