A Novel CAD/CAM Base Metal Compared to Conventional CoCrMo Alloys: An *in-vitro* Study of the Long-term Metal-ceramic Bond Strength

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Abstract

Objectives: This study examined the metal-ceramic bond strength between different ceramics and a novel CAD/CAM base metal alloy as well as conventional CoCrMo alloys.

Methods: Three different base metal alloys were tested: a novel CAD/CAM milled and sintered alloy (MS), a. laser sintered alloy (LS), and a cast alloy (C). Specimens for the Schwickerath crack initiation test according to ISO 9693:2012 were prepared and veneered using the following veneering ceramics: i. Creation (Willy Geller), ii. VITA VM13 (VITA Zahnfabrik), and iii. Reflex (Wieland + Dental). The specimens were subsequently thermally aged (5,000 cycles, 5°C/55°C, dwell time: 20 s) and the bond strength was measured in a universal testing machine (Zwick 1445, 1 mm/min). Data were analyzed using two-way and one-way ANOVA, followed by a post hoc Scheffé test.

Results: For the veneering ceramics VITA VM13 and Reflex, no impact of the used alloy on bond strength results was found ($p=0.124 - 0.393$). In contrast, the veneering ceramic Creation showed significantly higher bond strength values in combination with MS and LS than with C ($p=0.001$). For MS ($p<0.001$) and LS ($p=0.001$) alloys veneered with Reflex, significantly lower bond strengths were observed than for specimens veneered with Creation or VITA VM13. Within C alloy ($p=0.012$), significantly lower bond strengths were measured for specimens veneered with Reflex than for VITA VM13.

Conclusion: The novel CAD/CAM base metal alloy Ceramill Sintron showed comparable metal-ceramic bond strength compared to conventional CoCrMo alloys.

Key words: CAD/CAM, Base metal alloy, Crack initiation test, Metal-ceramic bond strength, Schwickerath test

Introduction

Introduced in 1956, metal-ceramic restorations are still the golden standard in fixed prosthodontics [1,2]. In general these restorations combine good mechanical and optical properties after veneering with dental ceramics [3–5]. Base metal alloys are used extensively in dentistry due to their favorable chemical and physical properties, as well as their high cost-efficiency [6]. In the past, chrome-cobalt-molybdenum alloys (CoCrMo) were fabricated in Computer-aided-Design (CAD)/Computer-aided-Manufacturing (CAM) manufacturing centers. Two different approaches to CAD/CAM processing have been reported for these alloys, namely additive, using laser sintering, or subtractive, on massive, costly milling machines from the end-strength material. Only a few CAD/CAM systems for the dental laboratory were designed for the processing of materials milled in final densely-sintered stage. These CAD/CAM systems are associated with high acquisition and maintenance costs. The development of a novel CoCrMo material (Ceramill Sintron, AmannGirrbach, Koblach, Austria) allows this alloy to be processed on in-house on desktop milling machines with reduced manufacturing time and costs. The processing steps are quite comparable to those of pre-sintered zirconia. The soft CoCrMo blank is processed in a material pre-state by dry milling. The material contains adhesive agents such as organic binders and is milled in a “green state”. Subsequently, the milled reconstruction must be sintered to full density in a special, high-temperature sintering furnace under an argon protective gas atmosphere at 1300 °C. During the sintering process, the organic binder burns out and the metallic powder particles are sintered (caked) without creating a fused phase. This leads to a decrease in volume of approximately 10%. After sintering, this novel CoCrMo alloy exhibits comparable mechanical properties to the conventionally produced CoCrMo reconstructions (*Table 1*). From present knowledge, the novel sintered CoCrMo base metal alloy is suitable for long-span Fixed Dental Prostheses (FDPs) of up to four units.

The manufacturer recommends conventional veneering of the novel CoCrMo alloy analog to previously used veneering techniques for CoCrMo alloys after milling and sintering processes. As the coefficient of thermal expansion (CTE) of the new alloy is $14.5*10^{-6}$/K, all veneering ceramics designed for this CTE can be used. The longevity of the metal-ceramic system depends on the formation of a stable bond between the alloy and the veneering ceramic that can withstand stresses common in the oral cavity [7]. The combination of framework and veneering ceramic is of paramount importance for the overall mechanical strength of the restoration. The metal-ceramic bond is based on a combination of chemical, mechanical, and van der Waals forces, and a slight CTE mismatch contracting force to fuse the veneering ceramic to the alloy [8,9]. Chemical bond occurs by an oxide layer formed on the alloy during firing, developing metallic, ionic, and covalent bonds with the oxides in the veneering ceramic opaque layer [10-13]. The mechanical bond is primarily determined by the roughness of the alloy surface and the van der Waals forces [7].

