

Comparison of the mechanical properties and microstructures of fractured surface for Co-Cr alloy fabricated by conventional cast, 3-D printing laser-sintered and CAD/CAM milled techniques

Yun-Jung Choi¹, Jai-Young Koak^{1*}, Seong-Joo Heo¹, Seong-Kyun Kim¹, Jin-Soo Ahn², Dong-Soo Park³

¹Department of Prosthodontics, ²Department of Dental Biomaterials Science, School of Dentistry, Seoul National University, Seoul, Republic of Korea

³US Army Carius Dental Clinic, Dentac-Korea, 618th Dental Command, Seoul, Republic of Korea

Purpose: The purpose of present study is to compare mechanical properties and microstructural characteristics of fractured surface for cast, 3-D printing laser sintered and CAD/CAM milled cobalt-chromium (Co-Cr) alloy specimens and to investigate whether laser sintered technique is adequate for dental applications. **Materials and methods:** Thirty six flat disc shape Co-Cr alloy specimens were fabricated for surface hardness test and divided into three groups according to the manufacturing methods; 12 specimens for casting (n=12), 12 specimens for laser sintered technology (n=12) and 12 specimens for milled technology (n=12). Twelve dumbbell shape specimens for each group were also fabricated for a tensile test. Statistical comparisons of the mechanical properties for the alloys were performed by Kruskal-Wallis test followed by Mann-Whitney and Bonferroni test. The microstructural characteristics of fractured surfaces were examined using SEM. **Results:** There were significant differences in the mean Vickers hardness values between all groups and the cast specimen showed the highest (455.88 Hv) while the CAD/CAM milled specimen showed the lowest (243.40 Hv). Significant differences were found among the three groups for ultimate tensile strength, 0.2% yield stress, elongation, and elastic modulus. The highest ultimate tensile strength value (1442.94 MPa) was shown in the milled group and the highest 0.2% yield strength (1136.15 MPa) was shown in the laser sintered group. **Conclusion:** Different manufacturing methods influence the mechanical properties and microstructure of the fractured surfaces in Co-Cr alloys. The cast Co-Cr alloy specimens showed the highest Vickers hardness, and the CAD/CAM milled specimens revealed the highest tensile strength value. All alloys represent adequate mechanical properties satisfying the ISO standards of dental alloy. (*J Korean Acad Prosthodont* 2014;52:67-73)

Key words: Co-Cr alloys; CAD/CAM dental; Dental laser sintering; Mechanical property; Microstructure

Introduction

Cobalt-Chromium (Co-Cr) alloys have been widely used in dentistry for removable partial dentures, metal frames, and porcelain-fused-to-metal crowns.¹ Because the alloys are strong, resistant to corrosion and relatively inexpensive compared to gold alloys and some all-ceramic materials.¹⁻⁵ However, the fabrication processes for the alloys, such as casting, cutting and plastic works, are usually difficult because of their high melting point (1349-1449 °C), hardness, and limited ductility.⁶ Casting has been the most common method to fabricate dental alloy for many decades,⁷ but errors accumulat-

ed in the series of laboratory steps are inevitable. Simplification of procedure can reduce these errors along with costs that are related to expensive devices.

CAD/CAM technique was also introduced in dentistry more than 20 years ago.⁷ One major advantage of using milling technology is that some disadvantages of casting, such as several clinical appointments needed including impression taking procedure, casting-induced flaws and porosities which can degrade the quality of the reconstructions, can be avoided. Therefore, it can be both time-saving and cost-effective compared to conventional casting technology.

