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Research Article

In Vitro Corrosion Behavior of Lingual Orthodontic Archwires

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Objectives. To investigate the in vitro electrochemical corrosive behavior of archwires used in lingual orthodontics and the effects on the phase transition temperatures. *Materials and Methods*. Six different types of archwires of stainless steel, titanium-molybdenum, nickel-titanium and nickel-titanium-copper were used. Corrosion tests were performed following ISO-standard 10993-15:2000. Differential scanning calorimetry and scanning electron microscopy were used. *Results*. The stainless steel archwires showed an E_{pit} around −600 mV, and those of titanium alloys showed E_{pit} values around 1000 mV. Differential scanning calorimetry detected a rhombohedral phase in nickel-titanium archwires, while it was not detected in nickel-titanium-copper wires. A difference of 2°C to 3.5°C from the manufacturer's claim was found in the as-received and polarized samples, respectively. *Conclusions*. The 0.016 stainless steel archwires were found to be the less resistant to corrosion. A rhombohedral phase was detected on the nickel-titanium archwires. No major differences were observed among groups concerning phase transformation temperatures.

1. Introduction

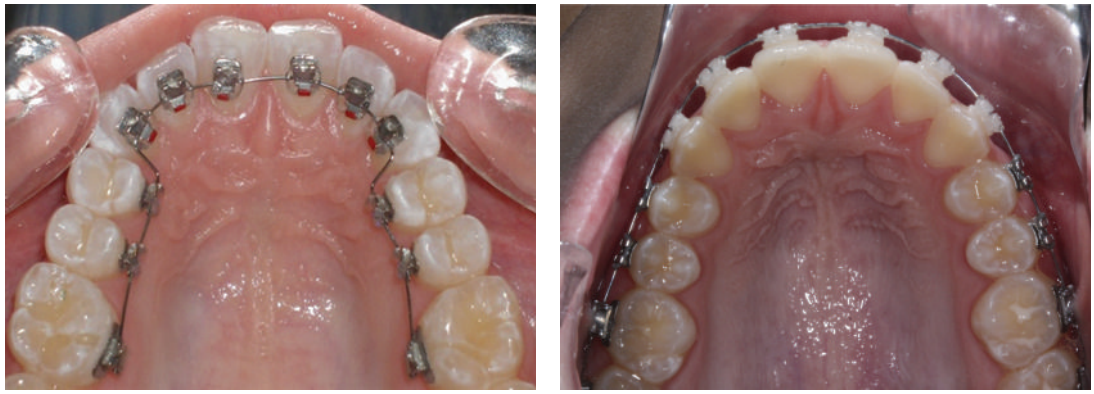
A new technique to treat malocclusions was introduced in the late 1970s by Kurz et al. [1] and Fujita [2, 3]. The main feature consisted of bonding the brackets on the lingual and palatal surfaces of teeth (Figure 1) instead of bonding them on the buccal surfaces. This bonding approach was developed in order to render the treatment invisible in the case of Dr. Kurz and to avoid orthodontic patients getting injured by the appliance while practicing martial arts in the case of Dr. Fujita.

Initially, conventional brackets and archwires were adapted for lingual orthodontic treatment. Nowadays, specific bracket systems and archwires are available from different manufacturers to perform lingual treatments. Most probably, the main advancement in this technique has been the improvement of laboratory techniques that allow an accurate bracket placement by means of indirect bonding. Since its introduction, the technique has proven to be effective to treat all kinds of malocclusions.

Biomechanics of lingual orthodontic appliances differ from those of conventional appliances [4, 5]. Changes in the point of force application and the different shape form of the archwires used in lingual orthodontics play an important role in the biomechanical differences.

The oral cavity represents a harsh environment for the orthodontic appliance of any kind [6]. Corrosion of orthodontic appliances has been thoroughly studied [7–12]. Two main concerns are directly related to the effects of corrosion: biocompatibility and appliance performance [13]. The most important aspect is the interaction that the appliance may have with the patient in terms of absorption of corrosion products and the systemic reactions that may arise. Attention has been focused on nickel [14–23] given that it is commonly found in most of the alloys used in orthodontics like stainless steel (SS), nickel-titanium (NiTi) and copper-nickel-titanium (CuNiTi).

