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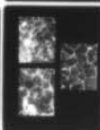
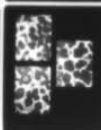
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OBSERVATIONS ON GOLD-PALLADIUM-SILVER AND GOLD-PALLADIUM ALLOYS--ETC(U)  
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Observations on gold-palladium-silver and gold-palladium alloys

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Commercial materials and equipment are identified in this report to adequately specify the experimental procedures. Such identification does not imply recommendation or endorsement or that the materials are necessarily the best available for the purpose.

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The increased popularity of the porcelain-metal restoration has been accompanied by the introduction of a seemingly endless number of palladium-rich white casting alloys. Accordingly, the use of these materials in lieu of "ceramic-type" alloys of higher gold content has grown markedly in recent years.

Unfortunately, the paucity of data on many of the white precious alloys makes the selection of specific products for clinical use arbitrary rather than rational. This alone, obviates the need for increased effort in the characterization of the palladium-rich proprietary alloys.

The present report is based upon data and observations on three high-palladium-content alloys (Neydium,<sup>\*</sup> JPW<sup>+</sup> and Olympia<sup>#</sup>) marketed for use in the porcelain fused-to-metal technique.

#### MATERIALS AND METHODS

Fabrication and conditioning of specimens. Castings for determination of tensile properties, hardness and microstructure were made by conventional dental laboratory procedures. Wax patterns for all test pieces were invested in a phosphate bonded refractory material.<sup>§</sup>

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\* The J. M. Ney Company, Hartford, CT.

+ Jensen Industries Inc., North Haven, CT.

# J. F. Jelenko and Company, Pennwalt Corp., New Rochelle, NY.

§ Ceramigold Investment, Whip-Mix Corp., Louisville, KY.

The resultant molds were placed in a cold gas oven, heated slowly to 1,500° F. and held at temperature for 45 minutes to insure heat saturation and total wax elimination. The alloys were melted and cast with the use of an automatic induction casting machine.<sup>¶</sup> Casting temperatures for Neydium, JPW and Olympia were 2,375° F., 2,390° F. and 2,550° F., respectively. All cast molds were bench cooled to room temperature before divestment of the test pieces.

Dimensions of the specimens for measurement of tensile properties were within the tolerances prescribed by American Dental Association Specification No. 14 for dental chromium-cobalt casting alloy.<sup>1</sup> Six specimens of each alloy were subjected to heat treatment by a simulated porcelain firing cycle<sup>Ω</sup> prior to testing. A like number of specimens

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¶ Electromatic Casting Machine, Howmet Corp., Chicago, IL.

Ω (1) Degassing: Specimens were heated from 1,200° to 1,950° F., held at 1,950° F. for 5 minutes, removed from the furnace, and cooled in open air. (2) Simulated application of opaque porcelain: Specimens were heated from 1,200° to 1,825° F., removed from the furnace immediately on reaching 1,825° F., and cooled to room temperature in open air. (3) Simulated application of body porcelain: Specimens were subjected to two successive firings from 1,200° to 1,775° F. Specimens were cooled to room temperature in open air after each firing. (4) Simulated application of glaze: Specimens were heated from 1,200° to 1,800° F., removed from the furnace immediately on reaching 1,800° F., and cooled to room temperature in open air.

of each material was reserved for measurement of as-cast tensile properties.

Hardness and metallographic specimens were 13 mm X 3 mm discs. The cast pieces were mounted in plastic and polished sequentially with 240 to 600 grit abrasive papers, diamond paste ( $0.25\text{ }\mu\text{m}$ ) and alumina ( $0.05\text{ }\mu\text{m}$ ). Metallographic specimens were etched by immersion in a solution of 92 percent HCl, five percent  $\text{H}_2\text{SO}_4$  and three percent  $\text{HNO}_3$ , by volume.

Upon completion of the examination of as-cast microstructure and the measurement of as-cast hardness, the discs were broken from their mounts and subjected to time and temperature controlled heat treatments. All heat treatment of metallographic specimens was accomplished by the simulated porcelain firing cycle. Some hardness specimens were exposed to the thermal cycle employed in the treatment of tensile bars and metallographic specimens. Others were subjected to serial heat treatments for determination of softening and hardening temperature ranges of the test alloys.

