

## **Mechanical properties and microstructural analysis of an AgPd alloy cast under different temperatures**

### ***Propriedades mecânicas e análise microestrutural de uma liga de AgPd fundida sob diferentes temperaturas***

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#### **ABSTRACT**

The metal restorations are used in Dentistry a long time ago. Nowadays we have resources that can get casting more accurate, with new material and equipments and techniques more precise. The purpose of this study was to evaluate the metallurgical and mechanical aspects of the AgPd dental alloy when it was submitted to different casting temperatures. It was used 30 specimens, divided in three groups (n=10): a) control group (no cast); b) casting temperature in accordance with the manufactures' instructions (T1); c) casting temperature above manufactures' instructions (like a torch) (T2). It was evaluated chemical and metallographic aspects, mechanical properties and Vickers hardness. The results showed a microstructure similar to T1 and T2 conditions, but with greater amount of light phase and particles in this last one. It was observed that the rupture tensile strength for the T1 condition was greater than the T2. The alloy in the no cast condition presented greater hardness but there was no statistically significant difference between T1 and T2. Supporting by the metallographic and mechanical results, it may predict that when elevated temperatures was used, above manufacture's recommendations, it can occur failures in the prosthesis, like porosities, fissures or cracks.

#### **UNITERMS**

Dental alloys; dental casting technique.

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## **INTRODUCTION**

Alloys are composed of two or more elements, and sometimes non-metal elements are also included. The casting of two elements will originate a binary alloy and the combination of three elements is called ternary alloy<sup>1,2,5</sup>

The firstly employed alloys were gold-based alloys, whose main characteristic was their resistance

to oxidation, which was more noticeable than the other properties, almost unknown at that time<sup>3</sup>. With the improvement on research methods, it was verified that besides resisting oxidation, the different gold-based alloys presented mechanical and biological properties perfectly compatible for oral use. However, due to the worsened economical situation and consequent increase in the price of gold, the use of these alloys became impractical and inaccessible for many practitioners<sup>6,9</sup>.

The choice of an alloy is based on several factors. Cost is a serious consideration due to the high price of gold. Other factors that shall be considered are biocompatibility and corrosion resistance<sup>18</sup>. These factors in particular limited the use of alloys for dental prostheses, and the choice of an specific application is primarily determined by their mechanical properties, such as hardness, mechanical strength and ductility<sup>14</sup>.

We may also emphasize the information provided by the ADA in 1980 stating that metal alloys composed of less than 75% of weigh in gold, platinum and other noble metals present staining and corrosion, and because of that they are used as alternative alloys<sup>10</sup>.

The inferior qualities of non-noble metal alloys when compared to gold-based alloys led to the introduction of quantitative and qualitative modifications in their chemical composition, either in making and casting procedures or in laboratory techniques, all of which had the objective of allowing their use as eventual substitutes for cast gold restorations, at the same

time that *in vivo* and *in vitro* studies were performed with comparative purposes<sup>11,17</sup>.

The objective of this study was evaluate the metallurgical and mechanical aspects of an AgPd alloy used for fixed partial and single prostheses using different casting temperatures.

## MATERIALS AND METHODS

The Palliag M (Degussa-Huls, Hanau, Germany) alloy used for this study is specifically applied for dental use in Fixed Partial Prostheses. This alloy has 58.5% of silver (Ag) and 27.4% of palladium (Pd), and has a specific mass of 11.1 according to data supplied by the manufacturer.

Twenty specimens were obtained by means of lost wax casting technique and divided in two groups of ten, so that each one was submitted to a different temperature: T1 – recommended by the manufacturer (1100°C), and T2 – above the recommended temperature (1300 ± 20°C) (Table 1).

**Table 1 – Temperatures used for alloy casting in the two studied conditions**

Alloy	Casting Temperature* (°C)	Above casting temperature (°C)
Palliag M	1100°	1300° ± 20°

\* recommended by the manufacturer

The templates that originated the specimens in this study were built in a laboratory specialized in dental prostheses using blue wax for inlay casting in a usinated stainless steel matrix, according to the International Standards Organization (ISO) specification number 6871, as it can be seen in Figure 1.

