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Flexural Behavior of Thermocycled Polyether ketone ketone (PEKK) and Micro-Strains Induced on its Application as Double Crown-Retained Removable Partial Denture in Kennedy Class I: In Vitro Study

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Aim: This study aimed to evaluate the effect of thermal aging for 5000 and 10000 thermocycles on the flexural strength and flexural modulus of Polyether ketone ketone (PEKK), as well as the micro-strains induced on the supporting abutments by its use as double crown-retained removable partial dentures (RPDs) combined with zirconia primary copings in Kennedy Class I.

Materials and Methods: The study was conducted using PEKK for fabrication of flexural strength specimens and double crown-retained RPD framework, and zirconia (ZrO2) for the construction of primary copings in maxillary Kennedy class I models. The study involved three groups according to time of testing (n=8): Group I included testing at 24 hours of storage, group II included testing after 5000 thermal cycles, and group III included testing after10000 thermal cycles. The flexural strength and flexural modulus were evaluated by a three-point loading test using a universal testing machine. The micro-strains were analyzed using the strain gauge technique.

Results: Results of flexural strength revealed group (I) to have the highest value followed by group (II), then group (III). For flexural modulus, both groups (I) and (II) showed no significant difference between them, with significantly higher values than group (III). Regarding micro-strain results, group (III) revealed the highest value, followed by group (II), and then group (I).

Conclusions: PEKK could be considered a polymeric material having a shock- absorbing property, but when subjected to thermal aging, it deforms and its flexural modulus and strength decrease, with more stress transferred to the supporting structures.

Keywords: Thermocycling, Flexural characteristics, CAD/CAM, Telescopic dentures.

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Introduction

Recent innovations in the dental field always seek tooth preservation, demanding treatment of partially rather than completely edentulous patients. RPDs are considered one of the most common prosthetic options in cases of bilateral distal extensions.¹ Double crown-retained RPDs are considered an esthetic and biomechanical alternative method; because they can attain excellent retention, achieve transference of occlusal loads toward the long axis of teeth, and eliminate the unacceptable metal display of clasps.2 Double-crown attachments constitute a primary coping that is cemented to an abutment tooth and a secondary crown, which is incorporated within the denture. Double-crown-retained RPDs can be constructed from precious or non-precious metal alloys.3 Also, can be formed of zirconia $(ZrO₂)$,⁴ titanium,⁵or high-strength achieve le polymers.6

ings in their structures.
 \blacksquare the \blacksquare \blacksquare PEKK is one of the high-strength polymers, which is a methacrylate-free, thermoplastic high-performance material. Recently, it has found a wide range of usage in prosthetic dentistry.7 PEKK and polyether ether ketone (PEEK) are the two highly recognizable members of the polyaryl ether ketone (PAEK) family. The PAEK family is characterized by satisfactory mechanical properties and chemical stability. PEEK and PEKK have aromatic rings in their structures. The difference between them is that PEKK has a second ketone group, which is reflected in its higher polarity and backbone stiffness compared to PEEK. This resulted in an increase in the melting temperature, achieving higher thermal stability as well as compressive strength superior to PEEK. $8-10$ Pekkton® ivory (Cendres * Métaux, SA, Switzerland) is a product of PEKK containing titanium dioxide $(TiO₂)$. It presented high hardness, wear resistance, and compressive strength. $8,11,12$ Regarding the biological activity of PEKK, it was

previously revealed to encompass very low bacterial adhesion on its surface and less inflammatory response.^{13,14} PEKK can be thought to be successfully used in dentistry as prosthetic and implant biomaterial. 8

Computer-aided design (CAD) and computer-aided manufacturing (CAM) technology revealed increased feasibility and accuracy of modern prosthetic material fabrication.^{15,16} Recently, CAD/CAM technology has been employed in the construction of PEKK prosthetic appliances.17,18

 $\frac{1}{\sqrt{2}}$ as a fluctuations in the oral cavity, they can The clinical oral performance of the material used in the construction of prostheses is related to its mechanical behavior; therefore, a mechanical assessment should be performed. A rigid framework for a removable prosthesis is mandatory to achieve less stress transfer on the supporting abutments and a more favorable stress distribution. This implies the need for a material with high flexural characteristics. Thus, studying the flexural behavior of prosthetic materials is crucial.¹⁹The final mechanical performance of any prosthetic appliance is highly related to the surrounding thermal effects. As dentures are subjected to heat fluctuations in the oral cavity, they can show deteriorated mechanical properties on the long run. 20