Degradation of the orthodontic appliance is related to a decrease in the performance of its mechanical properties [7, 24]. Shape memory properties of NiTi and CuNiTi archwires



(a) (b)

Figure 1: (a) The upper arch being treated with lingual orthodontic appliances. (b) The upper arch being treated with vestibular orthodontic appliances.

can also be distorted by corrosion in terms of changes in phase transformation temperatures. Some studies with differential scanning calorimetry (DSC) have already dealt with the effect that varying forces and temperatures may have on shape memory of superelastic and nonsuperelastic NiTi-based archwires [25, 26] and also differences that could be due to degradation [27].

Corrosion potential determines the tendency for degradation of a material. It is defined as the potential of a corroding surface in an electrolyte relative to a reference electrode measured under open circuit conditions. It is often referred to as resting potential or open circuit potential (E_r, OCP) [28]. OCP delimitates the anodic area at which pitting corrosion starts. Cyclic voltammetry (CV) is used to find the potentials at which pitting occurs using the OCP as the reference point. Pitting potentials are also referred to as corrosion potentials (E_{pit}) that characterize the material studied and allow comparison of corrosion resistance with other materials.

Much research has been focused on corrosion behavior and nickel release of conventional orthodontic appliances, but to the best of our knowledge there are no studies describing the behavior that lingual orthodontic archwires have in terms of corrosion and their electrochemical characteristics. Archwires characteristics such as chemical composition, microstructure, mechanical properties and surface treatment are not readily available from manufacturers. Although it could be suspected that the manufacturers use the same alloys for lingual appliances, this should be contrasted as often happens in orthodontic studies.

The purpose of this study was to investigate the electrochemical corrosive behavior of archwires used in lingual orthodontics in invitro conditions and to determine if in-vitro corrosion affects the phase transition temperatures of the archwires of NiTi and CuNiTi alloys through DSC.

2. Materials and Methods

The lingual orthodontic archwires used for the present study (Table 1) are a commonly used sequence of six different types of archwires produced by the same manufacturer (Ormco Corp., Glendora, Calif, USA). The archwires used had different cross-section dimensions and were made of different alloys as shown in Table 1.

2.1. Corrosion Testing. Archwires were sectioned in order to produce corrosion testing samples. A 25 mm straight distal end of each archwire was cut with a sterile orthodontic plier and was isolated with wax at the point of interphase between the testing solution and the air. The corrosion tests were performed following the ISO-standard 10993-15:2000 "Biological evaluation of medical devices. Part 15: Identification and quantification of degradation products from metals and alloys."

The tests were carried out with a Voltalab PGZ 301 potentiostat (Radiometer, Copenhagen, Denmark) controlled by Voltamaster 4 software (Radiometer Analytical, Villeurbanne Cedex, France). The testing solution was Hank's Balanced Salt Solution (HBBS, Sigma Aldrich Co., 3050 Spruce Street, St. Louis, Mo, USA) that was kept at a controlled constant temperature of 37°C. The reference electrode was an Ag/AgCl/KCl electrode (E° = 0.222 V). The auxiliary electrode used was a platinum electrode and had a surface of 240 mm^2 (Radiometer Analytical, Villeurbanne, France).

The OCP was monitored for 3 hours in order to allow a leveling off of the value before the polarization resistance test. The cyclic voltammetry assay was performed by scanning the potential of the alloy of the sample at 0.25 mV/s with the minimum current set at −1 A and the maximum at +1 A with a minimum range set at 100 μA between −300 mV and +2000 mV (the upper limit for TMA archwires was +2700 mV) around the OCP value. Breakdown potentials (E_b), corrosion potentials (E_{pit}), and corrosion currents (i_{corr}) were recorded for the different samples tested.

Samples of archwires were also prepared in order to study the nickel release pattern and the changes observed in their surface through an immersion test performed for 30 days. The methods and results have been presented elsewhere [29]. Immersed samples were analyzed and compared with as-received samples and with the samples that had undergone

Table 1: Archwires used in the study.

