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A multiscale reconstructing method for shale based on SEM image and experiment data

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Abstract

10 Owing to the presence of multiscale pore structures, characterization of laminated shales is both extremely difficult and substantially different from that of conventional reservoirs, and defies conventional methodologies. In this paper, a multiscale reconstructing method for shale is proposed to generate 3D layer representative elementary volume (/REV)-scale digital-experimental models to characterize the multiscale pore structure of the shale by means of the combination of a large area SEM image, nitrogen adsorption and pressure pulse decay experiment result. In this method an improved multiscale superposition algorithm is introduced to integrate the reconstructed complex models from nanoscale to mesoscale together, and it can preserve the details and main features enormously of each typical component (nanoscale organic pores in organic matter and pyrites, micro-nano inorganic pores and micro slits) in the shale.

15 Especially, to accurately reproduce the realistic morphology for shale, the proposed method uses the experimental pore size distribution and permeability as constrain conditions to adjust and optimize the /REV-scale digital-experimental model. Our proposed method was tested on Longmaxi and Wufeng shale samples, and the reconstructed /REV-scale digital-experimental model are proved to accurately describe the representative structure of the complex multiscale pore space of the typical layer of the shale. The success of this method provides a promising way for reconstructing more realistic model to continuously and systematically characterize the pore (slits) structure from the nanopore-scale to the /REV-scale. It can advance the understanding of the various gas transport mechanisms at different scales and will be helpful for understanding the quality of the shale reservoir.

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Keywords multiscale pore structure, multiscale reconstructing method, IREV, digital-experimental model, shale

1. Introduction

35 The morphology of pore structure can greatly influence the storage and migration of oil and gas in the reservoir, and therefore characterization of pore space is one of the most critical issues in the exploration of reservoirs. Although conventional reservoirs have been characterized through many methods, characterization of shale formations with low permeability remains as a major challenging topic because of the presence of their multiscale pore structure (Javadpour, 40 2009; Darabi et al., 2012; Tahmasebi et al., 2016). Considering that shale rocks are fine-grained, sedimentary laminated rocks with varying compositions, each layer in the shale has its own typical multiscale pore space. Thus the first and key issue of shale characterization is to modelling of the multiscale pore structure for the thin layer of shales. A typical multiscale pore structure in each layer of the laminated shale consists of nanoscale organic pores in organic 45 matter and pyrites, micro-nanoscale inorganic pores and micro slits in inorganic mineral, and all these pores (slits) together play a vital role in fluid flow (Gerke et al., 2015; Saraji and Piri, 2015). However, many conventional methods for modeling of porous media can only construct single-scale or single-component digital core and are unable to completely characterize the multiscale pore (slits) structure of the shale. Therefore it is of significant importance to develop 50 multiscale reconstructing methods to accurately reproduce the typical multiscale pore structure of the shale.

In recent years, several modern imaging techniques, such as focused-ion-beam scanning electron microscope (FIB-SEM) and X-ray computed tomography (CT), have been used directly to construct the three dimensional (3D) digital cores under different resolutions and provide a 55 prominent tool for shale characterization (Lemmens et al., 2011; Curtis et al., 2012; Bai et al., 2013; Saraji and Piri, 2015; Kelly et al., 2016). FIB-SEM and nano-CT can provide

high-resolution imaging of nano-scale pores in shale, however, it costs thousands of dollars and can only scan microscale volumes which is too small to represent bulk properties. Meanwhile, conventional CT and micro-CT can provide larger field of view, but they have relatively low
 60 resolutions (large than $1\mu\text{m}$) and are unable to reflect the nano-scale message of the shale. In a sum, the modern imaging techniques cannot satisfy the requirements of a sufficiently large field (a representative elementary volume, REV) and enough spatial resolution to image the multiscale pore structure of the shale.

On the other hand, there are also a number of reconstruction algorithms that can generate
 65 3D models and reproduce the pore structures by only using one or several 2D images. Among these methods, the process-based methods, the simulated annealing method, the sequential indicator simulation method (SISIM) and various hybrid methods (Bryant and Blunt, 1992; Strebelle, 2002; Øren and Bakke, 2003; Biswal et al., 2007; Hajizadeh et al., 2011, 2012; Yao et al, 2013; Strebelle and Cavelius, 2014) are commonly used to characterize the conventional
 70 reservoir and obtain good result. However, because of the aforementioned significant complicated of shales, they cannot be directly used to reproduce the multiscale pore structure of the shale (Tahmasebi et al, 2016). In view of this, Tahmasebi have combined the cross-correlation-based simulation (CCSIM) method and the iteration-based simulation together to generate a stochastically equiprobable digital core of shales (Tahmasebi et al, 2015). This
 75 hybrid method overcomes the shortcomings of the initial CCSIM method by removing artifacts and refining the spatial connectivity. Meanwhile, Ji (Ji et al, 2018) combines the CCSIM and the three step sample method together and obtains the CCSIM-TSS method to improve the vertical connectivity accuracy of the initial CCSIM. To reconstruct the multiscale pore structure of shales, Gerke et al (2015) has developed a method for integrating 2D categorical spatial data
 80 with different scales into a single dataset by means of stochastic construction algorithms with rescaled two-point correlation functions. However, the paper has only proposed this method and not applied it to actual 3D shale samples. Furthermore, the reconstruction method in this paper is based on low-order statistics and for complex shale pore structure higher-order statistics are required. Later, Tahmasebi et al (2015, 2016, 2018) has develop a three-step multiresolution
 85 reconstruction method to reconstruct a shale model with a bimodal distribution of the pore sizes