> supporting structures of RPDs can be evaluated by a variety of techniques, but no single technique met all the requirements for an illustration of the extensive physiological $involved.²¹$ Many studies consider strain gauge stress analysis to be an acceptable tool for recording the deformation of any object subjected to stress. $22,23$ Correlating the flexural behavior of prosthetic material to the stresses transferred through them could give a more

comprehensive assessment of the used prosthetic material.

By reviewing the up-to-date scientific literature concerning PEKK, little information was noticed concerning the effect of thermal aging on its mechanical properties, particularly the flexural strength, with insufficient data about the micro-strains A induced on the abutments in case they were used as double-crown-retained removable partial dentures. Therefore, this in-vitro study aimed to evaluate the effect of thermal aging for 5000 and 10000 thermocycling on the flexural strength and flexural modulus of PEKK, as well as the micro-strains induced on the supporting abutments by its use as double crown-retained removable partial dentures combined with zirconia primary copings in Kennedy Class I, using the strain gauge technique.

The null hypothesis set for this study that thermocycling would not affect either the flexural strength and flexural modulus of PEKK, or the micro- strains induced on abutments by its application as double crown- retained removable partial denture in Kennedy Class I.

Materials and Methods Sample Size Calculation

A pilot study was conducted on 15 samples (i.e., 5 samples per group) to determine the required sample size needed to achieve sufficient statistical power. Based on the flexural strength and strain results, a large effect size (f) was detected (i.e., 6.67 for flexural strength and 6.37 for strain analysis), so the initial sample size calculated was 15 samples (i.e., 5 samples per group), but sample size increased to 24 samples (i.e., 8) samples per group) to increase reliability of the study.

Study Design

This in-vitro study was conducted using PEKK (Pektone Ivory-Cendres+MMétaux Medtech, Switzerland,

Germany) for the fabrication of flexural strength and modulus specimens and double crown-retained RPD framework and $ZrO₂$ (Zirconia Ceramill Zirconia-Amann Girrbach AG, Koblach, Austria) for the construction of primary copings in maxillary Kennedy class I models.

block randomization was performed by collecting the 24 prepared specimens and double crown-retained RPDs and were randomly divided into three groups, so that each group included 8 specimens, that were put in similar envelopes and the envelopes were closed. Then random selection was further performed to name the envelopes according to the needed groups: I, II and III.

Group I: Included testing at 24 hours of storage (in distilled water in an incubator at 37ᴼ C) following construction of flexural strength specimens and double crownretained (RPDs), to be considered as a base line control group.

Group II: Included testing after thermal aging by applying 5000 thermal cycles, which is equivalent to six months of intra-oral service.

Group III: Included testing after طب الأسناك thermal aging by applying 10000 thermal cycles, which is equivalent to twelve months $\frac{15}{15}$ SD of intra-oral service.

sample size needed to specimens; water distillation was done by
stical power. Based on Stillo water distiller (Lab supply, Italy). The For the 24 hours storage of group, I specimens; water distillation was done by pH of the distilled water was checked to be 7 using Hydrion Insta-Chek pH Paper (0- 13, micro essential lab). The incubator used for specimens' storage was Titanox, ART.A3- 213-400I, Italy.

> The flexural strength and flexural modulus of PEKK were evaluated by a threepoint loading test. The micro-strains induced on the supporting abutments were analyzed by applying the strain gauge analysis. Tests were performed at 24 hours of storage, then and 10000 cycles of

thermocycling. A flowchart detailing the study procedure is shown in Figure 1.

Figure 1: Flowchart detailing the study procedure for this in vitro study.

