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An evaluation of the effect of porcelain firing cycles on the properties of co-cr casting alloys

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Abstract
To satisfy the demand of esthetic restorations, PFM prosthesis were introduced which also had sufficient strength. In the process of fabrication of PFM prosthesis, the alloy sbstructure is layered with various layes of powdered ceramics which is subjected to sintering process at high temperatures. At least three such firing cycles are needed which may result in alteration of properties of metal alloy sbstructure. Co-Cr is one of the most common base maetal alloy used for this purpose which is used in this study. The bar & disc shaped specimens were fabricated from Co-Cr alloys and were divided in various groups. The bar specimens were subjected to Ultimate Tensile Testing and disc specimens were subjected to X Ray Diffraction and Vickers Hardness Test. The UTS values of the Co-Cr ally decreases where as the HV values increases with increase in firing cycles. The changes in XRD patterns were also consistent.

Keywords: Co-Cr casting alloys, PFM firings, ultimate tensile strength, Vickers hardness, x-ray diffraction

1. Introduction
Esthetics has always been of paramount importance in the fabrication of indirect restorations and this has increased the demand for ceramic restorations since the introduction of ceramic as a restorative material in early 1900s [1]. The first published report describing the use of ceramic in PFM crowns was published in the mid-1950s [2].

The process of PFM prosthesis fabrication is quite complex. This process includes the fabrication of wax patterns which are then cast into metal alloy copings. In order to obtain a ceramic veneer, the copings are coated with various layers of porcelain and are subsequently subjected to numerous porcelain firing cycles. The firing process of porcelain fused to metal (PFM) prosthesis is conducted at a temperature range of 950 °C to 1010 °C in a vacuum in minimum three stages i.e. opaque, body and glaze [1].

Even though the majority of the functional stresses are borne by the ceramic layer in a PFM restoration, the mechanical properties of the alloy substructure play a major role in the success of the entire prosthesis.

The repeated firings to which the metal substructure is subjected to may cause some change in the internal structure, hardness, strength and the internal stresses developed in the metal alloy, the evaluation of which is necessary as it may affect the clinical success of the final prosthesis [3].

Ni-Cr alloys are the commonly used base metal alloys for dental prostheses. However, studies on Ni-Cr alloy have proven its allergic effects due to the release of Ni^{+} ions into the oral cavity during the corrosion process. To avoid this, cobalt-chromium (Co-Cr) alloys were developed as an alternative to Ni-Cr alloy for PFM crowns. In addition, Co-Cr alloys are stronger, harder, tougher, and are more resistant to corrosion than Ni-Cr alloys [4]. Thus it is important to study the properties of Co-Cr alloys and the effect of firing cycles on them.

Therefore, this study was planned to evaluate and compare the mechanical properties (tensile strength and Vickers hardness) of Ni-Cr and Co-Cr dental casting alloys before and after each porcelain firing cycle (fired once, fired twice, and fired thrice) and to relate these properties to the changes in XRD patterns of Cr-Co alloy that occur after every porcelain firing cycle.

2. Materials & Methods
For the purpose of this study, 20 bar shaped & 20 disc shaped specimens were cast from Cr-Co casting alloys:

Bar specimens: for tensile test. Dimensions: length-13/8 inch (35 mm), diameter- 0.09 ± 0.01 inch (2.5mm), and 12-24 threads at each end and a ¼ inch (6.35mm) radius connecting the bar and threaded portions.
Disc Specimens: for XRD study and Vickers Hardness. Dimensions:
5mm in thickness and 8mm in diameter.
The patterns of each specimen were prepared with Poly Lactic Acid (PLA) using a FDM type 3D printing machine. These patterns were sprued, invested and cast in Co-Cr alloy. All 40 Specimens were finished and polished and were divided in 4 groups. Group 1 (fired once), Group 2 (fired twice) and Group 3 (fired thrice) represented opaque, body & glaze firing respectively. Group C represented control group (unfired).

