

Mechanical properties of dental alloys according to manufacturing process

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Abstract. The purpose of this study is to compare how the fabrication method of dental prosthesis affects the mechanical properties. In order to compare the mechanical properties according to the manufacturing method, a test specimen of a dumbbell-shaped model conforming to the requirements of ISO 22674, an international standard for dental alloys, was produced using three methods: casting, milling, and 3D printing. Casting was produced using the lost wax casting method, and for milling, the shape of the specimen to be produced was designed using a CAD/CAM program, and then transferred to a milling machine to remove the metal block. The 3D printing method used the SLS technique to create a three-dimensional structure by sintering metal powder with a laser. When making the specimen, the specimen was oriented at 0, 30, 60, and 90 degrees. Various types of Co-Cr alloys such as pellets, metal blocks, and metal powders were used as materials to suit the manufacturing technique, and mechanical property evaluation was performed through tensile strength tests. Tensile strength was measured for yield strength, modulus of elasticity and elongation by applying a load until fracture of the specimen at a crosshead speed of 1.5 ± 0.5 mm / min ($n = 6$, modulus of elasticity $n = 3$). After the tensile test, the cross section of the fractured specimen was observed with a scanning electron microscope, and the statistics of the data were analyzed using Anova and multiple comparison post tests. As a result of comparing the mechanical properties of the three groups according to the manufacturing methods, it was shown that the yield strength was the highest at 1042 MPa at an angle of 0 degrees in the specimen produced by 3D printing method, and the elongation was the highest at 14% at an angle of 90 degrees in the specimen produced by 3D printing method. The modulus of elasticity was the highest at 235 GPa in the milled specimen. As for the shape of the fracture surface, a number of large and small pores were observed in the casting group, and a torn shape and rough surface were detected in the milling group, and irregular crack shapes were also found in the 3D printing specimen. The different result values between the three groups are considered to have an effect on the mechanical properties of the fabrication method. In particular, the 3D printing group showed a difference in yield strength and elongation according to the build direction.

Keywords Mechanical properties; Casting; Milling; 3D printing

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1. Introduction

For the fabrication of dental prostheses, precision casting has long been used. This is a method of dissolving a metal in a mold obtained by carving and embedding a wax pattern and injecting it to complete a cast body, which is also called a wax loss method or an investment method. Casting technology is a traditional dental prosthesis manufacturing method that has been used for a long time, but since all processes are performed by hand, it takes a long time, and incomplete casting may occur, and as a result, different outcomes may be obtained depending on the skill of the technician.¹⁻⁷

However, in recent years,, as the manufacturing technology of dental prosthesis is further developed and digital technology such as CAD/CAM is widely introduced in the

dental field, many changes have occurred in the manufacturing method of dental prosthesis. In particular, an oral scanner is used in the process of taking an impression, and CAD/CAM equipment has already been commercialized and used in the process of making a model and a wax pattern.⁶⁻⁹

The CAD/CAM system is a technology that produces a prosthesis in 3D form using a CAD program, saves it as an STL file, and then manufactures the prosthesis in the form designed with various CAM equipment. Milling and 3D printing methods using CAD/CAM systems are the technologies that have been introduced and actively used in the dental field because of their advantages that they are easier to manufacture than traditional casting methods and can more accurately control the appearance of the result of work.¹⁻²

The milling process solves the problem of porosity, which is a casting defect, in that the CAM process shortens the manufacturing time of the prosthesis and improves the precision of the framework. The CAM process uses a milling machine or computer numerical control (CNC) machining to cut the material with a machine, and all steps are controlled by a computer program.⁶⁻⁸ However, the milling method consumes a lot of raw materials for the metal block and, in the case of using a high-strength metal material, it has the disadvantage of frequent wear of the milling tool and restricts detailed works. There are methods such as heat treatment after processing.^{1-2, 8-9}

The selective laser sintering (SLS) method using 3D printing is a metal additive manufacturing (AM) process in which metal powders are melted layer by layer using a high-energy laser beam according to a computer design to create a three-dimensional model. This method has a fast production speed, less material waste, and a significantly lower incidence of porosity.^{6-7, 10-15}