1. Flexural strength testing 1.A. Specimens construction

For flexural strength and flexural modulus assessments, a total of 24 rectangular bar-shaped specimens $(15 \times 2.5 \times$ 1.5 mm) were designed with Exocad software (ExocadDentalCAD 2.4 Plovdiv, Exocad GmbH, Darmstadt, Germany), and the standard tessellation language (STL) file of the designed specimen was converted to the CAM software (CORITEC ICAM V5 SMART, imes-icore® GmbH, Eiterfeld, Germany) for nesting and preparation to be milled from PEKK blanks by a five-axis computer numeric control lab milling machine (Coritec 350i series, imes-icore® GmbH, Eiterfeld, Germany). 24

1.B. Thermocycling of specimens

Group II specimens were subjected to 5000 thermal cycles, and group III specimens were subjected to 10,000 thermal cycles. Thermocycling was performed in a thermocycling machine (Thermoscientific 1100, Mechatronic GmbH) between 5 °C and 55 °C. The cycles involved a dwell time of 30 seconds in each bath, with a transfer time of 10 seconds between baths. ²⁵

1.C. Flexural strength testing procedures

The test was carried out using a universal testing machine (Instron 3365, MA, USA, with a load cell of 5 KN). Each bar specimen was positioned horizontally on two supports that are separated by a 10 mm distance. Then each tested specimen was loaded at its center with a wedge-shaped load applicator having a curved edge and a crosssectional radius 0.5 mm. The test was run at a cross-head speed of 1 mm/min. until the specimens fractured. $26,27$ (Figure 2)

Figure 2: Figure 2a: Flexural strength testing by 3 point loading. Figure 2b: PEKK specimen fractured under 3- point loading.

The flexural strength (F.S.) in MPa was given by the used software (Instron Bluehill Software version 3.72, Norwood, MA, USA) in accordance with the following equation:

$F.S = 3 F L/2 W (Th)²$

Where F stands for the maximum applied load (in Newton: N),

 $\frac{1}{4}$ SDL: stands for the distance between the two supports (10mm).

blanks by a five-axis $\bigcup_{2.5}$ W: stands for the width of the specimen $(2.5$ mm).

> Th: stands for the thickness of the specimen (1.5mm).

1.D. Flexural modulus determination

The flexural modulus (E_{flex}) in GPa was given as an automatic reading by the software, determined from the slope of the initial linear segment of the stress-strain curve.25

As an equation, E_{flex} could be calculated by applying the following formula:

E flex = F $L^3/4$ W (Th)³ d

Where d (mm) represents the deflection at the maximum applied load.

2. Micro-strain testing

2.A. Models construction

simulating upper Kennedy Class I, with first premolars as last abutments were scanned using a desktop scanner (DOF Freedom UHD, 3D DDS, Seoul, Korea). The scanned file was then uploaded to the Mesh- Mixer program (Model Creator, Exocad GMBH, Darmstadt, Germany) and used to design the model's different details, including the detachable dies of the last two abutments on both sides. Mucosal space (2mm thickness at ridges and 1mm thickness at palate) and 0.25 mm periodontal ligament membrane simulating space around the roots of the detachable dies were cut back on the virtual model. Two grooves 1mm distal to the principal abutments were made for installing the strain gauges. A mucosal stent was designed with four stoppers on the ridge for later installation of mucosa.

The virtual design of the model and the mucosal stent were transformed into a Standard Tessellation Language (STL) file. Then they were 3D- printed into 24 models, using an additive manufacturing device (Dent 2 Mogassam, Mogassam Co., Delaware, USA), applying the digital light processing technique (DLP) for printing the model. Printing was conducted layer by layer via
ultravialet (UV) light polymorization from ultraviolet (UV) light polymerization from base to top. After 3D printing, the model was placed in a post-curing box to complete its polymerization. The last two abutments on both sides were prepared with a 1mm deep Chamfer finish line, 2.5mm occlusal reduction, and a 3-degree taper. 27

 Mucosa simulator material (GINGISIL, soft Endhärte Shore A45, Dent-E-Con E.K., Berlin, Germany) was injected around the roots of detachable dies, as well as and STL the formula of the double into the mucosal stent. Finally, the stent was pressed against the model.

2.B. Primary copings construction

An educational acrylic cast were designed by EXOCAD software with a The model and dies were sprayed with a scanner spray and scanned using a DOFF desktop scanner. The primary copings common path of insertion, conus in shape, and a zero-degree taper at the cervical third. The rest of the tooth was tapered to 4 degrees. The thickness of the copings was kept at the minimum thickness, which was 1 mm, with a 1mm thick finish line and a gap distance set at 0. 05mm. (Figure 3)

Figure 3: Virtual designing of double crown-retained RPD framework.