Archwire	Alloy	Section (inches)	Batch number	Lot
Respond	Stainless steel	0.0175	203-0007	05G18
D-Rect	Stainless steel	0.016 × 0.022	201-0023	04H206H
SS 0.016	Stainless steel	0.016	206-0001	05E5
SS 0.016 × 0.022	Stainless steel	0.016 × 0.022	206-0006	05J84
NiTi	Nickel titanium	0.016	205-0023/0029	05L 175L and 05L576L
CuNiTi	Copper nickel titanium	0.017 × 0.017	205-0075/0078	05E236E and 05L 188L
TMA	Titanium molybdenum	0.016	202-0025	05J37J

the corrosion testing in the following analytical studies of DSC and image analysis.

2.2. Differential Scanning Calorimetry (DSC). An examination through DSC was performed on NiTi and CuNiTi archwires of as-received samples, samples that had undergone only the immersion test for nickel release and samples obtained after corrosion testing. The aim was to determine the phase present and derived transformations that may occur after immersion and corrosion testing. For this purpose, 2 mm segments were cut from the samples with sterile orthodontic pliers. The samples were weighed and put in an aluminium crucible and sealed. An empty control aluminium crucible was used for reference measurement. The temperatures were scanned from −100 to 200°C at a rate of 5°C/min. The analysis was performed with a 2920 Modulated DSC V2.4F instrument (TA Instruments, New Castle, Del, USA). Two samples of each group were tested to determine reproducibility of measurements of A_s, A_f, R_s (M), R_f (M), M_s, and M_f for the different groups.

2.3. Scanning Electron Microscopy (SEM). As-received samples and polarized samples were examined through scanning electron microscopy (JSM-6400; JEOL Ltd., Tokyo, Japan) to examine surface changes before and after corrosion testing. Immersed samples were also analysed, and results obtained have been presented elsewhere [29]. Images obtained are presented at ×1500 magnification.

3. Results

The results obtained for E_{pit}, i_{corr}, E_b, and OCP during corrosion testing are presented in Table 2. Representative cyclic polarization curves are shown in Figures 2 and 3.

Results showed consistent measurements for E_{pit}. The archwires with the highest tendency for corrosion were found to be Respond > D-Rect > SS 0.016 × 0.022 with values around −600 mV. The SS 0.016 archwire was found to be less resistant to corrosion with an E_{pit} of −845 mV. The archwires of titanium alloys showed a clearly higher resistance to corrosion with NiTi archwires being the most resistant (presenting E_{pit} values around 1000 mV) followed by CuNiTi and TMA archwires.

With regards to i_{corr}, the order was the same as previously shown with the exception of TMA which showed a value of 1×10^{-5} mA/cm^2.

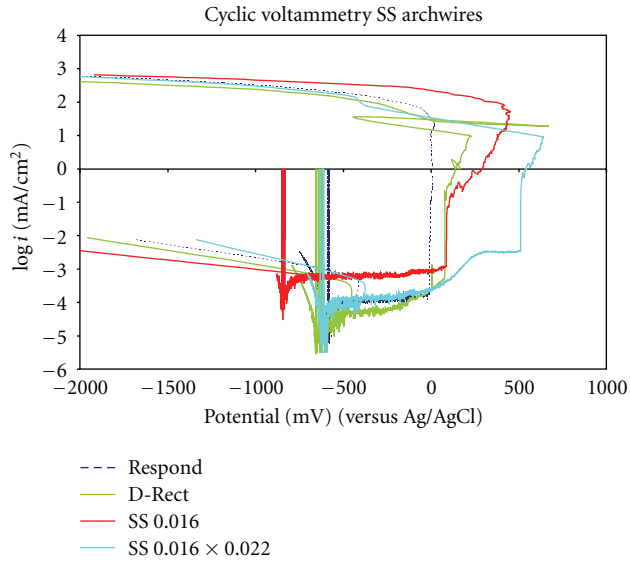


Figure 2: Cyclic voltammetry of Stainless Steel archwires.

E_b values correspond to the points of which a certain amount of electricity can be detected, but they do not correspond to values that describe the general tendency for corrosion. OCP values show a great variability among the measurements performed.

