Mechanical Properties of the Hard Sandstone Fracture Surface under Hydrochemical Corrosion

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1. Introduction

Geological conditions for the occurrence of coal seams in China are very complicated, where coal seams on the difficult-to-collapse hard roof in the overlying strata account for approximately one-third [1]. The difficult-to-collapse hard roof of the Tashan Coal Mine in Datong features many strata, large thickness, and high stratum height. It experiences large-area hanging in the borehole operation process, and intense periodic weighting occurs once it collapses, thereby causing coal mining accidents [2, 3]. Strong strata pressure behavior is prominent during the mining period of Carboniferous super-thick coal seam. The field test results showed that this problem cannot be solved only by elevating the supporting strength. Thus, a new challenge is formed for roof control.

In recent years, domestic and foreign scholars have achieved progress in research on the influences of solutions on rock mechanical properties. However, these studies mainly focused on failure characteristics under acidification effect on complete specimens as well as parameter analysis. Xinrong Liu [4] and Wen Yuan [5] studied the parametric change laws and porosity evolution laws of sandstones under the effect of drying and wet cycles at different pH values. The sandstone strength declined with increasing frequency of water–rock cycles. In the early phase of the drying and wetting cycles, the sandstone specimen was significantly influenced and the strength declined to a great extent. Subsequently, the influence of cyclic action was weakened and the strength declined to a relatively small extent. Liang Zhang et al. [6] conducted conventional uniaxial and triaxial compression tests of sandstone specimens under drying and wetting cyclic actions in the acid environment. All mechanical parameters of argillaceous sandstone significantly declined after the first saturation compared with the situation in which the influence of drying and wetting cycles was absent. As the drying and wetting cycles increased,
the decrement gradually slowed down. Wen Yuan et al. [7] conducted uniaxial and triaxial tests of sandstone specimens after different drying and wetting cycles in acid and alkaline environments to determine their internal cohesive force and internal frictional angle. Then, they obtained the change of sandstone shear strength with cycle index. Aggravation of sandstone shear strength was the most serious in the acid environment, followed by alkaline environment and neutral environment. Hongwei Deng et al. [8] conducted a cyclic test of freezing and thawing under $H_2SO_4$, NaOH, NaCl, and water conditions and tested the porosity of sandstone specimens after cycles of freezing and thawing, with the red sandstone in Shandong as an example. After cycles of freezing and thawing at different times, rock porosity distribution underwent evident changes. As the cycle index of freezing and thawing increased, the rock tensile strength declined. Shengjun Miao et al. [9] conducted uniaxial and triaxial compression tests and splitting test of granite specimens after erosion using different acid solutions. After treatment using acid solution, most parameters of granite declined to a certain degree. Moreover, the parameter declined greatly when the pH value was small or when the flow velocity was great.

The porosity and mineral composition of the rock will experience changes to different degrees after chemical corrosion. Therefore, the influence on rock mechanical properties cannot be neglected. Xie et al. [10] conducted conventional triaxial and rheological tests of porous limestone under the natural state and that after chemical corrosion. They also analyzed the influence of chemical corrosion on porosity change and ageing mechanical properties of limestones. X. T. Feng et al. [11] systematically studied the influence of corrosion on rock mechanical properties and believed that chemical corrosion weakened the bonding between rock mineral particles at a microscopic scale and corrosion effect on mineral particles or crystals while causing attenuation of rock mechanical characteristics at a macroscopic scale. N. Li et al. [12] used water–rock reaction rate to express chemical damage process of sandstone caused by corrosion, established the corresponding chemical corrosion-induced damage model, and predicted the strength and elasticity modulus of sandstone under chemical corrosion at different times.

Hydrochemical corrosion can also affect shear strength and crack propagation of the rock. Thus, B. K. Atkinson et al. [13] studied the influences of different water solutions on the crack propagation rate of quartz materials through the experiment. Chemical composition had controlling effect on crack propagation, and ductile damage value under wet conditions was lower and with faster crack propagation than that under dry conditions. Li Peng [14] carried out shear strength tests under different erosion conditions using solutions and analyzed the influence of action on sandstone microstructure. The chemical kinetics method was used by introducing rock hydrochemical damage degree to simulate the time-dependent process of sandstone damage degree under given hydrochemical environmental conditions to predict evolution laws of its shear strength.