The aim of this study was to investigate the metal-ceramic bond strength of three different base metal alloys combined

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Table 1. *Summary of mechanical properties and composition of tested base metal alloys.*

	Girobond NB	**Ceramill NP L**	**Ceramill Sintron**
Tensile strength (Rm)	850 MPa	800 MPa	900 MPa
0.2% yield strength (Rp0.2)	620 MPa	600 MPa	450 MPa
Modulus of elasticity (E)	210 GPa	170 GPa	180 GPa
Elongation at break	10%	10%	25%
Vicker´s hardness (HV 10)	320	320	270
Coefficient of thermal expansion (25-500°C)	14.6 * 10^{-6}/K	14.0-14.5 * 10^{-6}/K	14.5 * 10^{-6}/K
Specific weight	8.5 g/cm³	8.5 g/cm³	7.9 g/cm³
Composition	62% Co, 25% Cr, 5% Mo, 5% W, 1% Si, <0.1% Ce	62–66% Co, 24–26 % Cr, 5–6% Mo, 5–6% W, <1% Si, <0.1% Mn, <0.5% Fe	66% Co, 28% Cr, 5% Mo, <1% Mn, <1% Si, <0.5% Fe

with different veneering ceramics. The authors used the three most common veneering ceramics for base metal alloys at their institution. The null-hypothesis tested was whether the metal-ceramic bond of the novel CAD/CAM base metal alloy is similar to the bond strength of cast and laser-sintered CoCrMo alloys.

Material and Methods

The following CoCrMo-alloys were used for the metal-ceramic bond strength investigation: i. Ceramill Sintron (MS: solid state sintered, AmannGirrbach, Koblach, Austria), ii. Ceramill NP L (LS: laser-sintered, AmannGirrbach), and iii. Girobond NB (C: cast, AmannGirrbach,) and three different conventional veneering ceramics: a. Creation CC (Creation Willi Geller International, Meiningen, Austria), b. VITA VM13 (VITA Zahnfabrik, Bad Säckingen, Germany), and c. Reflex (Wieland+Dental, Pforzheim, Germany) (*Table 2*). Specimens were tested according to the Schwickerath crack initiation test for metal-ceramic dental reconstructions described in ISO 9693-1:2012 [14].

Specimens preparation

In summary, 135 rectangular alloy strips with final dimension of 25×3×0.5 mm were fabricated, 45 of each CoCrMo alloy.

For the preparation of the Ceramill Sintron specimens, blocks with dimensions of 27.5×3.3×12 mm were designed (Pro-engineer, Creo 2.0, Parametric Technology Corporation, Needham, MA, USA) and milled in the green state (Ceramill Motion 1, AmannGirrbach). The milled specimens were sectioned into strips with a thickness of 0.65 mm using a diamond circular saw under dry conditions (Diacut Vario, AmannGirrbach). Subsequently, all specimens were sintered under an inert gas (Argon 4.6) in a special furnace (Ceramill Argotherm, AmannGirrbach) according to the manufacturer's instructions.

For the laser-sintered Ceramill NP L specimens, small supports by means of software 45 specimens with dimensions of 25.5×3.05×0.6 mm were added. The alloy strips were laser-sintered layer by layer to the final dimensions (EOSINT M270, EOS, Krailing, Germany), processing loose CoCr powder (CoCr Sp2 powder, EOS) in rapid prototyping. The supports were removed from the strips using a wire cutter.

