Surface roughness analysis of NiTi alloy in electrical discharge coating process

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Abstract. Nickel-Titanium (NiTi) alloys, most widely known as nitinol, are presently employed in many micro-engineering applications such as coronary stents of medical implants due to their unique properties (shape memory effect and superelasticity). However, non-optimized surface finishing attributed a significantly high potential of nickel exposure after a long time of application. Releasing of nickel ion to the body environment can be harmful and toxicity resulting in adverse health as well as degrading the material biocompatibility. It is widely known that controlled surface roughness play a vital role in the formation of new bone ingrowths around implant. In this study, surface modification of NiTi alloy was used through electrical discharge coating (EDC); an adaptation of electrical discharge machining. The potential of EDC in which can facilitates the production of hard coatings may exploit the phenomena for the attachment of desirable materials onto the surface of materials. Therefore, the aim of this paper is to present a robust method (two levels of full factorial design and ANOVA) to determine the desired parameters and significant factors based on the surface roughness of the machined surface. Manipulation of parameters set up such as gap voltage, discharge duration and pulse interval and the current were employed and a reverse polarity was selected for this experiment. The results demonstrated that the most significant factors influence the surface coating performances are the discharge duration, current, gap voltage as well as the interaction between gap voltage and discharge duration.

1. Introduction
Electrical discharge coating (EDC) is an adaptation of electrical discharge machining (EDM) which can deposit high melting points materials, such as hard-wearing, electrically conductive ceramics, onto a substrate. Regardless of their mechanical properties of hardness toughness and strength, EDM is considered as a non-contact process machining [1]. EDC phenomena demonstrate the workpiece being held at the positive terminal and electrode is held at the negative terminal. Then, the sparks leading to the development of localised melt pools on the surface of the workpiece that combine with the element present in the sparking gap from the tool. Rapid cooling of the particles forming localised cermet deposits later allowing multiple, overlapping craters formation to form a continuum coating on the surface of the workpiece [2]. Based on above reasons, EDM process has promoted excellent interest for its novel applications emphasis on the potential of the surface modification also known as EDC. Surface modification can be conducted prior to the formation of recast layer that can adhere well to the substrate due to the metallurgical bonding that exists between them during coating process [3].

Over the years, nickel-titanium (NiTi) based alloy has been extensively used in biomedical application especially in a bone-implant application such as dental implant and bone tissue
engineering. The NiTi are particularly attractive due to their specific properties of shape memory effect (SME) and superelasticity (SE) which fits the stress-strain behaviour of human bone and tendons, makes it an excellent material to meet some of the challenges presented in biomedical implants [4]. However, the limitation of NiTi implantation is the harmful Ni ion release to the body. This, in turn, NiTi alloy implantation cannot highly induce the response of the bone-forming cell or effectively integrate with the surrounding bone tissue. This is leading to the need to modify the NiTi surface specifically via EDM to enhance the biocompatibility by reducing the nickel ion release then enhancing the osteoblast response of NiTi alloy [5].

This paper explores effect of EDC parameters impact the coating performance in terms surface characteristics. Based on literature, the desired surface characteristics of the material can be controlled by selecting appropriate operational parameters of EDC and electrode materials [6,7]. Prasanna et al [8] aimed at optimizing the surface quality through EDM coating in their study by varying the current, discharge duration, pulse interval and gap voltage. It was found that when current is increased, the surface finish was reduced. Chakraborty et al [9] also stated in their study that the increase current and discharge duration will increase the deposition rate and formation of craters making and increase in surface roughness.

Above all, there are many researchers have conducted experimental investigations on surface roughness for SME material surface modifications via EDM coating. Prakash et al [10]–[12] highlights how controllable surface roughness is desired since the porosity characteristics has a favourable impact on cell adhesion, proliferation and a new bone formation. However, there are limited information is available regarding the surface roughness of medical devices for NiTi alloy especially for biomedical application purposes. On top of that, other researchers have not systematically investigated the study on surface roughness specifically by EDC process. Therefore, the purpose of this study is to employed design of experiment (DOE) and ANOVA analysis in selecting the optimum parameter and identifying significant factors based on surface roughness response for surface modification of NiTi alloy. Moreover, the regression model was developed in this study based experimental parameters selected.