These studies on the corrosion effect of solution on sandstone mainly focused on the mineral particles of sandstone specimens, porosity change, and mechanical characteristics. Pad-acid injection through surface hydrofracturing mainly aimed to generate cracks at target strata, thereby changing the mechanical properties of the fracture surface through the corrosion effect of acid liquor on rock fracture surface and reducing rock strata strength. A quantitative expression of change laws of the shear strength of the fracture surface with hydrochemical damage can provide a theoretical basis for site construction. Therefore, a study on strength parameters of rock fracture surface after corrosion must be carried out.

On this basis, hard roof sandstone specimens containing hydraulic fracture surfaces in the Tashan Coal Mine of Datong were taken as the study objects. A magnetic resonance imaging (MRI) test system and 3D laser scanner were used to analyze change laws of sandstone porosity and roughness of its fracture surface before and after corrosion. A shear test of the postcorrosion specimen was carried out, shear strength and corrosion degree of the fracture surface under different conditions were discussed, and rock hydrochemical damage parameters were introduced to quantitatively describe the change process of shear strength of the sandstone fracture surface with damage. The obtained results provided a new idea for quantitatively studying the influence of hydrochemical action on mechanical characteristics of the rock fracture surface and for the hard roof weakening technology by surface hydrofracturing.

2. Water–Rock Chemical Reaction Mechanism

The main mineral compositions in the sandstone hard roof include quartz, feldspar, mica, and rock fragments as seen in Table 1. In the solution forms a complex together with Al, Si, and other ingredients in the specimen, dissolving the Al, Si compounds in the specimen and transforming the rock microstructure. These reactions above are as follows. HF and sandstone minerals form a new substance, which will react with HF as the third mineral [15):

$$HF + H_2O = H_3O^+ + F^-$$
$$SiF_4 + 2HF = SiF_6^{2-} + 2H^+$$

As HF is in incomplete ionization, silicate is dissolved when HF concentration is high; and silicon hydroxide sediment (silica gel) is generated under low concentration of HF. The chemical reaction equations are as follows, respectively:

$$SiO_2 + 6HF = H_2SiF_6 + 2H_2O$$
$$H_2SiF_6 + 4H_2O = Si(OH)_4 + 6HF$$

$$6HF + Si(OH)_4 = H_2SiF_6 + 4H_2O$$

(2)

The solubility product of fluoride and fluosilicate depends on the concentrations of Ca, Mg, and Na ions, etc. in the sandstone. When the solubility product is lower than the solubility limit, no sediment of fluoride and fluosilicate will be generated, with feldspar generating a large quantity of sediments in the residual solution. Reactions of HF with quartz, feldspar, etc. are as follows [16]:

$$HF + H_2O = H_3O^+ + F^-$$
$$SiF_4 + 2HF = SiF_6^{2-} + 2H^+$$

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(2)
3. Methodology

3.1. Testing Process and Method. Rock specimens used in the test were standard 40 mm × 40 mm × 40 mm cubic specimens prepared using the same rock mass. The MRI test of all specimens was carried out, the specimens with approximate porosities were selected, and their porosities were determined to analyze the influence of solution of specimen microstructure. A 3D topographical optical measurement scanner was used to scan the fracture surface, with a number of pixels of 2,000,000, scanning spot pitch of 0.1 mm, and scanning accuracy of 0.02 mm. Scanning was implemented thrice to obtain acceptable scanning data. Software was used to synthesize all scanning results and save the final point cloud data. Subsequently, the specimen was dried in a 105 °C drying oven for 48 h. Then, it was taken out for cooling. The sandstone specimen was placed in solutions with concentrations of 0% (pure water), 1%, 3%, 5%, and 10%, respectively, for 15 d and 30 d soaking, and the soaking process was completely sealed to avoid the contact of hydrochemical solution with external atmosphere during the test process. The test temperature was room temperature, the temperature difference was small in the entire soaking process, and the influence of temperature on the test was not considered. After the specimen was obtained, an MRI test was carried out again to change porosity change laws before and after corrosion. To compare the roughness changes of the same fracture surface, a fixed section was marked as the upper part in the direct shear test, and the mobile section was the lower part. Then, 3D scanning of the fracture surface was carried out. To better compare the change in roughness, the roughness was quantified. The roughness of the intercepted tangent was used to represent the roughness of the structural surface. Roughness along the shear direction was related to shear strength, and the tangential direction is along the shear direction given that roughness has directionality and size effect. After scanning, a contour line was intercepted in the middle of a fracture surface, two lines were intercepted again at the left and right by 1 mm spacing, 39 contour lines were intercepted on one fracture surface, the roughness profile index \( R_p \) on each contour line was calculated, and the mean values of all parameters were taken as data of the fracture surface. A direct shear test was carried out in the end, displacement loading control was adopted, the loading rate was 0.2 mm/min, and the normal stress was 3 MPa. Figure 1 shows the testing process and equipment.