For the cast specimens, forty-five specimens of the residue-free burn Ceramill PMMA (AmannGirrbach) 30×3×0.55 mm were milled (Ceramill Motion 1, AmannGirrbach), embedded into a phosphate-bonded speed investment (Giroinvest super, AmannGirrbach) and preheated, following the recommendations of the manufacturer. The molten non-precious alloy (Girobond NB, AmannGirrbach) was cast into the investment mold using an induction-casting machine (Heracast IQ, Heraeus Kulzer, Hanau, Germany). All molds were bench cooled after casting. Finally the alloy strips were divested, cleaned by means of air-abrasion with 110 µm alumina powder (Sandmaster, Hasenfratz, Aßling, Germany), and cut with a disc to the final length of 25 mm.

A visual inspection was performed on all alloy strips, and specimens with surface defects were replaced. Before the veneering process, all specimens were standardized air-abraded (110 µm alumina powder, duration: 20 s, angle: 45°, pressure: 0.3 MPa, distance: 10 mm; Sandmaster) and subsequently cleaned for 5 min using an ultrasonic bath filled with 80% ethanol. Thereafter, the alloy strips were randomly divided into 3 groups for the veneering ceramic. The specimens were veneered according to the manufacturers' instructions (*Table 3*; VITA Vacumat 4000 Premium, VITA Zahnfabrik, Bad Säckingen, Germany). An opaque wash was applied, followed by an opaque layer. The specimens were then placed in a special veneering device for the Schwickerath tests, condensing the dentine body ceramic to ensure a uniform shape and thickness. Prior to glazing, overhanging ceramic was removed with a disc (918 PB.104.220, Komet Brasseler, Lemgo, Germany) to obtain the correct final dimensions of the veneering ceramic.

Thermal cycling (DS Mechatronic, Holzkirchen, Germany) was performed with bath temperatures of 5°C and 55°C, respectively. The dwell time in each bath was 20 s, the transfer time between baths was 5 s, resulting in a frequency of 1 cycle/min. The specimens were aged for 5,000 thermal cycles.

Crack initiation test

The crack initiation test for metal-ceramic was performed in a Universal Testing Machine (Zwick 1445, Zwick, Ulm, Germany). The specimens were placed in the special device with the ceramic positioned symmetrically on the side opposite to the applied load (*Figure 1*). The specimens were loaded with a crosshead speed of 1 mm/min up to failure. Only those specimens which failed by debonding cracks occurring at one end of the metal-ceramic layer (crack initiation on one edge of the veneering ceramic at the metal-ceramic interface) were used for the calculation of bond strength.

The metal-ceramic bond strength (σ) for each specimen

Table 2. *Brands, batch numbers and manufacturers of tested framework and veneering materials.*

	Ceramill Sintron	Ceramill NP L	Girobond NB
Framework material	Ceramill Sintron LOT: 12D0151 (AmannGirrbach, Koblach, Austria)	EOS CoCr SP2 powder LOT: H401101 (EOS, Krailing, Germany)	Ceramill PMMA (AmannGirrbach, Koblach, Austria) Girobond NB LOT: 11-607 (AmannGirrbach, Koblach, Austria)
Devices and equipment	Ceramill Motion 1 Ceramill Argotherm Ceramill Argovent (AmannGirrbach, Koblach, Austria)	EOSINT M270 (EOS, Krailing, Germany)	Ceramill Motion 1 Heracast IQ (Heraeus Kulzer, Hanau, Germany)
	Diacut Vario (AmannGirrbach, Koblach, Austria)		Giroinvest Super Powder LOT: 2-52146-59 Liqiud LOT: 2-00071-60 (AmannGirrbach, Koblach, Austria)
	Air-abrasion device: Basis quattro (Renfert, Hilzingen, Germany) FINOX alumina powder with mean size of 110 µm hard metal burrs		
Veneering ceramic			
Creation CC Crea alloy bond LOT: A001-9 Opaque LOT: 1491/A208-1 Dentine LOT: 1440/A541-1 Glaze CC LOT: 9884 (Creation Willi Geller Int. Meiningen, Austria)	VITA VM13 Wash opaque LOT: 34510 Opaque LOT: 36870/28380 Dentine LOT: 34940/35260 Glaze VITA Akzent LOT: 16520 (VITA Zahnfabrik, Bad Säckingen, Germany)	Reflex Wash opaque NP safe LOT: 5/11 Opaque LOT: 41/11 / 9/10 Dentine LOT: 62/11 / 2/11 Glaze LOT: 6/10 (Wieland + Dental, Pforzheim, Germany)	

