EFFECT OF FLUORIDE AND ACID ON SHAPE MEMORY BEHAVIOURS OF NITI ORTHODONTIC ARCHWIRE

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DECLARATION

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TABLE OF CONTENTS

Contents **Pages**

LIST OF TABLES

Tables **Pages**

LIST OF FIGURES

Figures **Pages**

NOMENCLATURE

Nomenclature	**Description**
NiTi	Nickel Titanium
Sa	Austenite phase start
Fa	Austenite phase finish
Sm	Martensitic phase start
Fm	Martensitic phase finish
Ea	Austenite elastic modulus
Em	Martensitic elastic modulus
$C_3H_6O_3$	Lactic Acid
NaF	Sodium fluoride
DSC	Differential Scanning Calorimeter
SEM	Scanning Electron Microscope
ICP-MS	Inductively Coupled Plasma Mass Spectrometry

ABSTRAK

Nikel Titanium (NiTi) aloi memori bentuk (AMB) adalah sejenis bahan baru yang telah digunakan secara meluas dalam bidang ortodontik menggantikan keluli tahan karat. Hal ini adalah merujuk kepada sifat ketegangan elastik yang lebih besar semasa perubahan fasa martensit. Walau bagaimanapun, ciri-ciri wayar lengkung akan dipengaruhi oleh hakisan permukaan wayar dan akan menyebabkan kepada pelepasan Nikel yang boleh membawa kesan toksik kepada manusia. Hakisan pada permukaan wayar lengkung biasanya akan berlaku dalam persekitaran mulut kerana aktiviti pemakanan seharian individu atau aktiviti memberus gigi yang akan menyebabkan kepada pengurangan kelakuan suhu dan mekanikal wayar lengkung. Dalam kajian ini, natrium fluorida dan asid laktik telah digunakan untuk mengkaji kesannya terhadap tingkah laku suhu dan mekanikal wayar lengkung, NiTi. Hal ini mendapati bahawa masa pendedahan yang lebih lama dalam larutan berasid, sifat terma dan mekanikal akan berkurangan lebih bandingkan dengan larutan fluorida. Ini terbukti dengan morfologi permukaan dalm imej SEM, yang jelas menunjukkan bahawa terdapat hakisan pada permukaan dawai ortodontik yang menyebabkan pengurangan kelakuan suhu dan mekanikal dan menyebabkan kepada pelepasan ion Nikel ke dalam tubuh manusia. Dapat disimpulkan bahawa nilai keasidan yang lebih tinggi akan menyumbang kepada kadar hakisan yang tinggi keatas wayar lengkung ortodontik.

Kata kunci: Wayar lengkung ortodontik, kelakuan mekanikal, hakisan, pelepasan Nikel

.

ABSTRACT

Nickel-Titanium (NiTi) Shape Memory Alloy (SMA) is new material that had been widely used in orthodontic application that replacing stainless steel. It is refer to larger elastic strain relieved by martensitic phase transformation. However, these wire properties will influence by the surface erosion of the wire and lead to Nickel release that may toxic to human. The surface erosion of the wire normally occur in oral environment due to eating behaviour or tooth brushing activity that will reduce the thermal and mechanical behaviour of wire. In this research, sodium fluoride and lactic acid was used to study its effect toward thermal and mechanical behaviour of NiTi arch wire. It was found that the longer the exposure time in acidic solution, the thermal and mechanical properties will reduce more compare to fluoride solution. This is proven by surface morphology on the SEM image, that obviously show that the erosion on the surface of orthodontic wire will reducing the thermal and mechanical behaviour and also lead to Nickel ion release into the human body. It can be concluded that higher value of acidity contribute high rate of corrosion toward the orthodontic arch wire.

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Keywords: Orthodontic arch wire, mechanical behaviour, erosion, Nickel release

1.0 INTRODUCTION

Ni–Ti shape memory alloys (SMA) becomes are favorable material used in most orthodontic application compare to stainless steel [1] due to shape memory behavior as shown in figure 1 [2]. NiTi SMAs have greater strength and lower modulus of elasticity when compared with stainless steel alloys [3]. Ni–Ti shape memory alloy can exist in two different temperature-dependent crystal structures (phases) called martensite (stable at lower temperature) and austenite (stable at higher temperature) [4]. It used to level and align the teeth under bending conditions in the oral environment for a certain period of time [5].

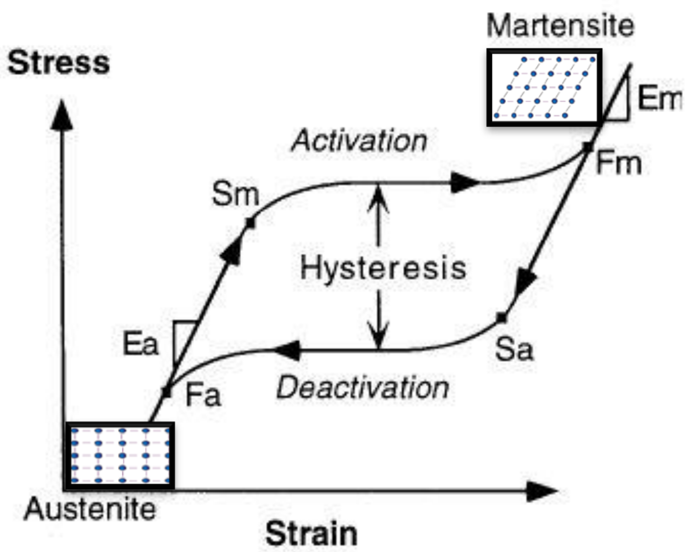

Figure 1. Stress-strain diagram of NiTi alloy with super elastic behavior

This shape memory behavior can be described on a stress strain curve. Application of stress leads to linear increase in strain up to point where martensitic transformation starts to occur (S_m). Slope is equal to austenitic modulus of elasticity. At the end of martensitic full transformation, F_m, it can be notice that increase in strain without associated increase in stress. When test piece is completely martensitic, relationship between stress and strain become linear again and start to undergo plastic region. Slope is now equal to martensitic modulus of elasticity (E_m). In deactivation, there will be a linear relationship between stress and strain down to point where austenitic transformation starts to occur (S_a). Therefore, there is deactivation plateau between S_a and point where restoration of austenitic structure is complete (F_a) and a linear relationship between stress and strain down from point F_a. The activation and deactivation plateaus have different stress magnitudes is called hysteresis [6].

