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Electric Discharge Machining of Ti-6Al-4V Alloy for Biomedical Use

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Abstract. Ti-6Al-4V alloy was subjected to electric discharge machining (EDM) as a surface treatment process. Several experimental investigations have been undertaken in order to assess whether EDM process is a viable surface treatment technique for application in orthopaedics. The surface roughness, chemical alterations of surface and osteointegration in vitro have been studied. EDM with relatively high peak currents induced sufficient surface roughness and created carbon enriched surface layer for improved osteointegration. Tensile properties are not substantially varied by EDM, however EDM has detrimental effect on fatigue endurance. Poor fatigue performance of material after EDM process irrespective of its original microstructure can be attributed to surface microcracks, brittle oxidized surface recast layer, internal tensile stresses and notch sensitivity of titanium alloys. These detrimental effects can be partly avoided by subsequent heat treatments. Microstructural changes of surface and subsurface layers have been observed by means of scanning electron microscopy. Fatigue performance is found to be the main limit for potential application of EDM process in orthopedics.

Introduction

Development of orthopedic implants is a complex and multi-field scientific issue. Wide spectrum of superior properties is required for implant material.

Concerning mechanical properties, the strength requirements of total joint arthroplasty restrict the suitable materials to metallic compounds only. Cyclical loading that is typical for implants during their life-time requires sufficient fatigue performance, specifically in the high cycle fatigue region. Low fatigue performance leads to early failures and decreased life-time of endoprostheses. The stiffness of implant material is another important issue. Typical elastic modulus of metallic materials is much higher than that of bone. This results in stress shielding and premature loosening of the stem of the implant [1].

Excellent corrosion resistance is a basic requirement for an implant material and also one of the principal concerns. Reliable passivation layer must be stable over a wide range of pH (pH 3–9). Along with corrosion, the biocompatibility, cells growth, cells adhesion and osteointegration are the surface related topics [2]. Implant materials can be divided into three generations. First generation materials aimed at reducing the immune response and the foreign body reaction to a minimum. Second generation biomaterials aimed at development of interaction with the biological environment to enhance the biological response, namely improvement of the implant/tissue interface. Third generation biomaterials are able to stimulate specific cellular responses at the molecular level [3]. In the case of orthopedic implants, the bioactivity is usually achieved by specialized surface treatment, plasma coating by titanium dioxide or by hydroxyapatite being the most popular [4].

Considering all following requirements, titanium alloys showed their superior mechanical properties, excellent corrosion resistance and favorable biocompatibility. The combination of properties makes the titanium alloys the material of choice for orthopedic implants [5]. Titanium as metallic element can be regarded as a first generation material. However, recent noncemented implant technology requires improved bone/implant interface that can be attained only by appropriate surface treatment.

Surface treatments of titanium alloys can be divided into two groups. The first are so-called 'fatigue enhancing' surface treatments, whereas the second are 'bioadhesion enhancing' surface treatments for biomedical use. Shot peening is typical example of fatigue enhancing treatment, whereas already mentioned hydroxyapatite coating is popular bioadhesion enhancing treatment [6].

In this study, the most common titanium alloy – Ti-6Al-4V alloy is used. The principal disadvantages of this alloy are its high elastic modulus and questionable effect of toxic vanadium. However, this alloy is still the mostly used orthopedic implant material and the mostly investigated titanium alloy.

Electric discharge machining (EDM) was employed as the surface treatment for orthopedic implants. We discuss its advantages and disadvantages with respect to the introduced property requirements. EDM is a common machining procedure, among others for titanium and its alloys. The spark between electrode and workpiece locally melts the material during the EDM process. Dielectric liquid then partly flushes the molten material away. The final surface structure then consists of debris and drops of solidified material. It has been reported previously that EDM affects the surface quality and the surface roughness [7]. The resulting surface roughness depends predominantly on the peak current of the EDM process [8]. EDM has already been proposed as a suitable surface treatment for biomedical application due to favourable biocompatibility and osteointegration [9, 10]. However, only a limited number of investigations of important properties that are required for implant material with suitable surface treatment have been performed yet.

Material and Experimental Procedures

Material Preparation

The investigated alloy Ti-6Al-4V might be prepared in three fundamentally different microstructural states. As-received material used in this study possesses fine grained (grain size ≈ 2 μm) globular (equiaxed) microstructure. The other two are lamellar microstructure and bimodal (duplex) microstructure. The details of preparation of the two latter microstructures can be found in [11]. Although these two microstructural states have been also prepared and investigated, this study is focused only on as-received material with globular microstructure.

