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Journal of Natural Gas Geoscience 1 (2016) 203−211

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Original research paper

Reservoir diagenesis research of Silurian Longmaxi Formation in Sichuan Basin, China☆

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Received 7 May 2016; revised 15 June 2016
Available online 21 August 2016

Abstract

The reservoir diagenesis of Silurian Longmaxi Formation in Sichuan Basin was studied based on a large number of thin section identification, scanning electron microscopy analysis, X-ray diffraction tests, and some other experiments. Seven diagenetic processes were identified, including compaction, cementation, clay mineral transformation, replacement, dissolution, organic matter thermal maturation, and tectonic disruption. Three kinds of cements (quartz, carbonate and sulfide) were recognized, while the source material of quartz cements and the main factor of forming abundant carbonate cements were summed up. According to the single well analysis of the Well N3, it shows that the best, the suboptimal and the none shale reservoir sections were subjected to different diagenetic transformations. As to best shale reservoir, except for compaction, all the main inorganic diagenesis were significantly related to organic matter maturation. Through comprehensive analysis of diagenetic indicators, it is observed that the reservoir has already been in period B of middle diagenetic stage to late diagenetic stage. The inorganic diagenesis has a significant impact on shale reservoir, because it not only controls the conservation, development, and evolution of porosity, but also the mechanical property and the adsorption capacity of rocks. The organic diagenesis is the source material of shale gas, and it generates a large number of nanoporosity in organic matter, which increases the total porosity and the adsorption capacity of the reservoir.

Keywords: Sichuan Basin; Longmaxi Formation; Shale reservoir; Diagenesis

1. Introduction

Silurian Longmaxi Formation is one of the major black rock series of the Paleozoic layers in the Sichuan Basin. Nowadays, with the development of shale gas exploration, it became the main gas producing shale in the Sichuan Basin [1]. Previous studies showed that the best reservoir in the Longmaxi shale is located in the bottom of the layer, with the characteristics of high GR value (higher than 150API), high TOC content (greater than 2%), and high gas content (greater than 2 m^3/t). It also had complex mineral composition, various rock types, poor pore structure, complex pore types, low porosity, and extremely low permeability and heterogeneity [1−10]. All these geological features show a strong particularity and complexity compared to conventional reservoirs.

Some research has been carried out, but specific work on diagenesis of Longmaxi Formation has not been put into practice yet. Aplin et al. [11] mentioned that many important physicochemical properties of mudstones are

☆ This is English translational work of an article originally published in *Natural Gas Geoscience* (in Chinese).The original article can be found at: 10.11764/j.issn.1672-1926.2015.08.1547.

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Peer review under responsibility of Editorial Office of *Journal of Natural Gas Geoscience*.

http://dx.doi.org/10.1016/j.jnggs.2016.08.001

strongly influenced by the mineralogy and size of the deposited grains, and by diagenetic changes. Therefore, such work will not only expand the understanding of the shale reservoir's geology and clarify the main diagenetic alterations and diagenetic stage, but it can also help find out the controlling factors and degree of diagenesis in terms of shale reservoir. Finally, deeper knowledge aids to provide reliable geological basis for shale gas development. In this paper, we focus on the diagenesis of Longmaxi Formation through the samples from coring wells and outcrops within the basin. Core samples were extracted approximately every meter, and the same set of sample were used in the same experiments including thin section identification, scanning electron microscopy, x-ray diffraction, organic carbon analysis, vitrinite reflectance measurement (equivalent vitrinite reflectance), and conventional porosity analysis. The equipment used including Axio Imager A2 polarizing microscope, XL30 scanning electron microscope, X'Pert MPD X-ray diffractometer, and CS-230 carbon/sulfur analyzer. All these analysis and testing were completed in accordance with the national and industry standards recommended. In addition, the gas content test in field was completed by Weatherford with the sampling density of 1–2 samples per 10 m.

2. Diagenesis of Longmaxi Formation

Worden et al. [12] summarized that diagenesis comprises a broad spectrum of physical, chemical, and biological post-depositional processes by which original sedimentary assemblages and their interstitial pore waters react in an attempt to reach textural and geochemical equilibrium with their environment. These processes are continuously active as the ambient environment evolves in terms of temperature, pressure, and chemistry during the deposition, burial, and uplift cycle of the basin history. Based on a large number of thin section identification, scanning electron microscopy analysis, X-ray diffraction tests, and some other experiments, it showed that the Longmaxi Formation suffered multiple diagenetic changes, including compaction, cementation, clay mineral transformation, replacement, dissolution, organic matter thermal maturation, and tectonic disruption (Table 1). Of all the aforementioned, compaction and cementation reduced the rock's porosity. Meanwhile, dissolution, organic matter thermal maturation, and tectonic disruption increased the porosity, while replacement and clay mineral transformation had a minor effect on reservoir porosity.