(organic pores and few inorganic pores), and the size of the final digital core is only $10*10*10\mu\text{m}^3$. As mentioned above, the complex multiscale pore structure in one thin layer of the shale consists of nanoscale organic pores, micro-nanoscale inorganic pores and micron slits, and the above reconstructed result is too small to contain enough information and characterize the multiscale pore structure of the shale. Furthermore, the reconstructed result is random and its physical properties may have some difference with the measured experimental data. In sum, the reconstruction of multiscale pore structure of shale is not yet a mature research field and requires more research. Aside from the previous studies for stochastic modeling of shale samples, in this paper, we present an alternative approach by which one can produce sufficient large 3D multiscale samples for shale systems while the experimental data (e.g. porosity and permeability) are integrated.

The rest of this paper is organized as follows. In the following section we will describe the definition of IREV, the newly developed multiscale reconstructing method and multiscale gas transport simulation method in detail. Section 3 presents the reconstructing process of the IREV-scale 3D model, which includes nanoscale organic pores, micro-nanoscale inorganic pores, micro slits, organic matter and pyrites, for the shale in Longmaxi and Wufeng Shale Formation based on a high-quality and large area SEM image and experiment data. Also, to validate the proposed method, it presents the comparison of several sets of simulations with the measured experimental data, including pore-size distribution and apparent permeability. The paper is summarized in Section 4, where the advantages of the multiscale reconstructing method are enumerated.

2. Methodology

2.1 IREV (layer representative elementary volume)

Shale rocks are defined as fine-grained, sedimentary laminated rocks with varying compositions. Some thin layers in shale rock may have similar mineral distribution and multiscale pore structure, while other layers may have different mineral distribution and multiscale pore structure. Thus in this paper, we proposed a conception of layer representative

elementary volume (λ REV) for the permeability of the shale, as shown in Fig. 1. λ REV is defined as the minimum sample size that is large enough to represent the heterogeneity which result from the organic matter, pyrites, organic pores, inorganic pores and slits in one layer of the shale rock. The permeability and porosity at the λ REV-scale will remain stationary variation and scale independent. In fact, the laminated shale rocks may have macroscale heterogeneity, which mainly result from the heterogeneity of different thin layers (Region 3). In this paper, our focus is on the multiscale pore heterogeneity in the typical thin layer of the shale rock, and we will reconstruct the λ REV-scale digital-experimental model to represent the multiscale pore structure in the layer of the shale rock. The multiscale pore space in other layers can be studied in the same way.

(a)

(b)

Fig. 1 The sketch of λ REV. (a) λ REV in the shale core sample, (b) The thin layer in the laminated shale core.

2.2 The multiscale reconstructing method

130 Because of the wide pore size distribution varying from nanometer to micrometer in the shale, it is complicated to consider different scale pores and components simultaneously and the combination of various algorithms is necessary to construct the 3D model of the shale. The main idea of the multiscale reconstructing method proposed in our paper is to characterize the 3D structure of different pores of the typical layer of the shale with different techniques and then
 135 integrate them together. It consists of three steps: typical components reconstructing, multiscale superposition and optimization. Each step depends on the result obtained from the previous steps, and improves it. In the following, we will describe them in detail.

2.1.1 Typical components reconstructing

The pore structure of shale usually has strong heterogeneity and complex connectivity.
 140 Moreover, most micro-nanopores exist in organic matter (OM) and pyrites, and thus the structure of OM and pyrites can significantly affect the distribution of pores. Thus the typical components of the shale reconstructed in this paper are pores in the OM and IOM, slits, pyrites, and organic matter. By analyzing the characteristics of each components, we can find that some components, such as organic pores and organic matter, have complex structure and their
 145 connectivity is good. On the contrary, some components, such as inorganic pores, slits and organic matter, have relatively simple structure and their proportions are small. Thus, to save computational time and improve the computational accuracy, the CCSIM-TSS and statistical analysis algorithm are combined together to reconstruct the 3D models of each typical component.