3.2. Preparation of Hydrochemical Solutions. A hydrofluoric acid (HF) solution of 40% concentration was selected for dilution. This test mainly aimed to study the influence of chemical corrosion on sandstone shear strength. Restricted by testing time, solution concentration was enlarged to reflect the corrosion effect of the solution on sandstone within a short time. Solution concentrations were selected as 0% (pure water), 1%, 3%, 5%, and 10%. Thus, the concentration range was large for the convenience of observation.

3.3. Specimen Preparation. The specimens used in the test were obtained from hard roof rocks after Permo-Carboniferous hydrofracturing in the Tashan Coal Mine in Datong, as shown in Figure 2. The particle size was within 0.1–0.5 mm, and the main compositions were quartz, feldspar, clay minerals, and mica. Discreteness of test results existed due to the individual differences of rock specimens. Then, an MRI test was carried out, and the specimens with approximate porosities were considered. A wet-type machining method was used to cut rock specimens containing hydraulic fracture surfaces into cubic specimens with specifications slightly larger than 40 mm × 40 mm × 40 mm. Six faces of each rock specimen were machined using a grinding machine to obtain end-face flatness, perpendicularity, and parallelism of each specimen that satisfy standards recommended by the International Society for Rock Mechanics (ISRM). Sandpapers (600, 800, 1200, and 2000) were used to conduct graded grinding of the surfaces of each specimen. Parallelism errors of two-end faces were controlled within 0.02 mm, and sandstone specimens after machining were maintained in the natural dry state. To guarantee a relative specimen consistency, all specimens were selected from the same rock mass and the integrity of the fracture surface was maintained undamaged. Figure 3 shows the cubic sandstone specimens.

<table>
<thead>
<tr>
<th>Mineral compositions</th>
<th>Clay mineral</th>
<th>SiO₂</th>
<th>Al₂O₃</th>
<th>MgO</th>
<th>CaO</th>
</tr>
</thead>
<tbody>
<tr>
<td>Content (%)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>12.6~18.6</td>
<td>72~75.9</td>
<td>12.7~15.2</td>
<td>0.15~0.24</td>
<td>0.29~0.42</td>
<td></td>
</tr>
</tbody>
</table>
4. Result Analysis and Discussion

4.1. Change Laws of Rock Porosity. Porosity changes of lower-part sandstone specimens measured after the test was completed are shown in Table 2. The table shows that, under the same hydrochemical environment, the longer the corrosion time, the greater the change of the specimen porosity. Under the same corrosion time, high concentration of the solution led to great change in the specimen's porosity.

4.2. Influence of Hydrochemical Solution of Roughness of the Fracture Surface. Lower fracture surfaces of specimens before and after corrosion were scanned. Figure 4 shows the effect of fracture surfaces before and after 15 d of corrosion under concentrations of 0% (pure water), 5%, and 10%. Through a comparison of lower fracture surface scanning graphs under different conditions before and after corrosion, when the solution concentration was 0% (pure water), relief change of each specimen before and after corrosion was minor. Under 5% concentration, particles on the fracture surface fell off after corrosion and the overall relief increased with evident projection and depression. Under 10% concentration, relief on the fracture surface was large with evident corrosion effect, and the framework of the fracture surface was damaged to different degrees. The four corners of the fracture surface were also damaged, thereby influencing the engagement in the closing process.

