

Corrosion Behavior of As-Received and Previously Cast Type III Gold Alloy

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Keywords

Cast; corrosion; as-received; recast; type III gold alloy.

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Accepted: April 15, 2008

doi: 10.1111/j.1532-849X.2009.00565.x

Abstract

Purpose: The rationale for using gold alloys is based largely upon their alleged ability to resist corrosion, but little information is available to determine the corrosion behavior of recast alloys. This study characterized the elemental composition of as-received and recast type III gold alloy and examined the in vitro corrosion behavior in two media using a potentiodynamic polarization technique.

Materials and Methods: Seventy-eight disk-shaped specimens were prepared from a type III gold alloy under three casting protocols according to the proportion of as-received and recast gold alloy (n = 26). (1) Group as received (100% as-received metal), (2) group 50% to 50% (50% wt. new metal, 50% wt. once recast metal), and (3) group recast (100% once recast metal). The surface structures of 20 specimens from each group were examined under scanning electron microscopy, and their elemental compositions were determined using X-ray energy-dispersive spectroscopy. Further, the potentiodynamic cyclic polarization between -1000 and +1000 mV (SCE) were performed for six specimens from each casting protocol in 0.09% NaCl solution (n = 3) and Fusayama artificial saliva (n = 3) at 37°C. Zero-current potential and corrosion current density were determined. The data were analyzed with 1-way ANOVA and the Ryan–Einot–Gabriel–Welsch multiple-range test t (α = 0.05).

Results: Elemental composition was significantly different among the casting groups (p < 0.001). The mean weight percentage values were 72.4 to 75.7% Au, 4.5 to 7.0% Pd, 10.7 to 11.1% Ag, 7.8 to 8.4% Cu, and 1.0 to 1.4% Zn. The mean values for Zero-current potential and corrosion current density for all casting protocols were not significant (p > 0.05); however, the difference between the electrolytes was significant (p < 0.001). Fusayama artificial saliva seemed to offer the most corrosive environment. **Conclusions:** Type III gold alloy in any casting protocol retained passivity under electrochemical conditions similar to the oral environment. Moreover, high-gold type III alloys from reputable manufacturers and recasting protocol tested should produce acceptable corrosion-resistant castings.

The rationale for using high-gold casting alloys in dental practice is based upon mechanical and manipulative features, biocompatibility, and tarnish and corrosion resistance.¹ There are reports that at least four generations of the alloys can be used, based on evaluation of various physical properties.²⁻⁶

As an economic measure, dental laboratories combine previously cast metal with new alloy received from the manufacturer. Textbook guidelines for recasting gold alloy vary from adding no new metal, to some new metal, to 50% new metal with previously melted buttons or sprues removed from castings.⁷⁻⁹ Nevertheless, manufacturers' product information on gold casting alloys also typically states that such scrap metals can be re-melted to fabricate clinically acceptable castings, provided that at least 50% new metal is used. The basis for this empirical guideline is that certain important secondary elements, present in small percentages in the original alloy compositions, may be lost during melting through volatilization or oxidation. One example is zinc, which acts as an oxygen scavenger during melting to minimize the oxidation of other elements in the alloy.^{10,11}

Ayad¹² compared the effects of three casting protocols on the composition stability and marginal accuracy of a high-gold alloy for all-metal restorations. He concluded that the composition of noble metals (Au, Pt, and Pd) and other major (Ag and

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Cu) and minor (Zn) elements changed during melting. Moreover, recasting conditions may adversely affect the marginal accuracy of complete cast crowns.¹² While the loss of major component elements in type III gold casting alloys is probably not a significant concern, minor constituent loss may affect other factors such as corrosion resistance and potentially the overall quality and longevity of the finished casting.

