

Electrochemical Corrosion Behavior of Dental/Implant Alloys in Artificial Saliva

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The corrosion behavior and passive film characteristics of various dental alloys such as Co-Cr, Ni-Cr, Cu-Ni-Al, and commercially pure Ti (c.p. Ti) were evaluated in artificial saliva medium by utilizing electrochemical impedance spectroscopy (EIS), Tafel polarization, and cyclic polarization studies. EIS studies were carried out for various durations viz. 1 h, 1 day, and 7 days to evaluate the stability of passive film and change in corrosion characteristics with respect to time. Electrochemical parameters such as E_{corr} , i_{corr} , corrosion rate, passive film characteristics with respect to time were obtained from various studies mentioned above. The corrosion resistance decreased in the order Cu-Ni-Al > cp Ti > Co-Cr (Commercial) > Ni-Cr > Co-Cr (DRDO developed) in artificial saliva solution.

Keywords artificial saliva, corrosion, dental alloys, EIS study, passive film

1. Introduction

Non-precious metals and alloys are rapidly coming in vogue replacing precious or noble alloys in dentistry. They are being used in full-cast and metal-ceramic restorations besides removable partial dentures. Approximately 90% of all removable partial dentures are now cast from non-precious alloys containing Co, Cr, Ni (Ref 1). These alloys possess advantageous mechanical properties, and therefore can be easily cast into desired thinner shapes viz. crowns, bridges, fixed or removable partial dentures without compromising the rigidity (Ref 2-4). In oral cavity, the salinity of saliva approaches that of seawater and tends to be highly corrosive to most non-noble metals (Ref 1). The pH of saliva may vary between 2 and 11 while the temperature in the oral cavity may be between 0 °C and 70 °C, all these variations mostly depend on the food intake (Ref 5).

Thus corrosion resistance, besides other considerations such as affordability and biocompatibility of alloys (Ref 6-8) play an important role. Although base alloys possess inferior corrosion resistance as compared to noble alloys (Ref 9), they outweigh latter in terms of mechanical properties. In designing non-precious alloys, chromium is added in the range of 15-30% to obtain an optimum value of corrosion resistance and mechanical strength (Ref 1). Ni addition is also performed to increase corrosion resistance and mechanical properties. It has been observed that in nickel alloys chromium content of more than

20% and a molybdenum content of greater than 4% can ensure adequate corrosion resistance (Ref 10, 11).

A review of literature reveals various studies on dental alloys. Ameer et al. (Ref 12) have studied potentiodynamic and electrochemical impedance behavior of Co-Cr and Ni-Cr non-precious alloys and concluded that Co-Cr-Mo alloys are more corrosion resistant as compared to Ni-Cr-Mo alloys. Huang (Ref 13) has investigated Ni-Cr alloy in artificial saliva using electrochemical impedance spectroscopy (EIS) study wherein the film formation characteristics were studied after 2 h of film stabilization. Manaranche and Hornberger (Ref 14) have carried out corrosion and biocompatibility studies for Ni-Cr and Co-Cr dental alloys using inductively coupled plasma technique among other methods. Takemoto et al. (Ref 15) have carried out electrochemical corrosion behavior and dissolution studies of cobalt-chromium alloy (Co-Cr) and commercially pure titanium (cp Ti) alloys in various gargle solutions. Viennot et al. (Ref 16) Eschler et al. (Ref 17) and Reclaru et al. (Ref 18, 19) Dong et al. (Ref 20) Lucas et al. (Ref 21) Elagli et al. (Ref 22) have carried out comparative electrochemical studies of cobalt-chromium dental alloys with those doped with precious metals in artificial saliva viz. Fusayama solution/corrosive medium. Duffo and Castillo (Ref 23) evaluated corrosion behavior of Cu-Ni-Al alloy in natural as well as in various artificial saliva solutions using electrochemical techniques. Johanson and co-workers (Ref 24) have studied corrosion behavior of copper, nickel alloys in artificial saliva and saline solutions. Taira et al. (Ref 25) have conducted study on Ti alloys and found that the alloys studied showed a stable passivity in the corrosion test.