Table 3. *Firing schedule for used veneering ceramics.*

	Pre-drying		Heating rate (°C/min)	Firing temperature (°C)	Holding time (min)	Vacuum
	Temperature (°C)	Time (min)				
Creation CC (Creation Willi Geller Int., Meiningen, Austria)						
Wash opaque	550	6	80	980	1	+
Opaque	550	6	80	980	1	+
1st dentine	580	6	55	920	1	+
2nd dentin	580	4	55	910	1	+
Glaze CC	600	2	55	900	1	-
VITA VM13 (VITA Zahnfabrik, Bad Säckingen, Germany)						
Wash opaque	500	2	75	940	2	+
Opaque	500	2	75	920	1	+
1st dentine	500	6	55	880	1	+
2nd dentin	500	6	55	870	1	+
Glaze	500	4	80	880	1	-
Reflex (Wieland+Dental, Pforzheim, Germany)						
Wash opaque	450	6	75	975	1	+
Opaque	575	5	75	930	3	+
1st dentine	575	7	75	900	2	+
2nd dentin	575	5	75	890	1	+
Glaze	575	4	75	880	1	-

was calculated according to the following equation, using the appropriate algorithm for the calculation of k:

$\sigma = k \times F_{fail}$, $k = f(d_m, E_m)$; (mm^{-2})

d_m: thickness of alloy strip (mm), E_m: modulus of elasticity of the alloy (GPa), F_{fail}: load at debonding (N).

Failure type analysis

For the fracture type analysis, the veneering ceramic of the alloy strips was carefully removed. Then, the interface was analyzed under a light microscope (Axioskop 2 MAT, Carl Zeiss, Oberkochen, Germany) at a magnification of 25 x. Two different types were classified: adhesive [no ceramic (opaque/dentin) remnants on the alloy strip] or cohesive [alloy surface covered with opaque/dentin ceramic].

Grain size and surface topography analysis

Additionally, a specimen of each alloy (10 mm×10 mm×10 mm) was fabricated. The specimens (N=3, n=1 per alloy) were polished up to 1 µm with a diamond suspension (Struers, Ballerup, Denmark) and ultrasonically cleaned in isopropanol. To visualize the microstructure of the alloys, the specimens were etched from 5 to 60 s using a freshly-prepared etching solution [6.5 g iron (III) chloride, 20 ml hydrochloric acid (37%), 0.5 ml nitric acid (65%)]. The etched specimens were analyzed using a light microscope (Axiograph, Zeiss, Germany). The surface topography was evaluated under a scanning electron microscope (SEM, Carl Zeiss Supra 50 VP FESEM, Carl Zeiss, Oberkochen, Germany) operating at 10 kV with a working distance of 7.2-9.1 mm.

Statistical analysis

Descriptive statistics, such as the mean, Standard Deviation (SD) and 95% confidence intervals (95%CI), were calculated. Normality of the data distribution was tested using Kolmogorov-Smirnov and Shapiro-Wilk tests. The tests showed that all groups were normally distributed. Therefore, two- and one-way ANOVA followed by the Scheffé post-hoc test were used to determine the differences between the groups at a level of significance of 5 %. The data were analyzed using SPSS Version 20 (SPSS INC, Chicago, IL, USA).

Results

Crack initiation test results

Descriptive statistics (mean, SD, 95% CI) of the measured metal-ceramic bond strength for each base metal alloy material in combination with the veneering ceramics are presented in *Table 4*. *Figure 2* shows the bar diagram of the bond strength results for each base alloy and each veneering ceramic separately.

The 2-way ANOVA showed an impact of the base metal alloy ($p=0.001$) and veneering ceramic ($p<0.001$) on the bond strength. Interaction between both parameters was not found ($p=0.411$). In general, the base metal alloys showed no impact on the bond strength if VITA VM13 ($p=0.124$) and Reflex ($p=0.393$) were used as the veneering ceramic. Alternatively, the veneering ceramic Creation showed significantly higher bond strength results when combined with Ceramill Sintron and Ceramill NP L than with Girobond NB ($p=0.001$).