Electric discharge machining (EDM) with comparatively high peak current of 29 A has been used for all test samples in order to impose high surface roughness. Graphite electrode and hydrocarbon oil as a dielectric liquid have been employed.

The samples for tensile tests according to A5 standard and the hour-glass shaped samples for fatigue tests have been machined prior to the EDM process. Simple flat samples with diameter of 19 mm were prepared for biocompatibility investigation, surface roughness measurements and scanning electron microscopy (SEM) observations.

Electrolytically polished (EP) samples served as benchmark samples for fatigue tests (polishing at temperature –20°C, 100 μm removed from the surface after machining).

Commercial process of plasma spraying with titanium dioxide has been used for benchmark samples for biological tests.

Mechanical Properties Testing and Microscopy

Tensile tests were conducted on Instron 5882 machine with the initial strain rate of 10^{-4} s^{-1}. Surface roughness was measured by a Perthen profilemeter. The vertical range of the measurement was 100 μm with vertical and lateral resolution of 2 μm. Roughness of all EDM processed specimens and plasma sprayed samples was measured on two flat samples in three different directions. The appearance of the surface layers after EDM process was investigated by scanning electron microscope FEI Quanta 200F equipped with an FEG cathode at the accelerating voltage of 20 kV. Chemical analysis of the surface was performed by the energy dispersive X-ray analyzer (EDX). Stress controlled high cycle fatigue tests were performed in rotating beam loading (zero mean stress, R = –1) at the frequency of 50 Hz. Cyclical loading was applied until complete fracture of the sample. The tests were undertaken in air. Complete Wöhler curves (S-N curves) were measured for both EDM processed and electro-polished samples.

Cells Growth Tests

Flat disc samples for biological tests were autoclaved (steam, 120°C, 0.1 MPa, 45 min), inserted into 12-well polystyrene multidishes (diameter 21 mm; TPP, Trasadingen, Switzerland) and seeded with human osteoblast-like MG 63 cells (European Collection of Cell Cultures, Salisbury, UK).

The cells were cultured in Dulbecco's modified Eagle's Minimum Essential Medium supplemented with 10 % fetal bovine serum and gentamicin in a humidified air atmosphere containing 5 % of CO_2. The cell seeding density was 50,000 cells/well (i.e., about 11,000 cells/cm^2), and each well contained 3 ml of the culture medium. Reference samples were represented by the bottoms of standard cell culture polystyrene wells. After rinsing with phosphate-buffered saline (PBS), the cells were released from the material surfaces with trypsin-EDTA solution (incubation 10 minutes at 37°C) and counted automatically in a Vi-CELL XR analyser. On each sample, 50 measurements were performed. The automatic cell counting also included an evaluation of the number of viable and dead cells by trypan-blue exclusion test [12].

Results and Discussion

Surface Roughness

Two roughness parameters R_{max} (the difference between the highest and the lowest point of the profile in the evaluated region) and R_a (average deviation of the roughness profile from the mean line) have been established. The evaluated region was always 2.4 mm. Electro-polished samples possess no roughness on such micro-meter scale. The roughness of EDM samples is characterized by R_{max} = 78 μm and R_a = 11.6 μm. Note that relative error is up to 15% due to statistical nature of the measurements. In the case of plasma sprayed samples the parameter R_{max} = 75 μm is almost equal as that of EDM samples, whereas R_a = 8.4 μm is substantially lower. This is caused by relatively 'wild' surface after plasma spraying with smaller peaks that are closer to each other. Higher surface roughness but relatively smooth profile (not shown here) after EDM process is one of the factors that positively affect the cell growth. EDM process with high peak current results in much higher surface roughness than previously reported [9].

Tensile Tests

The yield stress and the ultimate tensile strength (UTS) are only slightly lowered by EDM process. The yield stress is decreased from 860 MPa to 840 MPa and UTS from 920 MPa to 890 MPa. However, the elongation until fracture is substantially decreased from 15 % to 10 % only. This can be attributed to the presence of brittle surface layers due to oxidation during EDM process and internal tensile stresses.