However, in orthodontic application, erosion on the surface of wire occurs in oral environment [7], so that it will reduce the thermal and mechanical properties of wire. As a result, this factor will reduce the effective force use in tooth movement or alignment [8]. In orthodontic treatment, human tooth can be categorizes into several types of alignment such as tipping, translation, root up righting, rotation, extrusion and intrusion [9]. Tipping movement is produced when a single force is applied against the crown of a tooth. If two forces are applied simultaneously to the crow of a tooth, the tooth can be said bodily movement (translation). For rotational movement, a certain force is applied at different side of tooth so that the tooth will rotate on its crown. Extrusion and intrusion is almost same with rotation, movement of teeth normally produce no areas of compression within PDL, only tension [9]. Therefore, optimum force need for this type of tooth alignment is shown in Table 1.

Table 1: Variation of tooth movement and optimum applied force[9]

Optimum Forces for Orthodontic Tooth Movement

Type of movement	Force* (gm)
Tipping	35-60
Bodily movement (translation)	70-120
Root uprighting	50-100
Rotation	35-60
Extrusion	35-60
Intrusion	10-20

The focus of this research is to study the effect of acid and fluoride on the thermal and mechanical properties of the arch wire. Lactic acid was use to represent acidic food consumption of a person in a day and sodium fluoride was use to represent content of fluoride during the tooth brush in a day. The chemical reaction between two types of solution will cause the surface erosion on the orthodontic arch wire [10]. The comparison of the surface erosion can be analyzed under the SEM image. This effect of erosion can reduce the thermal and mechanical behavior of the orthodontic arch wire [4]. To study the thermal behavior of the orthodontic arch wire, DSC thermal analysis was done to detect the change in austenite phase transformation temperature, A_s and A_f, and martensitic phase transformation temperature, M_s and M_f. In this thermal analysis, the erosion of wire will lead to change in the phase transformation temperature [11]. The higher the erosion on the surface of wire occur, the start martensitic phase transformation, M_s temperature will increase compare to the result before the immersion [12]. Therefore, this transformation temperature will result in closed to human body temperature, (37˚C) so that the phase transformation from austenite to martensitic will finish at higher temperature. Besides, the change of thermal behavior will affect the effective force when a person takes a cold or hot drink [13]. The effect of these changes in thermal behavior will source the change in mechanical properties of the wire. This is because phase transformation for both austenite and martensitic will complete early if erosion occur. For mechanical behavior test, tensile test was use to analyze the stress-strain behavior of the wire under room temperature. Under stress-strain curve, the phase transformation behavior of austenite-martensitic can be represent in the forward and reverse stress value during activation and deactivation of wire [14]. The change in phase transformation temperature will affect the forward and reverse stress. In addition, the erosion of wire will exposed the oral tissues to Nickel release during chemical reaction occur on acid and fluoride [15]. As a result, severe allergic case was reported due to reaction of NiTi orthodontic wire in a patient allergic to nickel [16]. This is because the corrosive products of NiTi wire were toxic to the primary cultured aortic smooth muscle cells [17].

2.0 EXPERIMENTAL DESIGN AND PROCEDURE

Samples was prepared from one type of dental alloys wires 0.016 3M Unitek (OrthoForm™ III Ovoid Arch Form). The composition of wire is 55 % Nickel and 45% Titanium. The wire is circular type with diameter 0.406mm. The samples were cut from the arch wire along the straight part about 50 mm. For each set of immersion test, two pieces was used. One pieces of wire was used in tensile test and the other one is used in DSC and SEM.

For immersion solution, two types of solution were prepared, lactic acid and sodium fluoride. For lactic acid, 3 different pH value was prepared (pH2, pH 3, pH 5) and for sodium fluoride, 3 different fluoride concentration (1000ppm, 1300ppm, 15000ppm). For the existent situation in oral environment, content of fluoride representing tooth brushing activity and lactic acid, $C_3H_6O_3$ representing a person daily acidic food consumption.

Immersion test was done to resemble the application of NiTi arch wire in mouth application. It involved chemical reaction of acid and fluoride with NiTi wire. The immersion test was categorized into three categories, real immersion, simulation immersion, and continuous immersion. For the real immersion test, 2 samples of NiTi arch wire were immersed periodically into lactic acid and sodium fluoride on a certain time according to our daily activity. For simulation immersion test, immersion time was represented in Table 2. Real time immersion represented cumulative time of wire expose to acid and fluoride that is related to eating or brushing time taken by a person. For example, a person eats 3 times per day and it takes about 5 min per meals so for a month it will take about 7 hour 30 min cumulatively. For sodium fluoride, estimation time for a person to brush teeth is three times per day and it takes about 3 minutes per session so for a month it will takes about 4 hour 30 minutes cumulatively. For continuous immersion, two pieces of wire were immersed into three types of immersion solution, lactic acid with pH value 2 and 5, and sodium fluoride with concentration 1500 ppm continuously in one month.

Table 2: Immersion Time

Immersion Solution	Real Immersion Time	Equivalent Time
Lactic Acid	7 hour 30 minute 22 hour 30 minute 45 hour	1 month 3 month 6 month
Sodium Fluoride	4 hour 30 minute 13 hour 30 minute 27 hour	1 month 3 month 6 month
Lactic acid	1 month	N/A
Sodium Fluoride	1 month	N/A

Tensile test was done to analyze the stress-strain curve of NiTi arch wire. By using INSTRON universal testing machine, 1mm/min of load speed was applied for each of the specimens at room temperature and the experiment set up was shown in figure 2. 15 mm in length was used for each gripping and 20 mm was used as gage length. In this testing, cyclic method was applied in Bluehill 2 software to get the stress-strain curve. The wire was pulled at 1.5mm of extension and release until it reverse back to its original length.