For laboratory studies generally an artificial solution is used since natural saliva composition cannot be generalized. Literature survey reveals various studies which have proposed different compositions for artificial saliva solution. A comparative study of such solutions has been performed by Duffo and Castillo (Ref 23).

The presence of Cr improves the corrosion resistance of alloys in a corrosive environment due to the formation of a Cr-rich, passive oxide film which is highly resistant to acid attack. Similarly, presence of molybdenum in the Ni-Cr based

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alloy increases the resistance to localized corrosion in the chloride containing environment (Ref 26). Therefore, for Ni-Cr-based dental alloys, the addition of 12% Cr (minimum value) and 2-5% Mo to the alloy bulk is well recommended from the corrosion resistance point of view (Ref 3). cp Ti has been widely used as dental implant material (Ref 27).

Since dental alloy implants are always in touch with media (saliva), it is felt that Electrochemical Impedance Spectroscopy studies should be carried out for long duration, which can provide wealth of mechanistic information. However, no studies were reported on time-dependent corrosion characteristics of dental implants. In this study, the corrosion behavior and passive film characteristics have been studied on various alloys for different duration, which can throw light on the passive film stability for these alloys. These results were corroborated with cyclic polarization studies.

Present investigation is aimed at electrochemical corrosion study of various alloys viz. Co-Cr (developed by Defence Research and Development Organization, India) and Co-Cr (Commercially available), Ni-Cr, Cu-Ni-Al and cp Ti by using Tafel polarization, cyclic polarization and EIS methods. All studies were carried out in artificial saliva Solution.

2. Experimental

2.1 Materials and Methods

Four different types of dental base alloys for implant Supraconstructions along with cp Ti were used for the study. The composition of Ni-Cr alloy was 75.8% Ni, 15% Cr, 2.8% Mo, 0.7% Nb, 2.9% Al, and 1.5% Mn. The commercially available Be free Co-Cr alloy was of the composition 65% Co, 28% Cr, 4.5% Mo, and 1.6% Si. The composition of Cobalt-Chromium-Molybdenum-Silicon alloy (Indigenously developed by DRDO, India) was 61% Co, 31% Cr, 3.6% Mo, and 1.3% Si. The composition of Copper-Nickel-Aluminum Alloy (Cunial Alloy) is proprietary. Besides these alloys commercially pure Ti was also used in this investigation.

For all the experiments AR Grade chemicals/reagents were used and solutions were prepared in double distilled water. Experiments were carried out in a three cell assembly with Platinum as a counter electrode and standard calomel electrode (SCE) as a reference electrode. Potentiostat/Galvanostat model 283 coupled with frequency response detector model 1025 were both supplied by EG&G Instruments, USA was used in the studies. Data acquisition was done through a computer software M398, Version 1.30, EG&G PAR, USA. The experiments were carried out at ambient temperature. Samples were prepared by cutting the alloys into 1 cm² pieces and then mounting them into epoxy base, thus leaving only the test specimen area into the contact of the test electrolyte. Samples were polished with successively finer grade of emery papers (up to 800 grit) and then degreased with toluene. Modified Fusayama solution (Ref 28) was used as artificial saliva whose composition was NaCl (0.4 g/L), KCl (0.4 g/L), CaCl₂·2H₂O (0.795 g/L), NaH₂PO₄·H₂O (0.690 g/L), KSCN (0.3 g/L), and urea (1.0 g/L).

The following electrochemical studies were carried out:

(i) *Open Circuit Potential (OCP)*: Open circuit potentials were measured in the electrolytes before carrying out the experiments. The OCP was measured for duration of 3000 s.

