Potential of silicon powder-mixed electro spark alloying for surface modification of Titanium alloy for orthopedic applications

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Abstract

Among titanium alloy is the best choice to produce orthopedic implants, because of their excellent mechanical properties. The bone ingrowth and osseointegration around the implants depend on the surface chemistry and surface topography. Therefore, implant's surface needed mechanical treatments to increase its bioactivity and to mimic the surface morphology like natural bone. A novel approach powder mixed electric discharge machining (PMEDM) used for surface modification of Ti alloy in the present research study. A determined concentration of silicon powder is mixed into the dielectric fluid of EDM. The effect of silicon powder with other working parameters like peak current (I_P), pulse duration (T_{on}) and duty cycle (τ) on the surface characteristics of β-phase Ti alloy such as topography, surface roughness, recast layer thickness, micro cracks, and micro-hardness has been investigated. The machined surfaces were characterized using field emission-scanning electron microscopy (FE-SEM), energy dispersive X-ray spectroscopy (EDS) and X-ray diffraction analysis (XRD) and formation of oxides and carbide phases with micro/sub-micro/nano-scale porous morphology was confirmed.

Keywords

titanium alloy, surface modification, powder mixed EDM, nanoporous surface, biocompatibility, orthopedic applications

1. Introduction

*T*itanium alloy aroused more scientific interest for the production of orthopedic implants in present research scenario, because of their superior biocompatibility and excellent mechanical properties [1]. Although have excellent mechanical properties, they failed to crate bone regeneration and healing around implant because of its un-active surface [2]. The main event after implantation includes adaptation of surrounding tissues and start bone formation by the biological self healing process. Therefore, surface modification required to maximize bioactivity or bone-implant bonding ability [3]. Thus, numerous surface treatment/ modification techniques have been attempted in past for better stability and fixation of implant [4]. Previous studies demonstrated that the formation of porous layer of titanium oxide (TiO_2), zirconia (ZrO_2), titanium nitride (TiN), niobium pentaoxide (Nb_2O_5), titanium carbide (TiC), composite ceramic coating like TiO_2-SiO_2, hydroxyaptite (HaP) on implant surface increases bioactivity and biocompatibility by different techniques, chemical, electrodeposition, chemical vapor deposition (CVD), physical vapor deposition (PVD) and Sol–gel etc [3]. Even so, their application is limited by difficulties such as poor adhesion, uncontrolled coating layer thickness, low physical bond strength at the interface and enable to serve under cyclic loading condition [5]. I order to develop biocompatible surface, the potential of electric discharge machining (EDM) has been adopted to modify the surface of metallic implants [6-7]. A nano-porous TiO_2 layer has been created on Ti-6Al-4V alloy to enhance its biocompatibility [8]. Further, Lee et al. and yang et al. have demonstrated biocompatible TiO_2 layer provides a better vehicle for cell growth to the specins surface [9-10]. Bin et al. explored the capability of electro-spark alloying on the Ti surface by EDM to form titanium carbide (TiC) having a high surface hardness and wear resistance [11]. The carbide based surface found a favorable influence on cell attachment and proliferation. A potential application of EDM was reported to fabricate rough biocompatible surface for orthopedics application [12]. The authors reported that carbon enriched highly macro-roughness has a favorable and better surface for adhesion and proliferation of human osteoblast-like MG-63 cells as compared with the sample prepared by plasma sprayed with TiO_2. Harcuba et al. reported that high surface roughness leads to obvious surface crack which induce poor fatigue performance may cause clinical failure [13].

EDM has many downsides like high surface crack density, high surface roughness, low efficiency, producing deep and wide craters; thus resulting different kinds of surface and sub-surface defects in the machined part which restricts further application [14]. Numerous studies have focused on the enhancement of EDM performance, especially in areas of surface modification [15]. Recent studies have documented the beneficial effects of PMEDM on the generation of better surface quality and reduction of surface cracks.