Figure 5 shows the profile maps of different specimen parts along the shear direction under different conditions. In the 1% solution, the overall relief degree of the tangent line after corrosion was largely identical to that before corrosion.
Table 2: Comparison chart of porosity changes of specimens under different state conditions before and after corrosion.

<table>
<thead>
<tr>
<th>Specimen number</th>
<th>Hydrochemical environment (%)</th>
<th>Specimen status (d)</th>
<th>Initial porosity (%)</th>
<th>Porosity after soaking (%)</th>
<th>Porosity change rate (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0</td>
<td>15</td>
<td>2.7604</td>
<td>4.6299</td>
<td>0.6772</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>15</td>
<td>2.6137</td>
<td>5.1087</td>
<td>0.9545</td>
</tr>
<tr>
<td>3</td>
<td>3</td>
<td>15</td>
<td>2.8173</td>
<td>6.3733</td>
<td>1.2622</td>
</tr>
<tr>
<td>4</td>
<td>5</td>
<td>15</td>
<td>2.1207</td>
<td>7.9512</td>
<td>2.7493</td>
</tr>
<tr>
<td>5</td>
<td>10</td>
<td>15</td>
<td>2.1363</td>
<td>9.0723</td>
<td>3.2467</td>
</tr>
<tr>
<td>6</td>
<td>0</td>
<td>30</td>
<td>2.3703</td>
<td>4.3091</td>
<td>0.8180</td>
</tr>
<tr>
<td>7</td>
<td>1</td>
<td>30</td>
<td>2.9716</td>
<td>5.7341</td>
<td>0.9296</td>
</tr>
<tr>
<td>8</td>
<td>3</td>
<td>30</td>
<td>2.5059</td>
<td>7.6134</td>
<td>2.0382</td>
</tr>
<tr>
<td>9</td>
<td>5</td>
<td>30</td>
<td>2.9578</td>
<td>8.7809</td>
<td>1.9687</td>
</tr>
<tr>
<td>10</td>
<td>10</td>
<td>30</td>
<td>2.7126</td>
<td>9.2914</td>
<td>2.4253</td>
</tr>
</tbody>
</table>

Figure 4: Fracture surface morphologies before and after corrosion.

As the concentration increased, fine particles on the sandstone surface fell off, the rock framework was damaged, new grooves appeared, and the tangent relief degree considerably changed after corrosion. For better comparison of roughness change of the fracture surface, roughness was quantified, and the average values of all parameters were considered the roughness data of fracture surfaces. $R_p$ is calculated using the following formula:

$$R_p = \frac{1}{L} \sum_{i=1}^{N-1} \sqrt{(x_{i+1} - x_i)^2 + (y_{i+1} - y_i)^2}. \quad (6)$$

Hyun-Sic Jang, Seong-Seung Kang, and Bo-An Jang [17] simulated the relational expression between $R_p - 1$ and JRC using exponential equation (7) in 2014 and achieved favorable correlation ($R^2$=0.973).

$$JRC = 65.9 \left(R_p - 1\right)^{0.302} - 9.65. \quad (7)$$

Roughness profile indexes $R_p$ of the contour lines on fracture surfaces of 10 specimens were calculated, their average value was obtained to calculate the roughness coefficient, and the fracture surface roughness obtained after the hydrofracturing test was within 14.39-18.05. Figure 6 shows that the roughness indexes of fracture surfaces after corrosion increased to different degrees as the concentration of hydrochemical solution increased.

Roughness differences before and after corrosion were calculated to more intuitively express roughness change tendencies because the roughness indexes of fracture surfaces were not the same before the test. As shown in Figure 7, a negative value indicates that the roughness after corrosion was smaller than that before corrosion. Under concentrations 0% (pure water) and 1%, the roughness difference value was smaller than 1 when corrosion time was 15 and 30 d. The solution concentration was low, the sandstone corrosion degree was small, and the clastic particles fell off the fracture surface after corrosion. The fracture surface roughness gradually increased with increasing solution concentration. Under 0%-3% concentration, roughness did not have any evident change as the reaction time passed and the water–rock reaction tended to be stable.

