

## Does the casting mode influence microstructure, fracture and properties of different metal ceramic alloys?

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**Abstract:** The aim of the present study was to evaluate the tensile strength, elongation, microhardness, microstructure and fracture pattern of various metal ceramic alloys cast under different casting conditions. Two Ni-Cr alloys, Co-Cr and Pd-Ag were used. The casting conditions were as follows: electromagnetic induction under argon atmosphere, vacuum, using blowtorch without atmosphere control. For each condition, 16 specimens, each measuring 25 mm long and 2.5 mm in diameter, were obtained. Ultimate tensile strength (UTS) and elongation (EL) tests were performed using a Kratos machine. Vickers Microhardness (VM), fracture mode and microstructure were analyzed by SEM. UTS, EL and VM data were statistically analyzed using ANOVA. For UTS, alloy composition had a direct influence on casting condition of alloys (Wiron 99 and Remanium CD), with higher values shown when cast with Flame/Air ( $p < 0.05$ ). The factors “alloy” and “casting condition” influenced the EL and VM results, generally presenting opposite results, i.e., alloy with high elongation value had lower hardness (Wiron 99), and casting condition with the lowest EL values had the highest VM values (blowtorch). Both factors had significant influence on the properties evaluated, and prosthetic laboratories should select the appropriate casting method for each alloy composition to obtain the desired property.

**Descriptors:** Dental Casting Techniques; Dental Alloys; Hardness Test; Tensile Strength; Metal Ceramic Alloys.

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### Introduction

Among the innumerable advantages of Ni-Cr and Co-Cr alloys, the most outstanding are their low cost,<sup>1,2</sup> high modulus of elasticity<sup>3</sup> and being relatively easy to cast, using a blowtorch as a heat source.<sup>4,5</sup>

However, some studies have demonstrated that when noble<sup>6</sup> and basic alloys<sup>7</sup> are cast in an uncontrolled environment, the metals are exposed to deleterious gases and elements, such as nitrogen and oxygen which they may absorb from the atmospheric air. This gas absorption, which is extremely dependent on the heating time, alloy composition and casting methods,<sup>8</sup> may negatively influence their resistance to corrosion or mechanical properties, because it induces internal porosity. It is known that the casting process may increase the porosity of noble alloys, particularly those containing palladium (Pd) in their composition.<sup>9</sup>

Another source of alloy contamination during the casting process is

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the combustion gas used in the blowtorch, which leads to carbon formation.<sup>10</sup> As fuel for a (gas/oxygen) blowtorch, hydrogen derived from the decomposition of H<sub>2</sub>O may be used, since this gas has great affinity for Pd.

The process of casting these alloys may be improved with the use of casting machines requiring different heat sources, which have been developed for titanium alloy casting.<sup>11</sup> Machines that associate a controlled environment with inert gas as a heat source may be able to reduce the contamination of alloy.<sup>12</sup> In addition, they allow the temperature to be controlled, with the technical advantage of reducing heating time and eliminating the need for flame adjustment. Moreover, there is reduced contamination, less probability of burning the metal, and consequently, less porosity due to volatilization of components with low melting temperatures.<sup>13</sup>

However, the mechanical properties and microstructure of different alloys indicated for Ni-Cr, Co-Cr and Pd-Ag metal ceramics cast under different casting conditions are not known, and the object of this study was to investigate these factors.

## Methodology

Two Ni-Cr-based alloys (Wiron 99, Bego, Bremen, Germany; and Suprem Cast V, Talladium Inc, Valencia, USA), one Co-Cr-based alloy (Remanium CD, Dentaurum, Pforzheim, Germany) and one Pd-Ag-based alloy (W-1, Ivoclar Vivadent, Schaan, Liechtenstein) were evaluated. The composition of the alloys is presented in Table 1.