Corrosion resistance of dental prostheses is an important property for dental alloys, in addition to other properties, such as strength, ductility, and casting accuracy. Corrosion of dental alloys in the oral environment not only results in the deterioration of the restoration, but also involves a release of ions directly related to their biocompatibility.^{13,14} Corrosion behavior of alloys depends upon their compositions, their electrode potentials, the stress over the metals, and surface roughness, in addition to the specific characteristics of different oral environments.¹⁵⁻²²

The potentiostatic/potentiodynamic polarization test²³⁻²⁵ is the method commonly used to study the in vitro corrosion of dental alloys; however, this method is not generally accepted as being applicable and clinically relevant for every dental alloy.²⁶ Although several electrolytes have been used in the potentiostatic/potentiodynamic polarization test, Fusayama solution²⁷ has been considered the medium in which the electrochemical behavior of dental alloys most closely resembles that in natural saliva.^{28,29}

Several studies^{12,30} have characterized the material science aspects for laboratory specimens of gold alloy, but no mention is made as to what composition modifications might degrade the corrosion performance of cast dental gold alloys. Furthermore, little experimental data is available on the effect of recasting on corrosion resistance.³¹ The purpose of the present study was to characterize the elemental composition of as-received and recast type III gold alloy and to examine the in vitro corrosion behavior in two media using a potentiodynamic polarization technique. The null hypothesis was that combining as-received and recast type have no effect on corrosion resistance of type III gold alloy.

Materials and methods

Seventy-eight disk-shaped wax specimens (Gator wax, Whip Mix Corp., Louisville, KY), 6 mm diameter × 3 mm thick, were made with a brass split mold. The wax patterns were sprued, invested with carbon-free, phosphate-bonded investment (Cera-fina, Whip Mix Corp.), and cast with an ADA type III gold casting alloy (Ney-Oro-B2, Ney International, Bloomfield, CT) with a nominal composition listed by the manufacturer as 74% gold (Au), 4% palladium (Pd), 11% silver (Ag), and 9.5% copper (Cu).

Three casting protocols (n = 26) were compared according to the proportion of as-received and recast gold alloy. (1) Group as-received, all metal was used for the first time (100% as-received metal). (2) Group 50% to 50%, the sprue assembly was carefully removed from the cast crown, cleaned, weighed, and combined with an appropriate amount of new alloy to maintain an equal balance of new and recast material (50% wt to 50% wt ratio). (3) Group recast, the once-cast alloy was subsequently used to produce the second casting (100% recast metal). A peak burnout temperature of 650°C was chosen on recommendation from the alloy manufacturer. Each casting protocol was melted in an individual ceramic crucible with a gas-air torch (National Handle No. 2, Tip N-2, National Welding Equip. Co., San Francisco, CA) and a broken arm centrifugal casting machine (Kerr Mfg. Romulus, MI), following standard dental laboratory procedures.

The investment and casting protocol was established by pilot testing. Castings were recovered from the investment, bench-cooled to room temperature, cleaned in pickling solution (Jel-Pac, J.F Jelenko Co., Armonk, NY), and airborne-particle abraded with 50- μ m aluminum oxide for 10 seconds with a contra-angle micro-etcher (Model erc-er, Danville Engineering, Danville, CA) at 60 psi. To minimize the effect of variations in the casting procedure, the same clinician completed all castings.

For compositional measurements, 20 disc specimens of each casting protocol were used. Each disk was embedded in acrylic resin (LR white, London resin, Hampshire, UK) with a vacuum impregnation technique, and the resin was thermally polymerized to 50°C. Each embedded specimen was sectioned into halves with a low-speed, water-cooled saw (Vari/Cut VC-50, Leco, St. Joseph, MI), with a diamond-coated blade, and subsequently ultrasonically cleaned in ethanol for 2 minutes. The specimens were then wet-ground with 1000-grit (18 μ m) and 4000-grit (5 μ m) silicon carbide abrasive papers (Struers, Westlake, OH). Final polishing was performed with a series of metallographic abrasives through $0.05-\mu m$ Al₂O₃ suspensions (Liquimant, Vos and Van Eyck Metallurgie, Vianen, The Netherlands) in DP-Lubricant blue cooling solution (Struers). Between each polishing step, the specimens were rinsed copiously with water and ethanol (70%). After polishing, each specimen was again ultrasonically cleaned in both distilled water and ethanol for 5 minutes.