- (ii) *Tafel Studies*: Tafel plots of various alloys were obtained by exposing them into respective electrolytes and polarizing from $E_{\text{corr}} - 250$ mV to $+250$ mV vs. SCE with scan rate of 0.166 mV/sec.
- (iii) *Cyclic Polarization Studies*: The specimens were polarized in a cyclic manner from -250 mV from OCP to a vertex potential of 1.2 Volts and final potential of the cyclic scan was -250 mV vs. OCP. Scan rate during the experiment was 1 mV/Sec.
- (iv) *Electrochemical Impedance Spectroscopy*: Single sine AC Impedance studies were carried out in the frequency range 100 kHz-10 mHz. The AC signal imposed during the experiment was of 5 mV rms amplitude. Data were recorded at 5-frequencies/decade. In this article, bode plots are used for analysis of the corrosion behavior of various alloys. Bode plots are Log frequency versus phase shift of AC sine wave. In bode plots, it is easy to understand how the impedance/phase angle depends on frequency. At low and higher frequency the behavior of the cell (sample + electrolyte) is resistor like and phase angle is nearly zero. At intermediate frequency, the phase angle shifts toward 90° and for a perfect capacitor, the phase angle is 90°. Depending on the value of phase angle maxima, the information regarding equivalent circuit model can be deduced. For example, if there is only one phase maxima peak, then the equivalent circuit model corresponds to a simple Randles circuit. A detailed account of

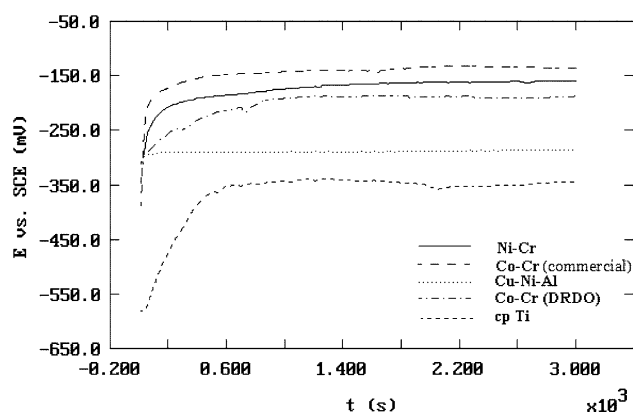


Fig. 1 Time potential curves of various dental materials

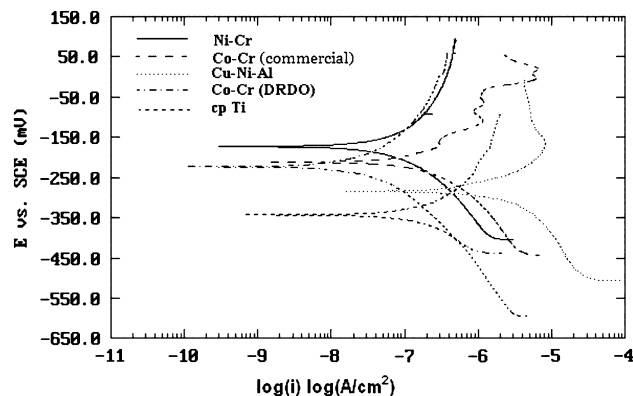


Fig. 2 Current density-potential curves (Tafel plots) of various dental materials

impedance plots may be obtained from the research papers of Walter (Ref 29) and Kolman (Ref 30). The plots were analyzed to obtain equivalent circuits by Zsimpwin software provided by EG&G.

3. Results and Discussion

Figure 1 shows the time potential plots of various dental materials viz. (i) Ni-Cr, (ii) Co-Cr (Commercial), (iii) Cu-Ni-

Al, (iv) Co-Cr (DRDO), and (v) cp Ti exposed to artificial saliva solution. Upon exposure of these samples in artificial saliva (modified fusayama solution), it was observed that all materials except Cu-Ni-Al showed a shift in the potential toward noble direction. The E_{corr} shift was maximum (251 mV) in case of Co-Cr (Commercial) while minimum E_{corr} shift was observed in case of Cu-Ni-Al (22 mV). This is possibly due to better passive film formation in Co-Cr (commercial) alloy. Finally, the order of E_{corr} after stabilization was Co-Cr (Commercial) > Ni-Cr > Co-Cr (DRDO) > Cu-Ni-Al > cp Ti.