2. Material and methods

2.1. Equipment, workpiece and tool electrode materials

The experimental setup for surface modification of NiTi alloy utilised an EA8 Mitsubishi EDM die sinker. The machine was equipped with an internal circulation system with the liquid capacity of 250L as shown in Figure 1. The EDC system used with deionised water as a dielectric fluid that was contained in a fabricated reservoir. The workpiece of nickel-titanium alloy was provided by Baoji Hanz Material Technology Co., Ltd, China in dimension size of $70 \times 70 \times 5$ mm. The samples have a standard specification of medical grade that corresponds to ASTM F2063-12. Meanwhile, pure Titanium with medical grade two was selected as a tool electrode in the form of cylindrical rod with $\emptyset 10$ mm $\times 10$ mm. At first, the samples were cut into desired dimensions, then they were grinded with 800, 1000 and 2000 grit of SiC papers until mirror finish. Next, the surfaces were polished by using monocrystalline diamond suspension. Lastly, etching process were conducted by using Kroll’s reagent (6 ml of HNO$_3$, 2 ml of HF and 92 of distilled water).

2.2. Experimental design/procedure

The experiment was performed to a systematic design layout by Design of Experiment (DOE) in $2^k$ factorial design. Four parameters were varied in this research paper; gap voltage, discharge duration, pulse interval and current. The design of experiment was a $2^5$ factorial design with three centre points of replications. Thus, the total number of trials were 19 runs. Table 1 shows the parameters settings during this study.
Meanwhile, the experimental conditions that was set up for the machining processes is outlined in Table 1. Essentially, surface modification of NiTi alloy are very amenable to production in which the process should underlines high accuracy and appropriate surface roughness (SR). The measurement data by means of measuring the surface roughness for the prepared samples were analysed via analysis of variance (ANOVA) in mean arithmetic (Ra) values. Handysurf E-35B portable surface roughness tester with stylus size of 2 µm/60° and 0.08mm cut-of-length. Five points area on the machined surface were measures and an average values were taken for analysis stage.

| Parameters          | Symbol | Factor level |
|---------------------|--------|--------------|
| Discharge duration, (µs) | A      | 50 295 540   |
| Pulse interval, (µs)  | B      | 6 7 8        |
| Current, (amp)       | C      | 80 170 260   |
| Gap voltage, (volts) | D      | 0 3 6        |
| Polarity : Reverse   |        |              |
| Machining duration (mins) : 180 minutes | | |
| Jump : OFF           |        |              |

3. Results and discussion
Prior investigations have implemented DOE of full factorial for surface modification of NiTi alloy in order to accomplish better understanding on the effect of discharge duration, pulse interval, current and gap voltage towards the performance of EDC.

3.1. Surface roughness
The effect comparison between parameters on the surface roughness is outlined with ANOVA table as shown in Table 2. These average values were calculated from the number of Ra readings measured according to procedure explained in section 2.0. Table 2 displays the ANOVA analysis results for surface roughness based on 95% confidence level. It is apparent that there are three factors namely; discharge duration, current and gap voltage have, expectedly, the strongest effect on surface coating quality (Ra). Resulting from the analysis, F-value of the factors is higher than critical Fisher ratio, $F_{(0.05,1,13)} = 4.67$. In this study, discharge duration, current, gap voltage, and interaction between discharge current gap voltage are found to be significant model terms. Nevertheless, factor B which is pulse interval has found to be insignificant in this study.
It is well established to use the multiple regression analysis (MRA) as a representation the relationship between surface roughness and variables of the coating parameters. The correlation between factors (discharge duration (A), current (C), and gap voltage (D)) and the surface roughness (Ra) for surface modification of NiTi alloy via EDC was achieved by regression model, Eq. 1. The R-squared of this model was 0.96 while the adjusted R-squared was 0.95 thus confirming the reliability and accuracy of the experimental data was within the acceptable range. Likewise, ANOVA results for the model as tabulated in Table 2, draw similar conclusion. Moreover, there is only 0.01% of chance that error existed in the model due to noise. F-value of 3.22 for “Lack-of-fit F-value” implies that the lack of fit is not significant relative to the pure error. There is a 26.07% chance that a lack of fit F-value this large could occur due to noise. Non-significant lack of fit is considered fit and acceptable which implies that the model is sufficiently fit.