*Corresponding Author: Jai-Young Koak

Department of Prosthodontics, School of Dentistry, Seoul National University,
275-1 Yeongeondong, Jongrogu, Seoul, 110-768, Republic of Korea
+82 2 2072 3027; e-mail, young21c@smu.ac.kr

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Laser sintering is a sort of additive manufacturing and relatively new method compared to both conventional casting and CAD/CAM milling technique. This is also called three-dimensional (3-D) printing or rapid prototyping (RP). Additive manufacturing can fabricate 3-D objects in a single stage, directly from their computer-aided design (CAD), for which X-ray CT and MR images are available.⁶ Different from CAD/CAM-based cutting technology, additive manufacturing technology creates products layer by layer on the basis of sliced data from the 3-D design.⁶ A laser is scanned metal powders according to the sliced data to obtain a layer of products. The powders for the next layer are covered on the melted layer, and the laser is again scanned according to the next sliced data. This sequence continues until the near-net-shape of the products is formed automatically.⁶ In addition, free form shaping can be achieved without mold and limitations from cutting tools in the process. Therefore, this process is expected to be applied for the fabrication of dental devices with complex geometry. It involves several advantages over the conventional CAD/CAM technique, and it also saves the raw materials and requires fewer tools to reduce costs.⁸

Both casting method and CAD/CAM techniques have been widely used for a long time to manufacture dental prostheses and many studies have been reported. However only few studies has been reported so far in order to address whether the properties of the prostheses fabricated using laser-sintering technology meet the needs of dental clinics. Basic research on Co-Cr alloys for dental applications has been reported using laser sintering technology. Akova *et al.*² reported that the bond strength of a laser sintered Co-Cr alloy to porcelain was not significantly different from that of cast Co-Cr alloy. The marginal and internal fit of metal-ceramic crowns fabricated by laser sintering technique is comparable to conventional production procedures.⁹ The mechanical properties and microstructures, which are the dominant factors for influencing mechanical properties of laser sintered Co-Cr alloy were also reported.^{6,10} However there were few studies about comparison of mechanical properties and microstructural characteristics for Co-Cr alloys manufactured by different methods. Therefore, investigation about the mechanical properties and microstructure of the specimens fabricated by laser sintered technique compared with those of conventional cast and CAD/CAM milled specimens is needed. The purpose of present study is to compare mechanical properties using microhardness and tensile strength test, and examine the microstructural characteristics of fractured surface for conventional cast, 3-D laser sintered and CAD/CAM milled Co-Cr alloy specimens. The null hypothesis was that the different manufacturing methods do not influence the mechanical properties and fractured surface characteristics of Co-Cr alloys.

Materials and methods

Thirty six flat disc shape cobalt-chromium (Co-Cr) alloy specimens (10 mm diameter and 3 mm thickness) were fabricated for surface hardness test and divided into three groups according to the manufacturing methods; twelve specimens for casting (n=12), twelve specimens for laser sintered technology (n=12) and twelve specimens for milled technology (n=12). All specimens were ground wet with a series of silicon carbide (SiC) abrasive papers (160, 320, and 800 grit) using a grinder-polisher machine (KDMT-300, Kyungdo Precision Co. Ltd, Seoul, Korea), and then ultrasonically cleaned in water for 5 minutes to remove the surface contaminants.

Twelve dumbbell shape specimens (3 mm diameter and 42 mm in length) for each group according to manufacturing methods were fabricated for the evaluation of the mechanical properties by a tensile test. All thirty six specimens were prepared according to ISO specification 9693: 1999(E) for metal ceramic restoration alloys (Fig. 1).¹¹

For fabrication of cast specimens, the data of disc and dumbbell shape was captured using software (3Shape D800, 3Shape A/S, Copenhagen, Denmark) and fabricated with castable pattern resin using three-dimensional system (ProJet[®] 3510 MP, 3D Systems, South Carolina, USA). These specimens were invested in a phosphate-bonded investment material (UNI VEST NON-PRECIOUS, SHOFU Inc. Kyoto, Japan) with metal casting, cast with the Co-Cr-based metal alloy (JEWOO S02, JEWOO M-Tech, Seoul, Korea). The composition of this Co-Cr-based alloy is provided in Table 1. Casting is usually carried out with induction heating in combination with either the centrifugal casting (Casting machine, Seki Dental Co. Seoul, Korea) according to the manufacturer's instructions. Cooling procedure, deflasking and blasting with 250 μm aluminum oxide at a pressure of 3 bar and 20 mm distance between nozzle and specimen surface with an angle of 45° were all carried out according to the manufacturer's instructions.