Figure 2 shows the current density-Potential characteristics (Tafel plots) of various dental materials exposed to artificial

Table 1 Various DC electrochemical parameters calculated from Tafel plots for various dental materials

S. No.	Material	$\beta_a, \times 10^{-3}$	$\beta_c, \times 10^{-3}$	$R_p, \text{ KOHms}$	$E_{\text{Corr}}, \text{ mV}$	$i_{\text{Corr}}, \mu\text{A}/\text{cm}^2$	Corr. Rate, $\times 10^{-3}, \text{ mpy}$
1	Ni-Cr	544.2	257.0	387.4	-172.8	0.198	88.43
2	Co-Cr (Commercial)	404.9	248.5	130.0	-207.9	0.4793	214.1
3	Cu-Ni-Al	8968	405.8	14.63	-283.8	11.200	5002
4	Co-Cr (DRDO)	273.3	182.3	909.8	-221.0	0.05442	24.30
5	cp Ti	439.3	414.0	128.8	-342.5	0.6980	311.7

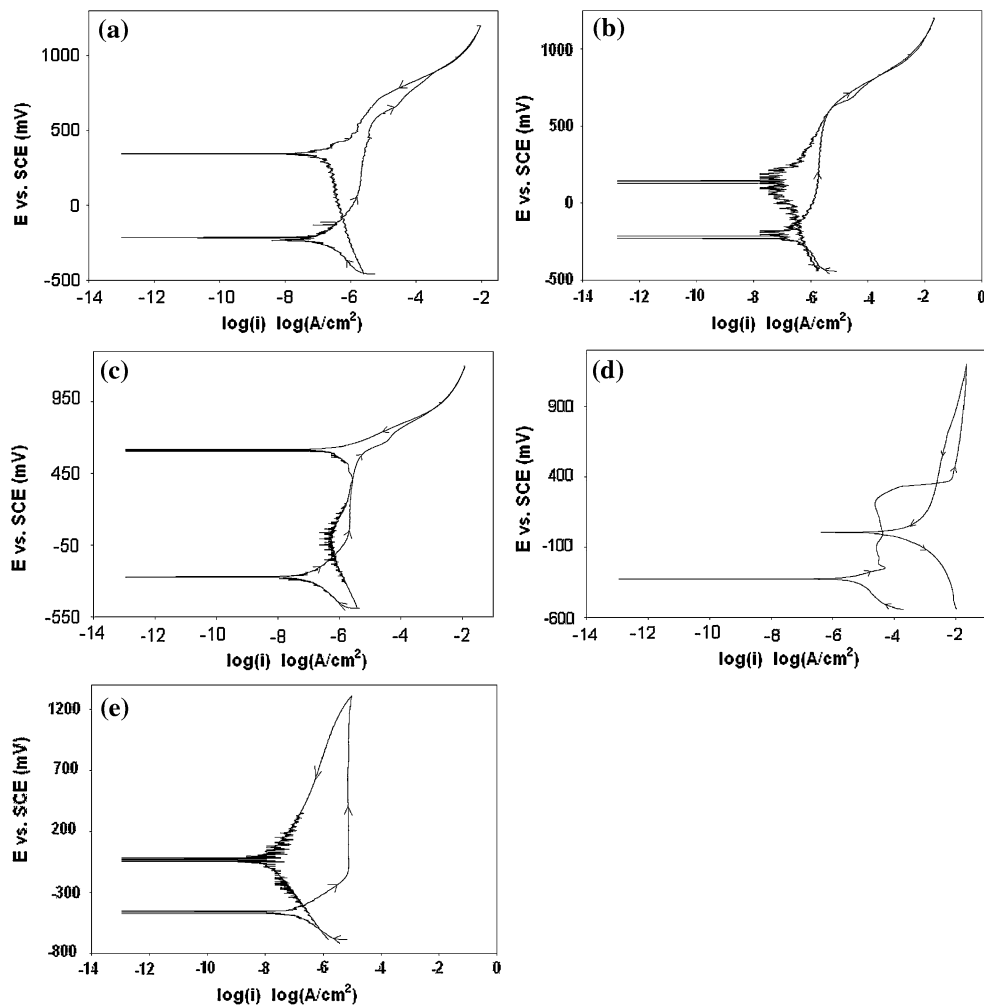


Fig. 3 Cyclic polarization curves of alloys. (a) Ni-Cr, (b) Co-Cr (Commercial), (c) Co-Cr (DRDO), (d) Cu-Ni-Al and, (e) cp Ti

saliva solution. It is evident from the figure that the Tafel behavior of all these alloys is highly distinct. Corrosion current was maximum in Cu-Ni-Al and it decreased in the order cp Ti > Co-Cr (Commercial) > Ni-Cr > Co-Cr (DRDO). The electrochemical parameters are compiled in Table 1.