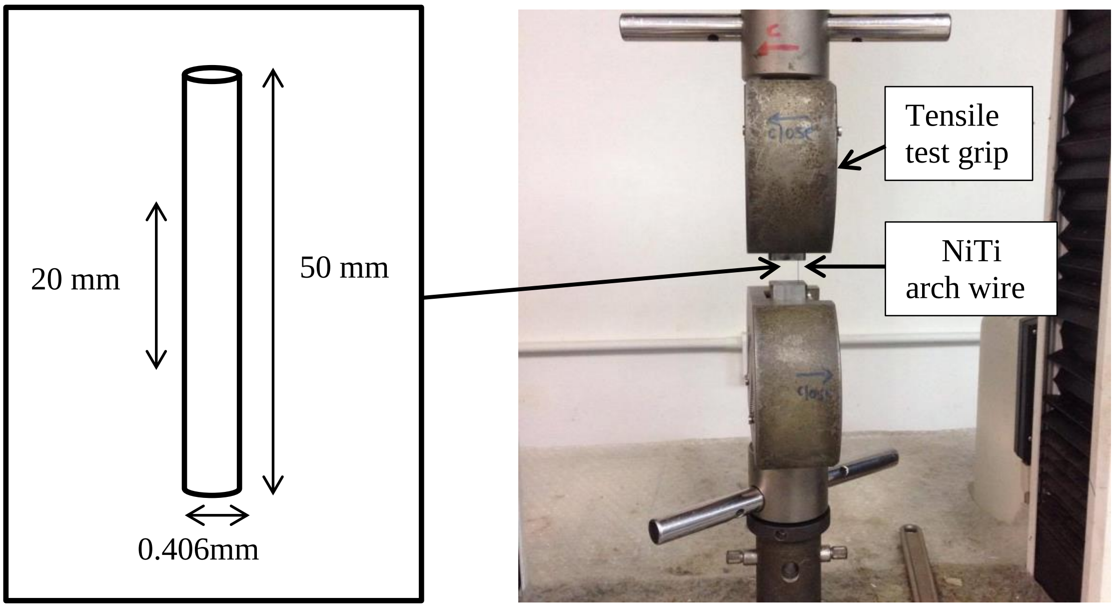

Figure 2. Tensile machine parts and set up of tensile test

For thermal analysis, 3–4 mm length of wire was cut from orthodontic wires by using diamond cutter for DSC test. This testing was carried out by using Thermal Analysis Q20 DSC instrument machine and liquid-nitrogen as cooling agent. Each sample was heated to 60 °C and cooled to –30 °C at the heating/cooling rate 10 °C /min to obtain the heating and cooling DSC curve. Therefore, each specimen experienced the austenite–martensitic transition twice in both directions (endothermic and exothermic).

Scanning electron microscopy (SEM) images of the specimen surface were recorded before and after immersion test. The affected area of the wires was studied under SEM images by using LEO 1530 instrument. Conventional SEM technique was used in this analysis under electron sources 15kV, 800x magnification, and secondary electrons, SE that are ejected from the k-shell of the specimen is less than 50 eV. For the metallic specimens, no sputtering was required. All the images data were collected over a selected area on the surface of the sample, and a 2-dimensional image is generated that displays spatial variations in these properties.

In this experiment, 9 samples (6 simulation immersion test and 3 continous immersion test) were selected to check the existing of Nickel in the solution. Method used is ICP-MS measurements, to check the metal elements in the solutions by using ICP-MS machine model, Varian 715-ES, ICP Optical Emission Spectrometer. Sample introduction was performed with a PFA (perfluoroalkoxy) micro flow auto-aspirating nebulizer combined with a double-pass spray chamber. For this purpose, 8 standard solutions were used to standardize the solution and the result from test solution was compared to this standard solution to get the concentration of Nickel in units of ppm.

3.0 RESULTS AND DISCUSSION

3.1 Thermal behavior of NiTi arch wire after immersion test

Figure 3 presents the comparison of DSC plots for both the heating and cooling curves for each orthodontic wire after real and simulation immersion test. All the curves on heating presented with temperatures A_f lower than 37 °C (body temperature), indicating an expression of the super elastic effect during an orthodontic treatment [12]. It is trend clearly showed that the transformation temperature of austenite finish, A_s is increase when there is longer the immersion time. From figure 3c, it clear shows that immersion in lower pH value will cause the drastic change in phase transformation temperature. For sample in figure 3c, martensitic phase transformation start, Ms is increase to 21.31°C from 15.6°C as a received. Therefore, orthodontic wire in figure 3c will fully transform into martensitic phase above 0 °C higher compare to orthodontic wire before the immersion. Besides, from the plot graph, it clearly show that the peak temperature decrease when the longer the immersion time.

Figure 4 presents the comparison of DSC plots for both the heating and cooling curves for each orthodontic wire in continuous immersion test. For cooling curves, for martensitic phase transformation temperature, it is clearly showed that the martensitic transformation start, M_s is increase when there is longer the immersion time. From figure 4c, it clear shows that immersion in lower pH value will cause the drastic change in martensitic phase transformation temperature. For heating curves, its trend clearly showed that the transformation temperature of austenite finish, A_s is increase when there is longer the immersion time with lower pH value (figure 4c). For wire in figure 4c, austenite phase transformation start, A_s is increase from 7.58°C to 12.71°C higher compared to figure 4b and 4d. Therefore, orthodontic wire in figure 4c will fully transform into austenite phase at 29.47°C almost closed to body temperature, 37°C higher compare to orthodontic wire before the immersion.