 $\frac{d}{dS}$ SD achieve its final shape. The fitting surface of ed layer by layer via **Departicles.** The primary crowns were then The virtual design was transferred to STL file to be prepared for milling from $ZrO₂$ blanks. Afterwards, complete sintering in the furnace at $1,350-1,500$ °C was done to each primary coping was sandblasted for 10 seconds with 110 μm aluminum oxide cemented to abutments.28

2.C. Double crown-retained RPDs

The model and dies were sprayed with a scanner spray and scanned using a DOFF desktop scanner. The double crownretained RPD framework was designed using EXOCAD software with a 1.5mm thickness. (Figure 3)

 The virtual design was converted to an STL file for milling from PEKK blanks. crown-retained RPDs frameworks were seated on the model to check its fit and accuracy. The secondary

crowns were veneered with a high-impact polymer composite, filled with a microceramic layer (Novo.lignVeeners, Bredent, Senden, Germany). (Figure 4)

 ${\sf MS}$

Figure 4: PEKK double crown-retained RPD framework seated on the model.

Afterwards, duplicate casts were fabricated from the models to complete the setting-up of teeth and waxing-up of double crown-retained RPDs. Then processed from heat-cured acrylic resin (Vertex Rapid Simplified, Vertex Dental, Soesterberg, Netherlands) via the conventional long polymerization cycle (9 hours at 74° C).^{29,30} 2.D. Micro-strain analysis and thermocycling.

The micro-strain analysis was performed on group I double crown-retained RPDs after milling within 24 hours, on group II double crown-retained RPDs after 5000 cycles of thermocycling, and on group III after 10000 cycles of thermocycling. Thermal cycles were performed in a thermocycling machine (Thermo scientific 1100, Mechatronic GmbH) at 5ºC and 55ºC, with 30 seconds dwell time in each bath, and 10 seconds of transfer between the two baths. The strain gauges (Strain gauges, kyowa, Tokyo, Japan) used in this study had a length of 1 mm, width of 2.4mm, and nominal and maximum
resistance of 120 Ohm. This choice was tested for resistance of 120 Ohm. This choice was based on the approach according to previous studies (Bahgat et al and Emera et al). The

strain gauges were installed in their created placement grooves, using a delicate layer of cyano- acrylate base adhesive cement.²⁷

Universal testing machine (Instron 3365, MA, USA, with load cell of 5kN) was used for applying vertical static unilateral load ranging between 0-100 Newton - on the right side of the denture, this load was applied in a point existing between second premolar and first molar using chisel shaped load applicator as recommended by previous studies. $27,31$ (Figure 5)

Figure 5: A chisel-shaped load applicator applying a load at a mid- distance between the second premolar and the first molar.

as **SDJ** After load application, a two channels and RPDs after 5000 **Dental Shamps at the loaded** and unloaded sides. strain-meter was used to assess the micro-Once the load was completely applied, the micro-strains readings were transferred to micro-strains units. Finally, micro-strains units' readings were analyzed using the software (Kyowa PCD-300A).

III. Statistical Analysis

Numerical data were presented as mean with 95 confidence intervals (CI), standard deviation (SD), minimum (min.), and maximum (max.) values. They were normality and variance homogeneity by viewing distribution and using Shapiro-Wilk's and Levene's tests,

respectively. They were found to be normally distributed with homogenous variances across groups and were tested using one-way ANOVA followed by Tukey's post hoc test.

Correlation analyses were made using Spearman's rank-order correlation coefficient. The significance level was set at p<0.05 within all tests. Statistical analysis was performed with R statistical analysis software version 4.4.0 for Windows.³²

Results

Descriptive statistics are presented in Table1. The results of intergroup comparisons are presented in Table 2.

Table 1: Descriptive statistics for flexural strength and modulus and microstrain showing mean, standard deviation, confidence interval, and maximum and minimum values.

SD: standard deviation, Min.: minimum, Max.: maximum.

Table 2: Intergroup comparisons between three groups showing the mean value, standard deviation, p and f values, and confidence interval.