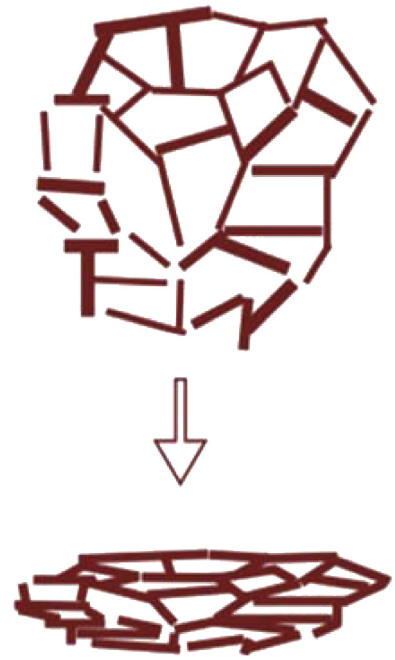


Fig. 1. Compaction of clay [13].

2.1. Compaction

Newly deposited mud consists of an open framework of clay mineral platelets with some silt, sand, pellets, and perhaps a small percent of organic matter; its original porosity value ranges between 60% and 80%. It collapses and deforms gradually as the burial proceeds (Fig. 1), hence, the porosity drops to less than 40% in the first few 100 m while the pore water is expelled. As it goes deeper, the final porosity reduced to only a few percent [13]. Thus, it can be inferred that compaction is the main reason for densification of the Longmaxi Formation. The common compaction signatures in the Longmaxi Formation include directional distribution of sheet-like minerals (Fig. 2) and the ruptured rigid particles.

Table 1
Diagenetic alteration of Longmaxi Formation.

Diagenesis		Diagentic alteration	Effect on porosity
Compaction		Clay mineral and mica bedding orientation	Negative
Cementation	Quartz	Quartz overgrowth, quartz cements precipitation in pore space	Negative
	Carbonate	Calcite and (or) dolomite precipitation in pore space	Negative
	Sulfide	A small amount of granular, nodular and berry pyrite precipitation in pore space	Negative
Clay mineral transformation		Smectite conversion to illlite	Minor
Replacement		Carbonate and (or) clay mineral growth within the body of feldspar	Minor
Dissolution		Feldspar and (or) calcite destroyed by interaction with acid fluid leaving behind a cavity	Positive
Organic matter thermal maturation		forming hydrocarbon and organic pore	Positive
Tectonic disruption		Forming fractures to connect pores	Positive

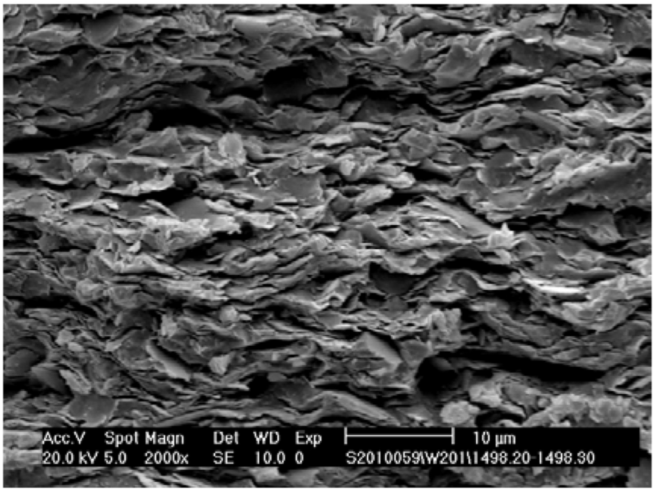


Fig. 2. Orientation of clays caused by compaction in Longmaxi formation in Well 1, 1498.20 m (×2000).

2.2. Cementation

Cementation is one of the most important diagenesis in Longmaxi Formation. The common cements in the Longmaxi shale include siliceous, carbonate, and sulfide. Regardless of the chemical composition, all of the cements will fill the pore space to cause rock densification it's another important reason to reduce reservoir porosity of Longmaxi shale.

Most of the siliceous cements in Longmaxi Formation are quartz, occasionally with a small amount of opal or chalcedony. Quartz exists in quartz overgrowth and (or) authigenic (Fig. 3a), and in the form of fillings of pores, fractures, and caves (Fig. 3b, c, d). Through comprehensive analysis, the source material of quartz cements mainly includes the following aspects: (1) The dissolution and clay mineral replacement of feldspar will result in a surplus of free silica in pore fluid, thus, becoming a potential source of siliceous cements [14]. (2) Illitization of smectite in shale will release a certain amount of silica, part of this diffusing to the adjacent layer with the compaction fluid while the remained precipitating in situ. Peltonen et al. [15] mentioned that the alteration of smectite to illite will result in the release of significant amounts of silica into solution and authigenic micro-crystalline quartz cement will precipitate within the clay matrix. (3) The lithology of the bottom Longmaxi is mainly black siliceous shale, which is rich in siliceous radiolarian and sponge spicules (Fig. 3e). The mentioned biogenic siliceous material may become the source of silica cements. (4) Bentonite always exists in the lower part of the Longmaxi black shale (Fig. 3f), because it can provide a quantity of Si^{4+} during the diagenetic alteration process, thus, becoming the source of siliceous cements.

