

## STUDY ON THE METAL-CERAMIC BONDING STRENGTH IMPROVEMENT BY UNCONVENTIONAL METHODS

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*This paper aims to develop practical research in order to enhance the bonding resistance between Ni-Cr / Co-Cr alloys and fused ceramic. The improvement of the ceramic-metal system bonding was made by the deposition of a thin indium layer at the interface between the metallic support and the ceramic. The bonding strength was determined by the three-point test bonding showing a high improvement for the indium deposited system case.*

**Keywords:** metal-ceramic bonding strength, Ni-Cr alloy, Co-Cr alloy, three-points bonding test

### 1. Introduction

Noble metal alloys have been widely used in dental ceramic systems. However, taking into account the fluctuations in the prices of noble metals, more attention was given to alternative alloys. The alloys from the Ni-Cr and Co-Cr systems have good mechanical properties, such as high hardness, low density, and high tensile strength [1]. Adding beryllium in these alloys improved their molding properties and the addition of indium conferred a higher resistance to the porcelain-alloy bonding [1-5]. Metal-ceramic compatibility is an important factor in ceramic dental restorations execution. The coefficient of linear thermal expansion (CTE), the thermal conductivity, and the nature and strength of the metal ceramic bond are factors that influence the cracking resistance ability during clinical use of restoration [6,7].

Numerous methods have been developed and selected by researchers to evaluate metal-ceramic bond. The minimum force value recommended by ISO9693 standard for metal-ceramic dental restorative systems is 25N for the

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three-point bending test. However, it can be said that this value is related more to the flexural strength of the metal substrate than metal-ceramic bond strength [8-10], causing difficulties when attempting a comparison between different metal substrates.

The three-point bending test has been suggested as a possibility of examination of the characteristics of the link, in particular the dependence of surface treatment of a metal for metal-ceramic system. This was considered as rupture strength, derived from the residual tension created at the metal-ceramic interface and the dependence of the alloys elastic modulus.

In this paper we propose to realize a practical research in order to improve the bonding strength between Ni-Cr / Co-Cr alloys and fused ceramics. Usually this is made by a surface oxidizing process that forms a thin oxide that enhances the bonding strength between the two components. In this study, the improvement of metal-ceramic system bonding was made by depositing a layer of indium by vacuum evaporation at the interface between the metal support and the ceramic. The bond strength was determined by three-point bending test.

## 2. Materials and Methods

Sheets of Ni-Cr and Co-Cr alloys were made according to ISO 9693 indications, with the size of  $(25 \pm 1)$  mm x  $(3 \pm 0.1)$  mm x  $(0.5 \pm 0.05)$  mm [11]. A set of test 40 samples, 20 of Ni-Cr alloy and 20 of Co-Cr alloy, were obtained by classical casting.

The chemical composition of the used alloys was determined by X-Ray analysis on a FEI Philips SEM microscope with EDAX spectrometer from University Politehnica of Bucharest is presented in Table 1.

Table 1  
Chemical composition of the alloys used in the experimental study

Alloy system	Ni	Cr	Co	Mo	Si	W	Fe	Ga
Ni-Cr	62.25	24.05	-	9.75	3.34	-	0.61	-
Co-Cr	-	26.01	61.88	4.52	1.75	5.83	0.50	2.90

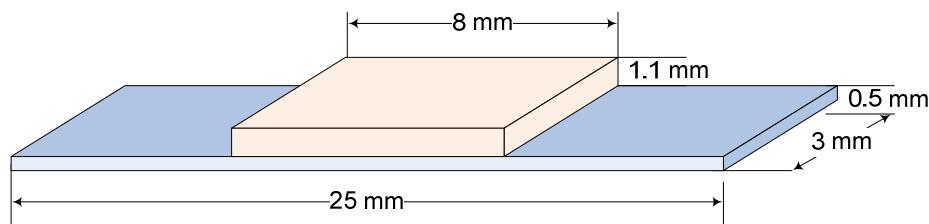


Fig. 1. Metal plates sizing for testing according to ISO 9693

The samples processing was made using a micromotor with 2,000rpm and a metal milling machine and then were grinded and polished using sandpaper (FEPA 80, 120, 320, 600, 800) to get rid of surface imperfections.

Before the ceramics deposition two surface treatments were made. The first one was blasting (for the increase of the link specific surface) using a sandblaster containing aluminum oxide (alumina) with 25-50 $\mu$ m grit, at a pressure of 80 bar. The other treatment was oxidation in a ceramics sintering furnace at a temperature of 960°C for 10 minutes, thus forming an oxide surface which improves the metal-ceramic bonding.

The oxide layer is distinguished by a matte surface of the casting, gray coloured. After the oxidation the ceramic burning follows.