Elemental analysis was performed with X-ray energydispersive spectroscopy (EDS) attached to a scanning electron microscope (SEM) (model JSM-820, JEOL Ltd, Tokyo, Japan). A Link eXL Microanalysis System with a silicon PentaFet detector and 7.6-µm beryllium window (Oxford Analytical Instruments, High Wycombe, UK) was used. Prior to microanalysis, standard characteristic X-ray spectra were acquired for Au, Pd, Ag, Cu, and Zn. During each experiment, EDS analyses were obtained under standard conditions at three sites along the specimen, and the data were averaged. Raster scans were performed at 5000× magnification with an accelerating voltage of 9 kV, a beam current of 3.0 nA, a live time of 200 counts/second, and dead time of approximately 50%. These conditions yielded a total number of 100,000 minimum X-ray photon counts from each site examined, which was considered necessary for an adequate elemental analysis.^{32,33} Sufficient Xray analyses were performed to obtain quantitative information about the elemental composition.

To avoid the possibility of spot overlapping and double counting during the EDS analysis, the depths of X-ray penetration were calculated along a line representing seven spots for each of the three locations. Again, the measurement data were averaged. The critical distance between two spots to avoid overlapping interaction volumes was computed to be less than 0.5 μ m. Consequently, the points selected for the EDS raster scans were at least 1 μ m apart. A computer software package associated with the analyzer converted the raw data, and the ZAF correction was performed.²⁴ Means and standard deviations were calculated for each group, and results were analyzed with 1-way ANOVA and the Ryan–Einot–Gabriel–Welsch multiple-range test ($\alpha = 0.05$). REGWQ was used, because it appears to be the most powerful yet valid step-down multiple-stage test in the current literature.³⁴

For measuring corrosion, six disk specimens of each casting protocol were used. A potentiostat (Model 273A Potentiostate/Galvanostat, EG & G Princeton Applied Research, Princeton, NJ) controlled by a personal computer with dedicated software (Model 352/252 SoftCorrTM II Corrosion Measurement & Analysis Software, EG & G Princeton Applied Research) was employed. A platinum plate was used as the counter electrode, and a saturated calomel electrode (SCE) was used as the reference electrode. NaCl (0.09%) solution and Fusayama artificial saliva were used as the test media (n = 3 per electrolyte). The corrosion media were maintained at 37°C.

The cyclic polarization scan was performed from -1.000 to +1.000 mV and back to -1.000 mV at 2 mV/sec, with 100 μ A as the selected current range setting. ZCP in the forward scan was determined from the cyclic polarization diagram. Corrosion current density (I_{CORR}) was determined using the curve-fitting routine of the corrosion software. New corrosion medium was prepared for each specimen tested. The specimen surface was re-polished and cleaned. Each test was repeated three times, and the data were averaged. After the test, the specimens were used for SEM examination to study corroded surfaces. The differences between mean values of the electrochemical parameters measured were analyzed with 1-way ANOVA and Ryan–Einot–Gabriel–Welsch multiple-range test at a significance level of ($\alpha = 0.05$).

Results

Table 1 lists the wt% of elemental composition of each group of castings. Castings in the once recast group had the lowest mean Au wt%; those in as-received had the highest Au wt%, which was almost equal to that of group 50% new, 50% once recast. For Pd, the highest wt% was obtained with the oncerecast group and the lowest with the 50% new, 50% once recast group. The highest total wt% for noble metals (Au and Pd) in combination were obtained with the as-received castings group

Table 1 Bulk chemical composition (%) for the ingot and casting protocols as analyzed by EDS (wt%; means \pm standard deviations, n = 20)

Element	50% new, As-received 50% once recast Once re				
Au	75.7 ± 0.7*	75.6 ± 0.7*	72.4 ± 0.5		
Pd	4.7 ± 1.2*	$4.5 \pm 1.3^{*}$	7.0 ± 0.7		
Ag	11.1 ± 0.3	$10.8 \pm 0.3^{*}$	10.7 ± 0.3*		
Cu	8.4 ± 0.7	$8.0 \pm 0.9^{*}$	7.8 ± 0.7*		
Zn	1.4 ± 0.1	1.1 ± 0.2	1.0 ± 0.2		

Values with^{*} were not statistically different at p < 0.001.