In general, there are noble metal alloys and non-precious metal alloys as metal materials for manufacturing dental prosthesis. Pure gold or Au alloy is used as a direct filling material, casting material, wire rod, etc. However, since pure gold is too expensive and has high ductility, it cannot withstand the occlusal force sufficiently, so it is mainly used in the form of an alloy to increase mechanical strength and lower the price. Ni-Cr and Co-Cr alloys, which are non-precious metal alloys developed as a substitute for gold alloys, have good mechanical properties, corrosion resistance, and excellent biocompatibility, so they are actively used as dental materials.¹⁶⁻¹⁷ In particular, Co-Cr alloy was used as a metal material in this study because it has excellent mechanical properties as well as corrosion resistance comparable to that of precious metal alloys in the oral cavity.¹⁷⁻¹⁸

The mechanical strength of a dental prosthesis is closely related to the masticatory pressure, and for clinically appropriate use, the mechanical properties requirements set out by international standards must be observed.^{7, 19} In order to find out how various prosthesis fabrication methods affect mechanical properties, a mechanical property test guided by ISO 22674, an international standard for dental alloys, was performed. The international standard (ISO 22674) categorizes dental metal materials into six types based on 0.2% permanent set yield strength, elongation, and modulus of elasticity, and for clinical use, the requirements set out in the international standard must be satisfied.²⁰

In this study, we would like to investigate the effect of different fabrication methods on the mechanical properties of dental prostheses by using casting techniques, milling using CAD/CAM systems, and 3D printing processing methods.

2. Materials and Methods

2.1. Specimen production

In order to find out whether the manufacturing method of the prosthesis affects the mechanical properties, the method of manufacturing the specimen was divided into three groups as shown in Fig. 1.

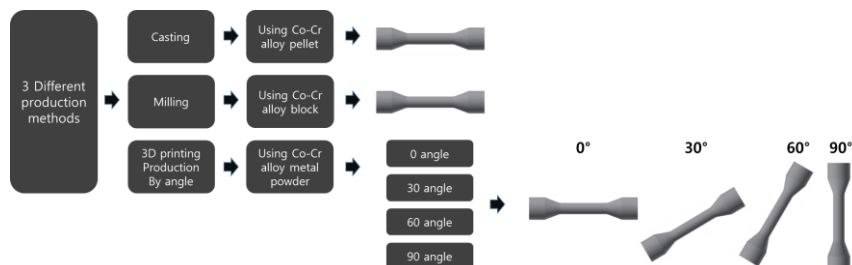


Figure 1. Three methods of manufacturing specimens for comparison of mechanical properties.

All specimens were made of Co-Cr alloy, and were manufactured in the form of dumbbell-shaped tensile specimens according to ISO 22674(Fig.2).

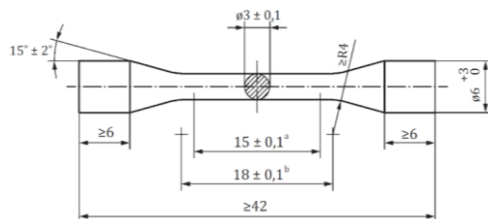


Figure 2. Dumbbell-shaped test specimens manufactured according to schematic drawing of ISO 22674.

The composition of alloys by group is shown in table 1.

Table 1. composition.

Group	Materials	Country	composition	Manufacturer
1	Wirobond®C	Germany	Co: 63.3%, Cr: 24.8%, W: 5.3%, Mo: 5.1, Si: 1%	Bego
2	Easymill	Korea	Co:>61%, Cr:>28%, Others, Co:Balance, Cr:28.7, Mo:6.1,	High dental
3	Cobalt alloys	Sweden	Mn:0.69, Si: 0.68, C:0.25, Fe:0.18, Ni:0.01	SANDVIK

The manufacturing process of the casting specimen is as follows. Dumbbell-shaped lead type waxes were produced using dental wax for casting. After the lead mold was produced, the injection line pin was attached to the thickest part of the mold, and was then embedded in an investment material made by mixing phosphate-based powder and water. To completely remove the lead, the mold was heated in an oven. When the mold reached the casting temperature, the alloy was melted and injected into the mold space. The mold was cooled after casting, and then the investment material was removed, the injection line was cut, and the surface was oxidized. The casted specimens were cooled to room temperature and cleaned by sandblasting with alumina particles (50 μm).