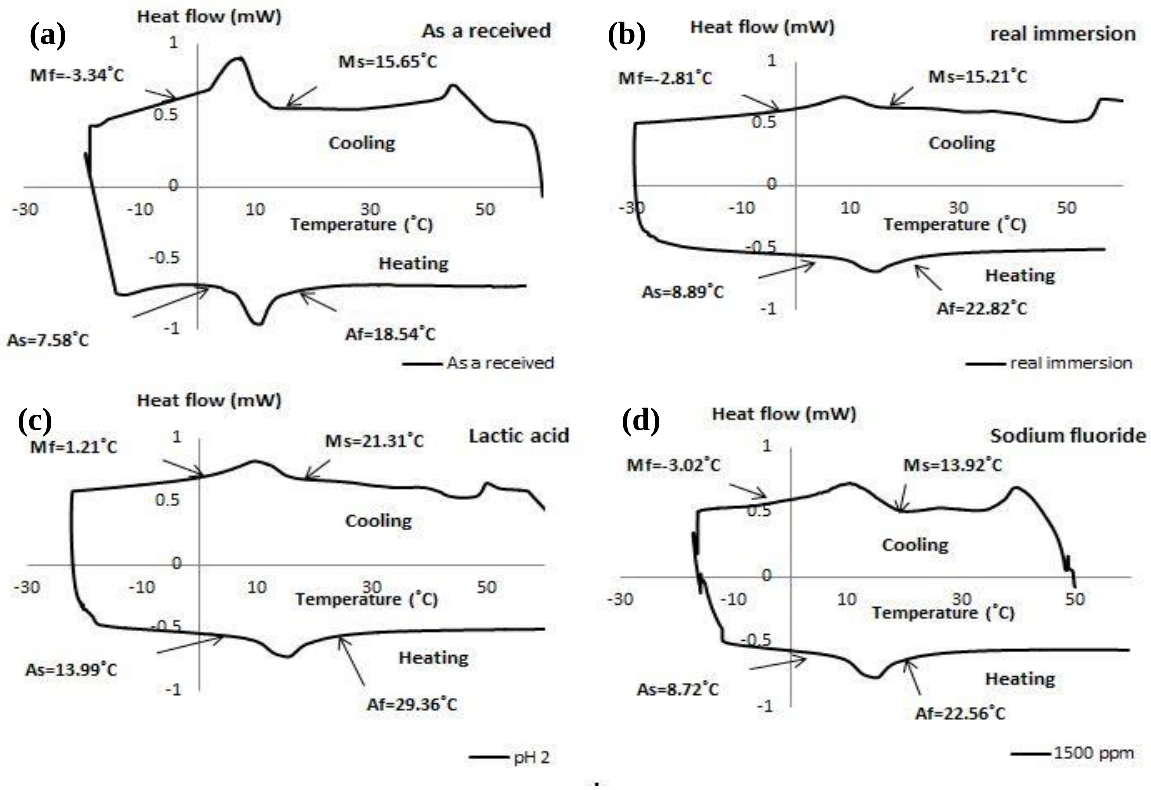

Figure 3. The DSC curves of orthodontic arch wire after 6 month simulation immersion:
a) before immersion b) real immersion test c) pH 2 Lactic Acid d) 1500 ppm NaF solution

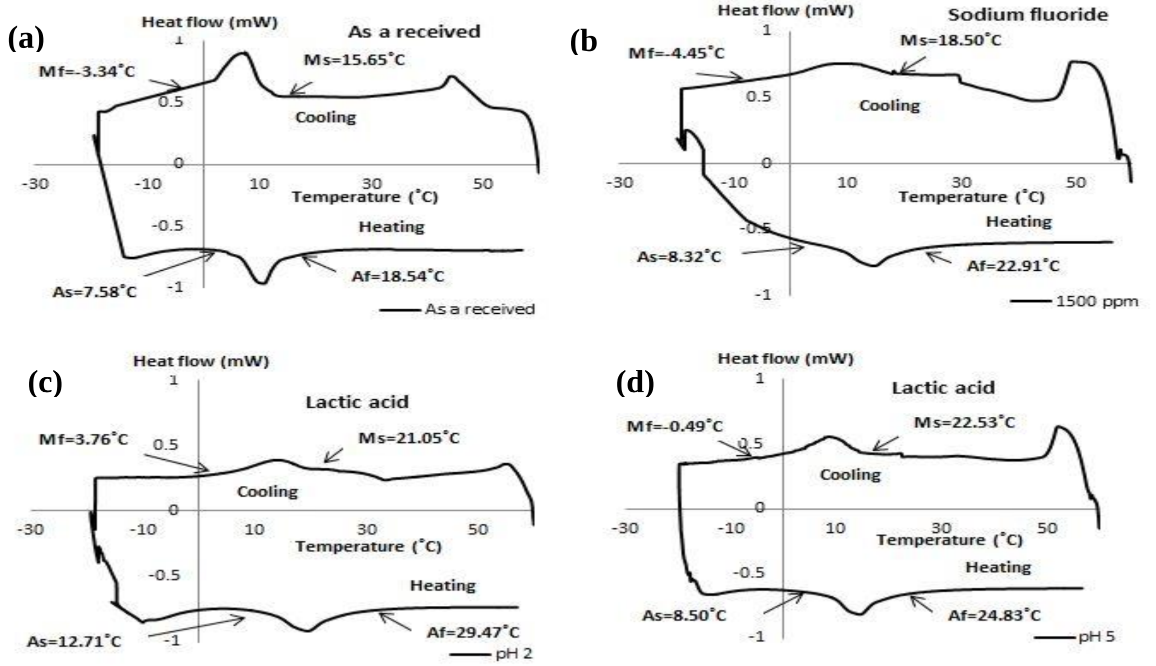

Figure 4. The DSC curves of orthodontic arch wire after continuous immersion test:
a) before immersion b) 1500 ppm sodium fluoride c) pH 2 lactic acid d) pH 5 Lactic Acid

3.2 Stress-strain behaviour of NiTi arch wire after immersion test

In the study of stress-strain curves of wires after immersion test at room temperature, it is observed that the condition of wire after immersed in the solution, do not show lower plateau stress (figure 5a) and significant residual strain is observed in the samples (figure 5c). Since that the goal of Ni-Ti orthodontic wires use is applying uniformed and permanent force to the teeth during treatment that requires a plateau in stress-strain curve of the wires. As a result change in thermal behaviour, austenite phase transformation will increase, it will affect the hysteresis value between upper plateau region of stress and lower plateau of stress. Due to martensitic phase transformation start early, about 3% transformation of plastic region formed in figure 5c compare to figure 5a. This because immersion in lactic acid will change the phase structure of NiTi arch wire so the martensitic phase is complete transform earlier. The super elastic properties decreased after immersion in acidic medium compare to fluoride medium. This is because more erosion occurs in acidic medium is higher than fluoride medium.

Besides, the drastic changes in elastic behaviour occur in longer the immersion time. It can be representing in the stress-strain curves in figure 6. In thermal behaviour during continuous immersion test in lactic acid pH 2, the martensitic phase transformation temperature change drastically. So, this will affect the mechanical behaviour of the wire as shown in figure 6c. It clearly show that the martensitic phase complete below 0.08 strain. This will cause the plastic region start occur at 0.07 strain show that all the austenite is complete transform to martensitic phase. Therefore, the recovery of the wire will reduce to 0.02%. From this result, it can be conclude that, the longer the immersion time in lactic acid, it will reduce the mechanical properties of the wire.