The first step in applying a coating of ceramics is the applying of an opaque that reduces transparency when applying ceramic metal. In this case, the layer of opaque, the dentin layer (ceramic) and the layer of coating, were applied successively on a length of  $(8 \pm 0, 1)$  mm in the middle of each plate of the Co-Cr / Ni-Cr alloys. The deposited ceramic layer was rectangular, with dimensions  $(8 \pm 0, 1) \times 5 \times (1, 1 \pm 0, 1)$  mm.

The samples on which indium (In) was deposited by the evaporation method, had no oxidation treatment and the deposition method of the porcelain was the same as for the free indium Ni-Cr / Co-Cr alloy samples.

The coatings of indium were made by vacuum evaporation. The deposition rate was 1-8 nm / s and the final thickness of deposition was 1  $\mu$ m. The bond strength test was tested with a 8801 Instron Universal three-point testing device. Samples were loaded in the center, with a rounded piston with a radius of 1mm. Fig. 2 shows the real testing conditions image.

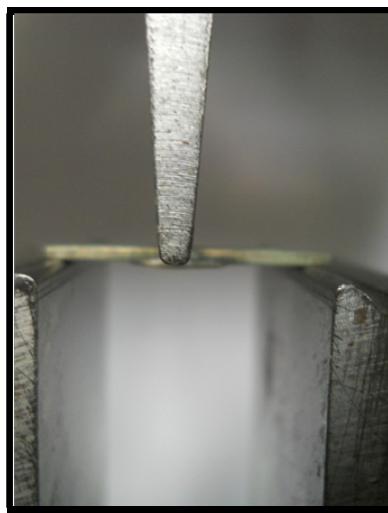


Fig. 2. Representation of the real three-point resistance test

The applied force had a maximum value of  $\pm 50$  kN and was applied at a constant rate of 1,5 mm/min and the values were registered until the apparition of a disturbance on the obtained loading curve, indicating link failure.

The rupture force  $F$  (Newtons) was measured for specimens that failed by a detachment of the crack that occurred at the end of the ceramic layer.

The breaking force  $F$  was multiplied by the coefficient  $k$ , that depends on the thickness of the metal substrate and the Young's modulus value of the used metallic materials. To read the value of  $k$  for a certain thickness a suitable curve of Young's modulus is selected first, then the value of  $k$  can be read from the thickness curve pick. Detachment force or initial cracking force  $\Gamma$  is calculated using the equation:

$$\Gamma = k \times F \quad (1)$$

Compared to other tests, such as shear tests, bending tests, simple torsion tests shoot tests, the flexural three point was selected being a widely method used, and properly described within German DIN 13927 and ISO 9693 standards [11].

### 3. Results

Metal-ceramic system Adherence, of Ni-Cr / indium deposition Ni-Cr, namely Co-Cr / indium deposited Co-Cr samples was determined by crack initiation mechanical test according to ISO 9693 standard.

The average values are shown in Table 2. There were tested 10 samples from each specimen type ( $n = 20$ ).

Table 1

Three-point bending test bonding strength values

	Fracture force (N)		Detachment / Crack initiation resistance (MPa)	
	Mean	S.D. (%)	Mean	S.D. (%)
NiCr	7, 3	0, 9	41,0	5, 9
NiCr (In)	9, 0	2, 8	41, 56	12, 6
CoCr	6, 86	0, 75	40, 64	5, 42
CoCr (In)	7, 94	1, 87	41,18	6, 45

Fig. 3 presents a graphic representation of the three points resistance test made on samples of Ni-Cr (Ni-Cr), and Indium deposited NiCr alloy (Ni-Cr1) for the determination of the bonding strength of the metal-ceramic system.

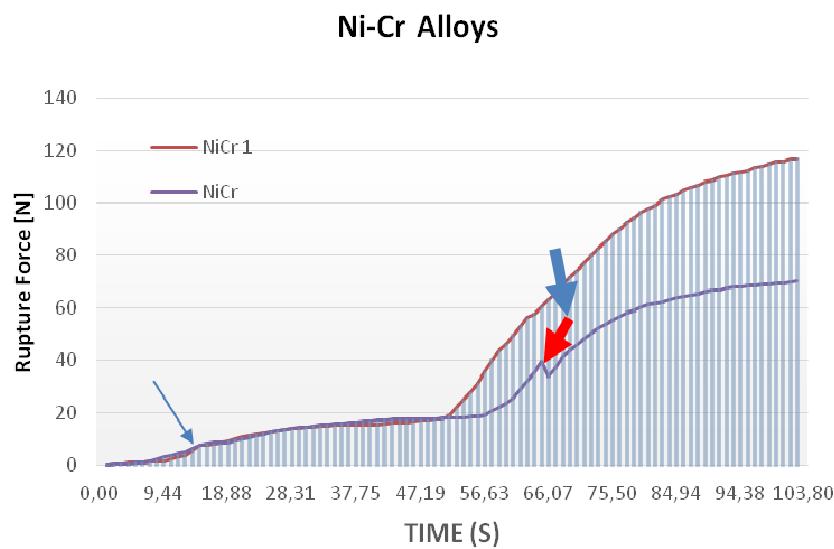


Fig. 3. Graphical representation of the bending test results on NiCr alloy (NiCr) and Indium deposited NiCr alloy (NiCr1) samples