A CAD/CAM program was used in the milling process. In the CAM process, a dumbbell-shaped specimen was designed according to the drawing in Fig. 2 using 3D data, and an STL file was created and then transferred to a milling machine (PM5-All, Pistis, Korea) to delete the metal block for processing. The milled specimen was cleaned by sandblasting with alumina particles (50 μm).

Specimens prepared using a 3D printer were printed by selective laser sintering (SLS). The STL file of the specimen to be manufactured was created through the

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CAD/CAM program used for milling, and then sent to a 3D printer (METALSYS 120D, Winforsys, Korea) and manufactured by the SLS method. In the SLS method, a laser is selectively irradiated on the metal powder applied to the bed, and the metal powder is sintered with a laser to form a three-dimensional object. The metal powder material (particle size: 1-250 μm) was irradiated with a laser at a power of 130 W and a speed of 800 mm/s; it was then solidified into a thin layer of 20 μm and was repeatedly stacked. The metal powder was repeatedly laminated onto the solidified layer to complete the result. When preparing a specimen by laminating metal powders, as shown in Fig.3 directionality was arbitrarily given to each angle at 0 degrees (tensile direction and horizontal), 30 degrees, 60 degrees, and 90 degrees (vertical to tensile direction). All the 3D printing specimen was cleaned by sandblasting with alumina particles (50 μm).

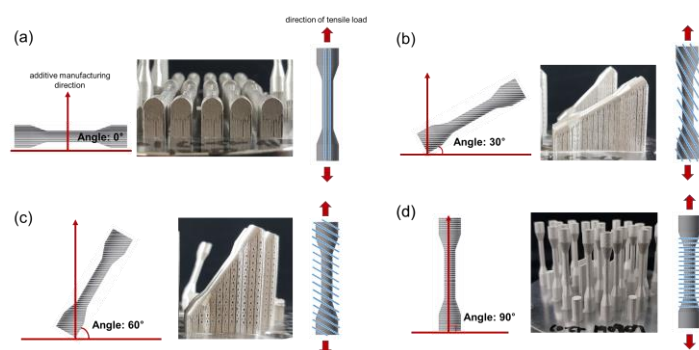


Figure 3. The additive manufacturing direction and the direction of tensile load: (a) 0°, (b) 30°, (c) 60°, (d) 90°.

2.2. Mechanical properties Test

Through the tensile test, 0.2% yield strength, tensile strength, and elongation were measured. For the tensile test, the gauge length of the test specimen was accurately measured within the gauge length interval to 0.01 mm using micrometers (MHT-2, Matsuzawa Seiki Co., LTD., Japan) or Vernier calipers (Mitutoyo, Co., Japan). A tensile load was applied to the test specimen using a universal material tester (Instron 3367, Instron Co., U.S.A.) at a cross-head speed of 1.5 ± 0.5 mm/min until the specimen broke. The elongation of the specimen was measured using an extensometer to obtain a continuous record without being affected by the tester compliance. The broken specimen was examined visually without a magnifying glass to determine whether the visible defects inside and outside the specimen were related to the damage of the specimen, in addition to whether the fractures occurred within the mark or the inscription line indicating the gauge length. If visible defects were observed or damage occurred outside the gauge length, the specimens and their results were rejected. The broken pieces were reattached and measured accurately to 0.02 mm with a portable microscope.

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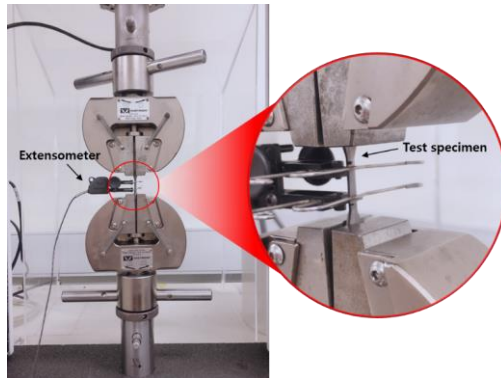


Figure 4. Tensile tests performed to evaluate mechanical properties.