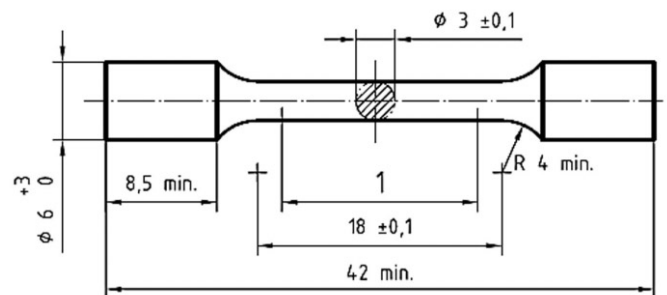


Fig. 1. Test specimen with radial shoulders for tensile strength test.¹¹

The laser sintered Co-Cr specimens were prepared from Co-Cr powder using direct metal laser sintering (DMLS) technology. The EOS CobaltChrome SP2 granule (Biomain AB, Helsingborg, Sweden) was used and its composition is provided in Table 1. The same 3Shape CAD data was sent to the production center (E-Master Dental Hub, Seoul, Korea) where the laser sintering was to be performed using direct metal laser sintering system (EOSINT M270, EOS GmbH - Electro Optical Systems, Krailling, Germany). The laser sintering procedure followed the recommendations of the manufacturer (EOS GmbH - Electro Optical Systems, Krailling, Germany). The specimens were fabricated under a laser power of 200 W and scan spacing from 0.1 to 0.2 mm. The laser scan speed and layer thickness were fixed at 7.0 m/sec and 30 μm , respectively. All specimens were sandblasted with 250 μm aluminum oxide at a pressure of 3 bar before the heat treatment. The heat treatment was performed in a furnace (LAB24 SF-25, Dongseo Science Co. Ltd, Seoul, Korea) at 800°C during 5 hours in air for releasing residual internal stress.

The 3Shape CAD data was sent to a communicating 5-axis milling machine (DNM-500, SMT Solution Co., Seoul, Korea) for the fabrication of CAD/CAM milled specimens from the Co-Cr alloy blanks (Starbond CoS, S&S Scheftner GmbH, Mainz, Germany Germany) according to the manufacturer's recommendation. The composition of this alloy blank is also showed in Table 1. No treatment after fabrication was performed.

Microhardness was measured using a Vickers indenter (Shimadzu HMV-2, Tokyo, Japan) with a 9.807 N·cm load and a loading time of 10s. Five measurements were made in each disc shape specimen.

Each dumbbell shape specimen was tested under tension at a cross head speed of 1 mm/minute according to ISO specification 9693 using a mechanical testing machine (Model 8516; Instron Co., Massachusetts, USA). Values of ultimate tensile strength (UTS), 0.2% yield strength (YS), elongation and elastic modulus were obtained with the aid of the mechanical testing machine software (Instron Series IX Software, Instron Co., Massachusetts, USA).

One of the fractured specimens for each test group used for the tensile mechanical property comparisons were subsequently used for fractured surface analysis. The fractured surfaces of the specimens for each group were examined using scanning electron

microscope (Mini SEM M-1, HyeJin Sys, Seoul, Korea) at two ranges of magnifications. Statistical comparisons of the mechanical properties between alloy groups were performed by Kruskal-Wallis test and followed by Mann-Whitney and Bonferroni test, the level of significance was set at $P=.05$. All statistical calculations were handled by the statistics software package (SPSS 19.0, IBM Co, NY, USA).