Table 1: Opaque, Body and Enamel firing cycles

Firing Cycle	Opaque Firing	Body Firing	Glaze Firing
Dry time	5 mins	3 mins	5 mins
Preheat time	3 mins	3 mins	3 mins
Low temperature	500 ºC	650 ºC	650 ºC
Heat rate	100 ºC/min	55 ºC/min	70 ºC/min
High Temperature	975 ºC	960ºC	935ºC
Vacuum on	500 ºC	650 ºC	None
Vacuum off	975 ºC	960 ºC	None
Holding time	2 mins	1 min	1 min

2.1 Tensile Test
All bar shaped samples were subjected to tensile test in an Universal Testing Machine with a 0.5 mm/min crosshead speed and a 50 kN load cell. Before starting the test, the gauge length of the tensile bar specimen was measured bu an Universal Testing Machine (UTM).

2.2 X-Ray Diffraction Study
All disc shaped specimens were first subjected to the X-Ray diffraction test. X-Ray Diffraction patterns were conducted at room temperature over 2θ range of 40° to 100° using a diffractometer. The diffraction was done with Cu kα radiation with step size 0.02 and the step time of 0.50s.

2.3 Vickers Hardness
Once the X-Ray diffraction study was done the disc shaped samples were tested for Vickers Hardness Number using a digital display Vickers hardness tester with a diamond pyramidal indenter. The hardness of the specimen was determined automatically using a digitized hardness tester by measuring the distance between the diagonals of the indentation.
The values of UTS & Hardness test were subjected to one way ANOVA to analyze the differences. The inter group comparison was later done using student’s t-test.

3. Results
3.1 Tensile test
The results of one way ANOVA for UTS showed highly significant (p=0.000) change in UTS values. The intergroup comparison showed decrease in UTS from Group C to Group 1 and from Group 2 to Group 3 was not significant (p>0.05). Whereas, highly significant (p=0) decrease in UTS was seen from Group C to Group 2, Group C to Group 3, Group 1 to Group 2 and Group 1 to Group 3.

3.2 Vickers Hardness
The results of one way ANOVA for Vickers Hardness showed highly significant (p=0.000) change in HV values. The intergroup comparison showed highly significant (p=0) increase in hardness values from Group C to Group 1, C to Group 2, Group C to Group 3. Significant (p<0.05) increase in the hardness value was seen from Group 1 to Group 3. However, increase in hardness value was not significant from Group 1 to Group 2 and from Group 2 to Group 3.

3.3 X-Ray Diffraction
The XRD patterns of the Control Groups showed that the pattern is similar to the major element i.e. cobalt. The XRD patterns showed variation amongst the groups. Group C showed peaks corresponding to 2Ө values of 44º, 47º, 51º, 75º & 91º which were related to (111), (111), (200), (220) & (311) planes respectively. However, after first firing i.e. Group 1, the peak at 47º disappears and the peak at 91º appears. Remaining three Groups (C1, C2, & C3) showed peaks corresponding to 2Ө values of 44º, 51º, 75º, 91º & 97º which were related to (111), (200), (220), (311) & (222) planes respectively. Also, all the peaks get intense from Group C to Group 3.

Table 2: Ultimate Tensile Strength values of Co-Cr bar specimens before and after each firing

UTS Values (MPa)		
Groups	**Mean**	**SD**
Control	601.3	4.13
1st PFM Firing	595.3	6.70
2nd PFM Firing	581.3	4.55
3rd PFM Firing	572.5	8.01

Table 3: One way ANOVA Comparison of Ultimate Tensile Strength

One way ANOVA for UTS					
	Sum of Squares	**df**	**Mean Square**	**F value**	**p Value**
Between Groups	2291.550	3	763.850	26.908	0.000 HS
Within Groups	454.200	16	28.387		
Total	2745.750	19			

HS- Highly significant, Sig- Significant, NS- Non-significant

Table 4: Vickers Hardness values of Co-Cr bar specimens before and after each firing

HV Values		
Groups	**Mean**	**SD**
Control	247.40	5.50
1st PFM Firing	264.00	3.54
2nd PFM Firing	268.00	4.53
3rd PFM Firing	269.20	3.27