2.2.1. Modulus of elasticity using tensile strain method

The modulus of elasticity was measured using the following ISO 22674 tensile strain method. Apply a strength equivalent to 60% of the 0.2% yield strength to the specimen, record the force and elongation, and then lower the force to 5% of the 0.2% yield strength. After repeating this procedure at least 4 times, calculate the elastic modulus according to the elastic modulus equation below. Measure two more specimens and record the average of the three most consistent values.

$$E = \frac{\Delta P}{A} \cdot \frac{L}{\Delta L} \quad (1)$$

where E is the elastic modulus (in GPa), ΔP is the change in the force (in N), A is the cross-sectional area (in mm²), L is the initial gauge length (in mm), and ΔL is the extension (in mm).

2.3. Observation of fracture surface

After the tensile test, the cross section of the test specimen was observed using a scanning electron microscope (SNE 4500M, Scanning electron microscope, SEM, SEC CO., LTD., Korea).

2.4. Statistical analysis

Data were analyzed with a statistical program (IBM SPSS Statistics 27; IBM SPSS Inc., Armonk, NY, USA). The homogeneity of variance was verified using one-way ANOVA (analysis of variation) analysis, and In order to identify significant differences between groups, post-mortem analysis was performed by applying a significance level ($\alpha=0.05$) adjusted by the Scheffe method in the post-analysis.

3. Results

Through the tensile test, the 0.2 % yield strength, tensile strength, and elongation measurement results for each group can be checked in Table 2. The 0.2% Yield strength was highest in 3D printing results at 1008 ± 59 MPa, followed by 501 ± 33 MPa in casting and 438 ± 15 MPa in milling. The elongation was 11 ± 3 % in casting, 12 ± 5 % in milling, and 9 ± 2 in 3D printing which showed the lowest value in elongation. The measurement results of the 3D printing group were 1042 ± 102 MPa at 0 degrees, 1022 ± 86 MPa at 30 degrees, 1002 ± 32 MPa at 60 degrees, and 966 ± 47 MPa at 90 degrees. The yield strength was higher in the horizontal than in the vertical build direction. The elongation tended to

be inversely proportional to the yield strength of $5 \pm 1\%$ at 0 degrees, $6 \pm 1\%$ at 30 degrees, $9 \pm 2\%$ at 60 degrees, and $14 \pm 2\%$ at 90 degrees

Table 2. Evaluation of mechanical properties according to the specimen manufacturing method.

Group	0.2% Yield strength (MPa)	Elongation (%)
	Mean $\pm$ SD	Mean $\pm$ SD
Casting	501 $\pm$ 33 ^a	11 $\pm$ 3 ^{ab}
Milling	438 $\pm$ 15 ^a	12 $\pm$ 5 ^b
3D printing *	1008 $\pm$ 59 *	9 $\pm$ 2 *
3D – 0°	1042 $\pm$ 102 ^b	5 $\pm$ 1 ^c
3D – 30°	1022 $\pm$ 86 ^b	6 $\pm$ 1 ^{ac}
3D – 60°	1002 $\pm$ 32 ^b	9 $\pm$ 2 ^{abc}
3D – 90°	966 $\pm$ 47 ^b	14 $\pm$ 2 ^b

* 3D printing result means the average value.

Values a,b,c are subsets of the significance level of 0.05, and the mean difference for each group is significant at the level of 0.05 ($p < 0.05$).

Fig. 5 shows the mechanical properties of the groups in a graph, and the difference between the graphs can be clearly confirmed. There was no significant difference between casting and milling in the overall average value of the yield strength of the 3 groups, but there was a statistical difference between casting-3D printing and milling-3D printing. In the 3D printing group by angle (0°, 30°, 60°, 90°), there was no significant difference.. When it comes to the elongation, there was a statistical difference in casting-3D 0°, milling-3D 0°, and milling-3D 30°. And the 3D printing group showed a statistical difference in 3D 90°-3D 0° and 3D 90°-3D 30°. As to the modulus of elasticity, the three did not show any significant difference within the group.

Table 3 shows the 60% and 5% of the yield load for each specimen calculated at 0.2% yield strength. Tensile deformation tests were performed using the values of the measured yield strength. The modulus of elasticity measured by the tensile strain method was similar in casting group 226 GPa, milling group 235 GPa, 3D printing group 0 degree 219 GPa, 30 degree 212 GPa, 60 degree 206 GPa MPa, 90 degree 198 GPa.