Carbonate cements in the Longmaxi shale include calcite and dolomite. Calcite commonly precipitates in early diagenetic stage to fill pores and fractures or to replace feldspar (Fig. 3g,h,i,j,k). Dolomite always appears in euhedral-subhedral shape (Fig. 3l) to replace the early calcite cements or clay minerals, or in the form of crack filling material (Fig. 3m). Through a large number of thin section identification, the positive correlation between content of calcite cements and particle size was found. In silty shale, silt strip or silt accumulation place due to bioturbation, the calcite cements develop well compared to where clay mineral accumulates (Fig. 3g and h). In addition, calcite cement is also closely related with the content of original calcite component. Cement precipitates from the pore water requires a nuclei to attach. If the calcite bioclasts or micritic carbonate exists in the original sediments they would provide the necessary precipitation nuclei for calcite cements (Fig. 3i). Thus, if the original sediments include calcareous components, the calcite cement would be more developed.

The content of pyrite cements is usually minor, but its appearance can vary one way or another. It can exist in granular, lamellar, aggregation, or fracture fillings (Fig. 3n,o, and Fig. 4a).

2.3. Clay mineral transformation

As the burial depth increases, diagenetic temperature, and pressure, the inter-layer water and cations in clay mineral layer would be expelled. Such process will result in the conversion of smectite to illite. The decrease in smectite and the increase in illite are in accordance with the progressive burial depth and temperature [16]. The XRD analysis shows clay mineral composition in the Longmaxi shale. Illite is the most common clay mineral, and the I/S average content ranges 24.03% with I/S·S% only 10% or sometimes none, not to mention, there is no kaolinite and smectite. The clay mineral composition indicates that Longmaxi shale undergoes intense diagenetic alteration.

2.4. Replacement

One mineral growth within the body from a former existing mineral is called replacement. The common replacement in the Longmaxi shale include dolomite replacing clay mineral (Fig. 4b) or feldspar (Fig. 4f), pyrite replacing siliceous bioclasts (Fig. 4c) or early calcite cements (Fig. 4h), calcite replacing feldspar (Fig. 4d), and clay mineral replacing feldspar (Fig. 4e).

The dolomite formed during replacement in the Longmaxi shale mostly exists in the bottom of the formation. It appears in euhedral-subhedral shape, and cloudy cores with limpid rims. There exists the same kind of dolomite in Barnett Shale. Its average TOC content was 2.7% in samples dominated by euhedral dolomite. The average TOC content was 3.2% in samples dominates by anhedral dolomite [17].

2.5. Dissolution

Dissolution is closely related with the acid pore water produced during the organic diagenesis. The maturation of organic matter brings about the pyrolysis of kerogen released in large amounts of CO_2 and carboxylic acid. Dissolution will occur when these acidic fluids in contact with the soluble

