Supplementary file

Microstructural damage evolution in coal under supercritical CO_2–water exposure: A multiscale indentation study incorporating indentation size effect

Bozhi Deng[1], Bingyang Jiao[1], Baisheng Nie[1,*], Changbao Jiang[1,*], Minghui Li[2,3], Yumeng Zhao[4]

[1] *State Key Laboratory of Coal Mine Disaster Dynamics and Control, Chongqing University, Chongqing 400030, P. R. China*

[2] *State Key Laboratory of Intelligent Construction and Healthy Operation and Maintenance of Deep Underground Engineering, Shenzhen University, Shenzhen 518060, P. R. China*

[3] *Guangdong Provincial Key Laboratory of Deep Earth Sciences and Geothermal Energy Exploitation and Utilization, Shenzhen University, Shenzhen 518060, P. R. China*

[4] *Department of Civil and Environmental Engineering, University of Nebraska-Lincoln, Omaha 68106, USA*

E-mail address: dengbz@cqu.edu.cn (B. Deng); jiaoby@stu.cqu.edu.cn (B. Jiao); bshnie@cqu.edu.cn (B. Nie); JCB@cqu.edu.cn (C. Jiang); mhli@szu.edu.cn (M. Li); yzhao52@unl.edu (Y. Zhao).

[*] Corresponding author (ORCID: 0000-0001-5612-4983 (B. Nie); 0000-0003-0520-9754 (C. Jiang))

Deng, B., Jiao, B., Nie, B., Jiang, C., Li, M., Zhao, Y. Microstructural damage evolution in coal under supercritical CO_2–water exposure: A multiscale indentation study incorporating indentation size effect. Advances in Geo-Energy Research, 2025, 17(3): 212-225.

The link to this file is: https://doi.org/10.46690/ager.2025.09.04

Appendix A

The invasion depth was then calculated as follows:

The microstructure surface before soaking treatments

$$RMSD = \sqrt{(\sum_{i=1}^{32\times32}(h_i - h_{ave})/1024)} \tag{1}$$

$$k_0 = (\sum_{i=1}^{32\times32} RMSD)/(32\times32) \tag{2}$$

The microstructure surface after soaking treatments

$$h_0 = (\sum_{i=1}^{n}(\sum_{i=1}^{32\times32} h_i)/(32\times32))/n \tag{3}$$

$$h_e = (\sum_{i=1}^{m}(\sum_{i=1}^{32\times32} h_i)/(32\times32))/m \tag{4}$$

$$m + n = 32\times32 \tag{5}$$

$$h_i = h_0 - h_e \tag{6}$$

In these formulas, the mean value of the RMSD for each microstructure of the coal sample in its original, untreated state was calculated and designated as k_0 (Fig 1); n represents the number of matrices whose RMSD is less than K_0; m represents the number of matrices whose RMSD is greater than K_0; The h_0 represents the height of the matrices selected as the datum surface. h_e represents the height of the matrices selected as the erosion surface; h_i represents the height difference between the datum and the erosion surface, which is the inversion depth.