Table 3. Elastic modulus result value of tensile strain method measured through 0.2 % yield strength.

Group	Yield load (100%)	yield load (60%)	yield load (5%)	Elastic modulus [GPa]	p-value
Casting	3500.45	2100.27	175.02	226	p>0.05
Milling	3152.41	1891.45	157.62	235	
3D - 0 °	6905.63	4143.38	345.28	219	
3D - 30 °	6760.25	4056.15	338.01	212	
3D - 60 °	6617.40	3970.44	330.87	206	
3D - 90 °	6387.27	3832.36	319.36	198	

The results of the elastic modulus measurement did not show any significant difference between the groups ($p > 0.05$).

Fig.6 and Fig.7 show scanning electron microscope(SEM) images of cross sections of the fractured portions of the specimens after the tensile strength test. After the tensile test, only specimens in which the fracture occurred within the gauge section were accepted. The observed microstructure of the fracture surfaces was slightly different, depending on the crystals produced during the fabrication of each specimen

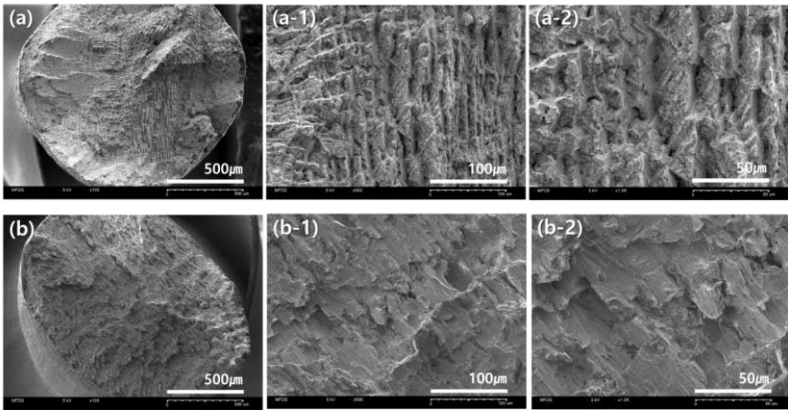


Figure 6. Observation of microstructure of fracture surface after tensile test : (a)~(a-2) casting specimen, (b)~(b-2) milling specimen.

In Fig. 6, (a) is the fracture surface of the specimen manufactured using the casting method and (b) is the surface of the specimen by means of the milling method. Large and small pores were observed in the cross section of the casting specimen, and a rough surface shape was observed in the cross section of the milling specimen as if the surface was torn to one side.

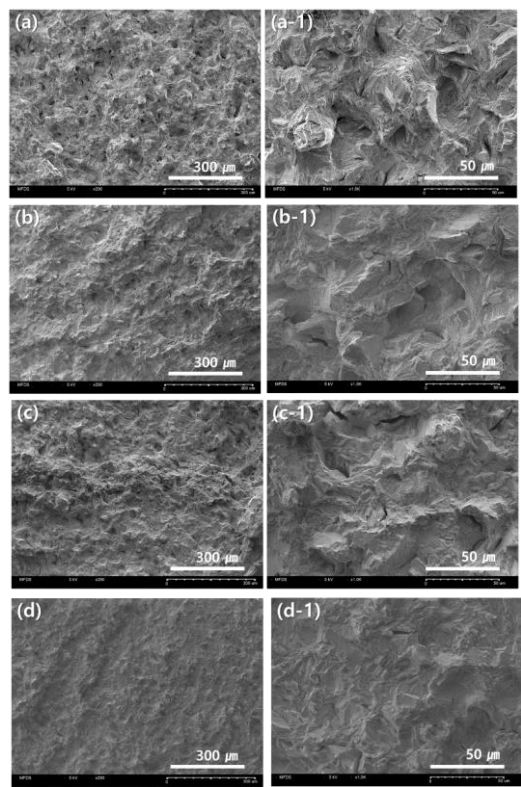


Figure 7. Observation of microstructure of fracture surface after tensile test of 3D printing group : (a)~(a-1) 0 degree, (b)~(b-1) 30 degree, (c)~(c-1) 60 degree, (d)~(d-1) 90 degree.

Fig. 7 is a cross section of a 3D printing group specimen. Regardless of the build direction, the images of the fracture surfaces of the specimens produced by 3D printing were similar, and it was observed that the irregular crack surface and gaps were formed in various places of the fracture surface.