Under 5% concentration, the roughness index increased by 22.71% and 33.56% when compared with those under
Figure 5: Continued.
the initial state when corrosion proceeded until 15 d and 30 d, respectively. Under 10% concentration, the roughness index increased by 21.89% and 28.38%. The fracture surface roughness gradually increased with time, and the increase in amplitude was enhanced. A large quantity of HF reacted with minerals in the sandstone, carbonate and clay minerals were
reduced, and rock frameworks were corroded to different degrees.

When the concentration of hydrochemical solution is less than 5%, the chemical reaction is more intense with the increase of concentration. The minerals in the specimen are dissolved in the solution or deposited on the fracture surface, resulting in the gradual increase of the roughness of the fracture surface. When the concentration is 5%, the roughness is the largest. After that, the concentration of solution continues to increase, with the HF content increasing, a large number of cements and silicates dissolved, the structure of sandstone destroyed, new fracture surfaces formed, and the roughness changes reduced.

4.3. Analysis of Specimen Shear Strength. Under a certain normal load effect, the asperities on the fracture surface were engaged to different degrees, and different engagement degrees influenced shear strength and tangential deformation of the fracture surface. Figures 8 and 9 show the direct shear strength curves of specimens after 15 and 30 d of corrosion, respectively. Figure 10 shows the change graphs of shear strengths on fracture surfaces of all specimens after corrosion.

As shown in Figures 8–10, peak shear strengths changed slightly with time after soaking with pure water at 26.67 and 25.97 MPa. Under solution concentrations of 1% and 3%, shear strength declined to a great extent. The strengths declined by 25.5% and 43.2% when compared with that in the pure water circumstance at a concentration of 1%. Shear strength declined to half of the original value as the time passed. When the concentration reached 3%, shear strength reached the minimum value, and then it fluctuated within 5-7 MPa. After 30 d of corrosion, strength slightly declined.

When the concentration was smaller than 3%, the roughness of the fracture surface did not change evidently, indicating that the soaking effect of water on sandstone played a dominant role in the solution. Water molecules would continuously permeate into the sandstone due to the adsorption effect, and clay minerals in the sandstone would experience softening and argillization when contacting water, thereby reducing the shear strength of the fracture surface. In the shear slipping process, the shear strength of asperities constituted one part of the strength of the fracture surface, and the shear stress was mainly the shear force for asperities at the time.

Under 3% concentration, shear strength was the minimum, acidification effect was the best, and roughness slightly changed with time. Cements and clay minerals in the sandstone reacted with HF; thus, the fracture surface became rough. At a microscopic scale, the corrosion effect of hydrochemical solution on the sandstone specimen caused changes to its mineral compositions and internal pore structure. At
4.4. Influence of Hydrochemical Damage on Sandstone Shear Strength. Appropriate damage variables should be selected to study the damage and describe the influence of damage state change and its influence on the mechanical characteristics of specimens, and appropriate state variables are selected from microscopic and macroscopic angles in common definitions of damage variables [14, 18, 19]. The porosities of rock specimens relatively increased after corrosion using hydrochemical solution, and the internal pore structure also changed. Therefore, porosity change is used to establish damage variables.

Damage variables are as follows:

\[ D = \frac{\phi - \phi_0}{1 - \phi_0} \times 100\% \quad (8) \]

where \( \phi_0 \) is initial porosity and \( \phi \) is porosity after corrosion.

Li Peng [14] obtained linear relations of damage variables with cohesive force and internal frictional angle through regression analysis. When the normal force is fixed, the shear strength of the fracture surface is related to damage variables.

\[ \tau = \sigma_n \tan \varphi + c \quad (9) \]

where \( \sigma_n \) is normal stress; \( \varphi \) is basic frictional angle; \( a_1 \) is coefficient under different states.

The chemical and physical actions exist in the water–rock reaction. In the aspect of physical action, the particles fall off the fracture surface, the roughness index is changed due to the dissolution effect of the solution on the rock, and the splitting effect is generated on the micropores. In terms of chemical action, water–rock reaction changes the rock mineral compositions and porous fracture structure. Their combined action finally gives rise to the strength change of the rock fracture surface.

