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Electrochemical determination of the corrosion resistance of noble dental casting alloys

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Forty-four dental casting alloys and pure metals have been evaluated for corrosion resistance in an artificial saliva. The electrochemical tests included measurement of the rest potential and tracing of polarization curves. Qualitative comparisons of the normalized polarization curves, and quantitative determination of corrosion resistance, and of corrosion and breakdown potentials, provided complementary means of evaluation. Alloys were assigned two families: alloys for crowns and bridges (C&B alloys), and alloys for ceramic veneering (PFM alloys). Among these two families, groups were based on the gold-content (high and low), and on the main non-gold component: silver for C&B alloys, and palladium for PFM alloys. Pure metals and some non-noble alloys have been included as references. From this study, it clearly appears that the PFM alloys, having a higher content of noble metals, are more corrosion resistant than the C&B alloys containing copper and silver. Some relationships between noble-metal content and electrochemical parameters such as rest potential and corrosion resistance, have been identified, but the strength of the correlations is somewhat lowered by the influence of additional parameters, such as the presence of non-noble components or the nature of the microstructure. The present investigation is part of a larger study including other electrochemical parameters as well as various biocompatiblity tests on the same set of 44 alloys.

1. Introduction

Gold-based alloys are used in dentistry, among other reasons, because of their good resistance to corrosion in the buccal environment. The early compositions based on gold and copper have later been modified to meet new criteria, such as increased melting range for the porcelain–metal technique, or replacement of gold in various extents by other noble elements to produce less-expensive alloys. These changes have resulted in several investigations to evaluate their influence on corrosion resistance. Many papers have been published and later reviewed in new papers. The latest were published in 1992 by Canay and Öktemer [1], and in 1994 by Geis-Gerstorfer [2] and by Nakagawa *et al.* [3].

The purpose of the present study is to evaluate the corrosion resistance of a set of 44 dental alloys and pure metals, and is part of a broader investigation aimed at relating the corrosion resistance with the biocompatibility of the same set of metals and alloys. Some results on biocompatibility testing have already been published by Craig and Hanks [4, 5], using cell culture tests. These investigations are part of the efforts of the manufacturer (Métaux Précieux SA/Métalor, Neuchâtel, Switzerland) of these dental alloys to better understand the behaviour of the products in conditions approaching those encountered in the mouth.

2. Materials and methods

2.1. Materials

Alloys and pure metals were selected to cover the widest range of alloys used in dentistry and of reference materials. The 44 alloys and pure metals tested in this study belong to the main classes described in Table I, including conventional casting alloys (high- and low-gold content, silver-based, and solders), alloys for the porcelain technique (high- and low- gold content, and palladium-based), and non-noble alloys and pure metals, used as references. The main components of each alloy are given in Table I in atomic per cent.

All the casting alloys were cast according to the manufacturer's instructions specific for each alloy, and, when applicable, submitted to a simulation of the thermal treatments needed in practice (porcelain bakes). Alloys such as Ti 6Al 4V and stainless steel, and all pure metals were machined to the shape used in this study, which consists of a short cylinder (6 mm in diameter and 10 mm in height), to which an electrical lead has been soldered. The sample and the lower part of the lead were first covered with an epoxy glue, and then embedded in a mounting resin; the section of the cylinder was metallographically polished.

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TABLE I Classes and composition of tested alloys and references, in atomic per cent