Confidence interval (CI) values with different superscripts within the same horizontal row are significantly different, *significant $(p<0.05)$.

flexural strength, A statistically significant difference $(p \le 0.001)$ was present between the three groups. Group I showed the highest mean value (184.27 ± 4.75) and Group III showed the lowest mean value (145.09±5.39).

Regarding flexural modulus, comparisons showed Groups I and II (4.21 ± 0.10) (4.04 ± 0.18) to have significantly higher mean values (p<0.001) than Group III $(3.29 \pm 0.11).$

ASDJ between the three groups. Group III showed Ain Shams Dental Journal (195.40±4.62). Micro strain values were Regarding micro-strain, a statistically significant difference ($p \le 0.001$) was present the highest mean value (271.58 ± 10.54) , and Group I showed the lowest mean value negative denoting development of compressive stresses.

> The correlation matrix presented in Table 3 showed a strong positive correlation between flexural strength and modulus. Additionally, they also showed that there were strong negative correlations between Micro-strain and both other variables.

AMS.I

*Significant (p<0.001)

Discussion

Tooth-tissue-supported RPDs have a higher risk of abutment loss due to the difference in viscoelasticity between the mucosa and the abutments, which leads to more stresses on the abutments and deformation, or fracture, of the material used in the construction of the denture. The study was performed on a maxillary model due to the need to achieve esthetic restoration on the maxillary arch rather than the mandibular. High-performance thermoplastic polymers (e.g. PEKK) have been recommended to be used in dentistry due to their favorable mechanical and shock-absorbance ability. $8,10,33$ In this context, this study was conducted to assess the flexural strength and flexural modulus of PEKK, as well as the micro-strains induced on the supporting abutments by its use as double crownretained RPDs combined with zirconia primary copings in Kennedy Class I before and after thermocycling. The null hypothesis of this study was rejected as thermocycling had an effect either on the flexural characteristics of PEKK or on the microstrains induced on abutments by its application as a double crown-retained removable partial denture in Kennedy Class I.

Based on the continuous demand for long-lasting durable materials, any prosthetic material should be assessed for its behavior when subjected to temperature fluctuations

Table 3: Correlation matrix showing the positive and occurring in the oral cavity, as this could $\left\{ \begin{array}{c} 1.6692\end{array} \right\}$ temperature changes in the oral cavity, with a induce structural changes in the material that will influence the clinical performance of the material. A research gap was noticed concerning the long-term evaluation of PEKK. Therefore, thermal cycling was applied in the current study as an accelerated aging technique for simulating the few cycles of 5000 and 10000 that are respectively equivalent to 6 months and 12 months of intra-oral service. For the control group specimens, storage was done in distilled water (pH 7) in an incubator at 37 °C, simulating the intra-oral conditions of pH and temperature.³⁴

> $\frac{1}{2}$ SD vary from being the maximum at the neutral e as double crown-
bined with zirconia $\bigcup_{n=1}^{\infty}$ manner of stress distribution greatly Testing the flexural strength of a prosthetic material is essential, as it describes the material's ability to resist permanent deformation or fracture under loading, thus persuading clinicians when choosing among different prosthetic materials. The universal testing machine is commonly used to measure flexure strength. In the present study, flexural strength was assessed using a 3-point bending test, where the maximum stresses concentrate under the loading tip in the middle and significant tensile and compressive stresses are created in the tested specimen, in addition to shear stresses that axis of the specimen to being zero at the outermost surfaces of the specimen. This resembles the intra-oral situation, particularly in cases of long-span prostheses.³⁴ It was previously reported that rigid framework materials possess high resistance to bending forces during functional movements. This fact may also have an impact on the amount stress and magnitude of loading transferred to the supporting structure. In relevance to that, it was found crucial to assess the flexural modulus of PEKK before and after thermocycling.36

The terminal abutment of an attachment system is subjected to greater risk and stress compared to the mesial abutment. For this reason, it was advised that at least two abutment teeth should be splinted when attachment prostheses are used, to improve the stress pattern. 37 On this backdrop, this study used two abutments on each side.