Test specimens (cps) were obtained by means of wax patterns measuring 25 mm long and 2.5 mm in diameter (n = 16). The wax patterns were included in a phosphated lining (Micro-fine 1700, Talladium Inc, Valencia, USA) immediately after fabrication,

using a metal mold. After final setting of the lining, each ring was put into a furnace (Ovmat-7, F.lli Manfredi, Torino, Italy).

After this, each of the alloys was cast according to the casting conditions to be tested:

1. (Flame/Air) gas/oxygen blow torch (Kerr Manufacturing Co, Orange, USA);
2. (Induction/Argon) electromagnetic induction (Easyti System, F.lli Manfredi, Torino, Italy), in an environment controlled with argon (Air Liquide, Sao Paulo, Brazil);
3. (Induction/Vacuum) electromagnetic induction under vacuum;
4. (Induction/Air) electromagnetic induction without control of the environment.

Each sample was tested under tension at a cross-head speed of 1 mm/min in a testing machine (Kratos, Sao Paulo, Brazil). Ultimate tensile strength and elongation to failure were determined. In addition, two of the fractured tensile specimens from each group were investigated by scanning electron microscopy (SEM) (JEOL JSM 6300, Tokyo, Japan) to characterize their fracture mode. The fracture mode results were evaluated only qualitatively.

After the tensile test, one half of two different fractured specimens were embedded in bakelite for metallographic sample preparation. The prepared surfaces were examined under a stereomicroscope (100×, Shimadzu HMV-2, Tokyo, Japan).

Microhardness was measured using a Vickers indenter (Shimadzu HMV-2, Tokyo, Japan) with a 1000 g load and a loading time of 10 s. Four measurements were made in each cast specimen.

## Statistical analysis

The tensile strength (MPa), elongation (%) and

**Table 1** - Composition of alloys used in this study (wt%).

Alloys	Ni	Co	Cr	Nb	Mo	Be	Si	Al	Sn	Ti	Ag	Pd	Others
Wiron 99	65.0	–	22.5	1.0	9.5	–	1.0	2.2	–	0.4	–	–	
Suprem Cast V	74.0	–	14.0	–	8.5	1.8	–	1.7	–	–	–	–	
Remanium CD	–	65.0	28.0		4.5		1.6		–	–	–	–	Fe, Mn, Al, La, Ce
W-1	–	–	–	–	–	–	–	–	8.5	–	37.7	53.3	In, Ru, Li

Vickers microhardness (VHN) data were subjected to a two-way ANOVA (Alloy versus Casting condition) and Tukey's test for pair-wise comparisons (0.05). The microstructure and fracture pattern results were evaluated only qualitatively.

## Results

### Tensile strength

Two-way ANOVA detected interaction between alloy and casting condition ( $p < 0.05$ , Table 2). For the Suprem Cast V and W-1 alloys the different casting procedures did not influence the tensile strength means. Suprem Cast V alloy showed the highest tensile strength means under all conditions ( $p < 0.05$ ), whereas the lowest tensile strength means were observed for the Wiron 99 and Remanium CD alloys when cast under the induction/argon and induction/vacuum condition respectively.

### Elongation

Only the main factors were statistically significant ( $p < 0.05$ , Table 3). With regard to the factor "alloy", the following materials were ranked in as-

ending order: W-1 < Suprem Cast V < Remanium CD < Wiron 99. As regards the casting condition, the lowest elongation values were found for Flame/Air, with the highest values being obtained when casting was performed with Induction/Vacuum and Induction/Argon ( $p < 0.05$ ).

### Vickers microhardness

Only the main factors were significant ( $p < 0.05$ , Table 4). For the factor alloy, there were significant differences for all the materials, in the following decreasing order of values: Suprem Cast V > Remanium CD > W-1 > Wiron 99. In the case of heat sources, the lowest microhardness values were found for Induction/Argon casting and the highest for Flame/Air casting ( $p < 0.05$ ).