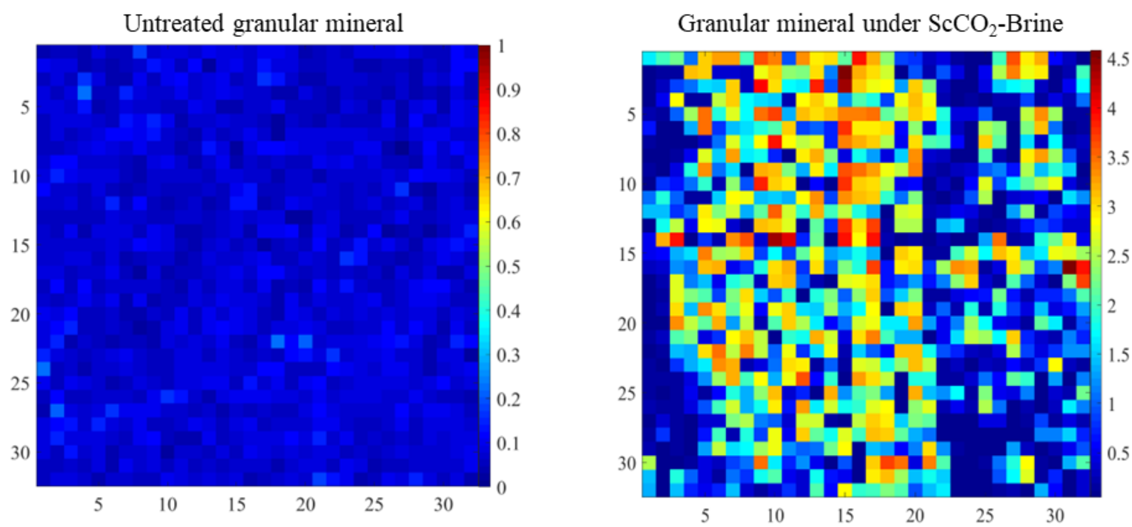

Fig 1. RMSD maps of granular mineral under different treatments

Appendix B

In this context, $P_{\max}$ represents the maximum load achieved during a single loading and unloading cycle. Similarly, $h_{\max}$ denotes the maximum indentation depth, while h_f signifies the permanent indentation depth after unloading. Additionally, h_r is the residual depth of the unloading curve, h_c is the contact depth under peak load, and S represents the slope of the initial section of the unloading curve, also known as the contact stiffness (Fig 2).

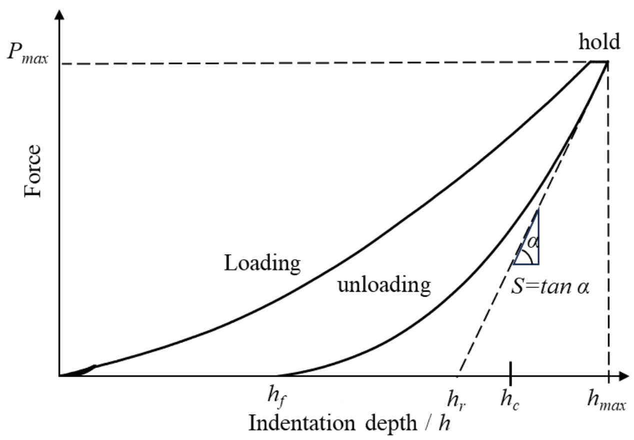

Fig 2. Schematic of typical load-displacement curve.

According to the Oliver-Pharr method (Joslin and Oliver, 1990), the unloading curve can be described by a power function, calculated as follows:

$$F = a(h - h_f)^b \tag{7}$$

In this formula, F represents the load, h represents the displacement, h_f signifies the permanent indentation depth after unloading, and a and b are both fitting parameters.

According to the definition of contact stiffness, it is defined as the tangent slope at the initial stage of the unloading curve. The specific calculation of contact stiffness, denoted by the symbol S, is given by:

$$S = \left(\frac{dF}{dh}\right)_{h=h_{\max}} = ab\left(h_m - h_f\right)^{b-1} \tag{8}$$

In this formula, S represents the contact stiffness, h_m represents the max of displacement.

The contact depth h_c can be calculated using the following equation:

$$h_c = h_m - \varepsilon\left(h_m - h_r\right) \tag{9}$$

Where ε is related to the indenter geometry, for Berkovich indenter, $\varepsilon = 0.7268$, h_r is the residual depth of the unloading curve.

The experiment was performed using a Berkovich indenter, which has the following formula for the contact projected area A_c:

$$A_c = 24.5h_c^{\ 2} + C_1h_c^{\ 1} + C_2h_c^{\ 1/2} + \cdots\cdots + C_8h_c^{\ 1/128} \tag{10}$$

Only the first term in equation (4) may be used in the calculation.

According to the Oliver-Pharr method, the hardness is calculated as follows:

$$H = \frac{F_m}{A_c} \tag{11}$$

In this formula, H represents the hardness, F_m represents the max of load.

Based on contact mechanics, the following formula for the converted modulus E_r can be obtained:

$$E_r = \frac{\sqrt{\pi}S}{2\beta\sqrt{A_c}} \tag{12}$$

where β is a constant related to the shape of the indenter, β for Berkovich indenter is taken as 1.034, and S is the contact stiffness.

The converted modulus represents the combined modulus of the indenter and the sample. The elastic modulus of the sample can be calculated using the following formula. If the effect of the non-rigidity of the diamond indenter on the elastic modulus of the sample is ignored, the converted modulus can be directly used to express the elastic modulus of the material.

$$\frac{1}{E_r} = \frac{1-v^2}{E} + \frac{1-v_i^{\ 2}}{E_i} \tag{13}$$

where E and v are the elastic modulus and Poisson's ratio of the sample, and E_i and v_i are the elastic modulus and Poisson's ratio of the indenter, E_i=1141GPa, and v_i.=0.07.

References

Joslin, D. L., Oliver, W. C. A new method for analyzing data from continuous depth-sensing microindentation tests. Journal of Materials Research, 1990, 5: 123-126.