Application of Large Area Electron Beam Irradiation for Surface Modification of Implant Materials

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Abstract – A new surface finishing process for implant materials of bio-titanium alloy with large-area electron beam (LAEB) irradiation is proposed. In LAEB irradiation equipment, high energy density electron beam can be generated without concentrating the beam. The LAEB provides about 60 mm of effective irradiation diameter, which can be used for instantaneous melting and/or evaporating of metal surface. Experimental results show that the surface roughness becomes smaller with increasing beam energy density. With a proper irradiating condition, surface roughness of titanium alloy implant machined by endmill, can be decreased from 10 μmRz to 0.8 μmRz in a few minutes. After the irradiation, about 3 μm re-solidified layer is formed on the surface and corrosion resistance can be greatly improved. The electron beam irradiation owns a possibility to be applied as new method for high efficient surface smoothing and surface modification of bio-titanium implant materials.

1. Introduction

Medical implant products such as artificial joints, artificial bones, fracture fixing plate, and so on, usually have very complex shapes. Most of them are made from titanium alloy, stainless steel, and cobalt-chromium alloy, because not only high strength but also high corrosion resistance is required to keep the human body safe. In general, those products are firstly machined by multi-axis milling or formed by lost wax casting. After that, surface finishing is processed with hand lapping in order to attain small surface roughness without defect layer. However, this process takes long time and needs special technical skills. Therefore, automatic high efficient process is strongly demanded.

In this study, a new surface finishing process for biomaterials with large-area electron beam (LAEB) irradiation is proposed. The LAEB irradiation equipment used here was recently developed for surface treatment or surface modification [1–3]. In this method, electron beam with high energy density can be generated without concentrating the beam. The LAEB with maximum diameter 60 mm can be used for melting and/or evaporating metal surface, and electron beam irradiation is carried out in a series of pulses.

In this report, the application of LAEB irradiation technique for surface finishing of bio-titanium alloy products is experimentally discussed. Moreover, the corrosion resistance of the irradiated surface is evaluated.

2. Experimental Procedures

Machining conditions are shown in Table 1. The EB irradiation duration time of one pulse is 2–3 μs, and the pulse frequency is 0.2 Hz. The effective EB diameter is 60 mm as mentioned above. At first, an optimum condition for surface smoothing is investigated by changing the energy density. As the workpieces, specimens made from titanium alloy (Ti-6Al-4V) are prepared. The titanium alloy surfaces are beforehand cut using ball endmill of 10 mm in diameter. Three kinds of workpieces with different surface roughness (5, 10, 20 μmRz) are prepared with different tool path pitch.

Table 1. Large-area electron beam irradiation conditions

Energy density Ed	**1.2 - 9.6 J/cm^2**
Number of pulse N	**30 shots**
Pulse duration τ	**2~3 μs**
Pulse frequency fp	**0.2Hz**
Beam diameter	**60 mm**

3. Results and Discussion

Optimum condition for surface smoothing is investigated by changing the beam energy density. Figure 1 shows SEM micrographs of the EB irradiated surface for various electron beam energy densities. The pulse number of irradiation is set to 30 shots for each energy density condition.

The surface before irradiation is also shown in Fig. 1 for comparison. Under small energy density condition of 1.2 J/cm^2, the surface melting can be confirmed obviously and it seems that the surface becomes smoother compared to the surface before irradiation. Furthermore, under larger energy density condition, the surface morphology completely differs from the surface before EB irradiation.

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Roughness profiles are shown in Fig. 2. In the case of before EB irradiation surface, the profile corresponds well with the tool path of machining. However, after EB irradiation the surface roughness becomes smaller with increasing energy density. At sufficiently large energy density, relatively smooth surface can be obtained.

Figure 3 represents graphically the variations of surface roughness and the glossiness with the energy density. The measurements of glossiness were carried out in accordance with JIS Z8741, in which, the glossiness of the perfect mirror surface is defined as 1000. In the case of surface roughness before irradiation Rzo is 10 μmRz, the roughness becomes smaller with increasing the energy density, and comes to minimum value of about 0.7 μmRz at 7.8 J/cm^2. On the other hand, the glossiness value becomes higher with increasing of energy density. The change of glossiness corresponds well to the change in the surface roughness. It means that very high efficient surface smoothing can be attained with LAEB irradiation, because the processing time under this condition of 30 shots takes only 150 sec.