### Microstructure

Figures 1, 2, 3 and 4 show the microstructure of the different alloys. No obvious microstructural differences were observed for the same alloy under the different casting conditions. Differences were only noted when different alloys were compared. All

**Table 2** - Means and standard deviations of ultimate tensile strength (MPa).

	Ultimate tensile strength			
	Flame/Air	Induction/Air	Induction/Vacuum	Induction/Argon
Suprem Cast V	1024 ± 58 <sup>a</sup>	1030 ± 54 <sup>a</sup>	1027 ± 65 <sup>a</sup>	1026 ± 74 <sup>a</sup>
Wiron 99	650 ± 64 <sup>d</sup>	623 ± 49 <sup>d,e</sup>	576 ± 34 <sup>e</sup>	572 ± 29 <sup>e</sup>
Remanium CD	864 ± 44 <sup>b</sup>	741 ± 55 <sup>c</sup>	659 ± 58 <sup>d</sup>	665 ± 43 <sup>d</sup>
W-1	622 ± 25 <sup>d,e</sup>	625 ± 48 <sup>d,e</sup>	618 ± 73 <sup>d,e</sup>	608 ± 51 <sup>d,e</sup>

Different letters indicate statistically significant differences (Tukey,  $p < 0.05$ ).

**Table 3** - Means and standard deviations of elongation (%).

Alloys	Elongation				Total mean of alloys
	Casting methods				
	Flame/Air	Induction/Air	Induction/Vacuum	Induction/Argon	
Suprem Cast V	7.25 ± 2.2	8.20 ± 3.0	9.44 ± 2.9	10.3 ± 2.1	8.80 ± 2.6 <sup>C</sup>
Wiron 99	46.2 ± 9.2	49.9 ± 8.1	51.3 ± 8.1	50.4 ± 5.4	49.5 ± 7.7 <sup>A</sup>
Remanium CD	12.1 ± 1.7	13.4 ± 1.9	12.9 ± 1.9	14.4 ± 2.9	13.2 ± 2.1 <sup>B</sup>
W-1	4.89 ± 1.1	4.89 ± 2.0	6.90 ± 1.5	7.33 ± 1.3	6.00 ± 1.5 <sup>D</sup>
Total mean of casting methods	19.1 ± 3.8 <sup>b</sup>	20.1 ± 3.6 <sup>a</sup>	20.6 ± 2.9 <sup>a</sup>	20.6 ± 2.9 <sup>a</sup>	

Different superscript lower case letters indicate statistical significance for the factor "casting methods", and different superscript capital letters indicate statistical significance for the factor "alloy" (Tukey,  $p < 0.05$ ).

**Table 4** - Means and standard deviations of Vickers Hardness.

Alloys	Microhardness				Total mean of alloys
	Casting methods				
	Flame/Air	Induction/Air	Induction/Vacuum	Induction/Argon	
Suprem Cast V	353 ± 17.1	371 ± 4.2	362 ± 9.6	352 ± 4.3	360 ± 8.8 <sup>A</sup>
Wiron 99	208 ± 5.0	209 ± 5.8	202 ± 15.3	194 ± 4.2	203 ± 7.6 <sup>D</sup>
Remanium CD	361 ± 13.7	337 ± 18.4	340 ± 3.9	340 ± 13.7	345 ± 11.7 <sup>B</sup>
W-1	242 ± 45.8	238 ± 4.4	238 ± 5.8	224 ± 13.8	236 ± 17.5 <sup>C</sup>
Total mean of Casting methods	291 ± 20.4 <sup>a</sup>	289 ± 8.2 <sup>a</sup>	286 ± 8.7 <sup>a,b</sup>	278 ± 9.0 <sup>b</sup>	

Different superscript lower case letters indicate statistical significance for the factor "casting methods", and different superscript capital letters indicate statistical significance for the factor "alloy" (Tukey,  $p < 0.05$ ).