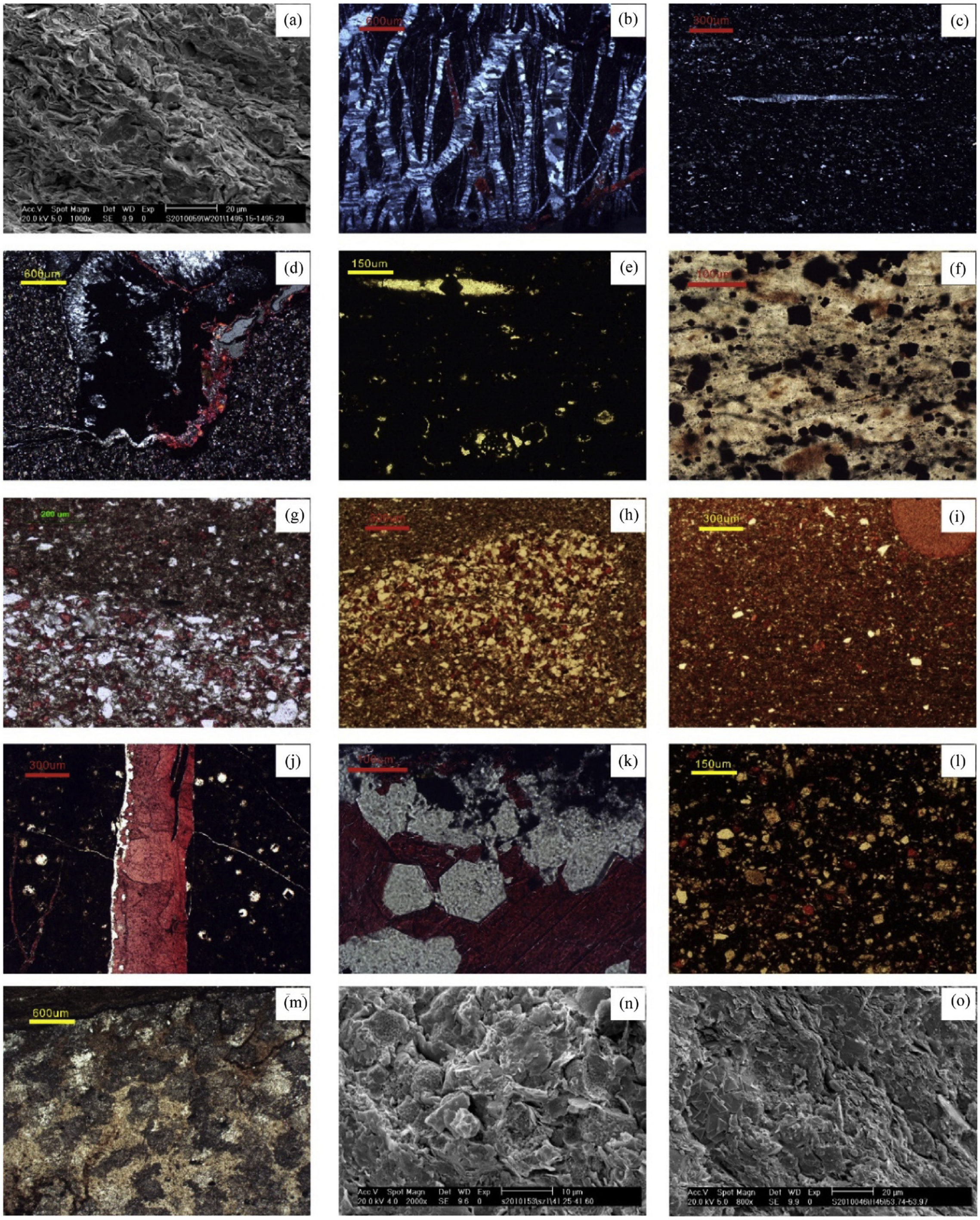


Fig. 3. Photographs of diagenetic alteration of the Longmaxi Formation (a) Well W1, 1495.15 m, ×1000, authigenic quartz; (b) Well HY2, 118.28 m, +, ×2.5, microfractures filled by quartz cements and minor calcites; (c) Outcrop sample from Tianba, +, ×5, quartz cements filling contraction microfracture; (d) Outcrop sample from Huangcao, +, ×2.5, Chalcedony, calcite and pyrite filling a cave; (e) Well N8, 1308.23 m, —, ×10, siliceous shale with radiolarians and sponge spicules; (f) well HY2, 103.22 m, —, ×20, bentonite; (g) Well N3, 2098.04 m, —, calcite cements well developed in silt strip; (h) Well N3, 2118.17 m, —, ×5, calcite cements well developed in silt accumulations formed by bioturbation; (i) Well N3, 2189.26 m, —, ×5, nuclei for calcite cements precipitation provided by micritic carbonates; (j) Well HY2, 122.92 m, —, ×2.5, calcite cements filling the microfracture; (k) Well HY2, 122.92 m, —, ×20, authigenic quartz, calcite and pyrite filling microfracture; (l) Well N3, 2389.65 m, —, ×10, euhedral-subhedral dolomite; (m) Well HY2, 39.55 m, —, ×2.5, dolomite cements filling microfracture; (n) Well LT1, 41.25 m, ×2000, berry pyrite; (o) Well HY2, 53.74 m, ×800, pyrite cements.

components of the rock to form secondary pores. Additionally, the mainly soluble components in the Longmaxi shale are feldspar and carbonate minerals (Fig. 4i). Dissolution has a positive effect on porosity, but it is not well developed in the Longmaxi Formation, because shale is a relatively closed diagenetic system, due to the extremely low permeability, pore fluid cannot flow smoothly within the system. The H^+ cannot get supplement and the reaction products cannot be brought