150 Concretely speaking, we use the CCSIM-TSS algorithm to generate the 3D organic pores and organic matter models. The CCSIM-TSS algorithm is a stochastic approach that integrates the cross correlation based simulation (CCSIM) and the three step sampling method together to produce more accurate realizations for the given heterogeneous porous media (Ji et al, 2018). The advantage of this method is that it can reproduce the complex connectivity of the pore space
 155 both in the horizontal and vertical direction and give sufficient approximation to the properties of the 2D images. Fig. 2 depicts the specific reconstruction procedure of the CCSIM-TSS

method. In the beginning, a 2D image that is typical enough to reflect the complexity and heterogeneity of the shale is selected. Then the right, back, left and front frames of the 3D model are reconstructed by the CCSIM. Finally, the internal structure in the vertical direction is generated plane by plane with the conditional data which is extracted in terms of the multiple-point connectivity probability function, the pore-size distribution and the porosity. The final reconstructed model can be obtained by stacking all the planes together.

Fig. 2 The process of the CCDIM-TSS algorithm

In our study, the statistical analysis algorithm is used to generate the 3D models for pyrites, slits and inorganic pores. The procedure of the statistical analysis algorithm is: 1) the diameter (length) distribution of the component is calculated based on the global SEM image at proper resolution; 2) random spheres (lines) whose sizes follow the distribution of the diameter (length) are constructed and the spheres (lines) are scattered randomly in the 3D model.

2.1.2 Multiscale superposition

As mentioned above, the shale samples have multiscale pore sizes which make the reconstruction more complicated. Meanwhile the shale reservoirs have very low permeability, even tiny pore structures play a vital role and must be considered in the generated model. In this study, to capture the pore structure at various scales, we proposed an improved multiscale superposition algorithm to integrate 3D models with different scale together and obtain the IREV-scale model considering the multiscale pores.

During superposition process, the resolution and the physical size of different component models should keep the same. First, the macro-component model with low resolution is refined into an image with high resolution using a cubic spline interpolation technique. During this procedure, each voxel in macro-component model is refined into $a1*a1*a1$ voxels ($a1$ is the ratio of resolution between the low and the high resolution model). It should be pointed out that

the cubic spline interpolation technique can keep the refined images smooth and preserve the details and main features of each component very well. Second, we stack $b1*b1*b1$ nano-component model with high resolution together to obtain a composition image with the same size of the macro-component model using the combination algorithm and image conversion, as shown in Fig. 3 ($b1$ is the size ratio between the macro-component model and the nano-component model). During stacking by image conversion of the origin nano-component model, we can keep the interface of the two adjacent digital core consistent and thus keep the connectivity of the nano-component in the composition nano-component image.

Fig. 3 The schematic illustration of the combination algorithm. 1, 2, 3, 4, 5 and 6 are the number of the surface of the nanopore digital core

Then a multicomponent superposition algorithm is proposed to integrate the 3D models for different component in shale. The detailed steps of the superposition operations for shale samples in this paper are as follows:

$$\begin{aligned}
 \Omega &= \Omega_1 + \Omega_2 + \Omega_3 + \Omega_4 + \Omega_5 \\
 \Omega^{skeleton} &= \Omega_1^{skeleton} + \Omega_2^{skeleton} + \Omega_3^{skeleton} + \Omega_4^{skeleton} + \Omega_5^{skeleton} \\
 \Omega^{OM\ pore} &= \Omega_1^{OM} + \Omega_2^{OM\ pore} \\
 \Omega^{IOM\ pore} &= \Omega_1^{skeleton} + \Omega_2^{skeleton} + \Omega_3^{skeleton} + \Omega_4^{skeleton} + \Omega_5^{IOM\ pore} \\
 \Omega^{Pyrite} &= \Omega_1^{skeleton} + \Omega_2^{skeleton} + \Omega_3^{Pyrite} + \Omega_4^{skeleton} + \Omega_5^{IOM\ pore} \\
 \Omega^{slit} &= \Omega_1^{skeleton} + \Omega_2^{skeleton} + \Omega_3^{skeleton} + \Omega_4^{slit} + \Omega_5^{skeleton}
 \end{aligned} \tag{1}$$

where $\Omega, \Omega_1, \Omega_2, \Omega_3, \Omega_4, \Omega_5$ indicate the shale multiscale model, the organic matter model, the organic pores model, the pyrite model, the slits network and the inorganic pores model, respectively. For the superposed shale multiscale model, $\Omega^{skeleton}$, Ω^{Pyrite} , $\Omega^{OM\ pore}$, $\Omega^{IOM\ pore}$ and

200 Ω^{slit} represent the skeleton, the pyrites, the organic pores, the inorganic pores and the slits, and their values are 0, 1, 2, 3 and 4. Fig.4 presents an example of the superposition of the organic matter model, the organic pores model, the inorganic pores model.