HS- Highly significant, Sig- Significant, NS- Non-significant

Table 5: One way ANOVA Comparison of Vickers Hardness

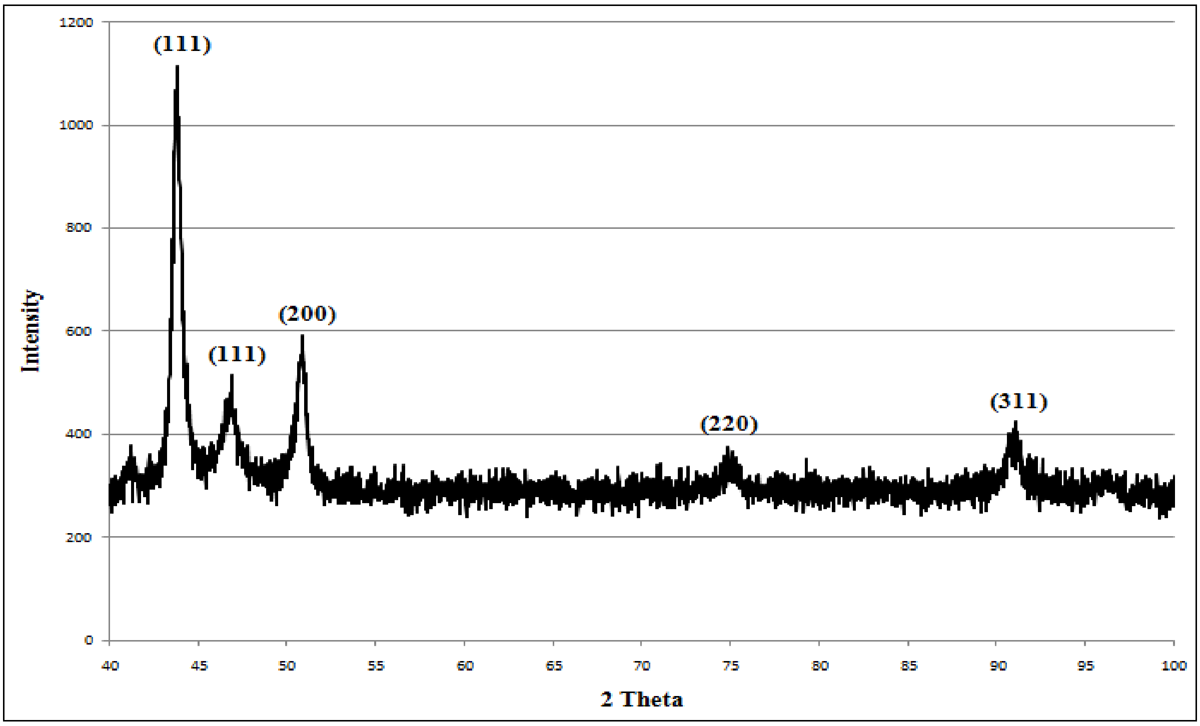
One way ANOVA for Vickers hardness					
	Sum of Squares	**df**	**Mean Square**	**F value**	**p Value**
Between Groups	1524.550	3	508.183	27.469	0.000 HS
Within Groups	296.000	16	18.500		
Total	1820.550	19			