alloys exhibited a solid solution matrix in a typical dendritic arrangement. The Suprem Cast (Ni-Cr-Be) microstructure (Figure 1) shows evidence of a dendritic microstructure and an interdendritic portion with lamellar eutectic structures, possibly Ni-Be. In Figure 2 [Wiron 99 (Ni-Cr)], a high volume of solid solution with few areas of interdendritic structures is observed. This is probably due to the high solubility of the alloying elements (Cr, Mo and Nb) in the Ni-matrix. The W-1 microstructure (Pd-Ag) presents white plates that represent the dendritic region, and black plates, possibly a large volume of segregated portion (Figure 3). Figure 4 shows the Remanium CD microstructure (Co-Cr). This alloy is characterized by a high volume of solid solution and low content of interdendritic portion and precipitates.

### Fracture mode (SEM)

The different casting conditions did not influence the fracture mode of the alloys. Suprem Cast V alloy showed a friable type of fracture due to the absence of cracks on the external surface of the test specimen. There was presence of globules in the inner portion of this alloy, showing evidence of absence of flow in the fracture region.

The Wiron 99 alloy test specimen showed a large external deformation, which resulted in loss of its circular shape. Its internal portion was marked by deformation or flow of dendrites.

The W-1 alloy test specimen showed a friable type of fracture due to absence of cracks on the external surface of the specimen. Its internal por-

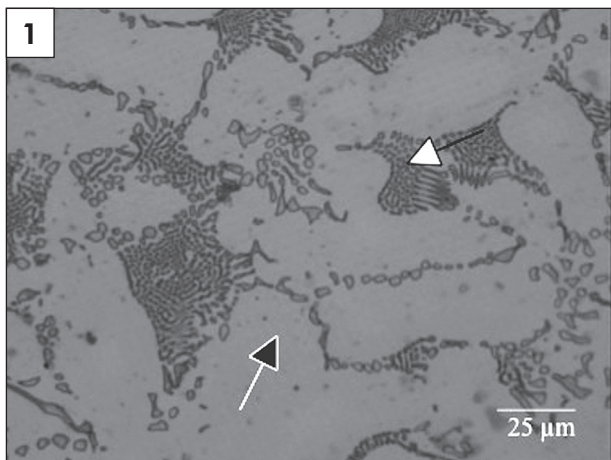
tion was characterized by the formation of dimples, which demonstrated low elongation. Remanium CD alloy was characterized by the scarce presence of cracks indicating some deformation before rupture. Its internal portion was marked by the formation of laminas of rupture, which was characteristic of low elongation.

### Discussion

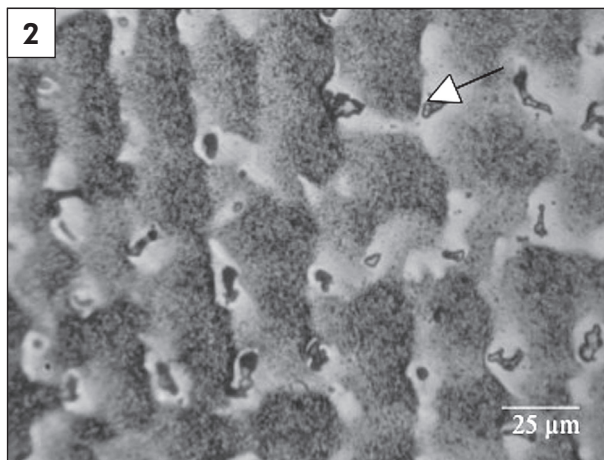
The microstructure of Ni-Cr-Be alloys shows a large volume fraction of Ni-Be in solid solution (Figure 1), as secondary phase located in the interdendritic portion, which promotes an increase in tensile strength and microhardness.<sup>10</sup> This can be observed when compared with other alloys without Be, such as Wiron 99, which showed complete absence of an interdendritic region (Figure 2).