Fig. 4 The schematic illustration of the superposition algorithm

205 Based on the superposition algorithm and the voxel refinement algorithm, a multiscale 3D model with multiscale pores and various components can be obtained. It should be emphasized that this multiscale model is not binary image, and different components have different values.

2.1.3 The optimization algorithm

In spite of the high accuracy of the reproduced statistical properties of the CCSIM-TSS and statistical analysis algorithm, the reconstructed multiscale models are random and their physical properties may be different from the experimental data (the pore-size distribution and the permeability). Therefore the reconstructed multiscale models should be optimized by using nitrogen adsorption and pressure pulse decay experiment result as constrain condition.

215 The specific procedure of the optimization algorithm is arranged as follows: First, 10 multiscale realizations are generated, and the pore size distribution of each realization is calculated. Then they are compared with the result from the nitrogen adsorption experiment, and the one that most approach the experiment data is chosen.

Second, the model obtained in the above step, named as initial multiscale model, is optimized to minimize the difference between it and the nitrogen adsorption experiment data by using the algorithm based on an improved simulated annealing algorithm (Mo et al, 2016; Ji et al, 2018). The objective (energy) function of the initial system E_0 is defined as the absolute difference of the pore size distribution between the initial multiscale model and experiment. Then new trial model can be obtained by exchanging the boundary points of two pores belonged

to two different sets selected based on the D3Q19 model and AB algorithm (Yi et al, 2017). One set contains the pores whose number in the trial model is larger than that measured by experimental data, and in contrast the other set contains the pores whose number is smaller than that of experimental data. Then, the objective functions of the trial model is calculated, and Metropolis criterion is used to determine whether the trial configuration is accepted. The above process is repeated until the objective function is smaller than the given value, as shown in Fig. 5. Finally, we can obtain the multiscale 3D model whose pore size distribution is close to the nitrogen adsorption result.

Third, the apparent permeability of the above 3D model is calculated by the multiscale gas transport simulation method, which will be introduced in detail in the following section. It should be pointed out that during using the multiscale gas transport simulation method, we assume that in each grid of the inorganic matter, the tortuosity of the inorganic pores is the same, and its value is determined by the permeability obtained from pressure pulse decay experiment. Up to now, we obtain the final multiscale digital-experimental model of the shale.

Fig. 5 The flow chart of the main procedure of the optimization algorithm

240 2.3 The multiscale gas transport simulation

Based on the multiscale digital core, the apparent permeability k can be calculated by the multiscale gas transport simulation introduced in our paper. The proposed method consists of three steps: First, the permeability distribution for organic matter is calculated based on the proposed model by Jiang (Jiang et al, 2016). Second, the permeability for the inorganic pores and slits are calculated. Third, the finite volume method is used to simulate the apparent permeability based on the multiscale digital core (Wu et al, 2016).

In the OM, the model proposed by Jiang (Jiang et al, 2016) is used to calculate the permeability:

$$\begin{cases} K_{om} = \frac{2R_{nt}\mu\phi_f}{3\rho_{avg}\tau} \left(\frac{d_m}{2R_{nt}} \right)^{D_f-2} \sqrt{\frac{8M}{\pi RT}} + K_{d,om} \left(1 + \sqrt{\frac{8\pi RT}{M}} \frac{\mu}{R_{avg}p_{avg}} \left(\frac{2}{\alpha} - 1 \right) \right) \\ R_{nt} = \left(\frac{1}{N} \sum_{i=1}^N \left(\frac{1}{r_i} \right)^{D_f-3} \right)^{-\frac{1}{D_f-3}} \\ R_{avg} = \left(\frac{1}{N} \sum_{i=1}^N \frac{1}{r_i} \right)^{-1} \end{cases} \quad (2)$$

250 where $D_f, N, r_i, \tau, \phi_f$ are the surface fractal dimension of organic pore, the throats total number, the tortuosity, the radius of throat i , and the flowing porosity. M, μ are the molar mass and the viscosity of the gas, R is the gas constant, T is the experimental temperature.

In the inorganic matter, the inorganic pores permeability K_{iomp} is estimated with the theoretical model proposed by Darabi (Darabi et al, 2012). Especially, here we assume that the D_f is 2, and the tortuosity of inorganic pores t will be determined by the permeability obtained from the experiment.

$$K_{iomp} = \frac{2R_{av}mMf}{3RT r_{avg}t} \sqrt{\frac{8RT}{pM}} + K_{d,iomp} \left(1 + \sqrt{\frac{8pRT}{M}} \frac{m}{R_{avg}p} \frac{2}{\alpha} - 1 \right) \quad (3)$$

where f, t, R_{av} are the porosity, tortuosity, average pore radius of the inorganic pores.

In the inorganic matter, the permeability of the slits K_{slit} is computed based on the theoretical model as (Singh et al, 2014)

$$K_{slit} = \frac{\phi \mu h}{3\tau} \left(\frac{h}{4\mu} + \frac{8}{\pi p_{avg} M} \sqrt{\frac{2MRT}{\pi}} \right) \quad (4)$$

where f, t, h are the porosity, tortuosity and the width of the slits. Especially, we assume that the width of slits don't change with the confining pressure.