HS- Highly significant, Sig- Significant, NS- Non-significant

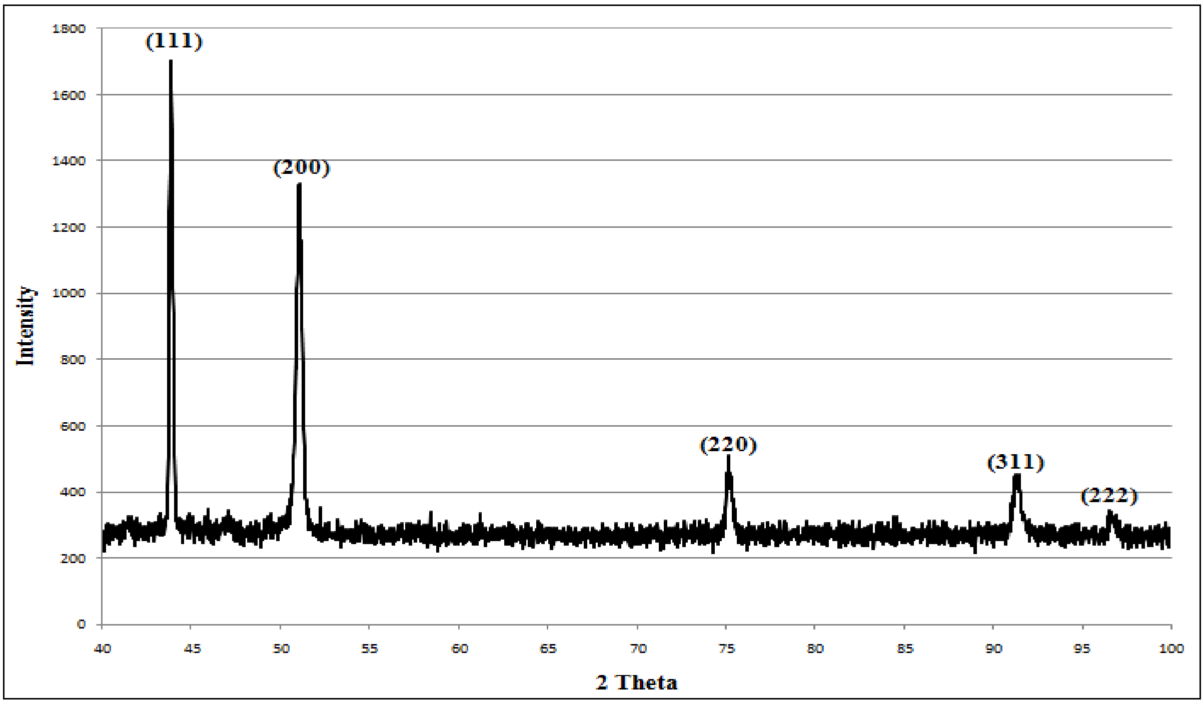
Table 6: Inter-group comparison of changes in UTS & HV using t-test

	UTS		HV	
Groups	**'t' value**	**'p' value**	**'t' value**	**'p' value**
Group C vs. Group 1	1.72	0.123 NS	5.67	0.000 HS
Group C vs. Group 2	7.28	0.000 HS	6.46	0.000 HS
Group C vs. Group 3	7.15	0.000 HS	7.61	0.000 HS
Group 1 vs. Group 2	3.85	0.004 HS	1.55	0.158 NS
Group 1 vs. Group 3	4.87	0.000 HS	2.41	0.042 Sig
Group 2 vs. Group 3	2.13	0.069 NS	0.48	0.643 NS

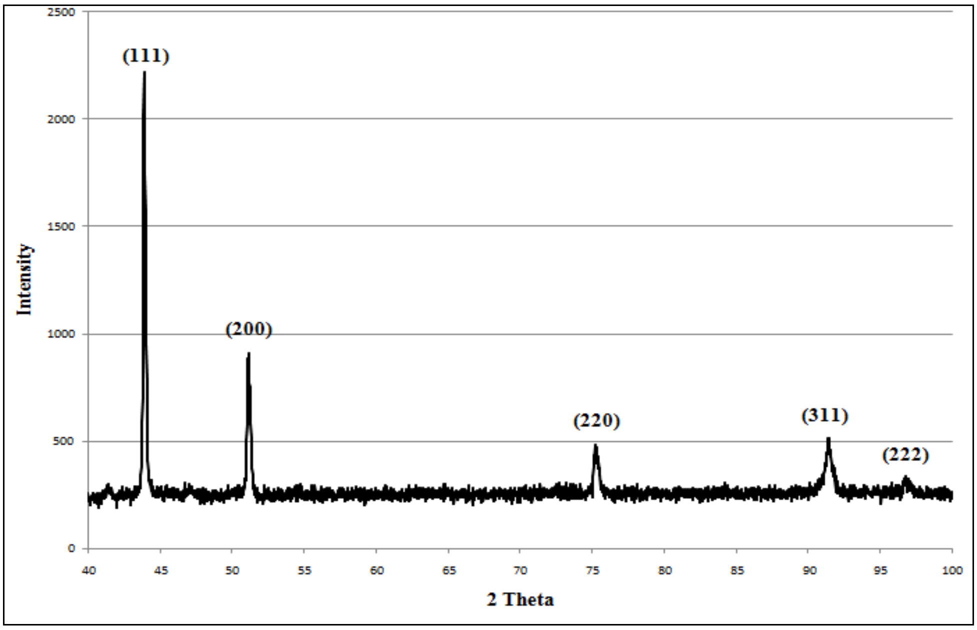
Student t-test, $p<0.05$; HS- Highly significant, Sig- Significant, NS- Non-significant