Results

The mean values and the standard deviations of the Vickers hardness for cast, laser sintered and CAD/CAM milled specimens were summarized in Table 2. Vickers hardness values are ranged from 410.40 Hv to 510.80 Hv in the cast specimen group, from 396.40 Hv to 426.80 Hv in the laser sintered specimen group, and from 227.40 Hv to 257.00 Hv in the CAD/CAM milled specimen group. The Bonferroni comparison test revealed that there were significant differences in the mean Vickers hardness values between all three groups according to the manufacturing methods ($P<.0001$), in the following decreasing order of values: The cast specimen (455.88 Hv) > the laser sintered specimen (413.10 Hv) > the CAD/CAM milling specimen (243.40 Hv). The cast specimen showed the highest mean value (455.88 Hv) which is higher than the mean hardness value of all alloys (370.79 Hv) while the CAD/CAM milled specimen showed lowest value (243.40 Hv) which is lower than the mean hardness value of all specimens.

Table 3 presents the comparison of the tensile mechanical properties for the three differently fabricated Co-Cr. Significant differences were found among the three groups for all mechanical properties, as noted by the Mann-Whitney and Bonferroni comparison test results ($P<.0001$). The mean ultimate tensile strength was

Table 2. Means and standard deviations of Vickers hardness test

Group	N	Mean (Hv)	SD
Cast	12	455.88	37.08
Laser sintered	12	413.10	8.77
CAD/CAM milled	12	243.40	8.97
Total	36	370.79	95.61

Table 1. Chemical composition of cast, laser sintered, and milled Co-Cr alloys as a percentage according to the manufacturer's instructions (wt %)

Alloys	Co	Cr	Mo	W	Si	Fe	Mn
JEWOOS02 (Cast)	63	28	5.5	etc. max. 3.5			
EOS CobaltChrome SP2 granule (laser sintered)	63.8	24.7	5.1	5.4	1	max. 0.50	max. 0.10
Starbond CoS (milled)	59	25	3.5	9.5	1	max. 1.5	

highest for the milled specimens (1442.39 MPa), and the laser sintered group showed 1411.12 MPa while the cast group showed the lowest value (831.51 MPa). The differences between groups were significant in all specimens ($P < .0001$). In 0.2% yield strength, the cast group showed from 770.18 MPa to 897.39 MPa, the laser sintered group showed from 1384.74 MPa to 1438.64 MPa, and the milled group represents from 1427.86 MPa to 1459.22 MPa which is the highest value. The cast specimens and the laser sintered specimens showed 0.59 mm and 0.87 mm elongation, respectively, which were lower values than total elongation (1.11 mm). There were significant differences in elongation comparing three groups

according to the post-hoc comparison results. The values of elastic modulus were the highest for the laser sintered group (67.0 GPa), the CAD/CAM milled group (61.0 GPa) and the cast group (59.0 GPa), and were significantly different ($P < .00001$).

Fig. 2 showed the scanned image of the fractured surfaces for the cast specimen, the laser sintered specimen and the CAD/CAM milled specimens at $1000\times$ and $2500\times$ magnification after tensile strength test. All alloys used in current study showed different characteristics of fractured surfaces according to their manufacturing methods. The fracture surface for the cast specimen showed the unique pattern and contained typical casting porosity as shown in some black

Table 3. Means and standard deviations of tensile strength test for the cast, laser sintered, and CAD/CAM milled specimens

Group		Ultimate tensile strength (MPa)	*0.2% Yield strength (MPa)	Elongation (mm)	Elastic modulus (GPa)
Cast (n=12)	Mean	831.51	620.67	0.59	59.0
	SD	41.10	20.06	0.11	.
Laser sintered (n=12)	Mean	1411.12	1136.15	0.87	67.0
	SD	17.00	49.10	0.14	.
CAD/CAM milled (n=12)	Mean	1442.39	1014.94	1.87	61.0
	SD	13.25	48.29	0.24	.
Total (n=36)	Mean	1228.34	923.92	1.11	62.33
	SD	286.06	226.78	0.59	.

* 0.2% yield strength ≥ 250 MPa will comply with the requirement of ISO specification.