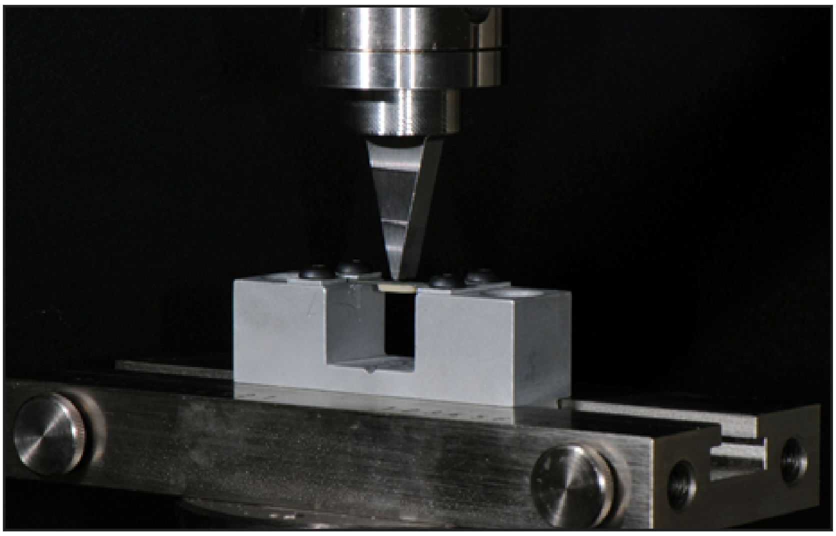

Figure 1. *Design of Schwickerath crack initiation test for metal-ceramic bond.*

Table 4. *Mean (SD) bond strength with confidence intervals (95% CI) and p-value significance for impact of base metal alloy.*

	Ceramill Sintron		**Ceramill NP L**		**Girobond NB**	
	Mean (SD)	95% CI	Mean (SD)	95% CI	Mean (SD)	95% CI
Creation	32.9 (4.4)^{b}	30.4; 35.4	30.8 (3.7)^{b}	28.5; 32.9	26.7 (4.7)^{ab}	24.0; 29.3
VITA VM13	29.7 (5.2)^{b}	26.5; 32.6	31.0 (4.7)^{b}	28.2: 33.6	27.5 (3.9)^{b}	25.1; 29.7
Reflex	24.8 (5.7)^{a}	21.5; 28.0	24.9 (5.1)^{a}	22.0; 27.8	22.5 (5.2)^{a}	19.5; 25.4

^{ab}different letters show significant differences between the veneering ceramics between one alloy type

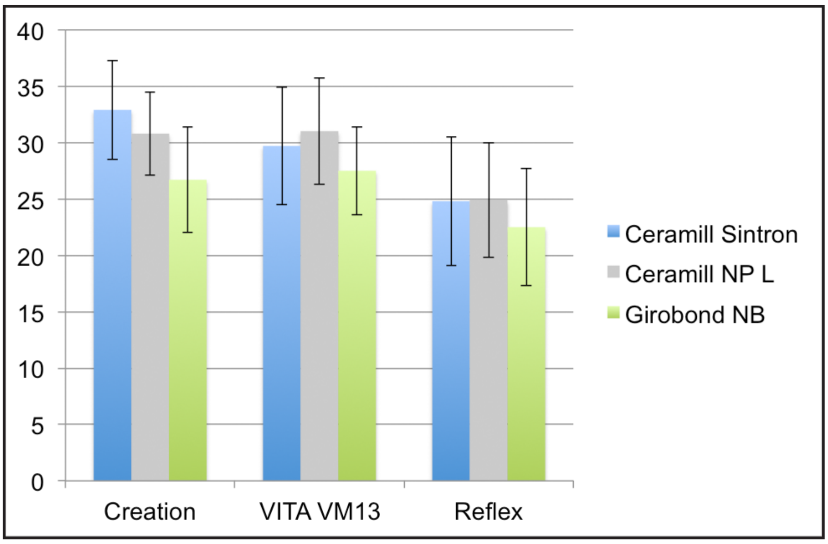

Figure 2. *Bar diagram for means of all tested metal-ceramic bond strength results (MPa), the error bar represents the standard deviation.*

For Ceramill Sintron (p<0.001) and Ceramill NP L (p=0.001) alloys veneered with Reflex, significantly lower bond strengths were observed than for those veneered with Creation or VITA VM13. Within the Girobond NB alloy (p=0.012), significantly lower bond strength values were measured for specimens veneered with Reflex than for VITA VM13. The bond strength results for veneering ceramic Creation was in the same statistical range as VITA VM 13 and Reflex.