A combination between Primary copings made from $ZrO₂$ and secondary crowns made from PEKK was used in this study to improve the performance of telescopic system. This combination was proven to have high biocompatibility and retention, better ability to absorb occlusal forces. 4,29,38

The Strain Gauge analysis method was adopted in this study to evaluate the strain on the distal surface of abutment teeth. This method is considered highly accurate as it can recognize very slight strains.39 The load application used for strain gauge analysis was at a point between the second premolar and the first molar, because this area is always subject to maximum occlusal forces. Additionally, a unilateral load was applied in this study, to simulate chewing activities that are usually carried out unilaterally.⁴⁰

Regarding the results of flexural strength in the current study, a significant difference was found between the three tested groups. Group I revealed the highest flexural strength value, while Group III revealed the its
lowest flexural strength value. This reflects lowest flexural strength value. This reflects the negative effect of thermocycling aging on the flexural strength of PEKK. To the best of our knowledge, no previously evident studies were directed towards investigating the effect of thermocycling on the mechanical properties of PEKK. Although highperformance polymers were known for their high thermal and hydrolytic stability, PEEK, as an example, when subjected to a hydrolytic environment with continuous thermal cycling could abate their stability, and a deterioration in flexural strength could

be observed. ⁴¹ This recognizable data for PEEK could also be employed for PEKK, both being very similar in structure. This result might be attributed to the possibility that the thermal stresses induced by thermocycling cause minute microcracks within the material, which consequently can result in increased water sorption and, therefore, increased material degradation. Hydrothermal degradation of PEKK can perhaps occur by breaking the weak bonds in the bridging groups found between the aromatic rings, 41 resulting in deteriorated mechanical performance, as revealed in the decreased flexural strength of the material within the course of the present study. Clinically, this finding could shed light on the liability of PEKK-formed prostheses for deterioration in their flexural strength over a long period of intra-oral service, leading to more forces being transferred to the underlying supporting structures. In addition to the possibility of the initiation of microcracks within the areas of prosthetic bars subjected to tensile or shear stresses, the area of cantilever.⁴³

ASD PEKK attributed to the presence of the Group III revealed the set its structure that kept resistant to
the value. This reflects the hydrothermal stresses up to 5000 thermal Regarding the flexural modulus results, thermal cycling for Group II revealed no significant difference compared to Group I. This could be referred to the rigid nature of aromatic rings with a second ketone group in structure that kept resistant to cycles. On the other hand, when the thermal cycles increased up to 10000 thermal cycles, a significant decrease in flexural modulus was observed, denoting the possibility of hydrothermal stresses to negatively affect the flexural modulus of PEKK at this number of cycles. The decrease in flexural modulus at 10000 cycles could be linked to the probable micro-structural degradation of PEKK, taking place by breaking the weak bridging bonds found between the aromatic rings, with more water sorption and so consequent

degradation of the material.⁴² This could reflect the liability of PEKK based prostheses for more flexural elastic deflections on the long run of intra- oral service, and so transferring more forces to the underlying supporting structures.

Correlating the results of flexural strength and modulus to the results of microstrains on PEKK application as a double crown-retained RPD, it was shown that there was a significant difference between the three groups: group I demonstrated the lowest stress level, while group III demonstrated the highest level. These results could be attributed to the dispute over the shockabsorbing ability of PEKK, but by thermocycling, it undergoes permanent deformation and the propagation of microcraks that lead to the transfer of more stresses to the abutments. $40,43$ These results agreed with a previous study that compared the Ti, Co-Cr, and PEKK bars supported on implants, and it showed that the PEKK bars transmitted the highest stress to supporting structures more than the other bars under dynamic load.⁴⁴

The limitations of this study are that it was applied in vitro, so it didn't replicate the exact complex nature of human supporting structures. Additionally, the load that was applied to the abutments was vertical, not complex, and dynamic like masticatory functions. All n Sharms

Conclusion

PEKK could be considered a thermoplastic polymeric material having a shock- absorbing property, but when subjected to aging by thermocycling, it deforms and its flexural modulus and strength decrease. In addition to that, it transfers more stress to the supporting structures.

Competing interests

The authors declare no conflict of interest.

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Ethics Approval:

 This invitro study was approved by Faculty of Dentistry, Ain Shams University of approvals: FDASU-Rec ER062401)

Availability of Data

All data included in this study are available from the corresponding author upon request.

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