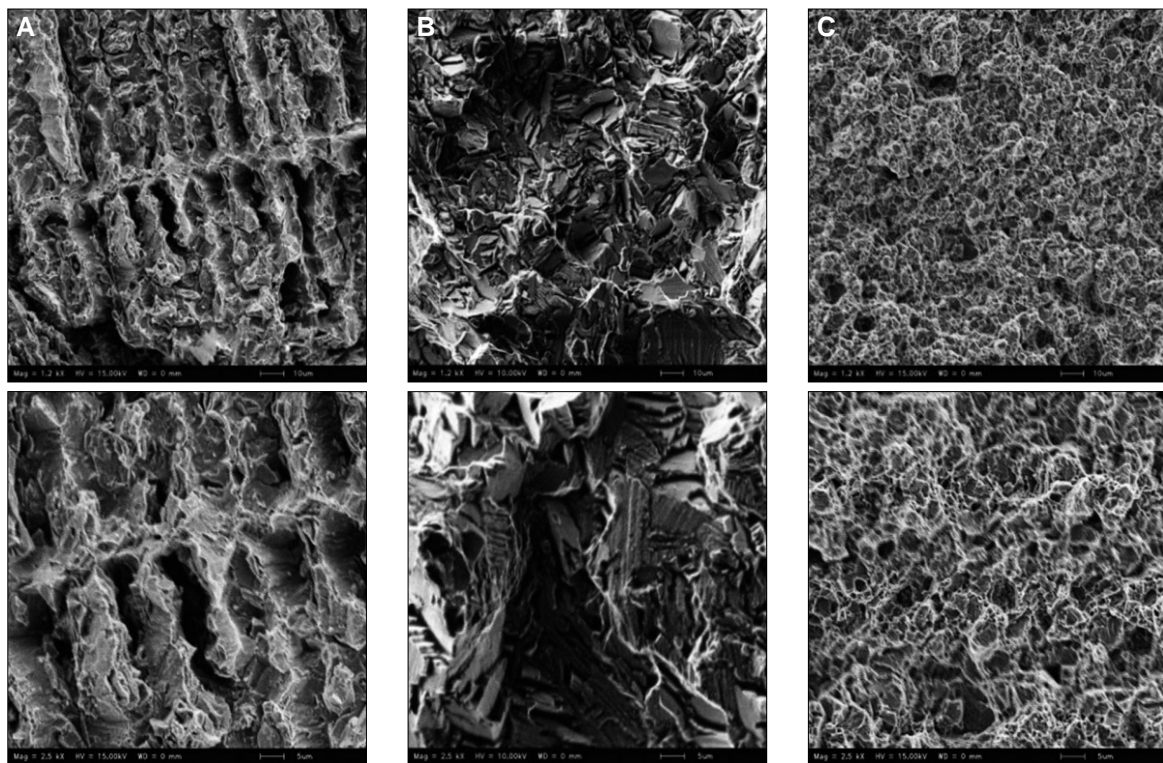


Fig. 2. Scanning Electron microscopy images of fractured surfaces for alloys. The characteristics of the cast specimen; unique loose pattern and typical casting porosity, A ($\times 1000$, $\times 2500$ original magnification), the laser sintered specimen; coarse granular structure with flat facets like characteristic features, B ($\times 1000$, $\times 2500$ original magnification), and the CAD/CAM milled specimen; homogenous and regularly dense microstructure, C ($\times 1000$, $\times 2500$ original magnification) are represented.

areas. The laser sintered specimens revealed more dense and coarse granular structure, and there are also characteristic features of flat facets with the wedge type cracks which may be formed by the cleavage fracture along the stacking faults were developed. The CAD/CAM milled specimens showed homogenous and regularly dense microstructure with small pores.