Moreover, comparing surfaces with different surface roughness before irradiation (Rzo), the surface roughness becomes smaller with increasing the energy density also for the cases of Rzo = 5 and 20 μmRz. The minimum surface roughness value is also about 0.7 μmRz in the case of Rzo = 5 μmRz. Therefore, the initial surface roughness up to about 10 μmRz can be decreased to less than 1 μmRz in just a few minutes by the LAEB irradiation. On the other hand, when the

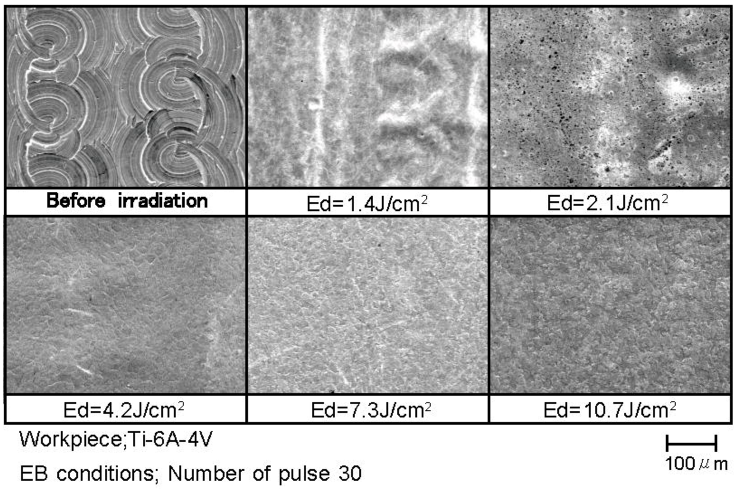

Fig. 1. LAEB irradiated surface for various energy densities of electron beam

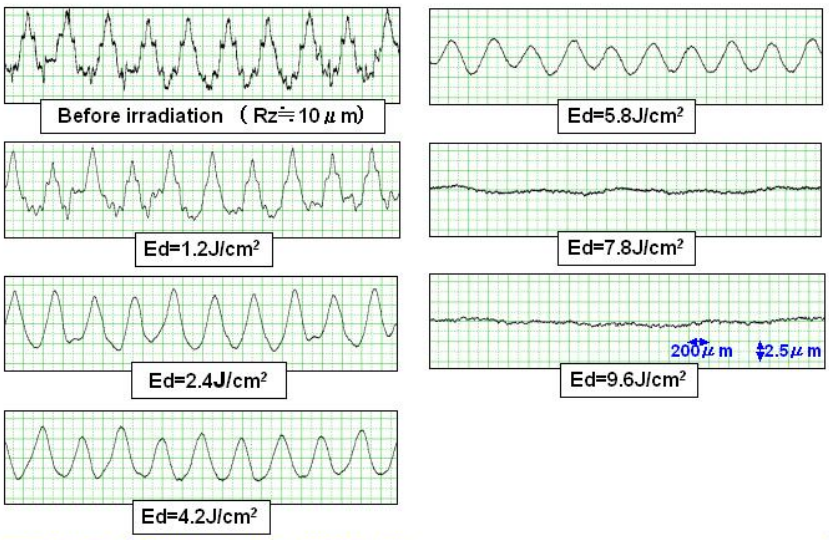

Fig. 2. Roughness profiles of LAEB irradiated surfaces

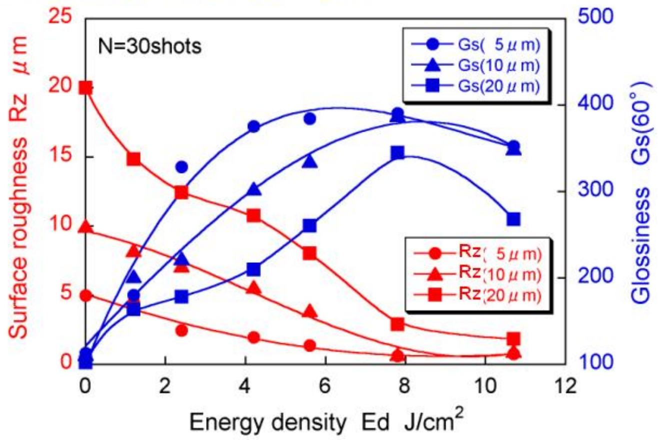

Fig. 3. Variations of surface roughness and glossiness with energy density

surface roughness before irradiation is 20 µmRz, the minimum value was about 2 µmRz under the conditions of N = 30 shots. However, it can be predicted that the roughness may become smaller under larger irradiation number.

Figure 4 shows the cross sections of the surface before and after LAEB irradiation. Ti-6Al-4V alloy has α-phase (white) and β-phase (black), and they can be seen clearly in the matrix. Surface smoothing can be confirmed by comparing the both surface undulations. From the SEM picture, thin re-solidified layer is observed, and the thickness is about 3 µm. In this EB irradiation system, EB irradiation time of one pulse is 2–3 µs. Therefore, it can be assumed that the effect of heat conduction on the surface is very small, and the heat energy could not transfer into the inside of material. The material modification through melting and evaporation occurs just near the surface.