In terms of the above equations for different pores (slits), the finite volume method is then used to simulate the multiscale gas transport. It should be pointed out that each grid size during simulation should be larger than the representative elementary volume for the spatial heterogeneous feature of organic pores and inorganic pores. The numerical tests are carried out under various average pressures.

3. Results and Discussion

To test the above proposed methodology, the shale rocks from Longmaxi and Wufeng shale formation of Lower Silurian in China, denoted as sample A and sample B, are used for reconstruction.

3.1 Sample A

The scanning electron microscopy (SEM) image of the typical thin layer of the sample A is used to investigate the spatial distribution and the shape feature of the multiple pores, as shown in Fig.6. The scanning area is $400 \mu\text{m} \times 400 \mu\text{m}$ with a maximum resolution of 4.0 nm. During reconstruction of the /REV-scale model, we have to get the characteristics of various pores at different scales, thus we should take images at different resolutions. We use a small high-resolution (4nm) image to capture the characteristics of nanoscale pores and a large low-resolution (about 100nm) images to capture the micron scale component.

Fig. 6 SEM image of the typical layer in sample A (400*400 μm^2)

3.1.1 3D models for typical components

At first, the 3D models for typical components of shale sample A are reproduced respectively. Inspecting Fig.6, we can extract five typical components (organic pores, inorganic pores, slits, pyrites and organic matter) from the SEM image. In the following we will reconstruct the 3D models for each component.

● Pores in OM

Organic matter has abundant nanoscale pores that connect the microscale pores and slits, and thus it is critical to construct an accurate interconnected nanoscale pore network. In order to accurately reproduce complex pore structure, the 3D models of organic pores are reconstructed by the CCSIM-TSS method.

To extract the accurate organic pore distribution, two typical area of kerogen aggregate (Fig. 7) are taken from the SEM image at the resolution of 4 nm. The modeling process of organic pores models is shown in Fig. 7 and the details are as follows.

(1) RES is the representative elementary surface, at which the porosity reaches a stationary variation and is converged. Inspection the SEM image, we can find that the distribution of organic pores is very heterogeneous in small region. In order to obtain the RES for the organic pores, we choose the four corners of the organic pore images, then expand the objective region from the selected points and calculate the porosity in the sub-square. The variation of the porosity will decrease gradually as the area of the sub-square increases. Inspecting Fig. 7, we can obtain that the sizes of the edge of the RES for the type I and type

II pores in OM are approximately 600 and 800, respectively.

305 (2) The RES images for the type I and type II organic pores with 600 pixels and 800 pixels are
exacted. The images are segmented to exact pores, and the final images are shown in Fig.
7.

(3) The CCSIM-TSS method is then used to generate the 3D realizations of pores in the OM.
The values of the data in the 3D models are 0 and 1, where 0 represents the organic matter
310 and 1 represents the organic pores. The final reconstructed organic pore models are plotted
in Fig. 7. Fig. 7 shows that the global pore structures of the 2D images are reproduced very
well and the connectivity of the pores is also very good. Therefore, we can conclude that
the CCSIM-TSS method can preserve the global complex pore structure both in vertical
and horizontal direction, which is very important for predicting the permeability accurately.

315 Finally, it should be pointed out that the type I organic pores are only distributed in the
zone marked by the pink circle of SEM image, and type II organic pores are distributed in other
organic matter except the marked zone.

**Fig.7 SEM imaging of the shale rock that depicts organic pore images and the model process of pores
structures in OM**

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- Spatial Distribution of OM

Nano organic pores are distributed in organic matter, and the structure of organic matter
has a significant effect on the connectivity of nanoscale organic pores. Thus in this section, we
325 will reconstruct the distribution of organic matter by using the SEM image at the resolution of

80nm using the CCSIM-TSS method.

First, the RES image of the organic matter is obtained as discussed in Subsection 3.1.1, as shown in Fig. 8. By calculation, it can be found that the content of organic matter will not significantly vary when the area of the 2D image is large than 3500×3500 pixels. Thus the number of pixels on the edge of the RES for organic matter is approximately 3500 pixels.

Like the reconstruction of pores in organic matter, the CCSIM-TSS method is also used to generate the 3D model of organic matter. The final organic matter model is shown in Fig. 8. The size of the reconstructed model is $320 \times 320 \times 320 \mu m^3$. It should be pointed out that 0 in the model is matrix and 1 is organic matter. It can be seen from the figure that the morphological feature of the 2D images was reconstructed and the connectivity of the organic matter is also well. The connectivity and the distribution of the organic matter model are very important because they can affect the distribution and the connectivity of organic pores in organic matter.