Classes	Groups	Code	Au	Ag	Pt	Pd	Cu	Zn	In	Sn	Ga	Others	Noble
C & B High-gold	1a	1	49.8	11.5	1.4	2.6	31.5	3.2				0.0	53.8
		7	42.8	24.8	0.3	3.8	25.2	3.1				0.0	46.9
		8	50.2	26.6		14.2		2.3	5.3			1.4	64.4
		14	55.0	16.7			28.3					0.0	55.0
		23	49.0	14.4	2.5	5.0	25.9	3.2				0.0	56.5
		24	52.4	18.4	3.3	2.6	20.0	2.7				0.6	58.3
		27	56.6	13.6		3.3	24.2	2.2				0.1	59.9
		28	43.0	31.0		4.5	20.0	1.5				0.0	47.5
C & B Low-gold	1b	2	31.9	30.9		8.1	27.2	1.9				0.0	40.0
		9	36.6	29.8	0.3	6.0	23.9	3.3				0.1	42.9
C & B Ag-based	1c	3	11.8	41.7		23.0		6.8	16.7			0.0	34.8
		4	1.6	62.7		22.5	10.6	0.8	1.8			0.0	24.1
		10		55.1		22.9	18.1	3.0				0.9	22.9
		11	2.2	50.7	6.2	30.3	7.5	2.5				0.6	38.7
		29	6.5	51.1		18.3	23.0	1.0				0.1	24.8
C & B Brazing alloys	1d	5	46.6	15.2			25.8	3.4				9.0	46.6
		6	51.5			3.8			3.9		12.6	28.2	55.3
		12	48.3	10.1		1.9	22.0	12.9	4.7			0.1	50.2
PFM High-gold	2a	A	68.8	1.5	9.3	15.6	0.6		2.1	0.6		1.5	93.7
		F	81.5		6.1	8.3			0.6	0.6		2.9	95.9
		I	83.8		9.7	2.3			0.6	0.6		3.0	95.8
		K	80.5	2.6	6.8	5.7			1.1	0.6		2.7	93.0
		L	61.6	1.5		28.9	1.1	1.2	2.8	2.7		0.2	90.5
		O	77.2	4.7	11.6	3.5			2.9			0.1	92.3
		P	79.3	2.5	6.1	8.2			0.6	0.6		2.7	93.6
		S	72.2		9.1	13.1			5.5			0.1	94.4
PFM Low-gold	2b	B	36.4			50.2			10.3		3.0	0.1	86.6
		C	36.8			43.3	8.2		8.9	0.7	2.0	0.1	80.1
		T	38.5	12.9		37.7	2.2		2.4	2.3		4.0	76.2
PFM Pd-based ...no Ag	2c	D	1.0			73.9	10.9		3.9	1.7	7.9	0.7	74.9
		M				81.0				12.7	5.8	0.5	81.0
		N	1.0			71.1	15.1	0.2			12.3	0.3	72.1
...with Ag	2c/Ag	E		31.5		57.3		1.6	5.5	1.8	2.3	0.0	57.3
		H		24.9		63.4		1.6		9.9		0.2	63.4
		R		1.7		73.5	10.8	0.2	3.9	1.7	7.8	0.4	73.5
		Code	Ti	Al	V	Fe	Ni	Cr	Mo	Be	Si	Others	
Non- precious	3	19	82.6	10.2	3.6							3.6	
		20				69.1	10.9	18.5	1.3			0.2	
		22	2.3				70.3	13.6	1.7	9.0	1.9	1.2	

Noble = sum of Au + Pt + Pd (at.%)

2.2. Testing medium

An artificial saliva derived from the one described previously by Fusayama, Katayori and Nomoto [6], was used. It is made of a solution containing NaCl 0.4 g/l, KCl 0.4 g/l, $NaH_2PO_4 \cdot H_2O$ 0.69 g/l, $CaCl_2 \cdot H_2O$ 0.79 g/l, $Na_2S \cdot 9\ H_2O$ 0.005 g/l, and urea 1 g/l. The pH of such a solution varies between 5.0 and 5.5. This electrolyte provides an electrochemical behaviour which is close to that experienced *in vitro* with a natural saliva [7–9].

2.3. Electrochemical measurements

These measurements were carried out with two different types of instrumentation: a group of single-purpose instruments (potentiostat, function generator, etc.) linked together (Potentiostat Model 550, AMEL, Milano, Italy), and an integrated electrochemical system (Model 273, PAR, Princeton Applied Research, Princeton, USA). The electrochemical cells were also different: in one case, the standard cell defined in ASTM Specification G 5-87 was used, and in the other case, a custom-made cell with functions similar to the ASTM standard, but with a slightly different geometry, was used. All tests have been performed at a standard temperature of 37 °C. A saturated calomel electrode (SCE), a platinum counter-electrode, and the working electrode made of the specimen to be tested, formed the measurement system.

The following cycle of measurements was used throughout the whole study:

(a) Measurement of the open-circuit potential over a 24 h period, followed by 2 h of de-aeration by nitrogen bubbling. The value obtained after 24 h in the aerated solution is referred to as "rest potential".