It can be seen from the figure that corrosion rate of Co-Cr (DRDO) was minimum possibly because of better spontaneous passive film formation due to much higher chromium content. The electrochemical reactions occurring on these alloys appear to be more or less mixed anodic and cathodic control except Cu-Ni-Al alloy where very high β_C was observed (Table 1).

Cyclic polarization study of Ni-Cr alloy exposed to modified Fusayama solution is shown in Fig. 3a. Ni-Cr alloy showed active-passive behavior with a pitting potential of 818 mV. On reversing the potential the reverse scan almost traces through the forward scan up to 900 mV and further reversal leads to shift of hysteresis loop toward lower current region indicating no pitting tendency under these conditions. The Co-Cr (Commercial) and Co-Cr (DRDO) also showed a similar behavior (Fig. 3b, c). However, in Cu-Ni-Al alloy on reversing the potential, the scan initially follows a lower current path and thereafter traces a higher current path (Fig. 3d) possibly indicating a poor repassivation by this alloy in artificial saliva. Therefore, this alloy has a tendency for localized corrosion. The

passivation current density of Ni-Cr, Co-Cr (Commercial), Co-Cr (DRDO), and cp Ti were 1.65, 1.7, 1.989, and 7.122 $\mu\text{A}/\text{cm}^2$, respectively. The lower passivation current in the first three alloys are due to chromium factor present. However, in cp Ti the pitting potential was attained up to a vertex potential of 1.4 V. Moreover on reversing, the scan traced through a lower current region with hysteresis loop area far higher than any material mentioned above indicating excellent repassivation and no tendency for pitting corrosion.

Most of the studies regarding dental alloys in saliva to the best of our knowledge, deal with potentiodynamic polarization of these alloys where almost all dental alloys showed active-passive behavior (Ref 31-33). However, cyclic polarization behavior was studied by Gil et al. (Ref 34) non-dental alloys where the passive behavior with respect to surface roughness in Hanks solution was evaluated. A linear behavior was observed when current density was plotted against roughness. However, our studies more or less deal with overall corrosion behavior of these materials in artificial saliva. A summary of various parameters obtained from cyclic polarization experiments is given in Table 2.

Figures 4-6 represent Bode phase plots of various dental alloys in artificial saliva (modified Fusayama Solution) for 1 h, 1 day, and 7 days duration, respectively. After 1 h of film stabilization, Ni-Cr alloy showed maximum phase angle 75.1°

Table 2 Various parameters obtained from cyclic polarization data

S. No.	Material	E_{pass} , mV	i_{pass} , $\mu\text{A}/\text{cm}^2$	E_{pits} , mV	i_{pits} , $\mu\text{A}/\text{cm}^2$
1	Ni-Cr	79.0	1.65	578	5.7
2	Co-Cr (Commercial)	102.0	1.7	638	7.733
3	Cu-Ni-Al	-190.0	31.11	274	42.11
4	Co-Cr (DRDO)	31.0	1.989	586	5.7
5	cp Ti	-134.0	7.122