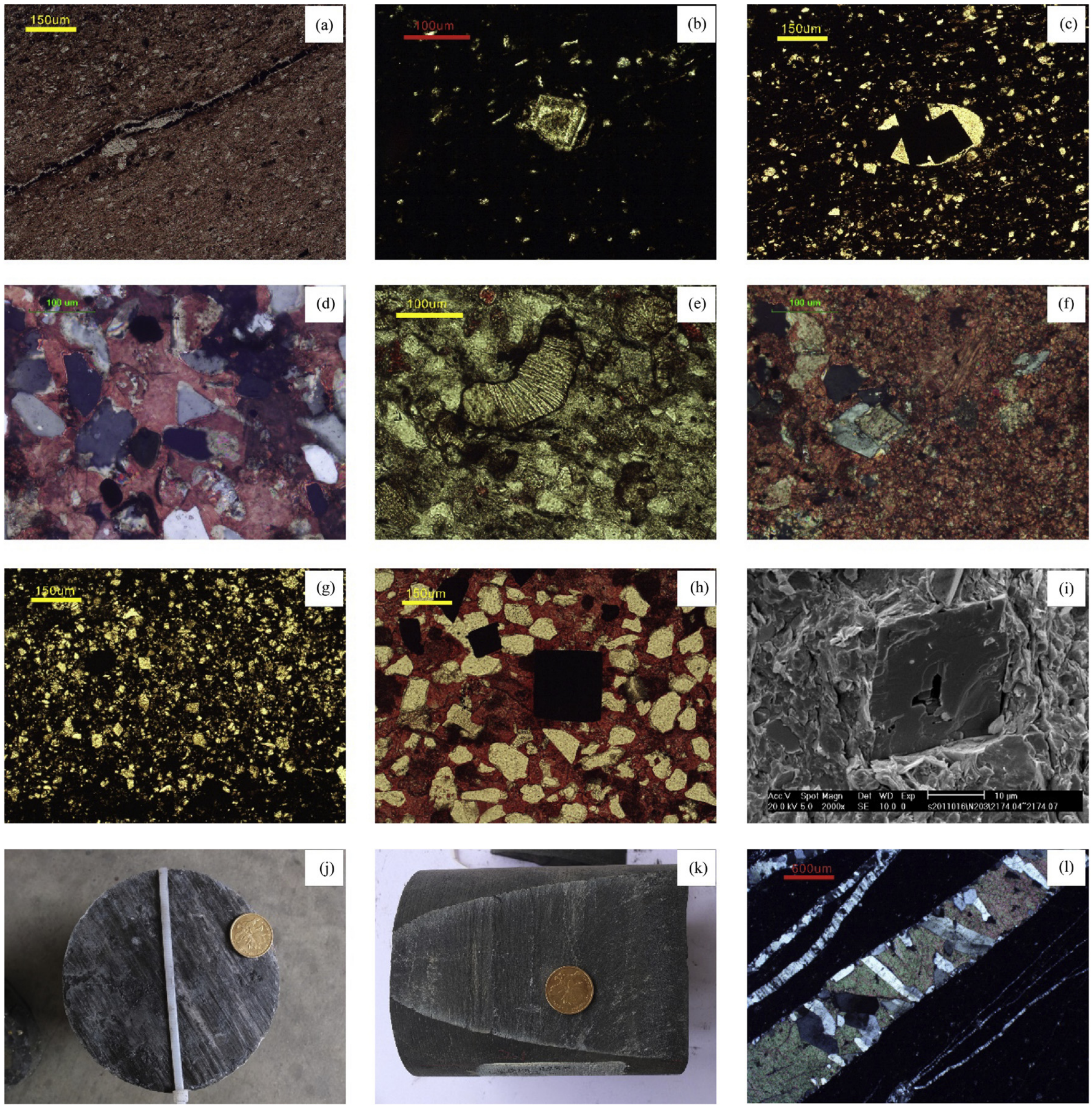


Fig. 4. Photographs of diagenetic alteration of Longmaxi Formation (a) Outcrop sample of Tianba, —, ×10, quartz and pyrite filling microfracture; (b) Well N8, 1292.56 m, —, ×20, dolomite replacing clay mineral; (c) Well N8, 1307.07 m, —, ×10, pyrite replacing siliceous bioclast; (d) well N3, 2210.85 m, —, ×20, calcite replacing feldspar; (e) Well N8, 1276.16 m, —, ×20, clay mineral replacing feldspar; (f) Well N3, 2216.33 m, —, ×20, dolomite replacing feldspar; (g) Well N3, 2377.86 m, —, ×10, authigenic euhedral-subhedral dolomite; (h) Well N3, 2234.03 m, —, ×10, euhedral pyrite replacing early calcite cements; (i) Well N3, 2174.04 m, ×2000, intragranular pore in dolomite; (j) Well N8, 1303.56 m, interlayer slip fractures; (k) Well N8, 1309.41−1309.54 m, high angle fractures; (l) Well HY2, 118.28 m, +, ×2.5, calcite and authigenic quartz filling microfracture.

out in time after the dissolution occurs. All these would prevent the further chemical reaction of dissolution.

2.6. Organic matter thermal maturation

Since there's no vitrinite in the Lower Paleozoic shale, we measured the reflectance of bitumen and converted it to equivalent vitrinite reflectance. The equivalent Ro value ranges 1.7%−3.0%, usually greater than 2.0%. It's in high-over mature stage. Organic matter maturation process will lower the pH value of pore water to form an acid diagenetic environment; contributing to dissolution and replacement. At the same time, a large number of organic pores are formed to increase the total porosity. According to the relationship between TOC content and rock porosity of Longmaxi Formation in Well N3 (Fig. 5), as for best shale reservoir, there is a significant positive correlation between them. Except for minor dissolution pores, the organic pores which are associated with organic diagenesis contribute much to the total porosity. As for suboptimal and none shale reservoir, this correlation is not significant.

