Impact strength of surface treated SS316L wires reinforced PMMA

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Abstract: Stainless steel 316L (SS316L) as a significant biomaterial, their wires were used to support the PMMA matrix. Two simple and low-cost surface pretreatments for SS316L wires were performed to enhance denture impact strength: mechanical scratching (treating SS316L wires with SiC powder inside a rotating container) and electrochemical anodizing. Three mechanical scratching samples for different periods of 60, 90 and 120min were prepared. Anodizing technique conditions were: Ethylene glycol with perchloric acid as an anodizing solution, 15V supplying and graphite rod as an anode. Anodizing process involved three pretreating periods of 15, 20, and 30min. All the prepared samples had dimensions of $65 \times 10 \times 3$ mm. SEM technique showed different morphology nature involved holes, scratches and pores with a density of $10^4/\mu m^2$ and a crack length of 60µm. The PMMA reinforced with scratched stainless steel 316L wire surface for 120 min presented the highest impact strength value (42 kJ/m²) with (450.91%) increment. Anodizing samples showed a fluctuating behavior of samples with enhancing in the impact strength of anodizing wire for 20min of about 26.99 kJ/m², which is still lower than that for scratched samples in average.

Keywords: stainless steel 316L wire; surface treatment; PMMA Impact strength

1 Introduction

Biomaterial acquires advanced rank of researcher attention to enhance the human life expectancy [1]. One of the candidate biomaterials for denture applications is the reinforced polymethyl methacrylate (PMMA) due to its simple molding, good looking and reasonable physical properties [2, 3]. Improving mechanical and biological properties were continued for the last decades to reach the perfect denture, which should be characterized by very good biocompatibility in the human mouth environment, an excellent match with natural tooth appearance, high Young modulus, impact, hardness and flexural strength, good binding with synthetic tooth and reformability [4–6]. Knowing that stress shielding can be created in the jawbone when a high modulus material is used as a dental implant [7, 8]. Owing to its unique properties, such as low density, aesthetics, cost-effectiveness, ease of manipulation, and tailorable physical and mechanical properties, PMMA has gained popularity for a variety of dental applications [9]. Although there are some drawbacks when using PMMA, such as denture fracture due to water sorption and poor impact and flexural strength, ongoing research has introduced a number of modifications to overcome and improve its properties such as its impact strength. Several studies reported improvements of PMMA material properties because of reinforcing with a variety of fibers [10, 11]. PMMA-based biocomposites with the addition of epoxy resins, polyamide, or butadiene styrene have also been reported to improve PMMA impact strength [12]. Some previous literature describes various methods to avoid or reduce the occurrence of denture fractures [13, 14], such as chemical changes in acrylic resin [15, 16], internal inserting of metallic structures [17, 18], and intrinsic fracture prevention. Embedding of fiberglass or fiber carbon and polyethylene [19, 20] or internal stainless-steel wire inserting [21]. Stainless steel is the first requested metal in most industries and applications [22]. Due to the faint cost, accessibility, smoothness of machining and high corrosion resistance, stainless steel 316L (SS316L) is considered as one of the great influencing materials for denture material [23, 24].

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In literature, anodization of ss316L metal was implemented as a working electrode and a platinum metal as the counter electrode in two electrodes cell for photo-electro catalyzing application. The anodizing solution was ethylene glycol containing a dilute solution of % HClO₄, methanol, water, and sulfuric acid and ammonium fluoride. The applied voltage was 40 V for 5 min at RT [25]. Another group was anodized SS316L for using as a substrate for a deposited antibacterial film. SS316L was electropolished in ethylene glycol (EG) solution containing 5 vol% perchloric acid at 20 V for 20 min. The electropolished sheets were used in the second anodization experiment in a 0.3 mol/L sodium dihydrogen phosphate solution at 5 °C and anodized at 30 V for 20 min [26].

The purpose of this article is to prepare a reinforced PMMA by surface pretreated ss316L wires for different biomaterial and denture applications, including unprecedented simple and low-cost surface modification methods (mechanical scratching and anodizing) to enhance the denture impact strength. Furthermore, the reasons that have improved the material properties of PMMA were studied and discussed. The novelty of this work is the use of SS316L wire with the thickness for reinforcing PMMA by a new simple low-cost mechanical method. Also, anodization was used on SS316L wire under conditions.