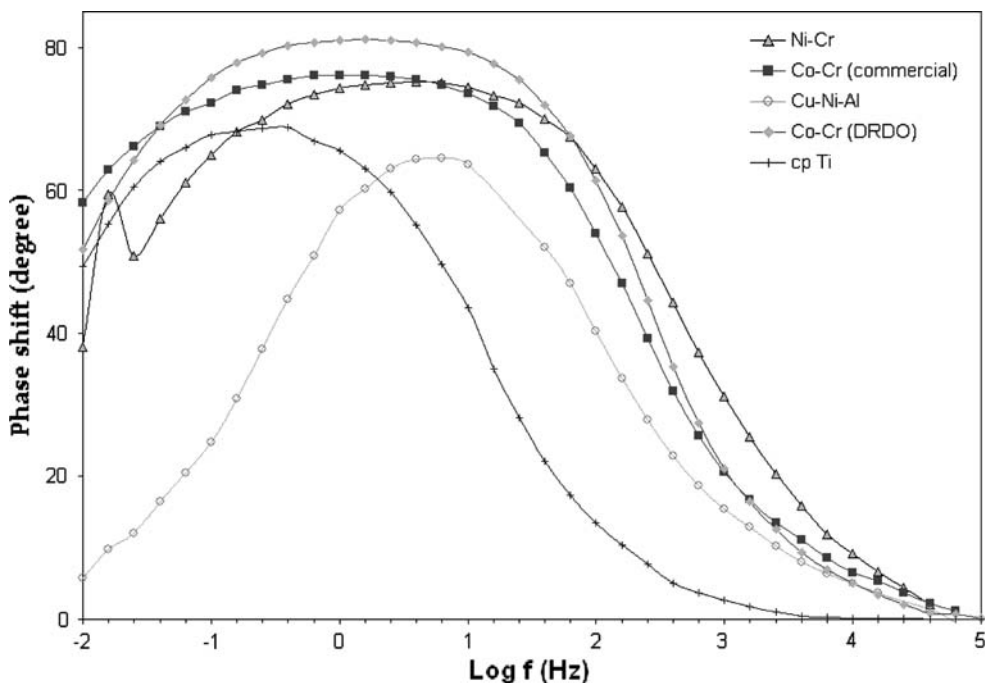


Fig. 4 Bode plots for various alloys after 1 h of film stabilization

at 3.98 Hz. Co-Cr (Commercial) alloy showed maximum phase angle 76.1° at 1.58 Hz. Cu-Ni-Al alloy showed maximum phase angle 64.5° at 6.31 Hz. Co-Cr alloy (DRDO) displayed maximum phase angle 81.1° at 1.58 Hz. cp Ti showed maximum phase angle 68.7° at 0.398 Hz. frequency. After 1 day exposure, Ni-Cr alloy showed maximum phase angle 75.2° at 0.398 Hz. Co-Cr (Commercial) alloy exhibited maximum

imum phase angle 74.6° at 0.631 Hz. Cu-Ni-Al alloy showed maximum phase angle 73° at 1580 Hz. Co-Cr (DRDO) showed maximum phase angle 79.6° at 0.631 Hz. Cp Ti showed maximum phase angle 66° at 0.1 Hz. After 7 days of exposure, as evident from Fig. 6, Ni-Cr alloy, Co-Cr (Commercial) and Co-Cr (DRDO) alloys showed maximum phase angles at 0.158 Hz. with the values being 75.4°, 76.1°, and 77.1°,