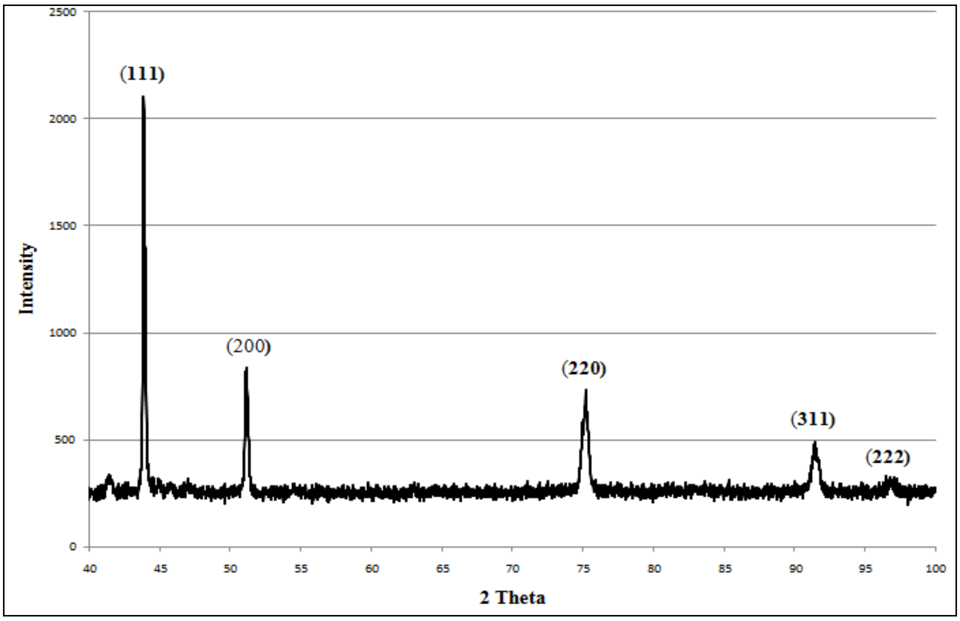
Graph 1: XRD pattern of Group C specimen



Graph 2: XRD pattern of Group 1 specimen



Graph 3: XRD pattern of Group 2 specimen



Graph 4: XRD pattern of Group 3 specimen

4. Discussion

The increased esthetic awareness in the present day population has increased the demand of esthetic rehabilitation [2]. Since early 1900s porcelain is being used in the process of fabrication of dental prostheses. They exhibit excellent abrasion resistance (hardness) as well as have better color and dimensional stability.

However, ceramics in general has poor tensile, impact and shear strength which often contraindicate its use in isolation. This problem was overcome by layering it on a substructure generally made up of cast metal alloy which has enhanced mechanical properties. Previously, more expensive metal alloys like gold etc. were used which were later replaced by less expensive base metal alloys. Ni-Cr alloys became most popular as they were stronger with lesser substructure thickness and more rigidity. However, Ni-Cr alloys have a major disadvantage of Ni being allergenic. Many cases of Ni allergies have been reported [11]. Thus, the use of Cr-Co alloys for PFMs became popular. Though, Co-Cr alloys are comparatively expensive, they possess superior mechanical and physical properties [12]. The clinical success of PFM restorations depend largely upon the mechanical properties of the alloy as well as that of the layering material [7]. It has been stated that, although considerable functional stress is borne by the ceramic portion of a metal-ceramic restoration, success of the entire prosthesis depends largely on the mechanical properties of the metal substructure [5].

During the process of ceramic layering, the alloy substructure has to pass through at least three heat treatments i.e. opaque firing, body firing and glaze firing [9]. Repeated PFM firing cycles have been shown to change the alloy microstructures, surface oxides, internal grain structure, corrosion, and mechanical and physical properties of the casting alloys [6].