DSC heating and cooling curves for the NiTi and CuNiTi samples in the as-received, immersed and corroded state are shown in Figures 4 and 5. Values for phase transformation changes in A_s, A_f, R_s (M), R_f (M), M_s, and M_f during heating and cooling are shown in Table 3. The phase transition temperature point has been determined by taking the intersection of the tangent to the slope of the heat flow curve with the corresponding hypothetical baseline value.

As can be seen from Table 3 and Figures 4 and 5, a rhombohedral phase (R-phase) was not detected for CuNiTi archwires, while NiTi archwires showed the R-phase. Phase transition temperatures are quite different when comparing NiTi and CuNiTi wires.

SEM images of the archwires in the as-received and polarised state are shown in Figure 6. Surface differences can be seen when comparing samples that underwent corrosive testing.

Table 2: Corrosion potentials and current densities.

	E_b (mV)		OPC (mV)		E_{pit}-E_{corr} (mV)	i_{corr} (mA/cm^2)
Archwire	Mean	SD	Mean	SD		
Respond	−72.5	3.54	−456.78	151.01	−600	3.16×10^{-5}
D-Rect	60	26.87	−17.47	153.31	−650	3.16×10^{-5}
SS 0.016 × 0.022	591.5	115.26	−500.91	109.82	−610	3.16×10^{-5}
SS 0.016	162.5	60.1	−365	45.43	−845	3.16×10^{-4}
NiTi	1068.5	167.58	14.69	359.65	1000	1×10^{-2}
CuNiTi	1163.5	33.23	−175.75	103.24	1000	1×10^{-2}
TMA			−226.66	85.07	1000	1×10^{-2}

Table 3: Descriptive values of DSC curves.

	NiTi			CuNiTi		
	As-received	30-day immersed	Polarized	As-received	30-day immersed	Polarized
As	−14,7	−15,29	−13,75	6,18	9,5	4,95
Af	17,22	17,27	18,2	32,17	32,96	31,5
R_s(M)	11,17	11,23	13,36			
R_f (M)	−0,16	−1,32	1,52			
M_s	−63,42	−69,12	−65,68	14,72	13,27	13,67
M_f	−105,13	?	?	−14,07	−13,81	−15,56

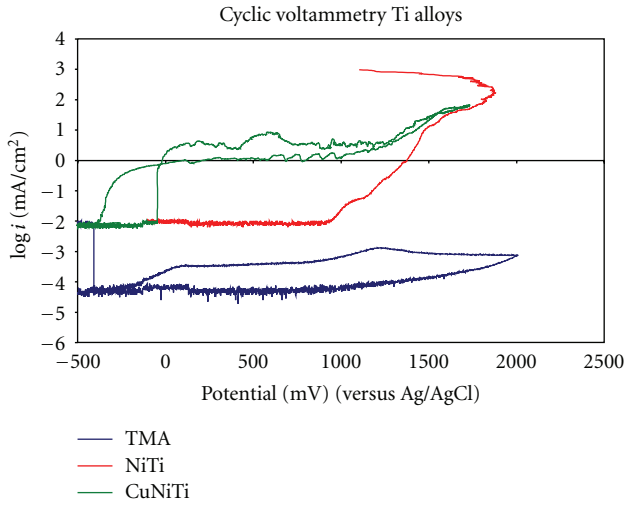


Figure 3: Cyclic voltammetry of TMA, NiTi, and CuNiTi archwires

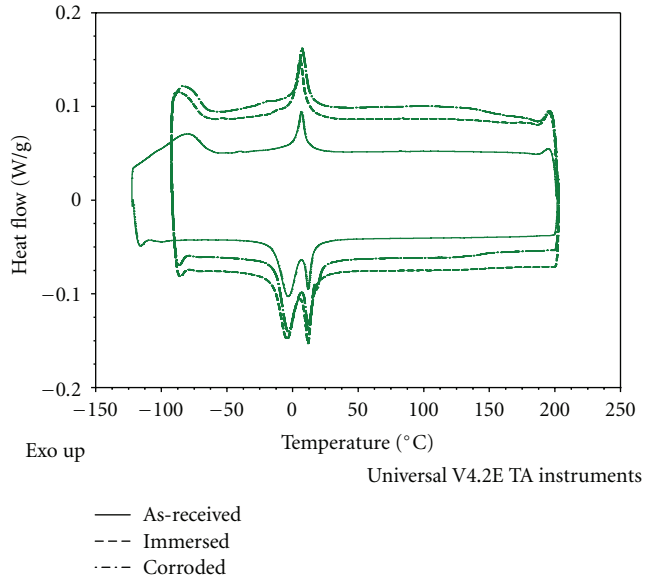


Figure 4: DSC heating and cooling curves of NiTi as-received, immersed, and corroded archwires.