2.7. Tectonic disruption

High-angled fractures are common in the rock core, its dip is generally above 70°, and it's usually half-filled or unfilled (Fig. 4k). Interlayer slip fractures can also be seen in the rock core (Fig. 4j). In addition, a small amount of microfractures

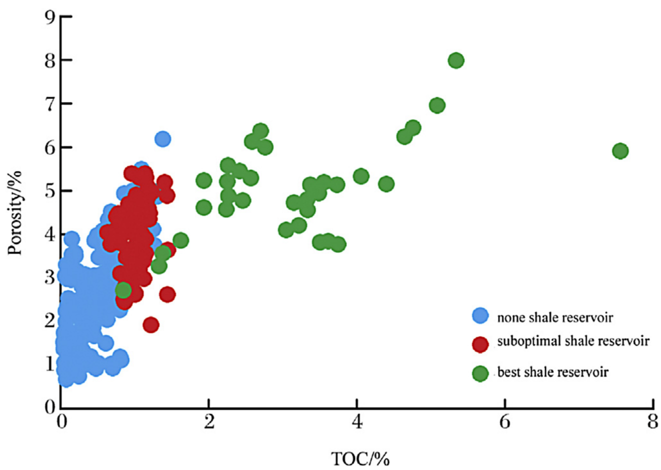


Fig. 5. The relationship between TOC content and rock porosity of LMX formation in Well N3.

are also found under microscope, and most of them are full filled with calcite, quartz and pyrite (Fig. 4l).

3. The diagenesis contrast of shale reservoir and non-reservoir in Longmaxi Formation

Overall, according to lithology and different reservoir characteristics, Longmaxi Formation can be divided into the best, suboptimal, and none shale reservoir sections from the bottom−up (Fig. 6). The main diagenetic events that they experienced and their reservoir parameters are obviously different (Table 2). We take the Well N3 as an example.

The best shale reservoir of Longmaxi Formation in Well N3 is located at the bottom of the layer. It mainly consists of organic siliceous shale and organic calcareous-siliceous shale. XRD analysis shows that the reservoir has high quartz content that averaged up to 55.55%. Thin section identification analysis shows that the terrigenous detrital quartz content is low, while the quartz cements and siliceous bioclasts contents are high. The dolomites are authigenic euhedral-subhedral which are formed in the late diagenetic stage, and it averaged up to 5.74%. The content of pyrite is also high, which usually appears as a strip or replacement of organic matter. The average TOC content is 3.18%, average porosity is 5.11%, and the average gas content is 3.2 m^3/t. Taking compaction, the shale undergoes a series of diagenetic alterations including organic matter thermal maturation, quartz cementation, dolomite and pyrite replacement, and occasionally dissolution. This series of inorganic diagenesis is closely related to the organic diagenesis. First, organic matter in the process of thermal maturation will release a large amount of organic acids, which lead to the dissolution of acid soluble minerals, while the pH value of the diagenetic environment gradually tends to be acidic. In an acidic medium conditions, the quartz is more prone to precipitation, therefore, the quartz cements in the best shale reservoir is more developed than the calcareous cements. As the acid fluid expels from the shale to the adjacent permeable layer and the consumption of H^+ due to dissolution, the pH value of pore water gradually increases, and the free Ca^{2+}, Fe^{2+}, and Mg^{2+} in pore water precipitate in ferroan dolomite. Pyrite is usually associated with organic matter. The organic matter content is higher, and the pyrite is more developed.

As to the suboptimal shale reservoir, its lithology is organic-rich silty shale. XRD results show that the average content of clay mineral value is 30.62%, quartz value is 45.71%, feldspar value is 8.22%, calcite value is 12.36%, and dolomite value is 2.95%. Based on thin section identification, the further analysis shows that terrigenous quartz is in vast majority of all siliceous components in the suboptimal reservoir, feldspar is also terrigenous, showing an increase of coarse silt content. The average TOC is 1.03%, average porosity is 4%, and the average gas content is 1.6 m^3/t. In comparison to the best shale reservoir, compaction and organic matter maturation is the dominant diagenesis, while quartz cementation, dolomite, and pyrite replacement which are related to organic diagenesis is not well developed.

The lithology of none shale reservoir includes inorganic calcareous shale and calcareous silty shale. XRD experiments show that the calcite content is higher than shale reservoir, and it averaged up to 35.77%. The identification of thin section further indicates that the occurrences of calcite are several, including calcareous bioclasts, micritics, calcite cements, and replacement products. The feldspar content is relatively low, with an average of only 2.81%, because most of them are replaced by calcite during the diagenetic process. The average TOC is only 0.42%, average porosity is 2.42%, and the average gas content is 1.1 m^3/t. The main diagenetic event is calcite cementation and replacement except for compaction. Due to the low content of organic carbon, the organic matter maturation is not well developed. The strata lacks of an acidic fluid from organic diagenesis and pH value does not show acid. It is conducive to calcite precipitation. The original micritic and calcareous bioclasts can provide the necessary nuclei for precipitation. Therefore, calcite cementation and replacement is more developed and it leads to a lower porosity when compared to the shale reservoir.