Fine conductive/semi-conductive powder is mixed into the dielectric fluid of EDM [16]. This process is termed as powder mixed EDM (PM-EDM) process [17]. Recently, potential of PM-EDM was acknowledged for surface modification by alloying of suspended powder particles from dielectric fluid on substrate surface with the aim of enhancing surface properties. This mechanism is called as "electro spark alloying" or "electric discharge coating (EDC)". Furutani et al. proposed the accretion of TiC by Ti suspended powder in EDM oil [18]. Janmanee et al. proposed a fabrication of hard layer of TiC on workpiece surface by EDC [19]. Chen et al. proved that the hardness of Al alloy was improved by EDC [20]. Arun et al. proposed the coating of nickel–tungsten by electro spark alloying from suspended Ni and W powder particles [21].

Their research created a new idea to modify the surface of implants material for biomedical applications. For the purpose of overcoming the limitations of the EDM alone, a simple, fast and low cost method silicon powder-mixed electro spark alloying (SPM-ESA) was developed to enhance the surface hardness and biocompatibility of β-phase titanium alloy in this paper. A composite layer of bioceramic oxides and carbides (TiO_2, Nb_2O_5, ZrO_2, SiO_2, NbC, SiC and TiC) on β-phase titanium alloy based implant was fabricated. However, there are no reports focusing on the effect of fabrication of nano-structured Nb_2O_5, Zr_2O and SiO_2 morphology with with micro-, submicro- and nano-scales surface porosity on biocompatibility of β-phase Ti alloy by PMEDM methodology. Therefore, the present study is the first of its kind to modify and to produce the nanoporous structure on β-phase Ti alloy by PMEDM for orthopedic applications.

2. Material and method

2.1. materials

The β-phase Ti alloy (53Ti–35Nb–7Ta–5Zr) of nominal chemical composition shown in table 1 was chosen as substrate surface. The specimens of 100 mm x 30 mm x 10 mm were cut from the cast ingot by wire-cutting EDM (W-EDM). Before PMEDM, the surface of specimens were grounded to a surface roughness of Ra 0.5 µm, and rinsed with ethanol and acetone. A commercial available pure Ti alloy (99%) rod with a diameter of 5 mm was used as the tool electrode. Pure silicon powder (99.7 wt. %) with particle size of <50 µm has been mixed into the dielectric fluid of EDM. The SEM micrograph of powder particles confirmed the shape and size as illustrated in Fig. 1.

2.2. Experimentation

The experiments are performed on die-sinking EDM machine (model SZNC-35-5030) made by Sparkonix (India) Pvt. Ltd., Pune. Fig. 1(a) illustrates the EDM equipment. A new experimental system for SPM-ESA was designed and fabricated as shown in Fig. 1(b-c). Considering the literature review survey, the process parameters such as peak current, pulse duration, pulse interval and powder concentration at tool electrode at negative polarity have favorable for the electro spark alloying or coating of foreign material on substrate surface. Fig. 2 illustrates the schematic diagram of silicon powder mixed electro spark alloying (SPM-ESA). The principle SPM-ESA is the mechanism of surface modification of substrate surface and has been used to the surface characteristics like surface hardness, wear performance and corrosion resistance Because of new material (*β*-phase Ti alloy), before conducting experiments pilot experiment have been performed to identify the range of input parameters. In order to investigate the effect of individual parameters, the experiments were conducting in various (i) concentration of silicon powder particles (0-12 g/l), (ii) peak current (5-25 A), (iii) pulse duration (5-100 µs), (iv) duty cycle (8-72%) at negative tool polarity (-ve). The effect of these process parameters on surface roughness, surface morphology, phase composition and mechanical properties (micro-hardness) has been investigated.