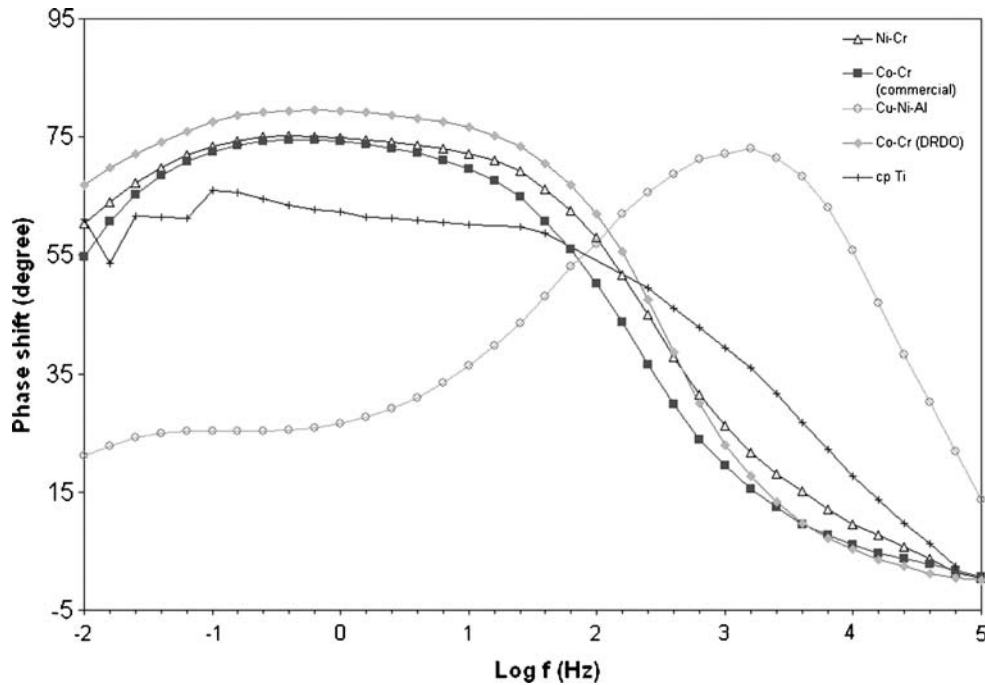


Fig. 5 Bode plots of various alloys after 1-day period

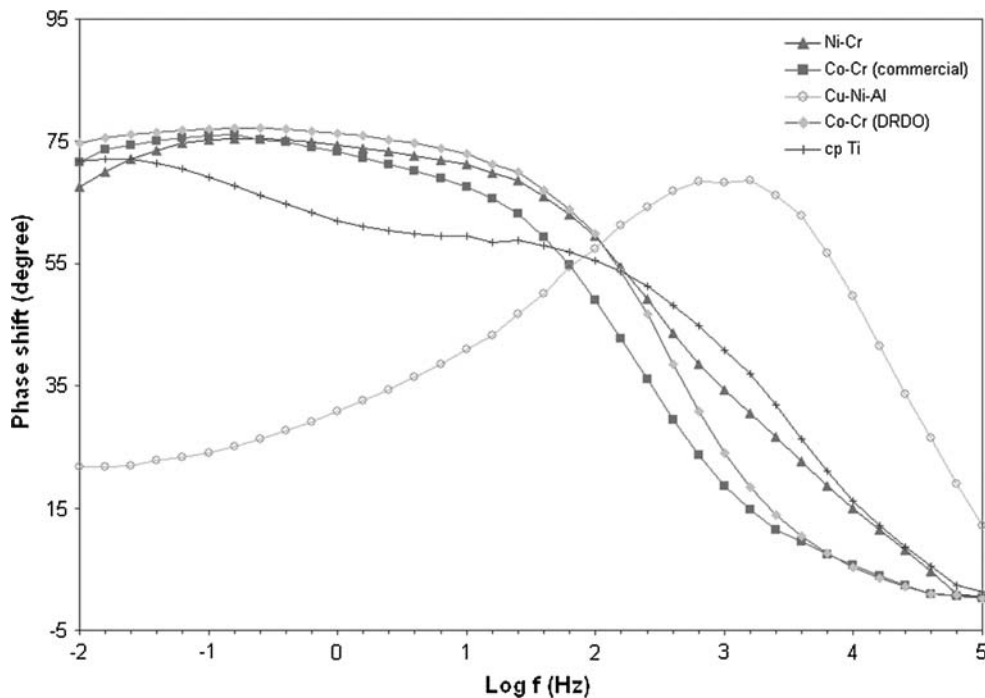


Fig. 6 Bode plots of various alloys after 7-day period

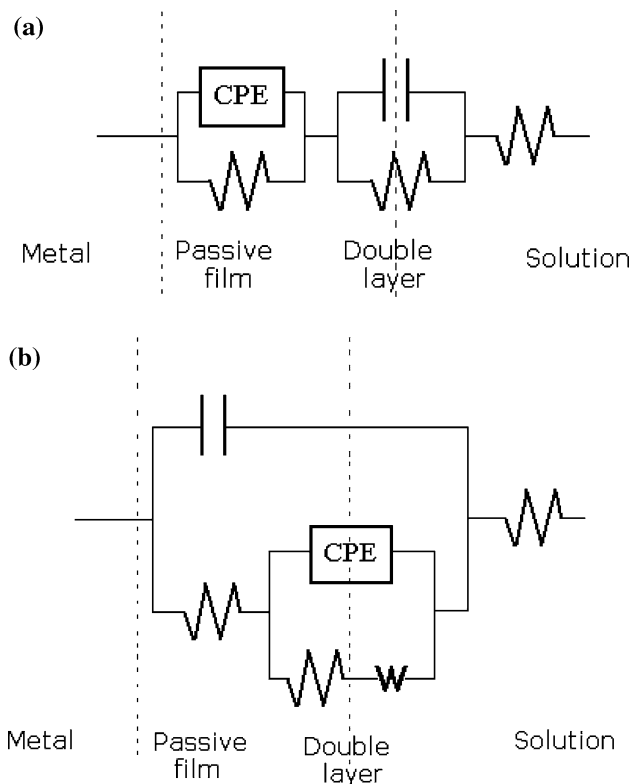


Fig. 7 Equivalent Circuit for Alloys in Fusayama solution, (a) for Co-Cr (Commercial), Co-Cr (DRDO), Ni-Cr, and cp Ti, (b) for Cu-Ni-Al alloy

respectively. Cu-Ni-Al alloy displayed phase angle maxima of 68.6° at 1580 Hz and cp Ti was seen to exhibit maximum phase angle 72.1° at 0.251 Hz.

It is clear from the above impedance results that phase angle maxima shifts to lower frequency with respect to time for Co-Cr (Commercial), Co-Cr (DRDO), Ni-Cr, and cp Ti showing an increased resistance to charge transfer supported by a stable passive film formation. However, in Cu-Ni-Al alloy the phase angle maxima shifts toward higher frequency region with respect to time indicating diffusion controlled electrochemical reaction probably due to the porous/heterogenous film formation.

The impedance behavior of dental alloys except Cu-Ni-Al alloy can be modeled by the circuit model seen in Fig. 7a. The proposed model features two time constants and a solution resistance. It can be seen from the bode phase plots (Figs. 4–6) that the two time constants are overlapped. Therefore, one of the non-ideal capacitive elements can be taken as constant phase element (CPE). At the higher frequency RC combination is attributed to passive film consisting of oxide film resistance and its capacitance. This combination is in series with an RQ combination due to double layer.