4. Diagenetic stage division of Longmaxi shale

The equivalent vitrinite reflectance of Longmaxi formation ranges between 1.7% and 3.0%. XRD experiments shows that I/S·S% is only 10% or none, and that the clay mineral composition is I/S + I + K + C or I/S + I + C. Calcite cements in Longmaxi shale are formed in the early diagentic stage, because it fills the primary interparticle pores and the grains are just like floating in it. Granular pyrites fill all kinds of pores and replace the early cements. All these characteristics show that the reservoir currently has already been in period B of the middle diagenetic stage to late diagenetic stage.

5. Effect of diagenesis on shale reservoir

Diagenesis has an important influence on conservation, development and evolution of reservoir porosity. At present, Longmaxi shale is in period B of the middle diagenetic stage

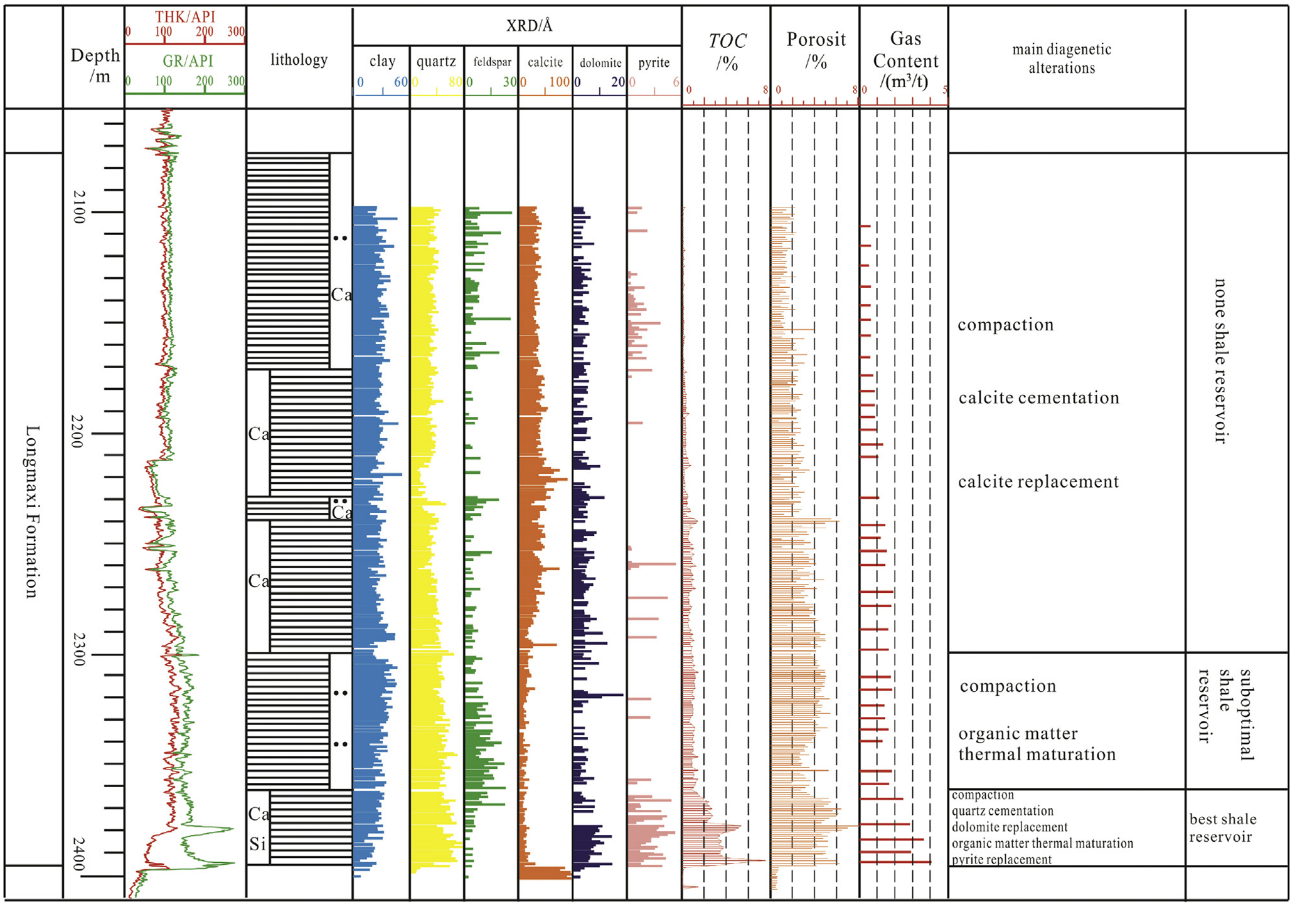


Fig. 6. The description of Longmaxi Formation in Well N3.

to the late diagenetic stage. In the lengthy geological history, it has gone through a variety of diagenetic alterations (Table 1). Compaction and cementation will cause the densification of the rocks to decrease the reservoir porosity, while dissolution, organic matter thermal maturation, and tectonic disruption will increase the total porosity. In terms of replacement, clay mineral transformation has a minor effect on reservoir porosity.

In addition to the impact on porosity, diagenesis also has an important influence on the mechanical property of the rock. Cementation will dense the rock which is the downside, but it also has a positive side. For the occurrence that both quartz and carbonate cementation will enhance the brittleness of the rock, thereby, facilitating fracturing of the shale reservoir.