(b) Tracing of a linear polarization curve between − 20 mV below the "rest potential" and + 20 mV above the "corrosion potential" (the potential obtained for $i = 0$ during the scanning of the potential range, in the deaerated solution, is called the "corrosion potential" E); determination of the polarization resistance R_p according to the method described by Mansfeld [10, 11].

(c) Tracing of the anodic potentiodynamic polarization curve at a slow rate of 1 mV/s and 0.03 mV/s, respectively, from -500 mV (SCE) up to $+1500$ mV (SCE), in order to possibly detect and measure the breakdown potential. This potential has been evaluated from these anodic polarization curves by fitting a tangent to the sharpest increase in current readings and measuring its intercept with the potential axis.

Some of the corroded surfaces were observed by scanning electron microscopy (CAMSCAN-24, Cambridge Instruments, Cambridge, England), and by optical microscopy.

3. Results

3.1. Rest potentials, breakdown potentials and corrosion potentials

Numerical data obtained during the electrochemical tests are reported in Table II. These values were all obtained with the PAR instrument, except for the breakdown potentials. For the same alloy, small differences are observed between the "rest potential"

TABLE II Electrochemical data

Classes	Groups	Code	Rest potential (mV SCE)	Breakdown potential (mV SCE)	Corrosion potential $E(i = 0)$ (mV SCE)	Polarization resistance R_p (kohm)
C & B High-gold	1a	1	102.0	905.0	67.0	1779.5
		7	97.0	920.0	93.8	816.8
		8	95.0	1325.0	77.0	712.6
		14	46.0	925.0	34.0	224.0
		23	94.0	980.0	107.8	1662.5
		24	108.0	940.0	113.3	2493.8
		27	136.0	1015.0	156.0	950.3
		28	104.0	880.0	108.5	1558.4
C & B Low-gold	1b	2	91.0	905.0	82.5	1756.1
		9	62.0	880.0	42.0	658.6
C & B Ag-based	1c	3	83.0	480.0	72.3	933.1
		4	83.0	400.0	66.2	852.9
		10	73.0	350.0	70.8	1257.3
		11	147.0	1160.0	117.0	1133.8
		29	122.0	415.0	118.0	553.3
C & B Brazing alloys	1d	5	80.0	855.0	75.5	1262.8
		6	77.0	430.0	38.5	483.0
		12	147.0	905.0	149.0	1565.7
PFM High-gold	2a	A	283.0	1515.0	260.6	1896.3
		F	114.0	1480.0	92.5	3543.2
		I	155.0	1480.0	155.8	1945.2
		K	225.0	1290.0	204.3	1780.3
		L	255.0	1490.0	261.5	1368.7
		O	180.0	1540.0	161.5	1065.1
		P	200.0	1440.0	211.5	2295.8
		S	97.0	1500.0	106.8	1900.0
PFM Low-gold	2b	B	223.0	1490.0	211.5	3073.6
		C	221.0	1460.0	167.0	3599.8
		T	149.0	1370.0	146.8	3444.0
PFM Pd-based ...no Ag	2c	D	253.0	1290.0	227.1	2777.0
		M	275.0	1350.0	217.3	2853.4
		N	215.0	1370.0	185.5	2215.0
...with Ag	2c/Ag	E	87.0	1425.0	70.1	1916.6
		H	234.0	1380.0	205.8	2468.0
		R	82.0	1285.0	55.3	1511.0
Non-precious	3	19	76.0	> 2000.0	102.8	1401.6
		20	− 32.0	275.0	− 20.0	476.0
		22	− 210.0	210.0	− 219.0	18.5
Pure metals	4	Au	213.0	1535.0	208.3	2506.6
		Ag	77.0	110.0	85.8	320.4
		Pd	30.0	1400.0	15.7	1518.7
		Cu	− 96.0	− 25.0	− 95.0	2.7
		Ti	32.0	> 2000.0	40.8	1755.3
		Ni	− 85.0	420.0	− 84.0	92.6

(measured with an open circuit) and the "corrosion potential" (measured during the short scanning in potential used to determine the polarization resistance). There is no systematic variation, either increase or decrease of potential from one condition to the other. When computing the average of these three parameters for each group of alloys, the following ranking is obtained:

Average rest potentials, ranked from lowest to highest:

3(− 55.3 mV) < 1b(76.5) < 1a(94.8) < 1d(101.3)