Failure types after crack initiation test

The representative failure types are depicted in *Figure 3a–d*. Predominantly the cohesive failure type (interface opaque - dentin ceramic) was observed for alloys veneered using VITA VM13 and Creation. Specimens veneered with Reflex only showed adhesive failures in the interface between the alloy and opaque ceramic for all three alloys.

Grain structure of tested alloys

The results of the microstructural analysis are given in *Figure 4a–c*. Evaluated at the same magnification, Ceramill Sintron showed significantly smaller grains compared to the etched metallographic cross-section of Girobond NB. For Ceramill NP L (*Figure 4c*) a typical flaky microstructure due to the layer-wise selective melting of the loose CoCr powder was observed.

In addition, Ceramill Sintron showed a homogeneous structure. This structure is caused in the solid state sintering process and shown in the uniform color of the metallographic surface of the cross-section. In comparison, the cast alloy Girobond NB has the dendritic structure that is specific for this group of alloys. Furthermore the microstructure of Ceramill Sintron differs from the other two alloys in its homogeneous and closed micro porosity, characteristic of the free sintering processes.

Surface topography of tested alloys

Three different surface topographies for the tested alloys were observed. The sintered alloy specimen was accompanied by uniformly distributed hollow opening in the microstructure (*Figure 5a*). The laser sintered alloy showed a very homogenous microstructure (*Figure 5b*). In contrast, the cast alloy showed typical dendritic growth in the microstructure (*Figure 5c*).

Discussion

The novel sintered CoCrMo alloy showed a similar metal-ceramic bond compared to the conventional alloys tested. Yet in combination with the veneering ceramic Creation, higher

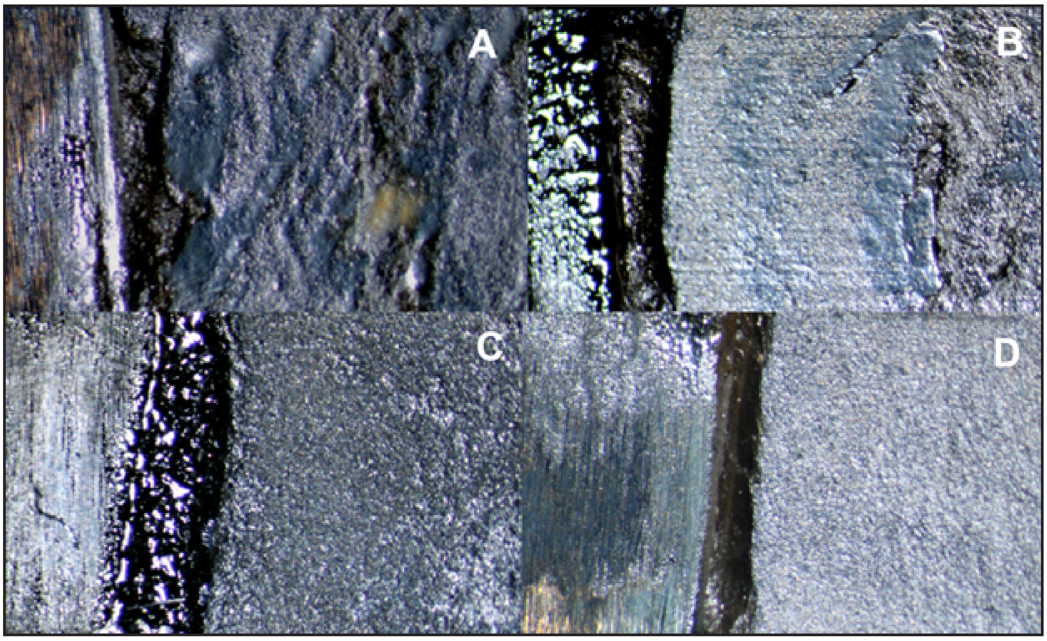

Figure 3. *Fracture type after crack initiation test. a. Ceramill Sintron veneered with Creation, b. Girobond NB veneered with VITA VM13, c. Ceramill NP L veneered with VITA VM13 (a-c: cohesive failure; alloy surface covered with opaque/dentin ceramic), d. Ceramill Sintron veneered with Reflex (adhesive failure; no ceramic (opaque/dentin) remnants on the alloy strip).*