Discussion

Based on the results of this study, the hypothesis that there would be no significant differences between differently manufactured Co-Cr alloy groups in mechanical properties and microstructural characteristics of fractured surfaces was rejected. The Vickers hardness test result revealed that microhardness of the cast specimens was significantly higher value. This finding does not substantiate the other study that explained laser sintered specimens showed higher hardness than the cast specimens because the laser sintered surfaces were homogenous and regular.¹⁰ The more homogenous is the microstructure, the harder the surface of the specimens.¹² However, in current study, the Vickers hardness value of the cast group and laser sintered group showed similar numeric level which can be ignored in clinical application. The CAD/CAM milled specimens showed the lowest Vickers hardness value which was lower than the total mean value of three Co-Cr alloys.

According to ISO specification 9693,¹¹ 0.2% offset yield strength for dental restorative alloys should be at least 250 MPa, while the percentage of elongation after fracture should be no less than 3%. All three group alloys used in the current study met these requirements and the differences were significant between all groups. The milled specimens revealed the highest values in all individual test results. The laser sintered specimens showed higher ultimate tensile strength, 0.2% yield strength, and elastic modulus compared to the cast specimen. This result was similar to that of the nickel-chromium dental alloy fabricated with laser sintered and cast technology,¹³ which indicated that the laser sintered alloy possessed excellent mechanical properties. The higher ultimate tensile strength of the laser sintered group may be due to finer grain size, cellular dendrite, and elongated precipitates which can work as obstacles for dislocation motion.⁶

The scanning electron microscopy observation revealed that the Co-Cr alloy specimens manufactured by different methods showed distinguished microstructural aspects. The fracture surface characterization of the cast specimens in this study was in accordance with the previous findings of other study about cast alloys,¹⁴ where the casting defects, like porosity, and other microstructural defects were considered to be responsible for the lower tensile strength value of the cast alloy than other alloy specimens. This would be expect-

ed, that the regions around pores have localized stress concentrations. The laser sintered alloy was dense and more solidified structure compared to the cast and CAD/CAM milled specimens. This may explain the higher mechanical strength and smaller standard deviation compared to the cast and milled specimens which indicated that the better forming consistency.¹³

The laser sintering technology goes through the post-heat treatment procedure for releasing residual internal stress of the products. It was reported that internal residual stress in the laser sintered alloy generates the strain and affects the accuracy of the product.^{15,16} By conducting heat treatment, residual stress could be released but microstructure was also changed. Further examination should be investigated on microstructure and mechanical properties of the laser sintered alloy after heat treatment.

Different alloys that are adapted to each of the technologies are developed for the various production techniques mentioned above. Hence, it is difficult to draw general conclusions about the properties of Co-Cr alloys based on only one of the manufacturing method. Furthermore, the various production techniques result in reconstructions with different microstructures, such as grain size and surface morphology, which make comparison even more difficult.¹⁷ However the results of this study revealed that all three manufacturing methods provided adequate mechanical properties and microstructures for the scope of dental applications.

It can be reported that, compared with traditional cast and CAD/CAM milling technology, the laser sintered Co-Cr alloys display proper surface hardness, tensile strength, and homogenous microstructure that meet with the demands of dental clinics. Thus from the viewpoints of the mechanical properties and structure, this newly introduced technique can be a promising candidate for use in dental devices. This technology can be utilized more widely accompanied by the development of direct oral scanning devices. In addition, the laser sintering technology has the advantage that human error can be minimized in the manufacturing procedure that can keep consistent quality of restorations.¹³ And the manufacturing costs of restoration might be reduced through large-scale production at one time. However, this was a basic comparison study of metal alloy using *in vitro* specimens, not clinically used forms. Further evaluation of the laser sintered Co-Cr alloy in the oral environment would be needed. Meanwhile, the clinical aspects, such as marginal and internal fit of restorations, fabricated by this technology must be investigated before full recommendation for dental restoration.

Conclusion

Within the limitations of this *in vitro* study, the following conclusions were drawn: the different manufacturing methods influence the mechan-

ical properties and microstructural characteristics of fractured surfaces of Co-Cr alloys. The cast Co-Cr alloy specimens showed the highest Vickers hardness, and the CAD/CAM milled specimens revealed the highest tensile strength value. However all alloys represent adequate mechanical properties satisfying the ISO standards of dental alloy. Different manufacturing methods are directly related to their microstructural characteristics of fractured surfaces as well.