2.3. Characterization of machined surface

The β-phase Ti alloy after PMEDM Machining was examined by various surface characterization techniques. The workpiece surface of machined samples was qualitatively examined using a field emission-scanning electron microscope (JEOL 7600F FE-SEM with EDS). The quantitative evaluation of element mapping is done by energy dispersive X-ray spectroscopy (EDS). X-ray diffractometer was used for phase identification. The incident angle was set two degrees. X-ray diffractometer with Cu Kα radiation was operated at 45kV and 40mA. The measurement of average surface roughness (Ra) is taken MITUTOYA surface roughness tester equipment using cut-off length of 0.08 mm and evaluation length of 0.4 mm. Measurement was repeated three taken to eliminate the measurement error. Micro hardness (HV) measurements were made on a MITUTOYA (model HM-125) micro hardness tester using an indentation load of 2.94N for holding time 10 seconds. Five hardness readings were recorded on each sample in a straight line from the recast layer to the base material.

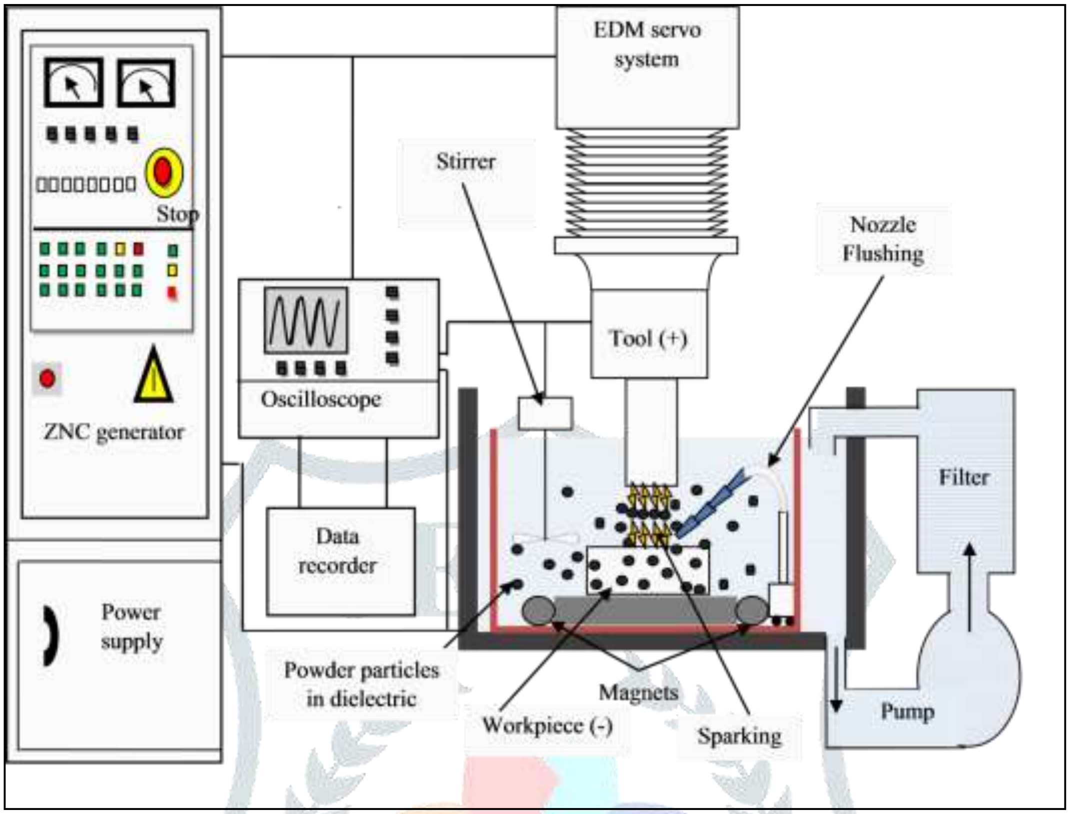

Figure 1. PMEDM Set-up

3. Results and discussion

3.1. Characterization for machined surface integrity

It is well known fact that the surface integrity of machined surface is a function of discharge energy, which is controlled by input parameters of EDM Process. The surface morphology of the EDMed and SPM-ESAed samples is shown in Fig. 3. The Figure shows the effect of duty factor on surface morphology of the machined surface. There was notable difference in the surface morphology between samples using EDM and SPM-ESA. The results shows that the larger and deep craters formed on surface that leads to higher surface roughness using EDM than those obtained using SPM-ESA (Fig. 3a). Moreover, higher surface cracks density around crater periphery and volcano like structure was observed on the EDMed surface (Fig. 3b). Whereas, relatively smooth surface morphology was obtained using SPM-ESA as compared to EDMed surface as can be seen in Fig. 3c. However, the volcano like structure was generated but cracks free. The re-solidified spalled cleave layer of molten metal, and chimneys formed by escaping of entrapped gases from the redeposit material was observed (Fig. 3d). The recast layer clearly evident the formation of oxide and carbide phases on the machined surface. Fig. 4 shows the SEM micrographs of machined surface at 8% duty factor EDM and SPM-ESA. The evidences of silicon elements, lumps (agglomeration) formation and poke like structure could be visualized when machining is performed at high concentration (8g/l) of powder particles as can be seen from Fig. 3a. By virtue of strong electric field, the silicon powder particles energized and get migrated toward the workpiece surface and penetrate into the molten pool before solidification [22]. This migrated layer of silicon particles collapses with carbon and oxygen (decomposed from dielectric) to form a thin layer oxides and carbides. This oxides and carbides will penetrate the molten pool before resolidification and fabricate a combined micro-/submicro- and nano-scale surface topography. On the other hand, at positive polarity a well-defined network of porous structure representing small pokes were observed on the top surface of the sample (Fig. 3c & d). The micro-, submicro- and