Softening temperatures were delineated by monitoring changes in the hardness of discs exposed to eight successive five-minute heat treatments at  $200^\circ$  intervals from  $400^\circ$  to  $1,800^\circ$  F. These treatments were terminated by water quenching. Hardness was measured after treatment of the castings at each temperature. Hardening temperature ranges were defined by serial reheat treatment of previously annealed specimens over the  $400^\circ$  to  $1,800^\circ$  F. temperature range. Again, hardness measurements were made on castings quenched from each of the eight treatment temperatures.

Measurement of mechanical properties. Tensile properties of as-cast and heat-treated bars were determined on a constant strain-rate testing machine<sup>\*\*</sup> at crosshead speed of 0.02 inch per minute. Elongation was measured over a one-inch gauge length with a breakaway extensometer.<sup>++</sup> All hardness (DPN) values were obtained through the use of a testing machine<sup>##</sup> and a 136-degree square-base diamond pyramid indenter.

Elevation of microstructure and chemical analysis. The acid-etched surfaces of metallographic specimens were observed with an optical microscope<sup>§§</sup> at a magnification of 450X. Constituents of the "as-received" materials were determined semiquantitatively by atomic absorption spectrophotometry.<sup>¶¶</sup> Definitive elemental analysis was accomplished by wet gravimetric procedures.

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<sup>\*\*</sup> Instron Universal Tensile Testing Machine, Instron Corp., Canton, MA.

<sup>++</sup> Strain Gauge Extensometer, Model LG-51-12, Instron, Corp., Canton, MA.

<sup>##</sup> Kentral Hardness Tester, Model MC-1, Riehle Testing Machines, East Moline, IL.

<sup>§§</sup> Vickers 55 Metallograph, Cook, Troughton, and Simms, Inc., Molden, MA.

<sup>¶¶</sup> Spectrophotometer, Model 403, Perkin-Elmer Corp., Norwalk, CT.

## RESULTS

Data on the mechanical properties of the high-fusing alloys are summarized in Table I. Values for the tensile properties of as-cast Olympia were significantly higher than those of either as-cast Neydium or as-cast JPW. However, cyclic heat treatment at temperatures employed in the application of dental porcelain elicited a discernible increase in the strength characteristics of the latter two materials. Neither the elongation nor the hardness of Neydium and JPW were altered appreciably by the treatment procedure.

Alterations in hardness of the alloys produced by softening and hardening heat treatment are depicted in Figures 1 and 2, respectively. Hardness of Olympia remained relatively stable over the entire range of treatment temperatures (Figure 1). Hardness of Neydium and JPW, however, declined markedly on treatment at temperatures in excess of 1,400° F. Values obtained for Neydium and JPW specimens quenched from 1,800° F. reflected a reduction in hardness of about 30 percent. Significant rehardening of annealed discs of Neydium and JPW was achieved with five-minute reheat treatments at 1,400° F.

Microstructures of the alloys are shown in Figures 3 and 4. As-cast structures of all three materials were cored (Figure 3). Although grain homogeneity improved significantly with heat treatment by the porcelain firing cycle, a subgrain network remained as a striking feature of the structures of Neydium and JPW (Figure 4). Overetching of heat-treated cast specimens was required to reveal the presence of subgrains in the structure of Olympia (Figure 4).

Findings from quantitative analysis of the test materials are presented in Table II. Compositions of Neydium and JPW were remarkably similar. The predominant components of these materials were gold, palladium and silver. Both Neydium and JPW contained a single base metal constituent (tin). Total gold-palladium content of silver-free Olympia was ten percent greater than that of the former materials. Other constituents of Olympia included indium, gallium and ruthenium.

#### DISUCSSION

From the experimental evidence, it would appear that Neydium and JPW are one-and-the same material. The response of these alloys to progressive aging treatments is probably the result of the precipitation of a tin containing compound. Also, it is likely that the increase in strength and hardness which accompanies heat treatment of Neydium and JPW by the porcelain firing cycle is a manifestation of the aging process.

Although the compositional features of Neydium and JPW vary widely from those of Olympia, the homogenized structures of all three materials exhibit striking similarities with respect to average grain size and grain configuration. This, in part, may account for the similarity in the strength and hardness characteristics of thermally cycled castings of the test alloys.