The specimens (bar and disc) in this study were fabricated from a commercially available Co-Cr alloy, BIODUR© *SOFT* (Co 61.0%, Cr 24.0%, W 8.0%, Mo 2.50%, Nb 1.0%, Mn 1.0%, Si 1.0%, Fe 1.0% and C in trace).

As previously mentioned, mechanical properties are of paramount importance for satisfactory clinical performance of the prosthesis. To avoid permanent deformation, the tensile yield strength of the alloy should never exceed the recommended value.

4.1 Ultimate tensile strength

Ultimate tensile strength (UTS) is defined as strength of an alloy at its fracture. The connector of a multiunit fixed dental prosthesis is the most common site for prosthesis fracture. Craig and Powers stated that an alloy must possess at least 300 MPa yield strength to avoid any permanent deformation clinically in the oral environment [10]. The results depict that the UTS of alloy decreases significantly after every PFM firing cycle becoming weaker and more susceptible to permanent deformation from clinical factors after ceramic built up. According to ADA specification no. 5, the recommended value of UTS should be within a range of 539 MPa to 919 MPa [1]. Samples of all groups fall within this range proving that, the Co-Cr alloy remain sufficiently strong even after all 3 firing cycles.

4.2 Vickers Hardness

Hardness can be defined as the surface property of a material to resist scratches. Hardness plays an important role in cases where lingual, occlusal or the linguo-cervical margins are left in metal. The hardness of the casting alloy must be sufficient enough to resist the wear from occlusal loads. However, the alloy should be soft enough to avoid wearing of opposite tooth as well. According to Seghi and Denry, the hardness of human enamel is 3.24GPa (330.4 HV) [12]. Thus, alloys with Vickers hardness <125 HV are can easily wear and alloys with hardness >330 HV can easily wear opposing teeth[8]. Although within clinically acceptable range, the hardness of Co-Cr alloys increased after every firing cycle. This was in accordance to the studies conducted by Qiu *et al.* [4]. Surface analysis of the Co-Cr alloys before and after firing revealed that the surface content of Co increases after PFM firings (as per as XPS analysis) which justifies the increase of hardness. Also, the homogenization of alloy wasn't observed and thus the dendrite-like structure becomes more pronounced and less homogenized after firing.

4.3 X-Ray Diffraction

Metals and metal alloys are in form of crystals and the properties of such alloy are altered by the changes in arrangement of these crystals. X ray diffraction or X ray crystallography is one of the methods in use to study the arrangement of crystals as well as to study the phases of metal and metal alloys. When a beam of X ray is projected on a crystal of a material, two phenomena occurs i.e. absorption or diffraction. The diffracted beams of X ray are detected by a specialized detector. The diffraction of X rays occurs at a specific angle termed as 2ϴ angle. The intensity of the X rays increases at few 2ϴ values which are represented as peaks in the XRD patterns. Higher peak indicates higher electron density. The indexing of XRD patterns is done from the 2ϴ values of all the prominent peaks obtained from the patterns [56]. The XRD pattern of control group specimen showed similarity to solid Co solution with FCC arrangement of its crystal lattice. The peaks appeared at (111), (111), (200), (220) and (311) planes of 2ϴs. Only one significant peak existed which represented γ phase of alloy. However a significant shift in peaks was seen in remaining three groups. The additional plane (111) disappeared and the patterns showed peaks at (111), (200), (220), (311) and (222) planes of 2ϴs. The disappearance of peak does not make much difference as the only significant peak is the 1st peak i.e. the peak representing the γ phase [1].

5. Conclusion

Based on the results of this study, following conclusions were drawn: The firing cycles did affect the UTS and Vickers hardness of both alloys:

A. The UTS values revealed that Co-Cr alloys get weaker after repeated PFM firings. Hence, additional firings should be avoided to maintain the strength properties of the alloys.
B. The values of Vickers hardness also proved that Co-Cr alloys get harder after repeated PFM firing making it difficult to polish. However, these can retain their polish for longer duration clinically.

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