Table 2 One-way ANOVA for each element among casting protocols

Source of variation	df	MS	F score	p value
Model (Au)	2	208.3892	525.45	< 0.001
Error	57	0.3966		
Model (Pd)	2	118.4226	63.08	< 0.001
Error	57	1.8772		
Model (Ag)	2	2.3906	34.61	< 0.001
Error	57	0.0691		
Model (Cu)	2	6.4579	6.95	< 0.001
Error	57	0.9287		
Model (Zn)	2	3.0349	94.26	< 0.001
Error	57	0.3966		

(80.4%) and the lowest with the once recast group. The wt% of noble metals in group 50% new, 50% once-recast was intermediate (80.1%). The ANOVA results in Table 2 demonstrate a statistically significant difference between casting protocols (p < 0.001). The Ryan–Einot–Gabriel–Welsch multiple-range test revealed no significant difference in the Au or Pd wt% of the as-received group and the 50% new metal-50% cast metal group; however, the AU wt% for the previously cast group was significantly lower (p < 0.001). A similar comparison for Ag and Cu elements showed that the highest wt% was recorded for castings in the as-received group (11.1% and 8.4%, respectively). These measurements were significantly different from the other casting protocols (p < 0.001). Comparison for the trace element Zn indicated significant differences among casting protocols, with the lowest weight percentage (1.0%)recorded for the previously cast group. The data recorded by EDS for the elemental analysis of casting protocols are shown in Figure 1.

Mean values and standard deviations for Zero-current potential (ZCP) and corrosion current density (I_{CORR}) in both the saline and Fusayama solutions are listed in Table 3. For



Figure 1 Sample intensities of characteristic X-ray for EDS analysis of as-received casting protocol of type III gold alloy.

Medium	ZCP (mV)	I _{CORR} (μA/cm ²)
0.09% NaCl	-134.5 ± 12.9	$19.9\pm4.1^{\rm a}$
Fusayama	-173.9 ± 5.7	20.3 ± 2.9^{a}
0.09% NaCl	-131.9 ± 17.8	$21.9\pm4.8^{\text{b}}$
Fusayama	-164.7 ± 8.2	23.1 ± 6.7^{b}
0.09% NaCl Fusayama	-137.8 ± 14.7 -168.4 ± 5.3	21.3 ± 5.5^{c} 24.8 ± 1.2^{c}
	Medium 0.09% NaCl Fusayama 0.09% NaCl Fusayama 0.09% NaCl Fusayama	Medium ZCP (mV) 0.09% NaCl -134.5 ± 12.9 Fusayama -173.9 ± 5.7 0.09% NaCl -131.9 ± 17.8 Fusayama -164.7 ± 8.2 0.09% NaCl -137.8 ± 14.7 Fusayama -168.4 ± 5.3

Table 3 Corrosion parameters among casting protocols for type III gold alloy (mean \pm standard deviation, n = 3)

Values with the same letters are not significantly different at p < 0.001.

saline solution, the ANOVA results demonstrate no significant differences between the mean values of ZCP (p = 0.896) and I_{CORR} (p = 0.874) for all casting protocols. Similarly, significant differences were not found between the mean values of ZCP (p = 0.297) and I_{CORR} (p = 0.477) for all casting protocols in the Fusayama solution.

All casting protocols showed significantly higher values of ZCP in the Fusayama solution than in the saline solution (p < 0.001). Similarly, the I_{CORR} values were not significantly different for all casting protocols (p = 0.893 for as-received, p = 0.813 for 50% new, 50% once cast, p = 0.348 once recast alloys). A representative cyclic polarization diagram for the as-cast alloy in the 0.09% NaCl and Fusayama solutions are shown (Fig 2). A similar cyclic polarization diagram was observed for the other casting protocols.

Discussion

This study supports the null hypothesis that combining asreceived and previously cast alloys has no effect on corrosion behavior of type III gold alloy. A 37°C standard temperature was chosen to represent the normal temperature of the oral cavity. Moreover, Fusayama solution has been considered the medium in which the electrochemical behavior of dental alloys most closely resembles that in natural saliva.^{19,28} A cyclic polarization test that provides general information on the state of the electrode at various potentials was used. The loss of major component elements in type III gold casting alloys is probably a significant concern. Many investigators^{4,5,12} have evaluated the effect of the repeated use of alloys on the characteristics of resultant castings. Tucillo et al⁵ discussed the possible ramifications of departing from the traditional high-gold alloy at a time when reduction in gold suggested that clinical failure would most likely be associated with tarnish and/or corrosion when the nobility was decreased or the silver–copper ratio was altered. Rosenstiel et al¹ recommended that at least 50% new metal be included in copings for metal–ceramic restorations. Although most manufacturers concur, there is little experimental justification for the 50% rule.