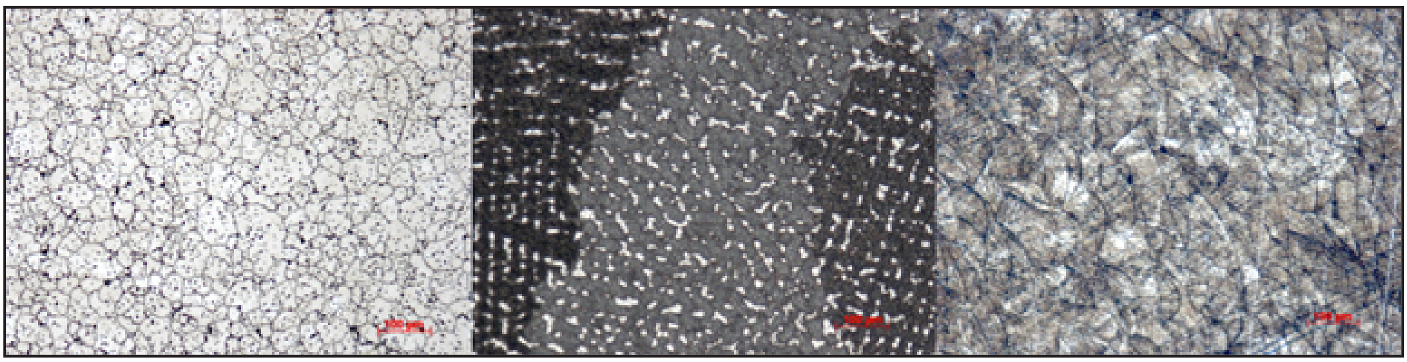

Figure 4. *Etched metallographic cross-sections of all three CoCrMo alloys: a. Ceramill Sintron, b. Ceramill NPL, c. Girobond NB (magnification: 100x).*

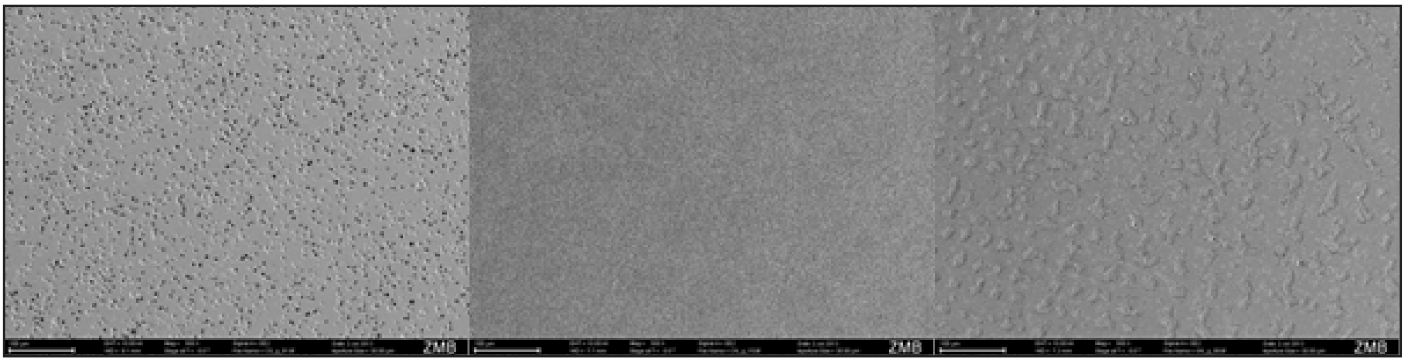

Figure 5. *SEM topography images of tested CoCrMo alloys: a. Ceramill Sintron, b. Ceramill NPL, c. Girobond NB (magnification: 500x).*

bond strength results were obtained for the novel CAD/CAM alloy than for the conventionally cast alloy. Therefore the tested null-hypothesis of this study is accepted.