4. Discussion

Electrochemical measurements of OCP and E_b showed a considerable variability among samples of the same alloy. Surface variations can influence the results obtained due to differences in surface finishing and surface defects [30–35]. All samples used in this study presented different kinds of surface defects as the SEM images of the as-received group showed (Figure 6). OCP and E_b are necessary values prior to performing the CV analysis given that they are used as the point around which the cathodic to anodic transition can be found or the corrosive attack begins. Therefore, the variability in OCP and E_b among samples of the same alloy is not important. The most important value in terms of corrosion analysis is E_{pit} given that it allows comparison between materials. Consistent results have been found when measuring E_{pit} of samples of the same alloy. OCP and E_b indicate at which point general corrosion starts, but it is E_{pit} that shows the capacity to withstand corrosion. Often in the orthodontic literature, OCP, E_b, and E_{pit} are used as the same measurement while in fact they are different concepts.

CV results showed similar corrosion resistances for Respond, D-Rect, and SS 0.016 × 0.022 archwires. The SS

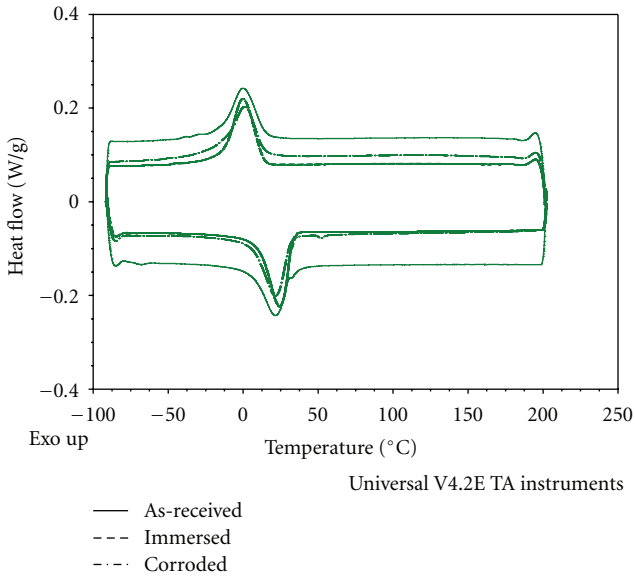


Figure 5: DSC heating and cooling curves of CuNiTi as-received, immersed, and corroded archwires.

0.016 archwire was found to be less resistant to corrosion. Similar corrosion potentials were expected for SS archwires given that they are all made of the same alloy and produced by the same manufacturer. SEM images showed numerous defects in the SS 0.016 archwires when compared to the SS 0.016 × 0.022 and Respond and D-Rect archwires. This fact could account for the differences found in CV when comparing archwires of the same alloy and manufacturer. The results obtained are comparable to those obtained by other authors [36].

NiTi and CuNiTi showed high corrosion resistance. TMA archwires were shown to be inert to corrosion at the conditions under which the test was performed. These results are similar to those found by other authors for archwires used in conventional orthodontics [37–39].

DSC testing has been previously performed on 35°C CuNiTi archwires of the same manufacturer as those used in the present research [27]. No statistically significant differences were found among the as-received archwires. Furthermore, no statistically significant differences were found when the authors compared as-received and retrieved archwires, except for a significant reduction in heating enthalpy associated with the martensite-to-austenite transition in the 27°C archwires.