Wax templates were invested in a metal ring previously underlain with amiantus. Then the phosphate-based investment (Micro- Fine 1700- Talladium - USA) was poured according to the manufacturer's recommendations. After the final investment setting, models were placed in an EDG furnace (model EDG-CON 3P – 3000) for wax elimination under temperatures between 600° and 900° C for 90 minutes.

Casting was performed in a centrifuge by induction (model DUCATRON – Series 3 – France) using an argon gas shielding and a digital optical infrared pyrometer (M-GULTAN- Pirograt- IS-3D- Germany) for temperature control.

Upon casting completion, rings were left at room temperature for cooling. Specimens were separated from their investments and submitted to blasting with

glass microballs (50µm) and subsequently with aluminum oxide (250 µm; pressure of 3.0 bar, distance of 2 cm). External machining using mounted tips and abrasive rubbers was performed for final finishing.

The chemical composition analysis of the alloys in this study was performed in the Centro de Caracterização e Desenvolvimento de Materiais (Center for Materials Characterization and Development) at the Universidade Federal de São Carlos-UFSCAR.

Samples with approximately 3mm of thickness were obtained from the tensile strength test specimens and embedded in transparent chemically activated acrylic resin (JET). They were then mechanically polished using 360, 400, 600, 1000 and 2000 mesh sandpaper followed by alumina polishing with 1 and 0.3µm granulations. This procedure aimed at obtaining a flat and polished surface. After that, samples were chemically etched using a 2:1 HCl and HNO3 solution for approximately 3 seconds for the AgPd alloy. The etching was controlled to produce a correct observation of phases and morphologies present in the microstructure.

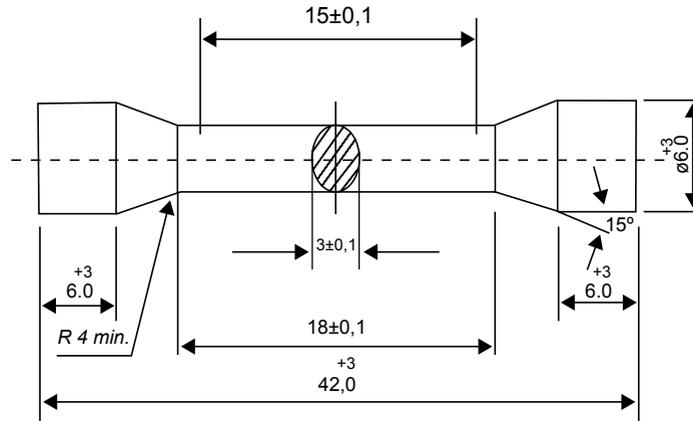


FIGURE 1 – Templates that originated the specimens in this study (ISO 6871).

Finally, they were submitted to metallographic examination using Scanning Electron Microscopy (SEM) in a JEOL-JSM microscope (T-330) coupled to an X-rays dispersive energy analyzer (XDE) and a camera with similar origin.

The tensile and elongation assays were performed using a Material Test System (MTS 810) equipment and data analysis was made using a specialized computer software (Test Star II) coupled to the system. Load cells of 100kN with 1.0mm/mim speed were used. Hardness measurements were obtained using a Micromet – 2003 – Buehler device under 500gf or 4903 mN strength.

Two-way analysis of variance (ANOVA) was used to detected significant differences among the conditions studied.

## RESULTS

There was no significant difference in chemical analysis among NC (no-cast), T1 and T2 groups. The Table 2 illustrates the results obtained in the chemical analysis performed for the three studied conditions using the AgPd alloy.

Table 2 – Chemical analysis performed for the three studied conditions using the AgPd alloy (% m/m)

Elements	NC	T1	T2
Au	1.25 ± 0.57	1.00±0.10	1.09±0.18
Pd	27.47 ± 0.58	26.10 ± 0.35	28.20 ± 0.14
Cu	12.34 ± 0.56	10.50 ± 0.32	10.80 ± 0.21
Ag	BALANCE	BALANCE	BALANCE

In the Figure 2 it can observe a brute fusion microstructure with the presence of particles in the entire alloy matrix. It also verified that the microstructure is oriented according to the direction provided during the lamination process.

It showed in the Figure 4 (T2 condition) a microstructure similar to that found for the T1 condition

(Figure 3), but with greater amount of light phase and less amount of dark phase and particles and porosities.