2 Materials and method

To prepare the denture material, Polymethylmethacrylate PMMA provided from (Superacryl Plus powder, SpofaDental a.s., Jicin, Czech Republic) was used as denture base material. Stainless Steel 316L wire (MOST FX 316L 0.8mm diameter, Warszawa, Poland) was used as reinforcing material. Silicon carbide (SiC) with a particle size in the range 0.2–0.7 mm was used as scratching powder.

A long wire was cut to pieces to form wire samples, each one of 65mm in length. A surface cleaning by ethanol and alcohol followed by distilled water was done for each wire group before and after each pretreatment process. The two pretreatment processes involved scratching and anodizing processes for stainless steel 316L wires. In the scratching process, a cylindrical Pyrex container with dimensions of 10 cm length and 4cm base diameter was used to contain the wires and SiC powder to achieve the scratching process. The wires were placed with the powder inside the Pyrex container, which was fixed strongly to an electric motor to perform the rotation process. The motor was running at 200 rpm for different periods, which are 60, 90 and 120 min. The container was placed horizontally to allow the powder and wires to move freely. The samples of the scratching process were divided into 3 groups. Each group has samples with identical preparation conditions including the same pretreating time. In the anodizing process, an Ethylene glycol electrolyte containing 5 vol% perchloric acid (HClO₄) in 100 ml beaker was used to anodize stainless steel 316L wires under applying 15 V for periods of 15, 20 and 30 min. A graphite rod (China supplies high density edm carbon graphite rod for battery, HUZHOU GREAT STAR TEXTILE CO., LTD. Zhejiang, China) of length of 77 mm and diameter of 1.8mm was used as an anode. Each denture sample had a dimension of (65×10×3) mm fabricated in a dental stone flask. The proportion of PMMA powder to monomer liquid (Respal NF: Heat Curing Resin (Liquid) – Spd, Dentalix, Madrid, Spain) was kept at 9 g/ 3.6 ml for all samples. To prepare the reference sample G1, the dough was installed at once in the flask cavity. Each treated or untreated wire was wetted with the monomer liquid before placing it in the dough. Reinforced samples with untreated and treated wires with different times (60, 90,120, 15, 20 and 30 min), denoted by G2, GM₆₀, GM₉₀, GM₁₂₀, GE₁₅, GE₂₀ and GE₃₀ min respectively, were prepared by putting only one wire in dough. A pressure of 0.5 Ton was applied by a hydraulic press (10t manual hydraulic press machine, Shanghai Ting Wang Science and Technology Ltd. Shanghai, China) on the flask for 5 min press time. The curing process was achieved by heating the flask in water at room temperature to be held at the boiling point for 60 min. Then, samples were grounded by sandpaper of grade size 400, 600 and 1000. The final samples are shown in Figure 1. Scanning Electron Microscopy (SEM) (Inspect S50) was used to

![Figure 1: Reinforced PMMA Samples: G1) reference samples, G2) reinforced with untreated SS316LSi wire, GM₆₀) reinforced with treated SS316LSi at 60 min, GM₉₀) reinforced with treated SS316LSi at 90 min, GM₁₂₀) reinforced with treated SS316LSi at 120 min, GE₁₅) reinforced with anodized SS316LSi at 15 min, GE₂₀) reinforced with anodized SS316LSi at 20 min, GE₃₀) reinforced with anodized SS316LSi at 30 min]
investigate the morphology of the treated stainless steel 316L wire surfaces. Impact strength test Charpy type was adopted to perform impact test by (ISO-179 impact tester N.43-1) tool using a pendulum of two Joules. The sample was positioned horizontally on its ends and struck by a free-swinging pendulum from a fixed height in the center. The impact strength value was measured in kJ/m² by applying the same formula in reference [25].