Explanations for the high tensile strength, microhardness and low elongation values perhaps reside in the interdendritic portion and presence of Be, an element that is a grain refiner and hardener of Ni-Cr alloys.<sup>14</sup> Moreover, there is the presence of other elements, such as C and Al. Aluminum provides an increase in mechanical properties in Ni-Cr alloys by forming a hardening compound ( $\text{Ni}_3\text{Al}$ ), and C in concentrations from 1% to 3% significantly increases the hardness values and diminishes elongation.<sup>15</sup>

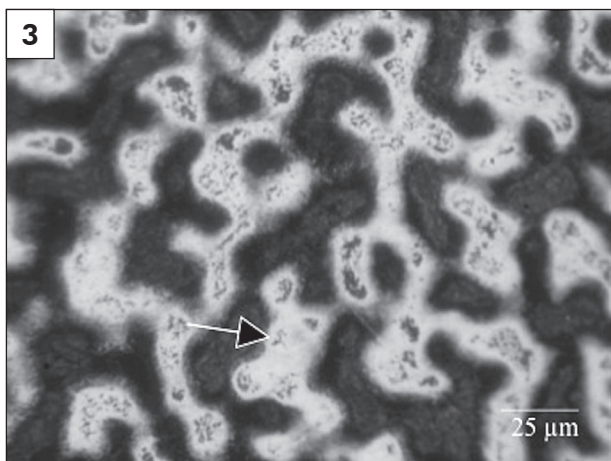
Wiron-99 alloy showed low tensile strength and microhardness values, but higher elongation values. These values can be explained when one observes the composition of this alloy, which contains elements with high solubility, such as Cr, Mo and Nb,<sup>10</sup> in the Ni matrix. Thus, hardness and tensile



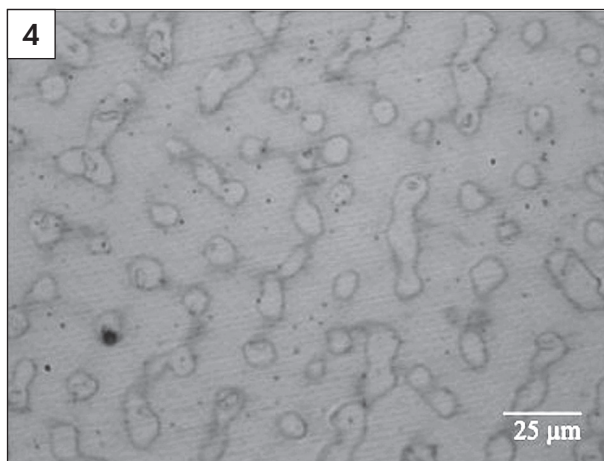
**Figure 1** - The Suprem Cast V microstructure showed the presence of a dendritic (white arrow) and an interdendritic portion with lamellar eutectic structures (black arrow), possibly Ni-Be.



**Figure 2** - Wiron 99 showed high volume of solid solution with few areas of interdendritic structures. Note the presence of precipitates (white arrow).



**Figure 3** - W-1 presented white plates that represent the dendritic region (black arrow) and black plates, possibly a large volume of segregated portion.



**Figure 4** - Reranium CD alloy showed a microstructure characterized by a high volume of solid solution

strength are increased with the inclusion of elements such as Si, C and Fe, which act as precipitates within the Ni matrix.<sup>16</sup> This is confirmed with the observation of a large volume of solid solution and absence of the interdendritic portion in the microstructure (Figure 2). Nevertheless, the Ni-Cr alloys may cause a hypersensitivity reaction to Ni.<sup>17</sup>

The Pd-Ag alloy (W-1) has a high modulus of elasticity, obtains excellent bond to porcelain, is high temperature resistant (*sag*), easy to use and resistant to corrosion.<sup>18</sup>