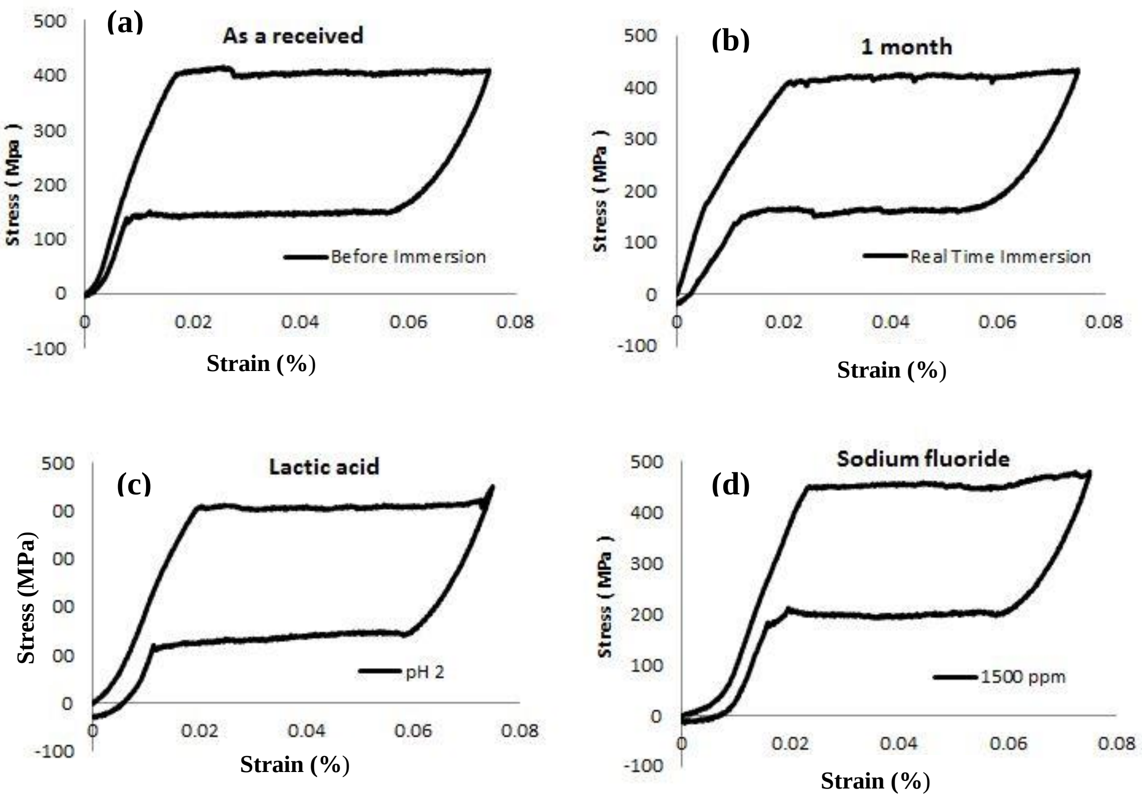

Figure 5. The stress-strain curves of orthodontic arch wire in simulation immersion test: a) before immersion b) real time immersion test c) pH 2 lactic acid d) 1500 ppm sodium fluoride

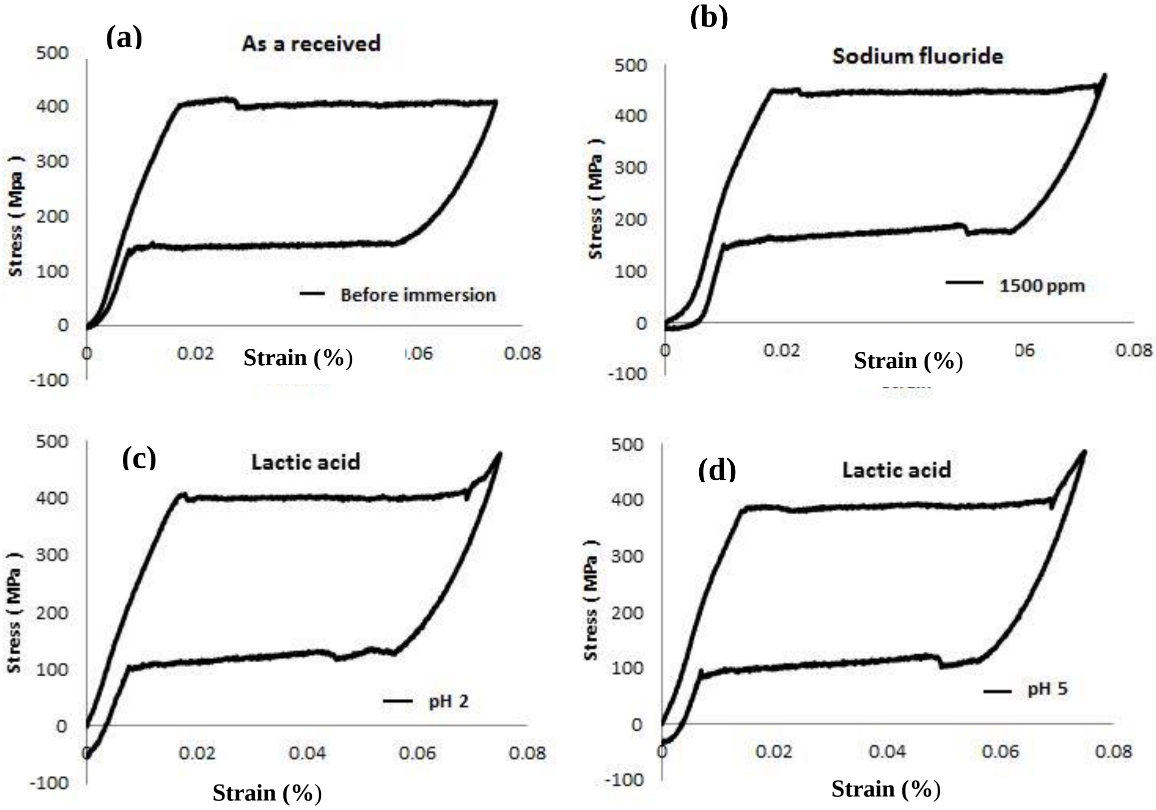

Figure 6. The stress-strain curves of orthodontic arch wire in continuous immersion test: a) Before immersion b) 1500 ppm sodium fluoride c) pH 2 lactic acid d) pH 5 lactic acid