= 1c(101.6) < 2c/Ag(134.3) < 2a(188.6) < 2b(197.7) < 2c(247.7)

Average breakdown potentials, ranked from lowest to highest:

1c(561 mV) < 1d(730) < 1b(892) < 1a(986) < 2c(1337)

< 2c/Ag(1363) < 2b(1440) < 2a(1467)

Average corrosion potentials, ranked from lowest to highest:

3(− 45.4 mV) < 1b(62.3) < 1d(87.7) = 1c(88.8) < 1a

(94.6) < 2c/Ag(110.4) < 2b(175.1) < 2a(181.8)

< 2c(209.9)

From these rankings, it appears clearly that:

(a) the non-noble alloys have the lowest rest and corrosion potentials;

(b) the crown and bridge alloys have rest and corrosion potentials in a mid-range from about 62 up to 101 mV; and

(c) the alloys for the porcelain-fused-to-metal technique (PFM) gave the highest values, from about 110 mV up to 248 mV versus SCE for the rest and corrosion potentials, and from a range of 561–986 mV up to a range of 1337–1467 mV for the breakdown potentials.

3.2. Polarization curves

The polarization curves of pure metals and of representative examples of the 44 tested alloys are reproduced in the Figs 1 and 2.

In Fig. 1, the noble metals Au and Pd show an extremely limited current below 1 V (SCE), whereas the base metals Cu and Ni start to corrode just below 0 V and at about 0.4 V respectively. Silver starts corroding at 0.1 V. Titanium does not show any sign of corrosion up to 1.8 V.

In Fig. 2, three broad categories of dental casting alloys are represented: the conventional precious alloys (classes 1a, 1b, and 1c), the precious alloys for the porcelain-fused-to-metal (PFM) technique (classes 2a, 2b, and 2c), and the non-precious alloys (class 3).

Alloys of classes 1a and 1b are characterized by a first peak of anodic oxidation at around 1.2 V, followed by the regular increase in oxidation above the breakdown potential at around 1.3 V. For the alloys of class 1c (silver-based alloys), a marked increase in the anodic oxidation is observed above 0.5 V.

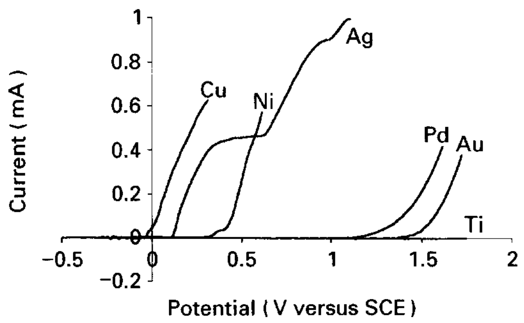

Figure 1 Polarization curves: pure metals.

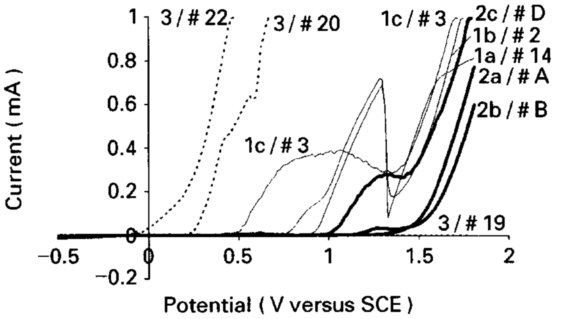

Figure 2 Polarization curves: one example for each class of alloys.

This can easily be correlated with the polarization curve of pure silver (Fig. 1).

The two classes of gold-based alloys for the PFM technique (classes 2a and 2b) have very similar polarization curves, characterized by an absence of oxidation up to about 1.2 V. For the class 2c (palladium-based alloys), an increase in oxidation may be observed above 1 V.

The three examples of non-precious alloys represented here exhibit fairly different electrochemical behaviour. The Ni-based alloy (class 3/#22) starts to corrode at − 0.1 V, whereas the stainless steel (class 3/#20) resists up to 0.2 V. In contrast, the Ti-based alloy (class 3/#19) behaves similarly to pure titanium, as shown in Fig. 1.