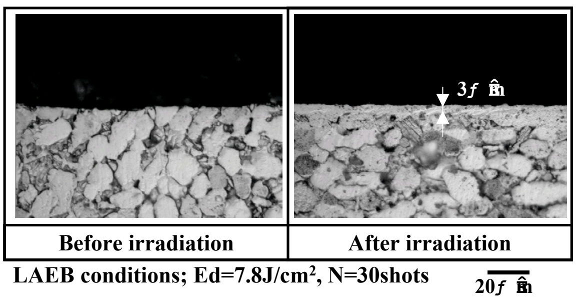

Fig. 4. Cross sections of surfaces before and after irradiation

Figure 5 shows Vickers hardness of the surfaces before and after LAEB irradiation. The hardness of Ti-6Al-4V alloy is about 340 Hv. The hardness of LAEB irradiated surface is almost the same as before irradiation in any cases for Rzo = 5, 10, 20 µmRz. In can be concluded that, for the titanium alloy, hardness does not change much with the LAEB irradiation.

To observe increase of corrosion resistance after EB irradiation, anodic polarization current measurement is conducted with an electrochemical analysis system shown in Fig. 6. The system consists of potentiostat with linear sweep voltametry (LSV), electrochemical reaction cell, reference electrode cell, and salt bridge. Anodic polarization current is measured when the voltage between counter electrode and work is increasing at constant potential scan rate using LSV.

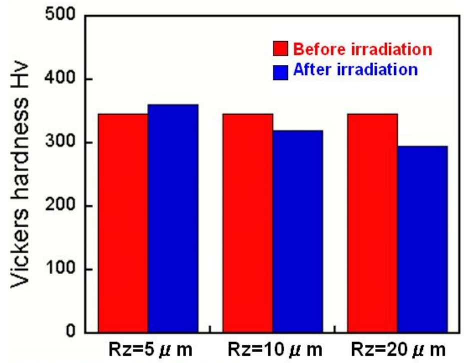

Fig. 5. Vickers hardness of surfaces before and after LAEB irradiation

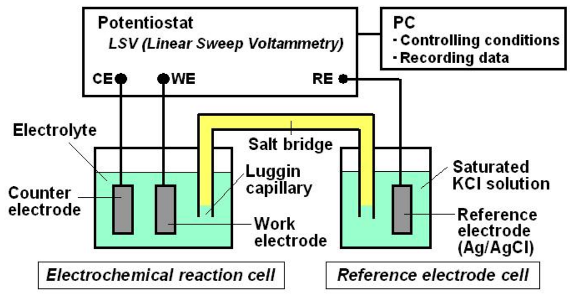

Fig. 6. Electrochemical analysis system for measuring anodic polarization curves

First, considering actual use in human body, Ringer's solution (artificial body fluid) was used as an electrolyte. However, as the result, the anodic polarization current was not detected up to 2 V in both cases of LAEB irradiated surface and no irradiated one, since Ringer's solution does not have high corrosiveness. Then, high corrosive 1N-HCl solution is used as electrolyte.

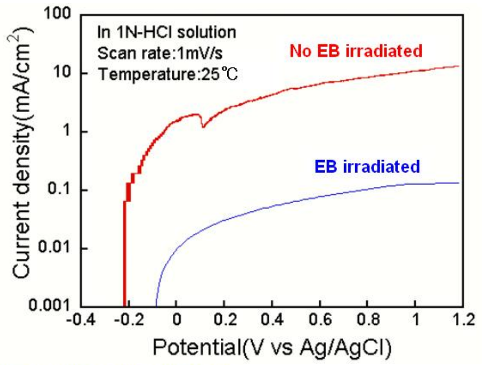

Fig. 7. Anodic polarization current curves using 1N-HCl solution

Figure 7 shows the anodic polarization current curves using 1N-HCl solution at temperature of 25 °C. The equilibrium potential for LAEB irradiated surface is smaller than that for no irradiated one. Also the current density for LAEB irradiated surface is much smaller than that of no irradiated one at any potential. Therefore, it is clear that the corrosion resistance of titanium alloy surface could be greatly improved by LAEB irradiation.

4. Conclusions

In this study, a new surface finishing process for bio-titanium alloy implant with LAEB irradiation is proposed, and the effects of irradiating conditions on the surface roughness and the surface modification are experimentally investigated. Main conclusions obtained are as follows.

1. Roughness of LAEB irradiated surface becomes smaller and the glossiness becomes higher with increasing the beam energy density.
2. Under proper irradiating condition, initial surface roughness about 10 μmRz can be decreased to less than 1 μmRz in just a few minutes by the LAEB irradiation. Therefore, very high efficient surface smoothing can be attained.
3. Thin re-solidified layer was formed on the LAEB irradiated surface for bio-titanium alloy. Moreover, the corrosion resistance can be improved.
4. LAEB irradiation method owns a possibility for high efficient surface smoothing and surface modification of bio-titanium alloy implants.

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