3.2 Comparison of forward and reverse stress after immersion test

Figure 7a shows the comparison of forward transformation stress after the immersion test in lactic acid and sodium fluoride. The graph shows the immersion in lactic acid with pH value 2 give the lowest forward transformation stress. Due the chemical reaction between the H+ ion and NiTi arch wire, the surface of wire had been erodes more in lactic acid solution compare to fluoride solution [18]. Therefore, the higher the concentration of hydrogen ion, H^+ in the immersion solution gives more erosion to the orthodontic wire [19]. As a result, the erosion will change the phase transformation of microstructure from austenite to martensitic during force is applied on the wires [20]. During meal, consuming food that contains higher content of acidic value, it will reduce returning force of the arch wire instantaneously [21]. These factors need to be considering in orthodontic treatment to ensure that the continuous forces of the application [22]. Normally, most of the SMA NiTi provides nearly continuous force in the transformations plateau. Figure 7b shows a comparison of the reverse transformation stress for each test set after the immersion test in immersion solutions, lactic acid and sodium fluoride. The graph shows the reverse stress for 1500 ppm concentration of fluoride give the lowest value in fluoride category. This is clearly shows that the longer immersion in the higher concentration of fluoride solution, the lower the stress value. This factor can be influence by our daily activity during mouth cleaning because the content of fluoride is higher (1500 ppm) so that it will give more corrosion to the orthodontic wires. Decrease value of reverse stress, it need less force during deactivation of martensitic transformation to austenite. Note that the lower the forward stress, there is decrease in the hysteresis where the activation and deactivation plateaus have different in the stress magnitudes [23]. Another difference in the stress-strain curves of different set of immersion test can be the difference in the amount of elastic strain energy and the amount of energy stored in the wires. Elastic strain energy acts as a potential energy and encourages a reverse transformation (martensitic to austenite) [24].

Figure 7b shows the comparison of reverse stress for each set of immersion test different concentration of fluoride and pH value of acid. The graph shows the immersion in lactic acid with pH value 2 give the lowest reverse stress compared to fluoride solution. The difference value of higher reverse stress and lower reverse stress is 8.238 MPa which mean it is decrease by 14%. This is because the wire had been corrodes more in lactic acid compare to sodium fluoride that will result reduce in stress. This condition can be presented by our daily activity. For the reverse stress, the lowest is 89.809 MPa compare to other immersion solution. This decrease in stress will reduce the restoration force in transformation of martensitic to austenite. The lower magnitudes of the reverse stress will result in incomplete phase transformation. So, this will reduce effectiveness of the orthodontic arch wire if the person consuming high food that contain lowest pH value [25].

For continuous immersion test, the extrapolation shows that the longer the immersion in lactic acid will reduce more of the reverse and forward stress values NiTi arch wire. So, it will conclude that the longer the immersion time in lower pH value will result in lower the mechanical behaviour of NiTi arch wire.

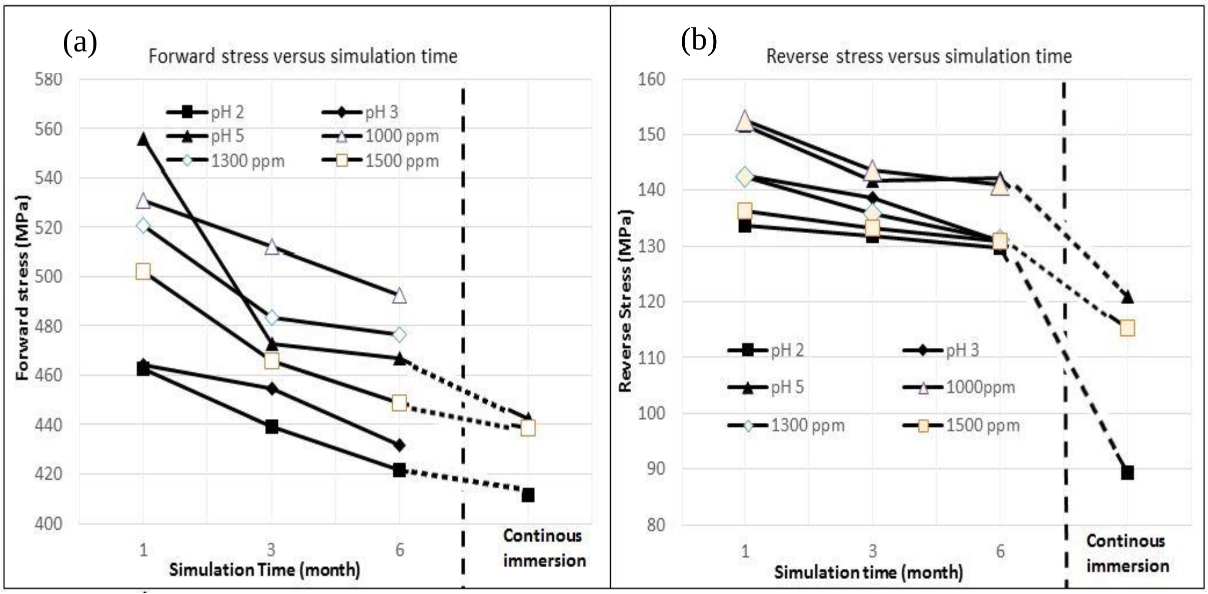

Figure 7. Effect of pH level and fluoride concentration toward forward and reverse transformation stress

3.3 Comparison of the surface image of NiTi arch wire under SEM after immersion test

This surface analysis was carried out to perform surface analyses on the NiTi alloys since they displayed the highest risk of corrosion. The samples were observed using scanning electron microscopy (SEM). The surface micrographs obtained are shown in Figure 8 and 9. For continuous immersion test in figure 8, it clearly show that the massive corrosion occur in lactic acid with lower pH value compare to sodium fluoride. This is because the chemical reaction between the orthodontic arch wires toward acid is more active if compare to sodium fluoride. As a result, this will reducing its thermal and mechanical behavior as discuss above. The wire corrosion will lead to reduce the stress value of the wire. From figure 8c, it clearly showed the porous shape formation occurs on the surface of the wire that lead to Nickel release into the solution. The comparison also shows that in pH 2 the surface erosion occurs more compare to pH 5 lactic acid. Thus, the pH value and fluoride concentration is the main parameter that leads to corrosion of the wire.