It was observed in Table 3 that the rupture tensile strength for the T1 condition is greater than the T2 for the AgPd alloy. On the other hand, the elongation and yield strength values were statistically similar.

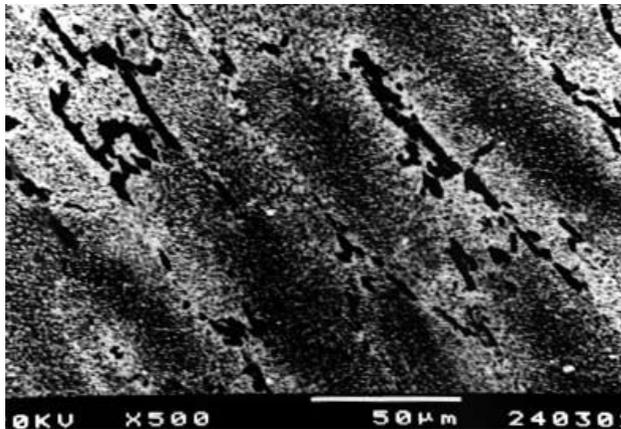


FIGURE 2 - Micrograph of the AgPd alloy in the no cast condition (NC), 500X.

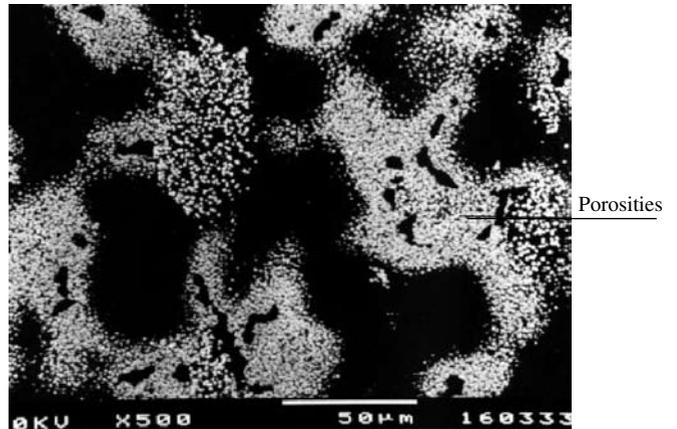


FIGURE 3 - Micrograph of the AgPd alloy in the T1 condition, 500X.

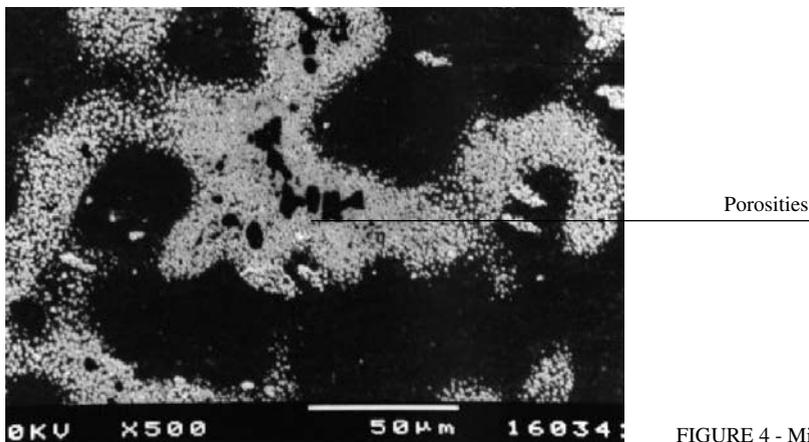


FIGURE 4 - Micrograph of the AgPd alloy in the T2 condition, 500X.

**Table 3 – Tensile strength tests and respective statistical data for the AgPd alloy**

	Elongation (%)		Rupture Tensile Strength (MPa)		Yield Strength (MPa)	
	T1	T2	T1	T2	T1	T2
Maximum Value	10.4	11.0	533.4	530.5	425	438
Minimum Value	4.9	3.7	438.3	414.4	360	387
Mean	7.1	6.6	492.9	473.5	410.8	417.2
Standard Deviation	1.8	2.3	38.8	37.3	19.9	17.04

The results obtained in the Vicker's hardness test for the AgPd alloy and corresponding statistical analysis

can be found in Table 4. There was no statistically significant difference between T1 and T2 (Table 4).