Although some conventionally performed DSC studies were unable to detect the R-phase on 35°C CuNiTi archwires [26, 40], other authors have observed it on a constant basis [27, 41, 42]. It seems that temperature-modulated DSC as used by Brantley et al. [41] may provide greater resolution that allows detection of the R-phase. Notwithstanding, conventionally performed DSC has also been able to detect an R-phase. Some studies suggest that the presence of an R-phase may be related to the amount of Cu present in the alloy. Some authors [43, 44] concluded that CuNiTi alloys that contain amounts of copper above 5% wt do not exhibit an R-phase, this being in agreement with Brantley et al. [41]. This could be the reason why the R-phase was not detected in the samples used in our study.

The main difference among CuNiTi samples can be seen at the immersed sample. The 50% austenite phase transformation temperature has significantly been moved towards a higher temperature (Figure 5). An increase in the hysteresis can be observed given that the 50% martensite phase transformation temperature remained unchanged.

DSC results for as-received CuNiTi and polarized samples were similar. It seems that although a lack of structure results after potentiostatic anodic polarization, CuNiTi archwires do not alter their phase transition temperature characteristics.

Biermann et al. [27] found that the A_f of the 27°C and 35°C CuNiTi archwires were within approximately 2°C of the manufacturer's claim, which was similar to the A_f found in other studies [42, 45]. Results obtained in the present research for CuNiTi archwires show similar outcomes, the A_f difference being within 2°C to 3.5°C from the manufacturer's claims considering as-received archwires and archwires tested for corrosion, respectively.

Clinical implications around this particular transition temperature of 35°C are quite important because a variation of 2–4°C in the transition temperature could mislead the clinician seeking a particular orthodontic effect at a given temperature.

All tested NiTi samples showed the presence of an R-phase prior to the austenitic and martensitic transformation. This is in agreement with other results obtained by several authors [41, 42, 46, 47]. Leu et al. [48] employed DSC to show that phase transformations were not direct processes and that they involved an immediate rhombohedral structure as described by Goldstein et al [49] and Otsuka [50].

The heating of the samples shows the endothermic peaks that belong to the martensite-to-austenite transformation.

There is an endothermic peak very close to 0°C that according to the literature and actual knowledge [47] is the rhombohedral phase (R-phase). More than 50% of the R-phase transformation is attained before reaching 0°C for all samples. The endothermic peak for the 50% R-phase shows slight differences toward higher temperatures but they have not been considered to be important.

The second endothermic peak for the 50% austenitic transformation is also the same for all specimens, and the value is 12.5°C with an As centered around 6.25°C, the point at which the R-phase is completed. NiTi archwires seem to have attained almost a fully austenitic phase at a temperature close to body temperature.

Martensite temperatures are low. NiTi-tested archwires are austenitic at room temperature. This is in agreement with previous studies done by Bradley et al. [46].

The image study through SEM allows differences to be determined when comparing polarized samples with as-received and immersed samples. As already shown elsewhere [29], image techniques that allow surface roughness measurement are needed in order to describe differences among samples that have undergone an immersion test and as-received archwires given that SEM images did not allow