For the simulation immersion test, corrosion in lactic acid with pH value 2 in figure 9b is lower compare to pH 2 in figure 8c. By comparing the SEM images in figure 8 and 9, it clearly shows that the longer of the wire expose to the acidic and fluoride solution, the higher the corrosion on the surface of wire occur. As the result, this erosion of wire will lead to Nickel release into human body. In fact, high amount of Nickel in human body will expose the patient to cancer and also cause some allergic problems toward patient during orthodontic treatment [26]. Besides, in sodium fluoride also shows the corrosion occur on the surface of the wire. So, it should be advised for patients wearing dental prosthetic reconstructions or who have titanium implants should not to use higher fluoride content toothpaste. In the future, it seems that the longer the immersion time will give a better result to compare the corrosion of wire.

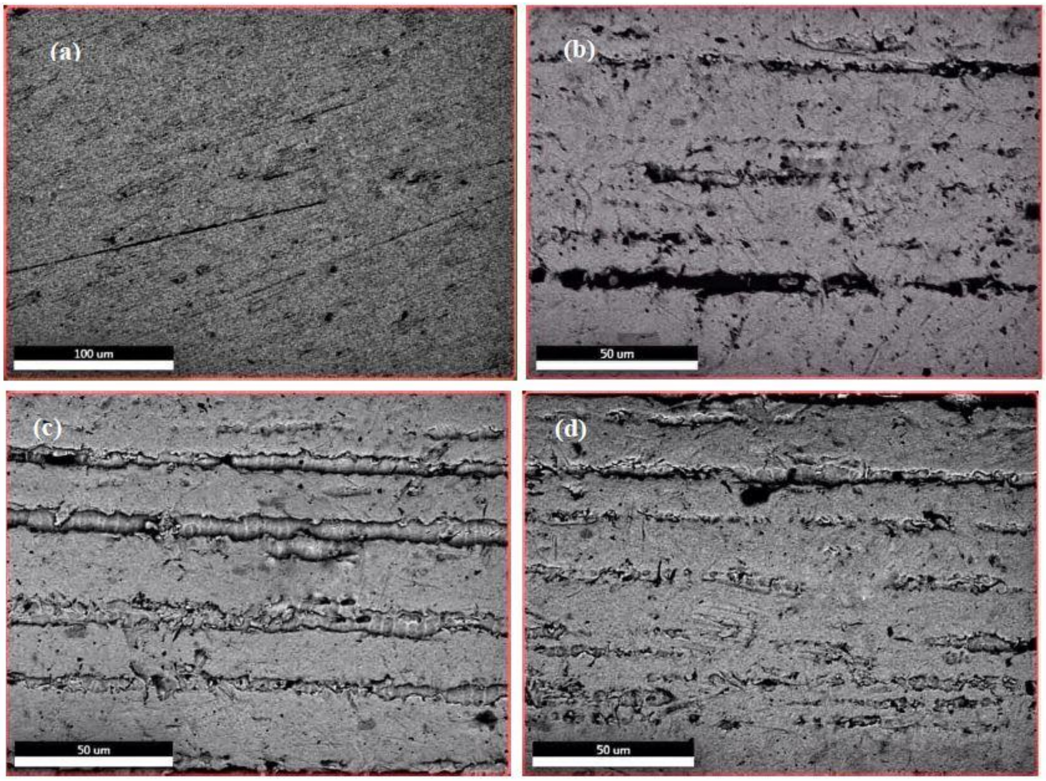

Figure 8. SEM images of wire surface after continuous immersion:
(a) surface of wire before immersion; (b) 1500ppm sodium fluoride; (c) pH 2 lactic acid; and (d) pH 5 lactic acid