nano-scale surface porosity and foamy like structure was observed on the top layer of machined samples of β-phase Ti alloy. A close examination shows that the surface obtained with machining of 8g/l Si powder mixed in EDM for longer pulse interval (8 % duty cycle) at negative polarity have a high pore density than the one obtained with positive polarity as can be seen in Fig. 3(b) and (d). This is because, at the larger pulse interval, the entrapped gases got sufficient time to escape from the molten pool. As a result, porosity builds on the top machined surface in larger density compared to the samples machined with a shorter pulse interval. Besides the ejection of gases, the grain pulls out from the host surface leave pits on the machined surface, causing micro-, submicro and nano-scales surface feature and results in an increase the biocompatibility of the surface. This represents the formation of nanopores on β-phase Ti alloy by PMEDM process.

It is clear that the workpiece surface after machining at positive polarity with silicon powder mixed EDM is smoother as compared to without silicon powder as seen in Fig. 2a. A highly magnified view shows the machined surface with thin redeposited overlapping layer and globules of debris melted from electrode. Moreover, cracks are not visible on the machined surface. On the other hand, very smooth surface free from cracks was observed on the machined surface with positive polarity. The machined surface has less melted drops due to good flushing. It was concluded that in EDM process, the material from workpiece surface was removed with large spatters causing very large craters; while with the addition of silicon powders into the dielectric fluid of EDM resulted in a decrease in the dimensional characteristics of the produced craters. With the addition of conductive powder particles into the dielectric fluid, the distance between tool and workpiece known as 'spark gap' has been increased; consequently the thermal energy generated by electrical sparks reduces and less material melts and evaporates from the workpiece surface. As a consequence, the size of craters formed over the workpiece surface decreases. Due to the enlarged gap between the tool and the workpiece, the eroded debris is flushed out more easily. The adhesion of resolidified debris on the machined surface is reduced, which results in a better surface topography.

4. Conclusions

In the present research work, an innovative method for fabrication of a nano-porous surface on β-phase Ti alloy by PMEDM has been reported. The effect of Si powder addition on the surface chemistry, surface topography, surface roughness, surface crack density and surface hardness was investigated. The presence of surface cracks, volcano-like re-solidified spalled cleave layer of molten metal and chimneys like surface was observed on the β-phase Ti alloy in the case of EDM process. With the addition of Si powder however, the crater size reduced and significantly improve the surface integrity. The density of surface cracks significantly decreased when Si powder used as dielectric additive. No evidence of regular formation of micro cracks observed when machining is performed at concentration of Si powder at 8 g/l Si concentration and longer pulse off-time for any value of current and pulse duration.

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