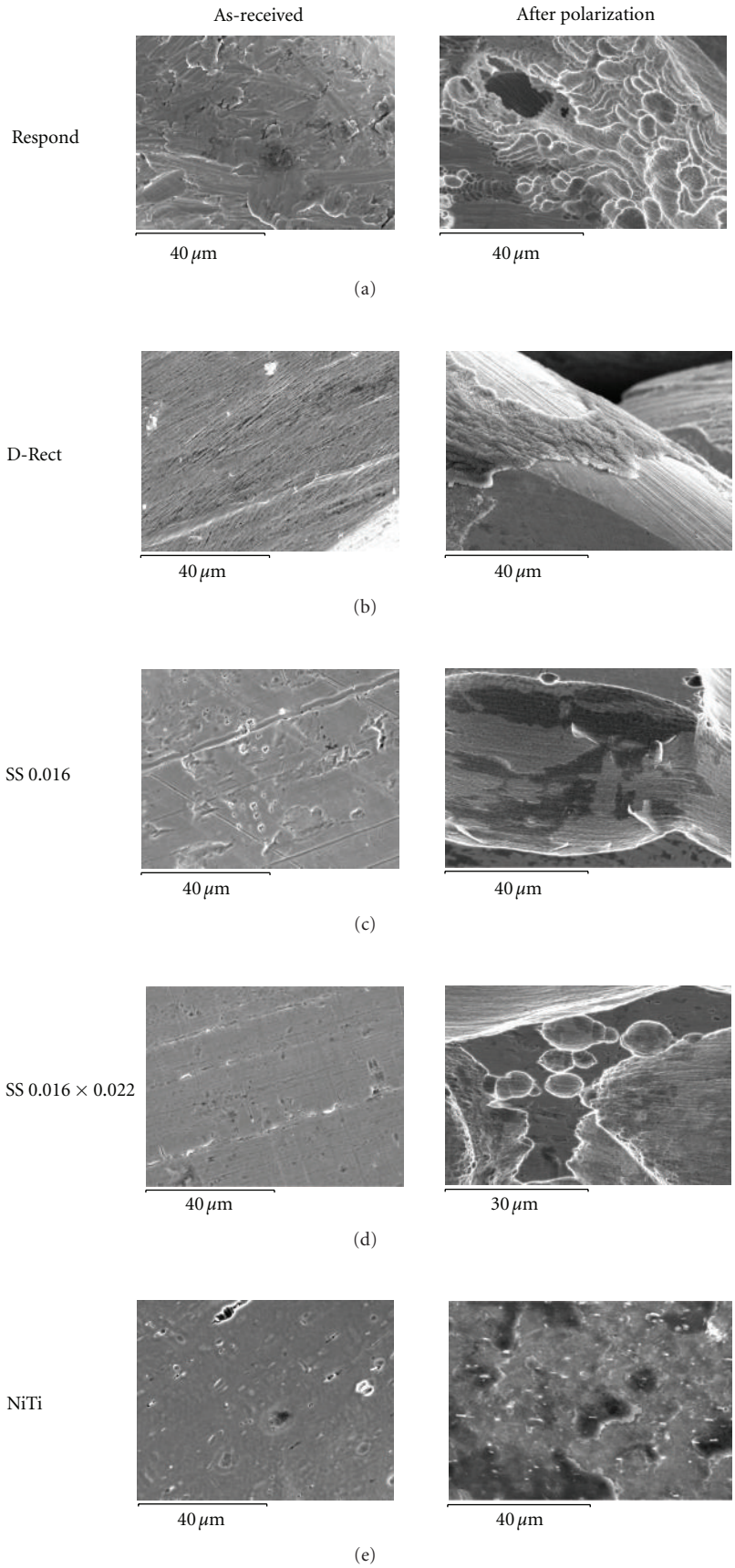


Figure 6: Continued.

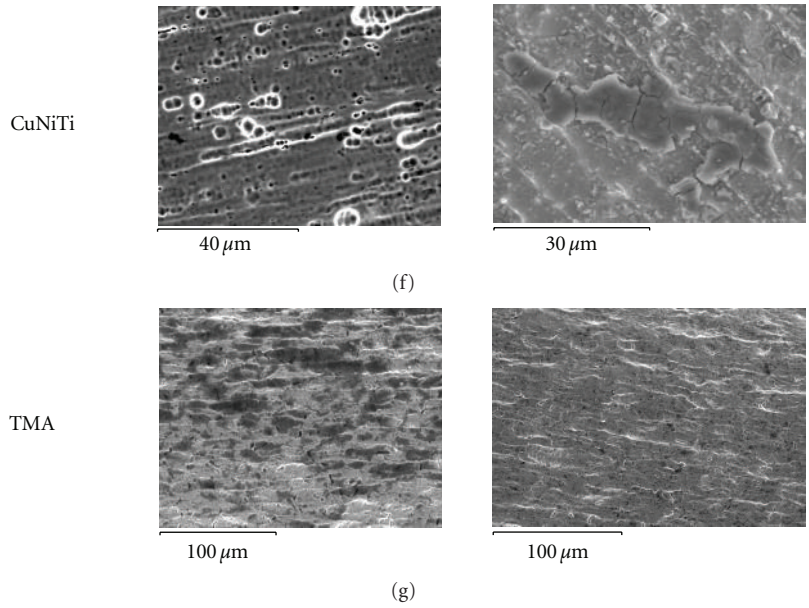


Figure 6: Scanning electronic microscope images of archwires in as-received state and after polarization (scale bars indicate 40 μm for all images except TMA for which they indicate 100 μm).

differences to be seen between as-received and immersion-tested archwires.