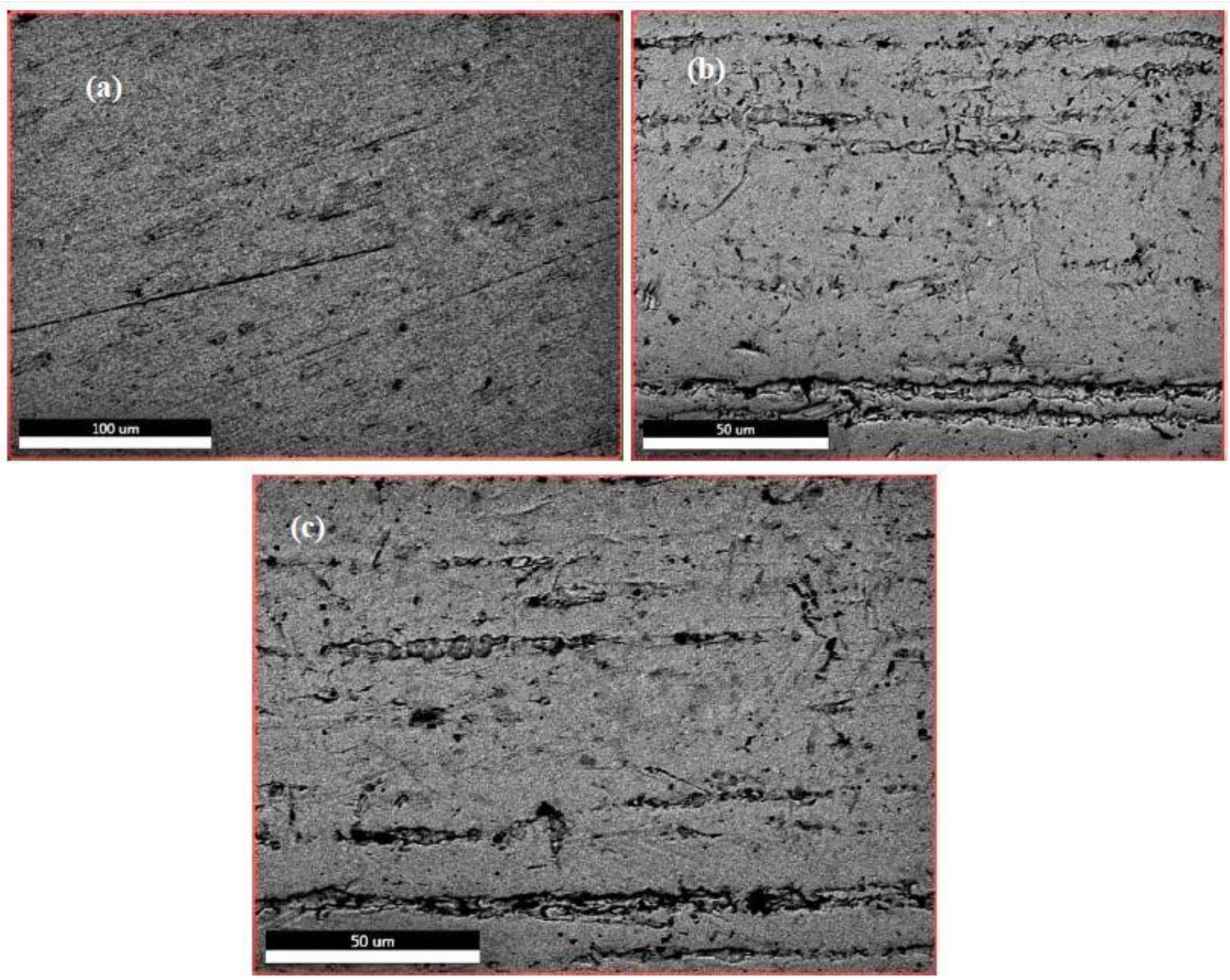

Figure 9. SEM images of wire surface after 6 month simulation immersion:
(a) surface of wire before immersion; (b) pH 2 lactic acid; and (c) 1500ppm sodium fluoride

3.4 Content of Nickel release after immersion test

It is known that erosion of orthodontic alloys occurs in the intraoral environment, and it is also known that the extent of manufacturing defects that may accelerate the process. From figure 10 and table 3, it showed the comparison of Nickel content between simulation immersion and continuous immersion test. The different increase content of Nickel in pH 2 lactic acid during continuous immersion is 0.04 ppm higher compare to simulation immersion. This shown that the longer immersion time increases the level of Nickel release in the solution. In sodium fluoride solution, the value of Nickel content increase by 13% in simulation duration of 6 month. However, the Nickel release in lactic acid is higher compare to sodium fluoride about 4%. So, it can be said that the reaction of NiTi arch wire toward lactic acid is higher compare to the sodium fluoride. This happen because of the surface of the wire become porous as seem in SEM images in figure 11c so that the Nickel will be release into the solution of immersion. It shown that the increase of Nickel ions release from this NiTi arch wires is enhanced by the surface corrosion. Nickel has been reported to be one of the most common causes of allergic contact dermatitis. The current consensus about the issue of orthodontics derived nickel as a sensitizing agent is that the risk is extremely low for patients who are not nickel hypersensitive at the start of orthodontic treatment [14]. This problem can only cause reactions in patients who are already sensitive to nickel. Therefore, the use of NiTi arch wires should be limited in nickel sensitive patients as even small amounts of nickel in saliva may cause reactions. However, the overall immersion result show that the Nickel content still not exceeds the safety limit of Nickel in human body that is about 58.7ppm content of Nickel in human body [27].

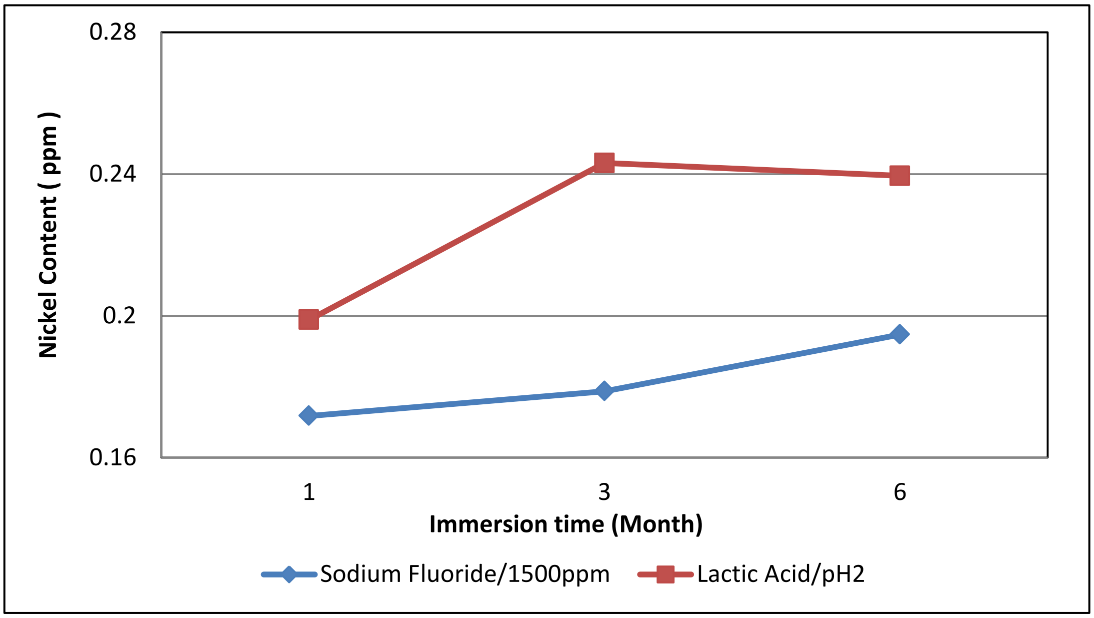

Figure 10. Effect of pH value and fluoride concentration toward Nickel release in simulation immersion test

Table 3. Effect of pH value and fluoride concentration toward Nickel release in continuous immersion test

Immersion solution	Nickel content (ppm)
Lactic acid pH 2	0.2886
Lactic acid pH 5	0.2510
Sodium fluoride 1500 ppm	0.1347

CONCLUSION

For DSC curve, it shown that immersion in acidic medium with lowest pH value will influence more of the transformation temperature of NiTi arch wire. Both of martensite and austenite transformation temperature is increase to 34% and 27%. This will result in reducing both forward and reverse stress about 25% and 40 % respectively. The highest Nickel content in this experiment is 0.2886ppm that is much lower than safe limit in body that is about 58.7ppm.

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