Tuccillo et al⁵ found that the elemental composition of highgold alloy remained stable during three melting procedures; however, the results of this study indicate that mixing new and previously cast metal can influence the compositional stability of a type III gold alloy, leading to changes in the amount of noble metals (Au and Pd) and the other major and minor elements (Ag, Cu, and Zn). In this study, changes in elemental concentrations were generally minimal, given that the practical accuracy of values determined by EDS is typically about 0.5 wt%.

The corrosion behavior of casting alloys has great importance, because of the oral galvanic actions that may cause a series of reactions including metallic taste, oral burning, oral pain, sensitizations, allergies, and other toxic reactions.¹⁶ Theoretically, the corrosion behavior of alloys can be predetermined by the standard electrode potentials of the alloy elements.¹⁸ If this standard electrode potential is positive, then the metal is regarded as noble and it hardly ionizes, but if it is negative, the metal is active and ionizes quickly. Therefore, the alloy composition is important. Further, for economic reasons, recycling of an alloy cast sprue is also a procedure in dental laboratories, and the composition change caused by multiple castings is a matter of interest.¹² In this study, the results of the bulk analysis (Table 1) show that the only compositional changes resulting from recasting were small decreases in the zinc and copper contents and an increase in minor elements, probably impurities. Zinc acts as an oxygen scavenger during melting to minimize



Figure 2 Sample cyclic polarization diagram for as-received gold casting alloy in the 0.09% NaCl solution.

The results for the alloy tested showed generally passive behavior, with no breakdown in the relevant range of potentials and low current density in the passive state. Mezger et al²¹ compared electrochemical properties of high palladium alloys with a gold-based alloy in the as-cast condition using an oxygenated 0.09% NaCl solution at 37°C. The corrosion current densities (I_{CORR}) in the high palladium alloys (0.017–0.065 μ A/cm²), determined using the Tafel extrapolation were similar to that of the gold-based alloy. Meyer and Reclaru²² reported OCP (82-275 mV vs. SCE), E_{CORR} (55-227 mV vs. SCE), and breakdown potentials (1285-1370 mV vs. SCE) of the high palladium allovs. In this research, the recast condition did not display any ionization (I_{CORR}) changes due to the media and recycling of the metals, and their corrosion tendencies remained comparable to as-received cast alloys. This result is in agreement with the electrode potentials of the elements in the alloy. The corrosion tendency of recast noble alloys was not reduced, although there was a diminishing percentage of the active metals in the alloy due to oxidation during the casting process. From the results, it is apparent that if the composition of the alloy is not changed, the corrosion will also be the same. Generally, in an alloy, if the noble metal percentage rises, the corrosion potential of the alloy also moves towards positive values, and the alloy corrodes less. Maintaining corrosion resistance for recast alloy may have been due to noble metal content remaining constant. Other possible causes include an absence of impurities and microshrinkage porosity. It has been reported that an increase in casting defects, principally porosity, decreases corrosion resistance for type III gold alloy.4

The results obtained from this research were only introductory and comparative. Further investigations are needed to determine how the loss of some elements from an alloy may cause alterations of its physical properties. Also, boundaries of acceptability for clinical use could not yet be formulated. Another limitation was the use of only one casting alloy system and having the corrosion test at lower temperature than the majority of corrosion studies reported.

Conclusions

Within the limitations of this study, the following conclusions can be drawn:

- 1. The elemental map showed decreased surface concentration of Au for the recast group. The bulk concentrations of Au, Ag, and Pd remained constant, but the copper and zinc contents slightly decreased.
- 2. Time and operating expenses required to prepare, weigh, and combine used metal with new metal for recasting were not cost effective considering the present cost of the alloy.
- 3. The results indicate that a dental alloy of this type can be safely recast.
- 4. Dental gold alloy in any casting protocol showed spontaneous passive behavior under electrochemical conditions similar to those of the oral environment.

5. An artificial saliva seemed to offer the most corrosive environment.

Acknowledgment

The authors are grateful to Ney Dental International for supplying the casting alloy.

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