4.4.1. Damage Variables. Damage variables are as follows:

\[ \varphi = a_1D + a_2 \]

\[ c = a_3e^{a_4D}, \quad (10) \]

where \( a_1, a_2, a_3, \) and \( a_4 \) are different coefficients; \( \sigma_n \) is normal stress; \( \varphi \) is internal frictional angle.

In the shear strength study of specimens containing fracture surfaces, the shear strength consists of two parts, namely, JRC-related part and surface friction-related part.

\[ \tau = \sigma_n \tan \varphi_0 + a_5JRC, \quad (11) \]

where \( \sigma_n \) is normal stress; \( \varphi_0 \) is basic frictional angle; \( a_5 \) is coefficient under different states.

In full consideration of damage variable and fracture surface roughness, their relationships with the shear strength of the sandstone fracture surface are as follows:

- **15 d specimens:**
  \[ \tau = 171.9445e^{-101.4002D} + 0.3129JRC (0.9969) \quad (12) \]

- **30 d specimens:**
  \[ \tau = 144.2008e^{-95.6043D} + 0.3129JRC (0.9969) \quad (13) \]

- **All specimens:**
  \[ \tau = 154.4041e^{-101.4002D} + 0.3557JRC (0.9587) \quad (14) \]

Figure 11 can be obtained according to the formula after all test data are fitted. Table 3 shows a comparison between the calculated value and measured value.

5. Conclusions

A self-developed coal rock shear test device was utilized to conduct indoor shear test after corrosion using solutions at different concentrations. Roughness and porosity changes of fracture surfaces were analyzed. The relationship between shear strength of the hydrofracturing surface and sandstone corrosion degree was discussed, and the following main conclusions were drawn:

1. Under sealed hydrochemical environment, corrosion effect was gradually enhanced, and the porosity and roughness of each sandstone specimen gradually increased as the solution concentration or soaking time increased. The fracture surface roughness reached the maximum value under 5% concentration.
Table 3: Specimen shear strengths under different states.

<table>
<thead>
<tr>
<th>Hydrochemical environment (%)</th>
<th>Specimen status (d)</th>
<th>Shear strength</th>
<th>Measured value (MPa)</th>
<th>Calculated value (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>15</td>
<td></td>
<td>26.67</td>
<td>27.666</td>
</tr>
<tr>
<td>0</td>
<td>30</td>
<td></td>
<td>25.97</td>
<td>25.612</td>
</tr>
<tr>
<td>1</td>
<td>15</td>
<td></td>
<td>19.87</td>
<td>17.905</td>
</tr>
<tr>
<td>1</td>
<td>30</td>
<td></td>
<td>14.75</td>
<td>14.495</td>
</tr>
<tr>
<td>3</td>
<td>15</td>
<td></td>
<td>7.49</td>
<td>10.169</td>
</tr>
<tr>
<td>3</td>
<td>30</td>
<td></td>
<td>5.76</td>
<td>6.7845</td>
</tr>
<tr>
<td>5</td>
<td>15</td>
<td></td>
<td>11.18</td>
<td>8.1872</td>
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<tr>
<td>5</td>
<td>30</td>
<td></td>
<td>8.58</td>
<td>8.8302</td>
</tr>
<tr>
<td>10</td>
<td>15</td>
<td></td>
<td>7.4</td>
<td>7.1632</td>
</tr>
<tr>
<td>10</td>
<td>30</td>
<td></td>
<td>6.7</td>
<td>7.9666</td>
</tr>
</tbody>
</table>

Figure 11: Relationships of specimen strength with D and JRC.

(2) When the concentration of the solution was larger than 3%, the peak strength of the fracture surface tended to be stable, the fracture surface roughness was the maximum under 5% concentration, and the shear strength increased with the shear displacement.

(3) The porosity change of the specimen after chemical corrosion was closely related to its strength. Porosity could be used to quantitatively describe the hydrochemical corrosion degree of the sandstone specimen, based on which chemical damage parameters describe the shear strength of the sandstone fracture surface with roughness.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that they have no conflicts of interest regarding the publication of this paper.

References


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