4. Discussion

This study was conducted to investigate the relationship between the manufacturing method and mechanical properties of dental prosthesis. Three methods of manufacturing were used: casting, milling, and 3D printing. In particular, the specimens produced by 3D printing were manufactured with various build angulations (0, 30, 60, 90degree). Mechanical properties were measured through a tensile test and statistically analyzed. The mechanical property test complies with the international standard ISO 22674 for dental alloys. According to the use of dental alloys, the relevant international standard is classified into 6 types, from type 0 to type 5, and based on mechanical properties, each type is classified according to 0.2% yield strength, elongation, and modulus of elasticity as shown in Table 4. The range of application is different for each type, and the highest type can include the low type, and the highest type, type 6, can be applied from veneer crowns to removable partial dentures, clasps, and other parts requiring high strength at the same time.

Table 4. Mechanical properties by type specified in ISO 22674.

Type	0.2 % yield strength [MPa]	Elongation [%]	Elastic modulus [GPa]
0	-	-	-
1	80	18	-
2	180	10	-
3	270	5	-
4	360	2	-
5	500	2	150

The results of mechanical properties (yield strength, elongation, and modulus of elasticity classified by the manufacturing method) in Table 2-3 showed similar trends in the casting and milling groups, and statistically, there was no significant difference between the groups ($P > 0.05$). The casting group had a yield strength of 501 MPa, an elongation of 11%, and a modulus of elasticity of 226 GPa, and the milling group had a yield strength of 438 MPa, an elongation of 12%, and a modulus of elasticity of 235 GPa. Mechanical properties were similar between the two groups. As shown in Table 4, it seems to be applicable to type 4-5 alloy.

Fig. 6 is an image of the fracture surface of the casting and milling group. In the specimen of the Casting group, several large and small pores were observed as a whole, and in the milling group, a rough surface with a tear in one direction was found. Despite the porosity that appeared in the process of casting²¹, the yield strength was slightly higher in the casting group compared to the milling group, and this difference is expected to be affected by the chemical composition of the alloy.

Co-based alloys have different mechanical properties depending on the content of the constituent alloy.¹⁷⁻¹⁸ W, Mo, Ni of solid solution strengthening element can improve the strength depending on the content.²² **In the case of 3D printed specimens, the yield strength was overwhelmingly superior to that of other groups, but it is difficult to judge that the mechanical properties are the highest because the elongation is low and brittle. The Co-Cr alloy used in the test is composed of various elements such as Mo, W, Ni, and Si, and the content of each element is affected by strength and corrosion resistance, but since the test specimen was manufactured in three ways, the composition It is somewhat difficult to judge the mechanical properties by itself. It is believed that the mechanical properties are affected by complex interactions such as the components that make up the alloy and the way the prosthesis is manufactured.** Therefore, if the specimen manufacturing process method and test conditions are the same, it is considered that the amount of the element constituting the alloy affects the mechanical properties.

In the case of the 3D printing group produced by selectively melting metal power with a laser, the yield strength was very superior to that of the previous two groups. Unlike the casting technique, the SLS technique melts and solidifies the metal powder with a high-temperature laser, so unlike the casting technique, the incidence of pores is significantly lower.^{10, 12, 23} Co-Cr alloy undergoes a phase transition to α phase (FCC) at high temperature and ϵ (HCP) phase at low temperature. In the 3D printing method of melting metal powder with a laser, α phase is predominant, and it is believed that the higher intensity was observed compared to the other two groups ^{21, 24}

The yield strength of the 3D printing group showed a statistically significant difference when compared with the other two groups (Casting, milling) ($p < 0.05$). The yield strength of 3D printing was the highest at 1024 MPa in the specimen manufactured at an angle of 0 degrees. In particular, at 0°, the highest yield strength was recorded among the all groups, but the elongation was the lowest. The 0 degree angle in Fig. 3(a) was parallel to the direction in which 3D printing laminates the metal powder and the loading direction in the tensile test. During the manufacturing process of the 3D printing specimen, the metal powder melts and a slight gap, such as pores, may be formed between the laminated layers, which may cause defects. These gap defects are expected to cause fracture during the tensile test, elongate along the loading direction of the force, and then break.²⁵ When