3.3. Polarization resistance

The average and extreme values are ranked by classes of alloys in Fig. 3. When averaging the values of the polarization resistance measured for the same alloys, and with the same equipment, the following ranking of these average polarization resistance values is obtained:

3(632.0 kOhm) < 1c(946.1) < 1d(1103.8) < 1b

(1207.3) < 1a(1410.2) < 2c/Ag(1965.2) < 2a(1974.3)

< 2c(2615.1) < 2b(3372.4)

As shown above with the potentials, a similar classification can be observed: the non-noble alloys exhibit the lowest polarization resistance (632 kOhms), the C & B alloys have intermediate values between 946 and 1410 kOhms, and the PFM alloys rank first with resistance between 1965 and 3372 kOhms.

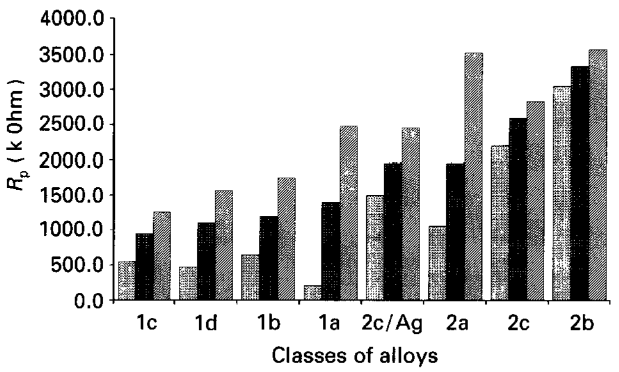

Figure 3 Polarization resistance: average and extreme values ranked by classes of alloys. (▤ low; ■ mean; ▩ high).

4. Discussion

4.1. Potentials

Three types of potentials are reported in Table II: rest potential, breakdown potential, and corrosion potential.

The *rest potential* gives some information about the electrochemical behaviour of the alloys. The more positive values indicate that these metals and alloys are more noble than those with a lower rest potential, and that, when placed in direct contact, a galvanic current starts to flow; its intensity increases as the difference between the rest potentials of the two alloys in contact increases. A high value is therefore beneficial for an individual alloy, since it means better protection against degradation when this alloy comes in contact with others. Since it has been measured in this study after a period of 24 h of immersion in an artificial saliva, it reflects a rather stable level.

The *breakdown potential* is defined by the intersection of a tangent to the polarization curve with the potential axis at current = 0, where the polarization curve starts to increase steeply in positive value. It is directly related to the composition of the alloys and is influenced by the component with the highest concentration. It marks the point beyond which degradation becomes massive; it should therefore never be exceeded, to avoid substantial damage to the alloy.

The *corrosion potential* was measured during linear polarization, which was performed in an interval starting at 20 mV below the rest potential, and increasing up to 20 mV above the potential at which the corrosion current is equal to zero (the corrosion potential). The rest potential and the corrosion potential are therefore directly linked. It is clearly evident that the crown and bridge alloys (classes 1a to 1c) and the brazing alloys (class 1d) have lower potentials than the ceramic alloys (classes 2a to 2c). A similar observation can be made with the breakdown potentials, which are more positive (and therefore more favourable) for the ceramic alloys than for the crown and bridge alloys (see Table II).

In the broad group of ceramic alloys, and for the rest and corrosion potentials, the two extremes are the palladium-based alloys without silver (class 2c), which have the highest potentials, and alloys of the same family, but containing some silver (class 2c/Ag), which show the lowest potentials among the ceramic alloys. The presence of silver in palladium-based alloys significantly affects the electrochemical behaviour of these alloys. On the other hand, the palladium-based alloys without silver (class 2c) definitely have a lower content in noble elements than PFM alloys with a high gold content (class 2a) (see their "compositional nobility" expressed as content in Au + Pt + Pd in Table I), but their potentials are markedly higher. A high palladium content (71 to 81 at% for class 2c, as compared to a Pd content ranging from 2 to 29 at% for the alloys of class 2a) seems therefore to contribute to improve markedly the "electrochemical nobility" of these alloys, that is their ability to resist oxidation.