3 Results and discussion

3.1 Morphological results

SEM test was done to investigate the effect of the scratching process on the forms and dimensions of scratching traces on wire surfaces. Figure 2 shows the SEM images for untreated and treated stainless steel wires. In the image (a), one can see a smooth surface that has a small surface area with few and small dimension cracks that are randomly distributed on the surface of the untreated wire. In the rest of images (b), (c) and (d), it is clear the effect of the scratching

| Sample | Scratching time /min | No. of Cracks in \(10^6 \mu m^2\) | Crack Length /\(\mu m\) | Width /\(\mu m\) |
|--------|----------------------|-------------------------------|---------------------|-----------------|
| a      | 0 (untreated)        | 3                             | 30                  | 0.5             |
| b      | 60                   | 5                             | 50                  | 12              |
| c      | 90                   | 5                             | 60                  | 35              |
| d      | 120                  | 10                            | 60                  | 40              |

Figure 2: SEM images of (a) Untreated SS316L wire surface, (b) scratched SS316L wire for 60min, (c) scratched SS316L wire for 90min and (d) scratched SS316L wire for 120min
Figure 3: SEM images of anodized SS316L for (a) and (b) 15min. (c) and (d) for 20min (e) for 30min, (f) SEM-EDS spectrum with elemental ration of the selected smooth lump area (sample GE 15)
process on the wire surfaces. The cracks are increased and their dimensions also increased. Besides, the surface area is increased gradually with the time of container rotation. Also, one can see shallow holes with different sizes are distributed on the surface of the treated wire. These holes are believed to have formed due to the meeting of multi cracks.

To understand the influence of scratching time on the crack dimensions, a statistical process was done on some pictures for each sample. Table 1 shows the results of these statics. It is clear that the number of cracks is increased as treating (scratching) time is increased. Furthermore, cracks dimensions, in general, are increased too. Crack dimensions depend on the grain size of the SiC powder, so it is believed a smaller particle size can produce a smaller scratch.

Figure 3 illustrates the SEM images for anodized samples. The images (a), (c) and (e) are related to anodizing periods of 15, 20 and 30 min. The sample GE 15 (15 min) did not show any clear surface porosity due to anodizing. This behavior is related to the short time for creating noticeable porosity where the induced reaction is just beginning on the surface. One also can recognize a “smooth lump” of the surface compared to the reference wire sample in Figure 3(a) and the magnified one in (b). It is believed that the smooth lumps are related to metal oxides (iron oxides) formation on SS316L surface that forms stain-like objects as shown in the image (c) [24]. As Fe is the predominant element in the wire material, and due to the stimulation of oxidation process as a result of existence the aqueous solution of perchloric acid, and a motivation electrolysis process, which they help to form the oxide, it is believed these particles are related iron oxide. SEM-EDS test was performed to the wire of the sample GE 15, as in Figure 3(f) for a selected area of the smooth lump and the analysis showed the presence of oxygen, which supports our explanation. As anodizing time increases, a clear porosity appears on the surface as shown in the image (e) as a result of removing oxides and inducing the anodizing process. Two pore sizes were recognized in an image (e). Ones belong to nano size (<200 nm), which are appeared as dots and bigger ones appeared as black dots (>2 µm). This inhomogeneity in size distribution may be attributed to non-uniformity in setting up and current density.

3.2 Impact strength test

Table 2 illustrates the Charpy impact (I.S.) results of the prepared dentures. The results display a high increase in the impact strength due to stainless steel wire reinforcement compared to non-reinforced one. Of course, such an increase can be attributed to the high strength and impact resistance of the stainless steel. The value of impact strength of the sample G2 that involves untreated wire is higher than the impact value of reinforcing PMMA with carbon fiber mat reinforced PMMA [28, 29]. This difference can be attributed to the kind of supporting material. Also, the impact value of the untreated wire is comparable to that obtained for stainless steel reinforced PMMA [30]. One can relate the small increase in the impact strength of the reference [30] to the difference in the thickness of wires used in both studies where this study used lower wire thickness.

The samples with surface treated wire, GM60, GM90 and GM120, show an additional increase in impact strength as seen in Table 2. The value of the impact strength is going to be saturated as the period of treating reaches 120 min. Here, it is believed as the time of scratching is increased, the number of cracks and crack dimensions are increased, therefore the surface area is increased as it was seen in SEM images in Figures 2(b), 2(c) and 2(d). Then, the cracks activate the wire surface and make high bonded to resin. Additionally, roughing the wire surface is raising the wire surface and affords more area with high friction between PMMA and the wire that they help in absorbing impact energy and increase the material to resist the impact [31]. The standard deviation the surface roughness. The mechanisms of bonding between the matrix and wires can be related to polymer diffusion to the wire, electrostatic attraction, chemical bonding, and mechanical overlapping.