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the stacking direction and loading direction are horizontal as shown in Fig. 3(d), this sagging is considered to be shorter. Fig. 7 is an image of the fracture surface of the 3D printing group, and irregular cracks and gaps can be observed in various places. The anisotropy of the material is thought to have an effect on the mechanical properties.^{12, 26} In the case of a prosthesis requiring high rigidity, it is advantageous to fabricate the specimen in the same direction as the tensile direction. Also, it is considered to be important to understand the difference between yield strength and elongation according to the build direction, and to manufacture a clinically appropriate dental prosthesis in consideration of the characteristics of mechanical properties.

The 0.2% yield strength of the alloy can prevent permanent deformation of the restoration, especially in fixed partial denture frameworks, and yield strength in excess of 300 MPa is sufficient to withstand permanent deformation.²⁴ All of the 3D printing groups were 500 MPa or more, suitable for use in type 5.

Elongation refers to the physical deformation of a material before fracture when subjected to tensile stress, and is related to the workability of the alloy.²⁷ Overall, the elongation was low in the 3D printing group, and a slight brittle property was also shown. Brittleness could not be overlooked even though the strength was much better than the other two groups. Despite this weakness the ductility properties can be restored without significantly changing the microstructure through heat treatment such as annealing^{26,28} In addition, when high-intensity ultrasonic waves are used, the microstructure of the metal can be converted from long columnar beta grain to equiaxed fine grain, thereby improving mechanical properties (yield strength and tensile strength).²⁹

The tensile strain method was used to measure the modulus of elasticity in this study. In 2016, ISO 22674 introduced three methods of testing the modulus of elasticity for type 5 alloys (tensile deformation method, bending method in three- or four-point bending mode, and acoustic resonance method). The test was performed using the yield load value of 0.2% yield strength measured through the tensile test (Table 3). The modulus of elasticity was the highest in milling at 247 GPa, and all three groups met the requirement of ISO 22674, which is 150 degrees. Previous studies have shown that the Co-Cr alloy used in the manufacture of dental prosthesis has a modulus of elasticity of 154–208.¹⁹ All three groups are considered to have similar results. Modulus measurements were repeated at least 4 times and the average of the 3 most similar values was calculated. As a result of measuring the modulus of elasticity, there was no statistically significant difference between the three groups ($P > 0.05$). Numerical consistency was maintained and large standard deviations did not appear, increasing the accuracy of the results.²³

In the case of casting, since the fabrication of the prosthesis is performed manually, the result of the work varies depending on the skill level of the dental technician, so the technique of the dental technician is important. In the case of milling, the accuracy of the prosthesis varies according to the size of the bur used. A bur with a large size has good durability and excellent cutting rate, but a bur with a small diameter is useful for reproducing fine parts.¹ This precise cutting is one of the important factors in the CAD/CAM milling process. In 3D printing, it was found that a number of factors such as the melting speed of the laser, the size of the metal powder, and the direction of the stacked build affect the mechanical properties.²⁶

Therefore, it is recommended that dental technicians and dentists who manufacture prostheses accurately understand the characteristics of the manufacturing method and select the appropriate method for clinical use.

The three methods performed in this study are considered to require more diverse studies such as the type of metal used as a material and the composition of the alloy.

5. Conclusions

The yield strength tended to be high in the 3D printing group, especially at an angle of 0 degrees, where the tensile and stacking directions were horizontal, and the results were different depending on the build direction. The 3D printing group showed brittle

properties due to low elongation compared to high yield strength, but this can be overcome by heat treatment such as annealing. The two groups, Casting and Milling, showed similar trends in mechanical properties. The elastic modulus measurement method performed in this study used the tensile strain method guided by ISO 22674, and the results of the tensile strain method were numerically consistent, and the standard deviation was not large and the accuracy was high. All three groups satisfied the requirements of the international standard. Different structures between the three groups were observed through the image of the fracture surface of the specimen observed with the FE-SEM. The structural difference is due to the manufacturing method, and it is believed that it affects the mechanical properties. Therefore, it is recommended to understand the characteristics of various manufacturing methods when fabricating dental prosthesis and to use it clinically.

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Conflicts of Interest: The authors declare no conflict of interest.

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