4.2. Polarization resistance

A similar pattern was found with the measurement of the polarization resistance. This value, obtained from the linear polarization test, is another way of describing the ability of an alloy to resist corrosive degradation. It is directly related to the corrosion current, which measures the intensity of the anodic current at the corrosion potential. The resulting current is equal to zero at this particular potential, but it is in fact the algebraic sum of the cathodic current (negative) and the anodic current (positive): when both currents have an equal intensity, the resulting current is null, and the corresponding potential is referred to as the corrosion potential. In fact, the anodic current still exists, and results from anodic oxidation (or corrosive degradation) of the tested alloy. With a high value for the polarization resistance Rp, the corresponding corrosion current will be low, and the alloy would endure very limited degradation. The ranking of the different classes of alloys, based on the average value of the polarization resistance, is presented in Fig. 3. The same discussion as above for the rest and corrosion potentials applies to this parameter.

4.3. Polarization curves

The anodic polarization curves are a descriptive means to compare the electrochemical behaviour of the tested alloys and metals. They do not produce quantitative data, but they are important in understanding the possible degradation, which may occur when the potential is raised. An alloy with no peak of anodic activity before the breakdown potential will be more suited for use in a degradative environment, since only severe polarization is able to initiate significant anodic oxidation. When this alloy is immersed in this particular environment, it will only suffer the limited anodic oxidation described by the corrosion current. The combination of a high breakdown potential, the absence of activity between the corrosion potential and the breakdown potential, and a high value for the polarization resistance, defines the optimal electrochemical behaviour of an alloy. From the numerical data presented in Table II, and from the polarization curves shown in Fig 2, it clearly appears that the PFM alloys, as a broad group, possess the best combination of electrochemical properties. Further nuances may be found, and finer rankings may be produced, but the overall image is maintained.

4.4. Relationship between "compositional nobility" and electrochemical parameters

The above discussion shows that the content of noble metals in an alloy markedly influences its electrochemical behaviour. Such observations have already been made by other researchers, [13–15], who have shown that the content in noble metals, eventually defining a "nobility index", can be used to explain the shape of the polarization curves or the amount of current flowing between two practical limits of the scanning potential (between − 300 and + 300 mV SCE) for dental gold-based alloys.

In this study, it has been attempted to correlate the various electrochemical parameters with the "compositional nobility". Several correlations were calculated, including rest or corrosion potentials versus noble metal content, and polarization resistance versus noble metal content. However, the coefficient of determination $r2$ was never greater than 0.40, which is rather low to describe a strong and statistically significant correlation between the two tested parameters.

4.5. Microscopical observations

Some samples were observed with a scanning electron microscope prior to and after the corrosion test. The main components of the various phases were identified by energy-dispersive spectrometry: for example, two alloys are presented in Figs 4 and 5, at a similar enlargement. In Fig. 4, alloy 2, a silver-based alloy for crowns and bridges (Group 1c), exhibits two different phases, 1 and 2, which are not clearly differentiated. The darker one is richer in noble metals (Au and Pd), while the lighter one, with a lower mean atomic weight, is richer in silver. After the corrosion test, corrosion products containing a large proportion of silver are located on top of the silver-rich phase, thus demonstrating the strong relation between silver-rich alloys or phases, and enhanced corrosion.

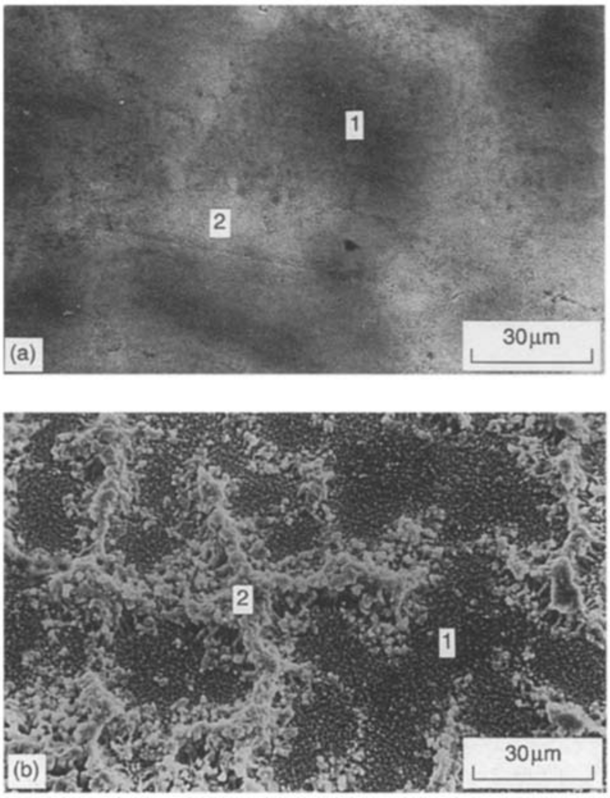

Figure 4 Alloy 2 (Group 1c: C and B silver-based) prior to (a) and after (b) corrosion tests