The CTE values of the three tested CoCrMo alloys are comparable and range from 14-14.6× 10^{-6}/K. It has to be noted that not only the thermal expansion properties, but also the mechanical and the chemical compounds, affect the bond strength. However, all alloys contain appr. 62-66% Co, 24-28% Cr and 5-6% Mo. With respect to the mechanical properties according to the manufacturer (*Table 1*), Ceramill NP L (800 MPa) presented a lower tensile strength (Rm) than Girobond NB (850 MPa) and Ceramill Sintron (900 MPa). In contrast within the 0.2 % yield strength (Rp0.2), the highest values are stated for Girobond NB (620 MPa), Ceramill NP L (600 MPa) and Ceramill Sintron (450 MPa), respectively. Therefore the alloys tested in this study can be assumed to be quite similar in terms of their mechanical properties having no impact on the bond strength results. The differences in modulus of elasticity between the alloys (Girobond NB: 210 GPa, Ceramill NP L: 170 GPa, Ceramill Sintron: 180 GPa) are respected in the formula for the crack initiation bond strength calculation.

Additionally, the formation of a strong bond between the opaque layer and the alloy is essential for the longevity of metal ceramic FDPs. The metal-ceramic bond is a result of chemisorption by diffusion between the surface oxides on the alloy and in the ceramic. These oxides are formed during wetting of the alloy with the veneering ceramic and the firing of the veneering ceramic. Several studies showed that the main factor in decreasing the metal ceramic bond strength in base metal alloys is an increased thickness of the oxide layer [15-19]. In this study, the highest oxide surface layers were observed for the cast alloy Girobond NB and the lowest for Ceramill Sintron. Hence the phenomenon that Girobond NB specimens showed tendencies in lower values that Ceramill Sintron or Ceramill NP L can be explained with the building of the oxide surface layer during the veneering process. In using the Creation veneering ceramic a significant influence of the different alloys on the bond strength was observed.

In general, specimens veneered with Reflex showed lower bond strengths compared to specimens veneered using Creation or Vita VM13, regardless of the alloy used. This statement was supported with the failure type analysis. Specimens veneered with Reflex showed adhesive failure at the interface between the alloy strip and opaque layer. In contrast, predominantly cohesive failures (interface opaque - dentin ceramic) were observed for alloys veneered using VITA VM13 or Creation. One possible explanation for this observation might be the inadequate bond between the alloy and the opaque layer of the Reflex veneering ceramic.

In this study, thermo cycling with 5,000 cycles was chosen to simulate a clinical situation. The stress for all specimens in the thermal cycling machine was standardized and reproducible. Other studies reported that the thermal stress applied in an aqueous solution negatively affects the metal-ceramic bond [20,21]. Therefore it can be assumed that the specimens without any further aging procedure might show higher bond strength results.

The bond strength between two materials can be tested using different test methods, such as shear bond strength, flexural, bending or torsion tests [22-27]. In this study, the metal-ceramic bond strength was determined using the crack initiation test according to Schwickerath. This test method is quite sensitive and was introduced into ISO 9693 [14,20,28]. By means of a finite element analysis the critical bending force value at which debonding occurred was transformed into an actual stress value at the point of debonding [20,28]. The critical bending force, where the specimen fails by debonding of the ceramic at one edge of the ceramic layer (F_{fail}), is measured and the bond strength is calculated by an algorithm, using the modulus of elasticity of the alloy and the thickness of the alloy specimen as variables [28].

Based on the findings of this study, CAD/CAM alloy Ceramill Sintron is comparable to conventional alloys, with respect to the metal-ceramic bond. The pre-sintered CAD/CAM-blanks allow manufacturing in small laboratory CAD/CAM-systems as the material is easily machinable. This might be an attractive alternative to dental laboratories with CAD/CAM-technology to mill base metal alloys instead of casting. However, prior to clinical recommendations the accuracy of this concept has to be evaluated. Furthermore, clinical studies are needed to support the use of this material in definitive restorations.

Conclusion

Within the limitations of this study it can be concluded that:

- The alloy and veneering ceramic used had a significant influence on the bond strength of base metal alloy to the veneering ceramic
- The metal-ceramic bond strength of CAD/CAM alloy Ceramill Sintron is comparable to those of cast alloy Girobond NB and laser-sintered Ceramill NP L.

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Conflict of Interest
No conflicts of interest declared. Rita Hoffmann and Falko Noack are employees of AmannGirrbach..

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