SEM Observations

Fig. 1 shows the typical appearance of the cross section of sample after EDM process obtained by means of SEM using secondary electrons. Surface consists mainly of the remnants (drops) of molten and solidified metal. Below such remnants, the recast layer is identified. This layer consists of α' martensite with typical needle-like microstructure and is often referred to as 'white' layer due to its appearance in light microscopy [8, 13].

Fig. 2 shows that surface of the samples after EDM process is carbon enriched. This is caused by mixing of dielectric liquid (hydrocarbon oil) with molten metal and subsequent solidifying. It has been found that whole surface remnants are carbon enriched. This chemical alteration along with high surface roughness is the most important factor for favorable cell growth on the samples after EDM.

Fatigue Performance

Fig. 3 shows the results of fatigue tests performed in rotating bending. Complete S-N curves have been measured for test samples after EDM and benchmark electro-polished samples. It is clear that EDM has a detrimental effect on fatigue performance of the material. The main reasons for such poor fatigue endurance are assumed to be brittle surface layer due to oxidation during EDM process, internal tensile stresses and microcracks created during EDM process. The microcracks have been observed by means of SEM (not shown here). However other two possible effects have not been investigated in a more detail yet. Poor fatigue properties after EDM limit the potential applications of the material. It has been found that fatigue performance changes only slightly for different microstructural conditions of original material.

Results of Cells Growth Investigation

Fig. 4 consists of two charts that show the number of the total/viable cells on different substrates. The polystyrene substrate is a standard benchmark substrate. However the comparison with the commercial plasma-sprayed surface is more important. Cell numbers on surface after EDM were significantly higher at all culture

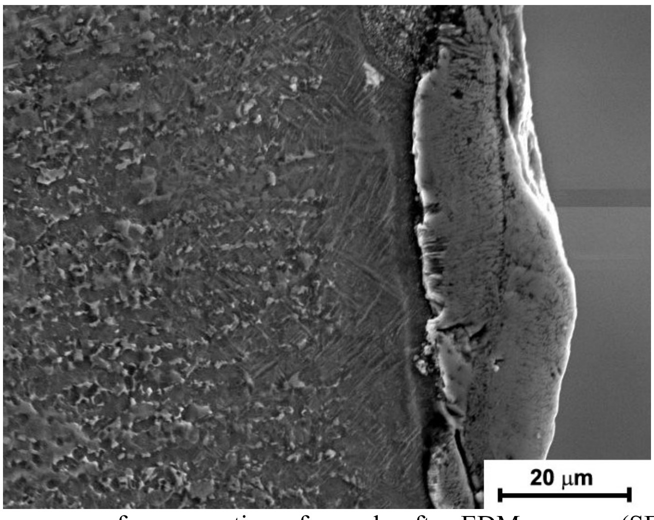

Figure 1. The typical appearance of cross-section of sample after EDM process (SEM, secondary electrons). From the left: bulk material, martensite recast layer and surface remnant of solidified metal are identifiable.

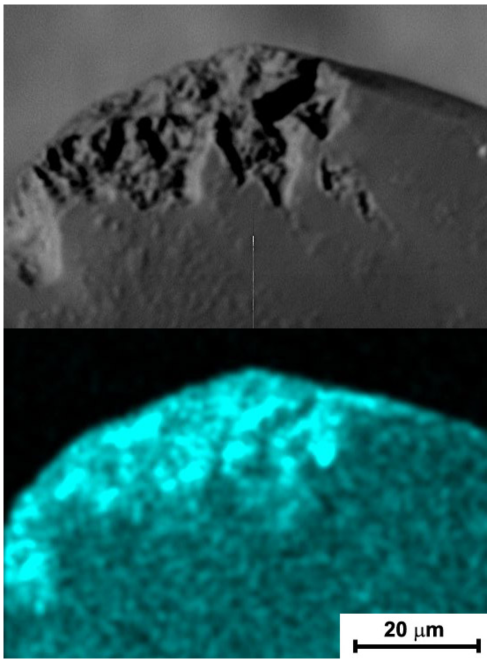

Figure 2. Upper panel shows both the surface and the cross-section of sample after EDM (SEM, secondary electrons). Lower panel shows the spatial distribution of carbon (light areas) (SEM, EDX analysis).