All SS archwires studied presented surface manufacturing defects that seem to be due to the drawing processes. As Hunt et al. [51] state, these defects may be the result of the archwires being drawn through diamond dies during production with the resulting longitudinal grooving. Depending on the final cross-section, different manufacturing processes are added. It seems that the higher the number of mechanical processes involved in the manufacturing of the wire, the higher the number of manufacturing defects found. This is in agreement with the observations done on the SEM images of the as-received SS archwires.

After polarization, Respond archwire shows a generalized affected surface. All the wires that form the strand have been severely damaged. D-Rect archwire seems to withstand polarization better than the Respond wire. Unpitted stainless steel can be observed, and pits seem to be mainly located at the edges of the wires that form this braided wire.

The 0.016 and 0.016 $\times$ 0.022 SS archwires seem to be similarly affected by polarization. The edge of the 0.016 $\times$ 0.022 archwire has been dramatically affected. Edie et al. [52] observed a similar finding noticing that pits occurred along the sharp edges of rectangular wires where the electric field would be the greatest.

NiTi, CuNiTi, and TMA archwires seem to behave differently from the SS archwires under corrosion testing.

Initial scans of the as-received archwires show that they have a rough surface finish. Surfaces do not present generalized grooving due to manufacturing processes as was observed with SS archwires.

The SS group of archwires shows low resistance to potentiostatic polarization as the scans show with severe pitting corrosion after polarization and high structural damage.

NiTi shows discrete grooving perpendicular to the longitudinal axis of the archwire that recalls the surface defects observed at the 0.016 SS archwires. Pickling of the surface is higher than that observed with the 0.016 SS archwire. The NiTi surface has a high density of pores and white spots that are highly oxidized points. According to Walker et al. [7], the bright white spots appear to be inclusions in the wire. NiTi scans are very similar to those obtained by other authors [7, 9, 12] with no significant differences to be highlighted.

Surface changes of NiTi can be readily seen after potentiostatic polarization. The polarized sample shows a regularly spotted roughened surface as described by other authors [7].

CuNiTi shows a characteristic surface topography with a rough surface, rougher than the NiTi archwire. It shows longitudinal grooves, and the characteristic surface of this archwire seems to be due to the recrystallized fine grains oriented in the direction of pull of the manufacturing process as described in other studies for conventional archwires (Fischer-Brandies et al. [40] and Walker et al. [7]). Fischer-Brandies et al. [40] characterized some inclusions in the archwire as Ti-rich inclusions of 2 μm length, C-rich precipitations, and some surface defects at the edges of this squared wire. The scan obtained by the authors is almost identical to the obtained in the present research.

Potentiostatic polarization of the CuNiTi archwire introduces clear changes of surface topography. The initial characteristic topography previously described disappears, and a very rough surface is presented. There is a roughening of the surface with an oxide layer present at the side shown on the scan. The edges of this squared wire are very damaged.

TMA scans show a very rough surface before and after polarization. Its surface is rougher than that of NiTi and CuNiTi archwires.

Although becoming rough after potentiostatic polarization, NiTi, CuNiTi, and TMA archwires did not alter their general shape, keeping their section after the corrosion tests and thus being more resistant than the SS archwires studied. The most resistant to potentiostatic polarization in terms of maintaining the original surface topography are the TMA archwires followed by the NiTi archwires and finally the CuNiTi archwires.

5. Conclusions

The lingual orthodontic archwires tested showed similar properties of corrosion resistance as those used in conventional orthodontics.

An R-phase was detected for NiTi archwires while, 35°C CuNiTi archwires did not show it. A difference of 2°C to 3.5°C from the manufacturer's claim was found in the as-received and polarized samples, respectively, for the CuNiTi archwires.

SEM analysis showed that manufacturing defects are frequent on SS archwires and are highly distorted when polarized. NiTi, CuNiTi, and TMA archwires show a high resistance to polarization with minimal structural damage.

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