| Group no. | Mean (kJ/m²) | Min (kJ/m²) | Max (kJ/m²) | SD |
|-----------|--------------|-------------|-------------|----|
| G1        | 7.43         | 7.21        | 7.65        | 0.45|
| G2        | 29.75        | 28.1        | 31.4        | 0.72|
| GM60      | 39.16        | 37.55       | 40.78       | 0.64|
| GM90      | 40.53        | 39.11       | 41.96       | 0.98|
| GM120     | 42.38        | 40.89       | 43.87       | 1.14|
| GE15      | 26.56        | 27.34       | 25.78       | 1.21|
| GE20      | 31.61        | 32.57       | 30.65       | 1.84|
| GE30      | 26.99        | 26.80       | 27.19       | 0.76|

In this study, diffusion is controlled by wetting ability. All treated wires were wetted with PMMA before the casting process as a result of verifying the diffusion mechanism. Surface energy and surface local electrostatic charges on wires and matrix are so important to enhance the mechanical properties through forming Van der Waals bonds. It is believed that mechanical surface treating (scratching) and
friction with SiC powder can provide such charges (negative and positive ions). Holes with different sizes on SS316L wires, which are generated by scratching and anodizing, provide good overlapping between matrix polymer and wires. In this mechanism, PMMA performs the cohesion by many meshing clamps due to permeation of the polymer inside the pores. So, the extra number of deep pores or holes on the wire surface produces better composite mechanical properties.

It is believed that the low pores depth and concentration of anodization samples for samples GE15 and GE20 compared to scratched ones is the main reason for making anodizing samples have lower impact strength. On the other hand, it was found that the anodized layer growing on stainless steel 316L releases nickel and chromium from the stainless steel structure, and in turn, the anodization process does not enhance longevity of stainless steel for mechanical application [32]. This may be the reason behind the impact strength was not as high as in scratched samples.

Table 3 shows the impact strength comparison of previous study results with the result of this study. In beginning, there was no previous work study the reinforcing PMMA with ss316L wire, as far as we know. Besides, this is the first time to treat the stainless steel surface in general, to be applied in the biomaterial. In Table 3, one can recognize the very high gotten impact strength by reinforcing PMMA with ss316L treated wire. Indeed, this high value relates to two reasons: the properties of ss316L and the PMMA-wire bonding due to wire surface roughness increases.

### Table 3: Comparison of the results of the previous study with the result of this study

| Literate study | Type of reinforcement | Dimension of sample | Impact strength test (KJ/m²) |
|---------------|-----------------------|---------------------|-----------------------------|
| [33]          | (PMMA) with Polyethylene fibers | 60 × 10 × 4 mm | 18.8707 |
| [28]          | 0.03 of Black chopped Carbon fiber + 0.02 of PVA + 0.06 of Tri calcium phosphate | 3 × 10 × 65 mm | 11.25 |
| [34]          | PMMA+ (8% chopped glass fiber) | 65 × 10 × 3 mm | 11 |
| This study    | PMMA reinforced with stainless steel 316L wire | 65 × 10 × 3 mm | 43.87 |

4 Conclusion

This study is depending on using ss316L wire for reinforcing PMMA for denture applications due to its well-known biocompatibility and mechanical property’s reliability. The proposed scratching method for ss316L wires showed a high enhancing in the impact strength despite its simplicity, availability, and low cost. The mechanical method produced cracks and shallow holes that were randomly distributed on the surface of ss316L wire and their surface density and sizes increased as treating time increased. The anodizing time increasing produced a clear porosity on wire surfaces and removed the previously produced oxides. The value of the impact strength is saturated as the treating period be 120 min. A low anodizing rate for ss316L wires can also enhance the impact strength of the ss316L reinforced PMMA samples but with a lower influence compared to the mechanical scratching method. The nature of the wire surface affects impact strength greatly due to most of the load being applied on the contact surfaces through compression, tension, and shear stresses. More energy is demanded to fracture for composite with good bonding between wires and matrix and very low energy consumption in slipping the wires with respect to host matrix. Generally, the two methods are active in improving the reinforced dentures.

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