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주조, 3-D printing을 활용한 laser sintered 및 CAD/CAM milled 기법을 이용하여 제작된 코발트-크롬 합금의 물리적 성질 및 파절 단면 관찰 비교 연구

최윤정¹ · 곽재영^{1*} · 허성주¹ · 김성균¹ · 안진수² · 박동수³

서울대학교 치의학대학원 ¹치과보철학교실, ²치과생체재료학교실,

³주한미8군 618치무사령부, 용산 케리어스 치과병원

연구 목적: 본 연구의 목적은 주조, 3D printing laser sintered 및 CAD/CAM technology를 이용한 milling 방법으로 제작된 치과용 코발트-크롬 합금의 기계적 물성을 비교하고 파절 단면의 미세 구조를 살펴보는 데 있다. 이를 통해 새롭게 소개된 제작 기법 - 3D-printing laser sintered - 이 치과용 합금 제작으로 적합한지 알아보려 한다.

연구 재료 및 방법: 36개의 flat disc 모양의 시편을 제작하여 제작 방법에 따라 세 집단으로 나누었다; 주조 방식으로 제작한 12개, laser sintering 방법으로 12개, CAD/CAM milling 방법으로 12개의 시편을 제작하여 표면 경도 시험을 시행하였다. 또한 각 집단 별로 12개의 dumbbell 모양의 시편을 제작하여 인장 강도 시험을 시행하였다. 통계량이 비모수적 통계 분포를 보였으므로 Kruskal-Wallis 검정을 이용하여 각 실험군의 인장 강도 시험 결과를 비교했으며, 통계적 유의 수준은 $P=0.05$ 로 설정하여 Mann-Whitney 및 Bonferroni 사후 검정을 시행하였다. 전자 주사 현미경을 사용하여 파절 단면의 미세 구조를 관찰하였다.

결과: Vickers hardness test에서 세 집단간에 모두 유의한 차이가 있었고, 주조 방식으로 제작된 실험군에서 가장 큰 표면 경도(455.88 Hv)가, CAD/CAM milling으로 제작된 실험군에서 가장 낮은 표면 경도(243.40 Hv)를 나타냈다. 최대 인장 강도, 0.2% 항복강도, elongation 및 elastic modulus에서 세 집단간에 모두 유의한 차이가 나타났으며, CAD/CAM milling으로 제작된 실험군에서 가장 높은 최대 인장 강도(1442.94 MPa)가, laser sintered 실험군에서 가장 큰 0.2% 항복 강도(1136.15 MPa)가 나타났다. 파절 단면의 전자 주사 현미경 관찰 결과, 주조 시편에서는 독특한 성긴 모양과 전형적인 주조 다공성 구조가 관찰되었고, laser sintered 시편에서는 편평한 면을 동반한 거친 결정 구조가, 그리고 milled 시편에서는 균일하고 규칙적인 치밀 미세 구조가 나타났다.

결론: 서로 다른 제작 방법은 코발트-크롬 합금의 물리적 성질과 파절 단면의 미세 구조에 영향을 미쳤다. 주조 방식으로 제작된 시편에서 가장 큰 표면 경도가, milling으로 제작된 시편에서 가장 큰 인장 강도를 나타냈으며, 본 연구의 모든 실험군에서 치과용 합금의 ISO 기준에 부합하는 물성을 보였다. (*대한치과보철학회지* 2014;52:67-73)

주요단어: 코발트-크롬 합금; 치과용 CAD/CAM; 치과용 laser sintering; 물리적 성질; 파절 단면의 미세구조

* 교신저자: 곽재영

110-768 서울특별시 종로구 연건동 275-1 서울대학교 치의학대학원 치과보철학교실
02-2072-3027; e-mail, young21c@snu.ac.kr

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