Rendering shows that the strength was bigger for the system with indium deposition, crack initiation occurred at a force value of 63.97 N and a time of 56.10 s. The visibility of the fracturing peak on the graph is very low, whereas Ni-Cr alloy with no indium deposition showed a weaker link, the crack initiation occurred at a 35.74 N force and a time of 65.02 s. The peak visibility peak of the crack initiation is more pronounced than in the previous case.

Fig. 4 shows the force of fracturing variation in time for the Co-Cr alloys system, the peak that appears when crack was initiated being highlighted. It is noted that the metal-ceramic adhesion is improved by indium deposition. For the Co-Cr alloys without indium deposition the crack occurrence can be observed at 53.05 N force value and a time of 72.61 s.

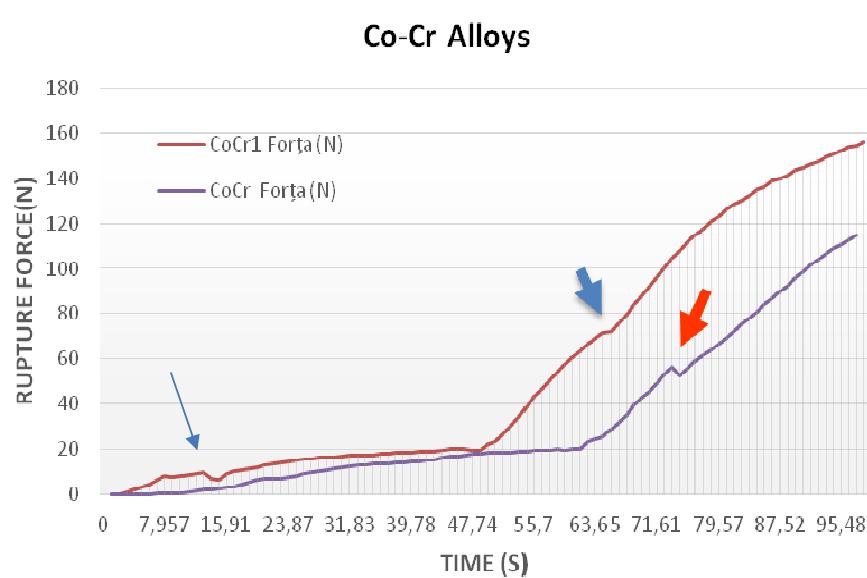


Fig. 4. Comparative graphical representation of the bending test results, made on CoCr alloy samples (CoCr) and Indium deposited CoCr alloy (CoCr1)

Crack initiation for the Co-Cr alloys with indium deposition occurred in at 68.74 N and a time of 63.65 s. In conclusion metal-ceramic bond system was optimized when indium was deposited on the surface of Co-Cr alloys and respectively Ni-Cr alloys.

## 5. Discussions

If the mechanical stresses are strong enough the porcelain will crack or separate from the metallic component. Even if the internal stresses are small and do not cause an immediate inability, they can weaken the bond strength. An essential role in adhesion plays the metal-ceramic interface topography or roughness [12].

Usually the metal-ceramic system compatibility depends on the harmony of the properties of the two materials.

The fracture localization knowing provides valuable information. In samples with the strongest metal-ceramic bonding the porcelain will fracture when tested. This is observed when the alloys are adequately prepared and fused porcelain properly applied.

Ceramic deposition parameters on test samples were made under standardized procedures, the porcelain application being identical in both cases (for alloys with indium deposition and no deposition). The surface treatment

before the ceramic firing is very important in terms of enhancing the bonding between the two components.

## 6. Conclusions

The three-point bending test results showed that the deposition of indium by evaporation on Ni-Cr and Co-Cr alloys was benefic in both cases, as confirmed by the visible improvement of metal-ceramic system bonding strength. Experimental data on the bonding strength resistance of the alloys were better for the indium deposited ones than for those without deposition showing that the metal-ceramic bond system was optimized when indium layers were made on the surface of Co-Cr alloys and Ni-Cr respectively.

## Acknowledgement

The work has been funded by the Sectoral Operational Programme Human Resources Development 2007-2013 of the Ministry of European Funds through the Financial Agreement POSDRU/159/1.5/S/ID134398

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