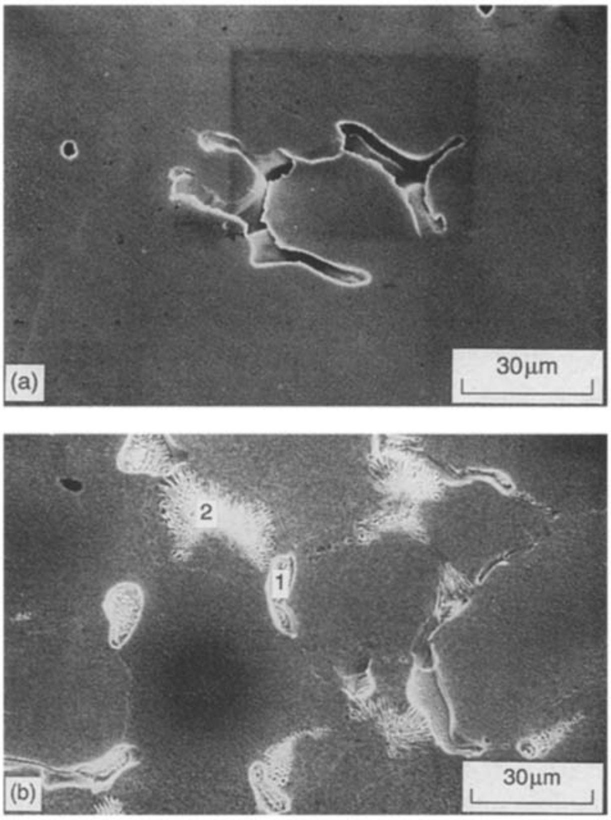

Figure 5 Alloy B (Group 2b: PFM low-gold) prior to (a) and after (b) corrosion tests.

For the second example, a ceramic alloy with a lower gold content (alloy B, group 2b) was selected. It appears immediately that degradation is much less extensive than with the previous alloy. After the corrosion test, no corrosion products are visible, and a slight attack can be observed at a localization corresponding to grain boundaries. The area marked "2" is richer in palladium than the core of the grain, which may indicate that some of the other elements present at the grain boundaries have been dissolved, leaving behind an area proportionally richer in Pd. However, the analytical instrument attached to the scanning electron microscope was not sensitive enough to clearly measure the slight differences in composition between the various zones detected after the corrosion test.

5. Conclusion

A large selection of dental casting alloys was evaluated for *in vitro* corrosion resistance, in an artificial saliva. Using various techniques, it was constantly shown that the alloys for preparing full-metal crowns and bridges (C and B alloys) are somewhat less corrosion-resistant than the newer alloys for the metal–ceramic technique (PFM alloys). This difference is easily explained by the higher content of noble metals in the

PFM alloys, or by the relatively high content of non-noble metals (silver, copper, zinc) in the C & B alloys. Within these two families, groups have been established, based on the gold-content, or on the main component (silver in some C & B alloys, and Pd in some PFM alloys). When decreasing the content of gold, the corrosion resistance decreases also, but not as much as when a non-noble metal such as silver becomes the main component.

Addition of secondary components such as indium or gallium in PFM alloys, or cadmium or nickel in some brazing alloys, has distinct effects: the presence of cadmium or nickel has a distinctive (and highly degradative) effect on the corrosion behaviour, whereas base-metals such as In and Ga do not affect significantly the resistance to corrosion. A relationship between nobility (expressed as the total amount of gold, platinum and palladium) and some corrosion indicators such as the rest potential or the corrosion resistance, has been observed, but the strength of the correlation between these two parameters is strongly affected by the influence of other components of the alloys, and probably by the metallographic structure of the alloys.

As part of a larger study on the same set of 44 casting alloys, including other electrochemical investigations as well as extended biocompatibility testing, this paper should provide basic knowledge on the corrosion behaviour of the tested products.

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