**Table 4 – Vickers hardness of the AgPd alloy**

	<b>NC</b>	<b>T1</b>	<b>T2</b>
Maximum	218.1	175.9	176.8
Minimum	209.3	171.1	169.3
Mean	213.8	173.4	172.1
Standard Deviation	2.5	1.4	2.7

## DISCUSSION

By observing the chemical analysis for this alloy in the no cast condition, we conclude that Table 2 is coherent with the composition supplied by the manufacture<sup>7</sup>. The results observed in Table 2 showed that there was no significant variation in the composition of Ag, Pd and Au under the three studied conditions, but there was a small reduction in the concentration of Cu under the T1 and T2 conditions. The reduction in Cu in this respective alloy may be due to an inefficient protection provided by the argon atmosphere during casting procedures, because this element is more reactive with oxygen. In the laboratories is very important that all the castings were performed using an argon atmosphere to avoid the contamination of the alloy and porosities<sup>4,8,13,15</sup>.

The dendritic microstructures found in the Au-Pd-Ag and Ag-Pd alloys by some authors is very similar to those obtained under the T1 condition, as long as they were cast according to the manufacturers' specifications and presented similar chemical composition as showed in the Figure 4<sup>4,16,19,20</sup>. Clinically, the microstructures without porosities are more resistant above mechanical forces.

The AgPd alloys presented a brute fusion microstructure with precipitates in the entire matrix for the no cast condition. When a temperature above the recommended by the manufacturer was used we could observe a double-phase dendritic microstructure (light and dark phases) with porosities<sup>12</sup>.

When the casting temperature is increased, like a T2 group, there is also an increase in the cooling speed which leads to a reduction on the time available for a phase transformation, which means the formation of dark phase and precipitates. Iridium is a noble metal with a high fusion point (2446°C) that can remain

as an impurity after the processing and refinement of gold, silver or palladium. This impurity can alter the structure of the casting and causes failure in the prosthesis<sup>16</sup>.

The rupture tensile strength for the T1 condition is greater than the T2 for the AgPd alloy. On the other hand, the elongation and yield strength values were statistically similar. Although the rupture tensile strength showed no statistically significant difference it can notice a more refined microstructure with greater amount of precipitate that leads to a better resistance under the T1 than the T2 condition.

The results obtained in the Vicker's hardness test for the AgPd are coherent with the observed microstructure. The alloy in the no cast condition presented greater hardness because it suffered a lamination process, and its microstructure was encruded (hardened due to a mechanical deformation). There was no statistically significant difference between T1 and T2.

## CONCLUSION

This study evaluated the metallurgical and clinical aspects of the different casting temperatures on an AgPd alloy. Within the limitations of this work, it can conclude that all the castings that were performed using manufacture's recommendations showed the best results. Supporting by results, it may predict that when elevated temperatures was used above manufacture's recommendations (more than 200°C), it can occur failures in the prosthesis, like porosities, fissures or cracks.

## ACKNOWLEDGMENT

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## RESUMO

As restaurações metalocerâmicas são usadas na Odontologia a várias décadas. Atualmente temos tecnologia que os fornecem fundições mais acuradas, com materiais e equipamentos novos e técnicas mais precisas. O objetivo desse trabalho foi avaliar aspectos metalúrgicos e propriedades mecânicas de uma liga odontológica a base de AgPd quando submetida a diferentes temperaturas. Foram usados 30 espécimes, divididos em três grupos (n=10): a) grupo controle (não fundido); b) grupo fundido de acordo com a temperatura recomendada pelo fabricante (T1); e c) grupo fundido com temperatura acima da recomendada pelo fabricante (simulando um maçarico) (T2). Foram avaliados aspectos químicos, metalográficos, propriedades mecânicas e dureza Vickers. Os resultados mostraram uma microestrutura similar nos grupos T1 e T2, mas com maior quantidade de fase clara e partículas neste último. Foi observado que a resistência à fratura no grupo T1 foi maior que no T2. A liga na condição não fundida apresentou o maior valor de dureza Vickers. A partir desses resultados, metalográficos e mecânicos, pode-se supor que quando se usa temperatura acima da recomendada pelo fabricante poderá ocorrer falhas na prótese, como porosidades, fissuras ou fraturas.

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Ligas dentárias; técnica de fundição odontológica.

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