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SR(R_a) = 1.70063 + 0.33362\text{(discharge duration)} + 0.43750\text{(current)} - 0.37837\text{(gap voltage)} + 0.030542 \text{(discharge current \times gap voltage)}
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(1)

Table 3 shows the effect list of surface roughness. It appears that the governing factors for Ra are discharge duration (A) that contribute 69.4% as well as 1.43% for interaction between AD (discharge duration and gap voltage). Figure 2 represents the interaction between those two factors. It can be observed that the correlative interaction between discharge duration and gap voltage where surface roughness increases as discharge current along with gap voltage increased in the range between 1.86 µm to 6.28 µm.
A previous study [13] has also found that gentle increase in the formation of craters on the machined surface is noticeable as current and discharge duration increases. It is worth highlighting that, the amount of tool-electrode material deposited on the machined surface is directly proportional to the amount of applied energy (regulate from the discharge duration and peak current) [14]. Therefore, surface roughness can be controlled by adjusting these two significant factors (discharge duration and gap voltage) to obtain a quality coating surface. Prior research by Shy Feng, (2016) has also clarified that surface roughness was increased with the increase in current and discharge duration. This is likely due to the generating sparks and struck the workpiece as the impact force repelled the molten material allowing the deep craters to remain on the surface. The high-energy discharge would also generate more heat in the coating zone, and influence the coating performance (Ra). According to in vivo and in vitro tests, the moderate surface roughness of implant is about 1-2 µm, and the range is also commonly employed in oral implant [15]. Therefore, it can be concluded that the setting Discharge duration 50 µs, Pulse interval 8µs, Current 80amp, Gap voltage 6 volts exhibits 1.87 µm are the desired coating parameters in achieving the requirement of implant application.

4. Conclusion
In conclusion, the present experimental investigation resulted in substantiating the possibility of modifying the surface of NiTi alloy under different setting of electrical discharge coating (EDC) parameters. Based on the two levels full factorial design with ANOVA results, EDC can be used to improve the surface characteristic of NiTi alloy or notinol for biomedical applications. The discharge duration is the dominant factor for surface roughness following by current and gap voltage. For achieving the implant requirement, lower discharge duration and current are preferred.

5. References
[1] Algodi S J, Murray J W, Clare A T, and Brown P D 2018 Procedia CIRP 68 28–33
[2] Algodi S J, Murray J W, Fay M W, Clare A T, and Brown P D 2016 Surf. Coatings Technol. 307 639–649
[3] Huang T S, Hsieh S F, Chen S L, Lin M H, Ou S F, and Chang W T 2015 J. Mater. Res. 30 3484–3492
[4] Mohd Jani J, Leary M, Subic A, and Gibson M A 2014 Materials and Design 56 1078–1113
[5] Jahan M P, Kakavand P, and Alavi F 2017 Procedia Manuf. 10 232–242
[6] Gill A S and Kumar S 2015 Mater. Today Proc. 2 1723–1730
[7] Öpöz T T, Yaşar H, Ekmekci N, and Ekmekci B 2018 J. Manuf. Process. 31 744–758
[8] Prasanna P, Sashank T V S S P, Manikanta B, and Aluri P 2017 Mater. Today Proc. 4 8517–8527
[9] Kar S, Chakraborty S, Dey V, and Ghosh S K 2017 J. Inst. Eng. Ser. C. 98 607–618
[10] Prakash C, Kansal H K, Pabra B S, Puri S, and Aggarwal A 2016 Proc. Inst. Mech. Eng. Part B J. Eng. Manuf. 230 331–353
[11] Prakash C, Kansal H K, Pabra B S, and Puri S 2017 Mater. Manuf. Process. 32 274–285
[12] Aliyu A A et al. 2017 Adv. Mater. Sci. Eng. 2017
[13] Dhakar K and Divedi A 2017 Mater. Today Proc. 4 5344–5350
[14] Chakraborty S, Kar S, Ghosh S K, and Dey V 2017 Surfaces and Interfaces 7 47–57
[15] Hsieh S F, Lin M H, Chen S L, Ou S F, Huang T S, and Zhou X Q 2016 Int. J. Adv. Manuf. Technol. 86 1475–1485

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