It should be emphasized that the regions which are filled with the type I organic pores have been recorded during reconstructing the OM model. When the OM model and the organic pores models are superposed together in the following section, the type I organic pores are only filled in the recorded region (marked by the pink circle in Fig. 8).

Fig. 8 The distribution of OM in the SEM image of the shale sample and the model process of OM structures

● Pyrites

Pyrites in IOM are scattered independently in the SEM image, and the connectivity of the pyrites is bad. Thus the model for the pyrites is constructed by the statistical analysis method. Firstly, the characteristics of the pyrites are investigated in the global SEM image with the resolution of 80nm. Similarly, the RES analysis and the distribution of diameter of pyrites are calculated.

We can obtain that the proportion of pyrites on the global SEM image is 0.9%, the RES for

pyrites is approximately 2500 pixels ($200\ \mu\text{m}$) and the distribution of the diameter of pyrites is shown in Fig.9. A large portion of pyrites has diameters that are smaller $2\ \mu\text{m}$. Then the statistical analysis method is used to construct random spheres whose sizes follow the distribution of the diameter, and the spheres are scattered randomly in the model, as shown in Fig.9. For convenience of superposition with OM models, the size of the reconstructed model for the pyrites is $320*320*320\ \mu\text{m}^3$, and the resolution is 80nm.

Fig.9 Modeling of pyrite parts.

● Pores in IOM

In this subsection the pore-size distribution of the inorganic pores is investigated. The inorganic pores are extracted in a relatively lower resolution (20nm) than that for organic pores. Fig.10a shows an example of the inorganic pores in part of the SEM image. It can be seen that the inorganic pores are bigger than organic pores, and they are scattered in the matrix of shales. The RES analysis and the pore-size distribution of inorganic pores are calculated based on the whole SEM image with the resolution of 20nm. The RES for pyrites is approximately 1500 pixels ($30\ \mu\text{m}$). It can be seen from Fig. 10c that most inorganic pores are smaller than 500nm and a large portion of inorganic pores is about 80nm. The porosity of inorganic pores on the whole SEM image is 0.03%. Like pyrites, the statistical method is also used to construct random spheres whose sizes follow the pore-size distribution, and the inorganic pores are scattered

randomly in the 3D model. Similarly, for convenience of superposition with other models, the size of the inorganic pores model is $320 \times 320 \times 320.0 \mu\text{m}^3$, and the resolution is 20nm. Note that the inorganic pores are too small to be seen in the model of $320 \times 320 \times 320.0 \mu\text{m}^3$, here we only plot a small part of the whole model, as shown in Fig. 10d.

Fig. 10 (a) An inorganic pore image in the SEM image of Sichuan shale sample ; (b)The pore size distribution of pores in the IOM; (c) A small part of the final model for inorganic pores

● Slits

Slits also play a vital role on the gas permeability of the shale. In this study, the distribution of slits is strongly heterogeneous and the apertures of slits are very small. Thus, the distribution of slits is observed at a relatively high resolution (10nm). Then we calculate the RES, the distribution density and the length distribution of inorganic slits in the whole SEM image with resolution of 10nm, as shown in Fig.11.

The RES for slits is approximately $180 \mu\text{m}$. The average aperture of slits in the SEM image is 35nm, and the density of inorganic slits is $30 / \mu\text{m}^2$. The length distribution is shown in Fig.

11. Then the statistical method is used to generate the slit network. Random lines (slits) whose lengths follow the length distribution are scattered randomly in the model. The aperture of each slit is set to be the average aperture (35nm). The size of the final model is $320 \times 320 \times 320 \mu m^3$ and the direction of slits is also random.

Fig. 11 (a) An slit image in the SEM image of Sichuan shale sample; (b) The RES analysis of the slits; (c) The length distribution of the slits; (d) The final model for slits

3.1.2 IREV-scale Model Construction

Up to now, five different models for typical components of shale sample are obtained. The models represent shale samples from microscale and nanoscale formations, respectively. Now we consider merging the five models with different size and resolution together (see Fig. 12). First, the kerogen solids (black) and organic pores (white) on the nanoscale image (Fig. 7) are merged with the organic matter model (Fig. 8) and the inorganic pores model. It should be noted that the nanoscale image of organic pores is only filled in the organic matter in the organic matter model and the inorganic pores are only filled in the skeleton in the organic matter model. Especially, the models for type I pores in OM are only filled in the marked regions of OM, and the models for type II pores in OM are filled in other OM regions. Then the models with organic matter and pores in the OM and IOM are integrated with the models for the pyrites and slits

together based on the multiscale superposition algorithm. From the reconstructing process of the
 405 3D models for each component, we can obtain that each model can represent the characteristics
 of the corresponding component. Thus the final superposition model can characterize the
 multiscale pore structure of the typical thin layer of the shale. Moreover, from the RES analysis
 for each component in the SEM image, we can obtain that each reconstructed model can
 represent the characteristics of the corresponding component in the thin layer, and thus we call
 410 the final superposed model as the *REV*-scale model. Inspecting Fig. 12b, we can find that it has
 multiscale pore structures.