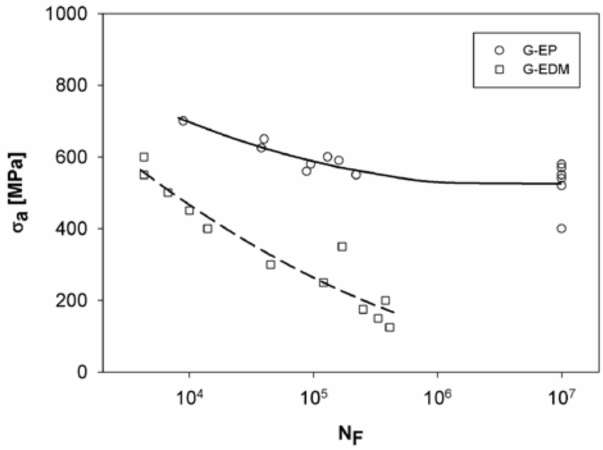

Figure 3. High cycle fatigue (HCF) performance of commercial material with globular (G) microstructure. EP refers to benchmark electrolytically polished samples and EDM denotes the test samples after electric discharge machining.

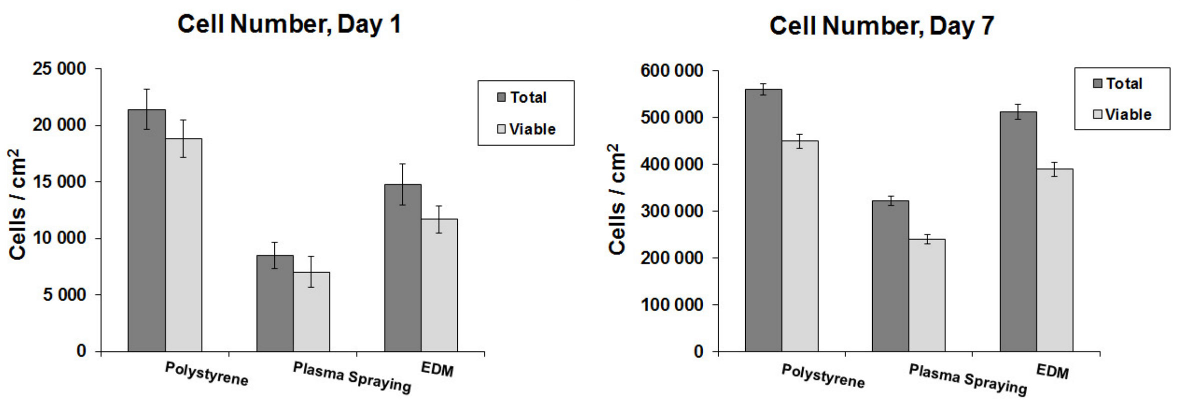

Figure 4. Number of human osteoblast-like MG 63 cells on day 1 and 7 after seeding on standard cell culture polystyrene dishes Ti-6Al-4V alloy modified by plasma-spraying with TiO_2 and test samples modified by electric discharge machining (EDM). Total number and number of viable cells are evaluated.

intervals than on the plasma-sprayed surface. This result suggests that EDM treatment seems to be a suitable modification which makes the material more attractive for colonization with bone cells than the conventionally used plasma-spraying. Higher attractiveness of EDM for cell colonization could be attributed to a more advantageous shape and organization of the microscale irregularities on the material surface. Another possible cause is the formation of carbon layer on the EDM-treated surfaces. In earlier studies, carbon-based layers, such as amorphous hydrogenated carbon, also referred as diamond-like carbon [14], pyrolytic carbon [15] or hydro-carbon plasma polymers [16] often acted as good substrates for the adhesion and growth of bone-derived cells.

Conclusion

Ti-6Al-4V alloy was surface treated by electric discharge machining (EDM) proposed for application in orthopaedics. The following conclusions may be drawn from this investigation:

- EDM with high peak current of 29A induces substantial surface roughness.
- EDM results in a slight decrease of the strength, while the elongation to fracture decreases significantly.
- Surface remnants and subsurface martensitic layer have been observed by means of SEM.
- Carbon enrichment of surface is well-documented by employing EDX analysis.
- Detrimental effect of EDM on fatigue performance has been found. Poor fatigue endurance is the main limit for potential application and fatigue enhancing processes should be investigated.
- Samples modified by the EDM provide better substrate for the adhesion and growth of human bone-derived cells than the alloy plasma-sprayed with TiO_2.

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