However, in the case of Cu-Ni-Al alloys the equivalent circuit model proposed can be seen in Fig. 7b. Here also two time constants were fitted due to porous film and double layer, respectively. The best fit was achieved when these time constants are in parallel. Moreover, Warburg diffusion impedance has also been depicted in the model.

The above observation can be corroborated with cyclic polarization studies where a stable passivity was observed on all dental alloys except in the case of Cu-Ni-Al alloy where

poor repassivation characteristics were observed. Therefore, all dental alloys except Cu-Ni-Al alloy form a stable passive film without any significant time-dependent dissolution.

4. Conclusion

From cyclic polarization studies it is clear that all the alloys showed active-passive behavior with large potential independent region terminated by a region comprising significant increase in current density except in cp Ti. Furthermore, cyclic polarization also reveals that all alloys are resistant to localized corrosion except Cu-Ni-Al alloy. Based on E_{pit} values, the tendency of pitting is in the order Cu-Ni-Al > Ni-Cr > Co-Cr (DRDO) > Co-Cr (Commercial) > cp Ti. The corrosion rates were observed in the order Cu-Ni-Al > cp Ti > Co-Cr (Commercial) > Ni-Cr > Co-Cr (DRDO).

Electrochemical impedance spectroscopy analysis revealed a stable passive film formation on all dental alloys except Cu-Ni-Al, where a porous film was formed. The equivalent circuit model for Co-Cr (Commercial), Co-Cr (DRDO), Ni-Cr, and cp Ti showed two time constants which are in series, whereas in Cu-Ni-Al alloys Warburg diffusion impedance was also observed along with two time constants in parallel.

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