Among all the alloys, W-1 was the one that

demonstrated the least elongation. This may be explained by the remarkable presence of C, an interstitial element that inhibits flow and deformation of the crystalline grid or the presence of grain refining elements, in this case Rutile.<sup>19</sup> A possible reason for the low elongation and high hardness of this alloy lies in the formation of a dendritic microstructure that has an interdendritic portion with a large volume, which makes stress propagation difficult.<sup>20</sup> The presence of a dendritic microstructure is of great importance in mechanical properties,<sup>21</sup> confirmed by the observation of the fracture pattern of



this alloy, which presented no crack formation on the external surface of the test specimen. Therefore, Co-Cr alloys have been proposed as an excellent alternative to high cost noble alloys and the biologic problems related to Ni-Cr alloys. Co-Cr alloys have good mechanical properties, such as microhardness and tensile strength, obtained due to the combination of a solid solution hardener and C, Cr, Mo and W precipitates in the pure Co matrix.<sup>22</sup> The elongation results obtained were shown to be superior to those of Suprem Cast V and W-1 alloys, completely compatible with the fracture pattern, which showed deformation of the dendrites and crack formation before rupture. The flame/air process significantly affected the tensile strength of Wiron 99 and Remanium CD alloys, which showed a trend towards elevation of values. Flame/air also presented the lowest elongation values, and highest Vickers microhardness values. This could be attributed to the incorporation of contaminants such as C, an interstitial element, arising from the combustion of gases arising from the heat source.<sup>10</sup> Casting by flame/air, combined with alloys that present elements such as Cr, Ti, Nb, Si and Mo in their composition, may lead to the formation of carbides of the MC, M<sub>6</sub>C, M<sub>7</sub>C<sub>3</sub>, M<sub>23</sub>C<sub>6</sub> and M<sub>2</sub>C<sub>3</sub> type.<sup>23</sup>

Furthermore, an increase in the concentration of C may increase the Vickers microhardness, tensile strength and diminish elongation, as the carbides are hard and friable. This results in the initiation of cracks within the carbide thus leading to a reduction in ductility.<sup>24</sup> Therefore, the hypothesis is that the high concentration of Cr in these alloys may have been a crucial factor in the sensitivity to the casting process. Therefore, basic alloys with low Cr content tend to behave indifferently, irrespective of how they are cast, in this case the Suprem Cast V alloy.

Alloys that contain Pd are known for their capacity to absorb gases from the environment during the casting process, particularly when heated with an inadequately set blowtorch.<sup>9</sup> Perhaps this capacity of Pd alloy to absorb gases did not have such a pronounced effect on the mechanical properties in this study. An advantage of controlling the casting

temperature is that it prevents the materials from being overheated, and consequently, precludes the creation of porosities by volatilization of components with lower fusion temperatures.

From a clinical point of view, the casting method can influence the surface roughness of the part due to oxidation of the metal casting under different conditions. This can lead to pathogenic microorganisms becoming attached to rough alloy surfaces, resulting in increased incidence of oral diseases<sup>25,26</sup> and acceleration of biocorrosion due to providing retentive niches.<sup>27</sup> Moreover, alloy casting done in a protected environment significantly decreased roughness and provided samples with smoother surfaces when compared with acetylene-oxygen flame casting. This is very important because the smoother surfaces facilitate finishing and polishing procedures, especially in areas of difficult access. Greater surface roughness requires additional finishing and polishing procedures, increasing the time necessary to complete these procedures.<sup>13</sup>

## Conclusion

The casting method without control of the environment, mainly using a blowtorch, tended to increase the microhardness and decrease the elongation of the alloys. With regard to alloys, Ni-Cr-Mo alloys showed the highest EL and lowest VM. Ag-Pd alloy had the worst elongation and Ni-Cr-Be the highest VM values. Regarding tensile strength, Ni-Cr-Be (highest UTS) and Ag-Pd (lowest UTS) alloys were not influenced by the casting methods, however, Ni-Cr-Mo and Co-Cr were significantly improved by using the blowtorch.

The composition of the alloys, as well as the casting methods significantly influenced the properties evaluated. Prosthetic laboratories should select the appropriate casting method for each alloy composition in order to obtain the desired property.

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