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Fig. 12 The process of merging various scale models together (a); An overview of the final *REV*-scale model. The green particles are organic matters, the yellow spheres are pyrites, and the red lines are slits (b).

3.1.3 Optimization and Validation

420 As mentioned before, the final IREV-scale models reconstructed in the above section are random and their physical properties may be different from the measured data (the apparent permeability and the pore-size distribution). Therefore the IREV-scale models will be optimized with experiment data in the following.

Firstly, we should obtain the experiment data. The permeability of the dry columnar sample
425 can be obtained by pulse-decay permeability measurement instrument at the temperature of 38 °C (Pan et al, 2018). The permeability at the pressure of 0.5MPa, 1.0MPa, 1.5MPa and 2.0MPa is measured. Meanwhile, the nitrogen adsorption experiment is used to measure the pore distribution of the shale sample.

It should be noted that the dry columnar sample used to measure the permeability contain
430 several thin layers in the vertical direction, thus here we assume that all these layers have similar multiscale pore structures with that of the IREV-scale models reconstructed in our paper. Thus the experimental permeability can be directly used to optimize the IREV-scale model.

With the above experiment result, the model is optimized by using the optimization algorithm of the multiscale reconstructing method, as shown in Fig. 13. 10 realizations are
435 generated, and the second one that is closest to the nitrogen adsorption result is chosen. Then after 185 iterations, the chosen model is optimized to minimize the difference between it and the experiment data. The result in Fig. 13 shows that the optimized model reconstructs the pore-throat size distribution of the real samples very well. Finally, the multiscale gas transport simulation method is used to calculate the apparent permeability of the model. During the
440 simulation, the model has 40*40 *40 grids, and each grid size is bigger than the representative elementary volume of OM and IOM. It should be pointed out that during using the multiscale gas transport simulation method, we assume that in each grid of the IOM, the tortuosity of the inorganic pores is the same. As mentioned above, we assume that the other layers in the shale core rock have the similar property of the IREV-scale model, thus the pulse-decay permeability
445 measurement result can be directly used to calibrate the model. Here the tortuosity of the inorganic pores is determined by the permeability obtained from experiment at 1.5MPa, and its value is 1.5. The optimization part takes 48 CPUs per realization. Up to now, we obtain the final

/REV-scale digital-experimental model for the shale sample.

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Fig. 13 The optimization of the /REV-scale model.

Now the geometrical and topological features of the /REV-scale digital-experimental model are evaluated.

First, the apparent permeability of the digital-experimental model at other pressures are calculated by using the multiscale gas transport simulation method, as shown in Fig. 14a (red lines). Fig. 14a demonstrates that the apparent permeability of the reconstructed model agrees very well with that from experiments when the average pressure is bigger than 1.0MPa. The

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difference of apparent permeability at 0.5MPa is relatively large, and this is because that the apparent permeability of IOM is calculated with the assumption that D_f is 2. Moreover, the resolution of the SEM image we use is 4nm, and the pores smaller than 4nm are not considered here, which may affect the apparent permeability at low pressure.

The good agreement of the apparent permeability when average pressure is bigger than 1.0MPa validated the reconstructed μ REV-scale digital-experimental model and the multiscale gas transport simulation method.

We perform a comparison of the pore size distribution between the μ REV-scale digital-experimental model and the nitrogen adsorption experiment. Fig.12b shows that there is a reasonable match.

Finally, we examine the relationship between the absolute permeability and the sample size in the μ REV-scale digital-experimental model. To calculate the smallest μ REV for the model, we choose eight corner points of the 3D model, then expand the research region from the source points of the μ REV-scale digital-experimental model, and calculate the absolute permeability in the sub-blocks. As Fig. 15 demonstrates, the absolute permeability fluctuates strongly when the volume is very small, and then reaches a plateau around the volume of $280 \times 280 \times 280 \mu m^3$. This means that the smallest μ REV size is $280 \times 280 \times 280 \mu m^3$.

Fig. 14 Comparison of the permeability (a) and the pore size distribution (b) obtained from experiment and μ REV-scale models of the shale sample A.

Fig. 15. Dependence of absolute permeability variation on the sample (image) size.

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3.2 Sample B

The second example, sample B, a typical sample from Wufeng Marine Shale Formation of Lower Silurian in the Sichuan Basin, is also used to test the described methodology in this paper. The scanning electron microscopy (SEM) image of the typical thin layer of shale sample B is shown in Fig.16. The scanning area is $400\ \mu\text{m} \times 400\ \mu\text{m}$ with a maximum resolution of $4.0\ \text{nm}$, too. Different from sample A, the multiscale pore structure for sample B is more complicated, and there are three different types of organic pores in this sample. Its digital core is reconstructed using the proposed method in this paper, as shown in Fig. 16. The size of the reconstructed model is $340 \times 340 \times 340\ \mu\text{m}^3$. It can be seen from the figure that the multiscale digital core provides the main pore networks that are connected by the small nanoscale pores.