Difficulty encountered in revealing the subgrain boundaries of Olympia is attributed to the effectiveness of ruthenium, and possibly indium, in the control of grain homogeneity. The extent to which the elongation of Olympia exceeds that of the gold-palladium-silver based

materials is compatible with the subtlety of its intragranular structure.

Experience gained through the fabrication of clinical prostheses from the test materials has failed to reveal differences in their handling characteristics. Well fitting, esthetically pleasing restorations can be made from any one of these materials through the use of state-of-the-art porcelain-metal techniques.

#### SUMMARY

Properties, heat treatment response, structure and composition of three high-fusing crown-and-bridge alloys were studied. Except for elongation, the properties of two gold-palladium-silver based materials were comparable to those of a gold-palladium alloy. The greater elongation of the latter material was attributed to its intragranular homogeneity.

REFERENCE

1. Guide to Dental Materials and Devices, 6th ed. Chicago, Am.  
Dental Association, 1972-1973, pp. 207-209.

#### LEGENDS FOR FIGURES

Figure 1. Effect of heat-treatment temperature on hardness of three high-fusing crown-and-bridge alloys.

Figure 2. Effect of reheat treatment temperature on hardness of three high-fusing crown-and-bridge alloys.

Figure 3. As-cast microstructures of three high-fusing crown-and-bridge alloys. (A) Neydium (B), JPW, and (C) Olympia.

Figure 4. Microstructures of heat-treated high-fusing crown-and-bridge alloys. (A) Neydium, (B) JPW, and (C) Olympia.

Table I. Mechanical properties of three high-fusing crown-and-bridge alloys

| Property   | Neydium                 |                         | JPW                     |                          | Olympia                  |                          |
|--|-------------------------|-------------------------|-------------------------|--------------------------|--------------------------|--------------------------|
|  | As-Cast                 | Heat-<br>*<br>Treated   | As-cast                 | Heat-<br>*<br>Treated    | As-cast                  | Heat-<br>*<br>Treated    |
| Tensile strength ( $\times 10^3$ psi)            | 86 $\pm$ 2 <sup>+</sup> | 97 $\pm$ 6 <sup>+</sup> | 85 $\pm$ 1 <sup>+</sup> | 100 $\pm$ 2 <sup>+</sup> | 109 $\pm$ 2 <sup>+</sup> | 115 $\pm$ 7 <sup>+</sup> |
| Yield strength <sup>#</sup> ( $\times 10^3$ psi) | 61 $\pm$ 2              | 71 $\pm$ 7              | 56 $\pm$ 2              | 77 $\pm$ 1               | 73 $\pm$ 1               | 79 $\pm$ 3               |
| Elastic limit ( $\times 10^3$ psi)               | 46 $\pm$ 3              | 57 $\pm$ 10             | 44 $\pm$ 3              | 58 $\pm$ 3               | 65 $\pm$ 0               | 69 $\pm$ 6               |
| Modulus of elasticity<br>( $\times 10^6$ psi)    | 17 $\pm$ 2              | 17 $\pm$ 2              | 17 $\pm$ 1              | 16 $\pm$ 1               | 18 $\pm$ 3               | 18 $\pm$ 1               |
| Elongation (percent)                             | 6 $\pm$ 0               | 8 $\pm$ 0               | 7 $\pm$ 1               | 6 $\pm$ 1                | 13 $\pm$ 1               | 20 $\pm$ 4               |
| Hardness (DPN)                                   | 199 $\pm$ 8             | 218 $\pm$ 18            | 187 $\pm$ 16            | 214 $\pm$ 8              | 213 $\pm$ 12             | 225 $\pm$ 7              |

\* Simulated porcelain firing cycle.

+ Standard deviation.

# 0.2 percent offset.

Table II. Compositions of three high-fusing crown-and-bridge alloys

| Element   | Percentages |       |         |
|-----------|-------------|-------|---------|
|           | Neydium     | JPW   | Olympia |
| Gold      | 48.98       | 49.14 | 51.76   |
| Palladium | 31.37       | 31.37 | 38.18   |
| Platinum  | 0.08        | 0.08  | ---     |
| Silver    | 14.89       | 14.72 | ---     |
| Tin       | 4.50        | 4.54  | ---     |
| Indium    | ---         | ---   | 8.61    |
| Gallium   | ---         | ---   | 1.40    |
| Ruthenium | ---         | ---   | trace   |