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In order to evaluate the reconstructed model, the pore size distribution and the permeability are calculated and compared with the results obtained from experiments. As shown in Fig. 17, the apparent permeability of the realization and the pulse-decay permeability measurement are compared. As sample A, the apparent permeability of experiment in 1.5MPa is used as the reference value to determine the tortuosity of the inorganic pores in the multiscale digital core

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and the tortuosity is 5.3. The figure demonstrates that the values of apparent permeability from the simulations agree well with the results from experiments. Fig. 17 also displays the pore size distributions of the realization and the nitrogen adsorption experiment. It is clear that there is a very good agreement between the realization generated by the proposed algorithm in this paper and the experiment. Thus it can be obtained that the method proposed in our paper can produce higher quality realization.

Fig. 16. The reconstruction of the multiscale digital core for the Sample B.

Fig. 17 Comparison of the permeability (a) and the pore size distribution (b) obtained from experiment and /REV-scale models of the shale sample B.

From the above study, we can conclude that /REV-scale digital-experimental model can be successfully reconstructed by using a large area SEM image, the experimental pore-size distribution and permeability. It should be noted that the SEM image should be large enough to contain the characteristic of the multiscale pore structure of the typical thin layer of the shale. The pore-size distribution and permeability are obtained from the nitrogen adsorption and pressure pulse decay experiment. The process of the reconstructing of the /REV-scale digital-experimental core can be summarized as follows: First, the RES (representative elementary surface) scale images of the typical components, such as pores in the OM and IOM, slits, pyrites, and organic matter, are extracted from the SEM image at different resolutions. Then the 3D models for each component are reconstructed by means of the combination of CCSIM-TSS and statistical analysis algorithm. Second, we use a novel multiscale superposition algorithm to integrate the 3D models for different components with different scale and physical size together to obtain the /REV-scale model. Finally, experimental data of the permeability and pore size distribution are integrated to optimize the reconstructed result and obtain the final /REV-scale digital-experimental core. All the steps are brought together to successfully simulate complex and multimodal shale samples, as shown in Fig. 18. In particular, it should be pointed out that other layers of the shale sample rock discussed in this paper is assumed to have the similar property as the typical layer we choose to reconstruct /REV-scale model. Thus it is reasonable to use the permeability obtained from the pressure pulse decay experiment directly to optimize the reconstructed model in the final step.

Fig 18. The schematic illustration of the process of reconstructing the /REV-scale digital-experimental model

4. Summary

In this paper, the multiscale reconstructing method, including the CCSIM-TSS algorithm statistical analysis algorithm, the multiscale superposition algorithm and the optimization algorithm, is used to reconstruct the /REV-scale digital-experimental model considering the multiscale pores. Some important conclusions are obtained as follows:

- 1) The representative 3D models of the typical components are reconstructed by the using the RES analysis, the CCSIM-TSS and statistical analysis algorithm. The RES analysis can help us to obtain the 2D representative image of each component. The combination of the CCSIM-TSS and statistical analysis algorithm can accurately reproduce the 3D characteristics of the typical components and meanwhile save lots of reconstruction time. Using the improved multiscale superposition algorithm, we superpose the five 3D models from nanoscale to mesoscale together and during this process the characteristics of the

545 inorganic pore, the organic pore, organic matter, pyrites and slits are preserved in good condition. The optimization method integrates the experiment result into the reconstructing process and helps us to produce higher quality realizations that are very close to the realistic sample.

2) The λ REV scale is introduced in this paper to describe the sample size that is large enough
550 to represent the heterogeneity of organic pores, organic matter, pyrites, inorganic pores and slits in the typical layer of the shale rock. With the multiscale reconstructing method, the λ REV-scale digital-experimental model is accurately reconstructed by means of the combination of a large area SEM image, nitrogen adsorption and pressure pulse decay experiment result. The success of this method resolves the duality between a sufficiently
555 large sample size and a sufficient spatial resolution which up to now has seemed insurmountable to modern imaging techniques. This model can help us to understand the multiscale gas transport mechanisms in the shale rock and will be helpful for understanding the quality of the shale reservoir.

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Highlights

- A multiscale reconstructing method for shale is proposed to generate μ REV-scale digital-experimental models.
- The method uses an improved multiscale superposition algorithm to integrate the reconstructed models from nanoscale to mesoscale together.
- The method integrates experiment results into the reconstructing process to produce higher quality realizations.
- The μ REV-scale model can continuously and systematically characterize the pore structure from nanopore-scale to μ REV-scale.