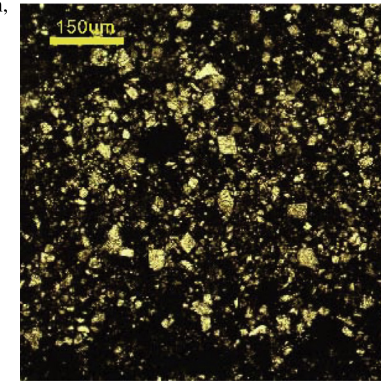
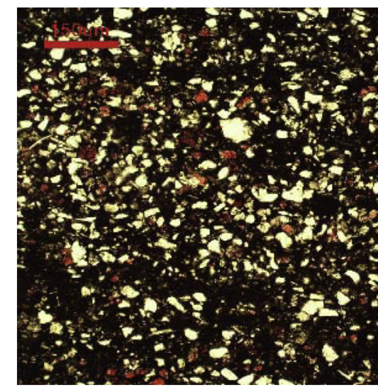
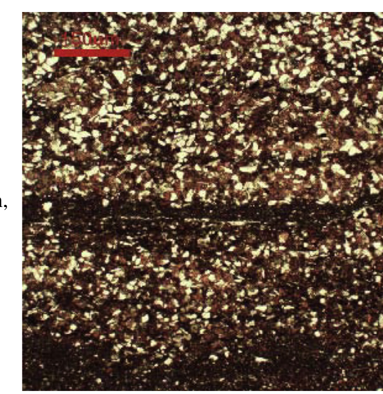
Diagenesis also affects the adsorption capacity of the rock. Adsorption capacity of illite is better than other clay minerals' the transformation of smectite to illite will somewhat increase the total gas adsorption capacity. On the other hand, due to the fine particle size of shale, the pore in it are always in micron-nanometer size, the cements in the pore space are in the same size or even smaller, so as to have a larger surface area. This is in favor of enhancing the adsorption capacity of shale reservoir.

Organic diagenesis is also of great importance to the shale reservoir. First, a large amount of hydrocarbons will be generated gradually during the organic matter maturation process, which is the material source of shale gas. Therefore, the formations which undergo a certain degree of diagenetic evolution to reach thermal maturity requirements are likely to become shale gas reservoir. Loucks et al. [18] mentioned that thermal maturation has to reach an R_O level of approximately 0.6% or higher before organic pores begin to develop; which is the beginning of the peak of oil generation. At levels of maturity less than 0.6% (R_O), organic pores are absent or extremely rare. Milliken et al. [19] further mentioned that both maturation and compaction may be factors in the creation, preservation, and destruction of organic matter hosted pores. High TOCs may cause low organic matter porosities, because the rocks suffers complete gas expulsion process as a consequence of greater organic matter connectivity and compactability. In addition, the organic matter maturation process brings about decarboxylation and releases large amounts of CO_2, resulting in the reduction of pH value of the pore water, thus, causing dissolution of the soluble components of the rock, and to form minor secondary pores. In the meantime, it helps to promote precipitation of quartz cements, thereby, increasing the brittleness of the reservoir.

6. Conclusions

(1) Several diagenesis were recognized in the Longmaxi shale, including compaction, cementation, clay mineral transformation, replacement, dissolution, organic matter thermal

Table 2
Statistics of reservoirs and main diagenetic alterations of different shale section of Longmaxi Formation in Well N3.

Shale section	Lithology	XRD/Å						TOC/%	Porosity/%	Gas content/(m^3/t)	Main diagenetic alterations	Typical photo under microscope
		Clay mineral	Quartz	Feldspar	Calcite	Dolomite	Pyrite					
Best shale reservoir	Organic siliceous shale Organic calcareous-siliceous shale	21.23	55.55	3.89	11.52	5.74	2.07	3.18	5.11	3.2	Compaction, Quartz cementation, Dolomite replacement, pyrite replacement, organic matter thermal maturation	
Suboptimal shale reservoir	Organic silty shale	30.62	45.71	8.22	12.36	2.95	0.14	1.03	4.00	1.6	Compaction, organic matter thermal maturation	
None shale reservoir	Inorganic calcareous shale, Inorganic calcareous silty shale	26.88	31.14	2.81	35.77	3.13	0.27	0.42	2.42	1.1	Compaction, calcite cementation, calcite replacement	

maturation, and tectonic disruption. The source material of quartz cements were summed up. It is related to the diagenetic reaction of feldspar, illitization, siliceous bioclasts, and bentonite. The main factor of forming abundant carbonate cements were also summed up. It is mainly related to particle size and available nuclei for cements to attach.

(2) There are significant differences in diagenetic alteration among the best, suboptimal, and none shale reservoir. As to the best shale reservoir, the main diagenesis includes compaction, quartz cementation, dolomite replacement, pyrite replacement, and organic matter thermal maturation. Except for compaction, all the other inorganic diagenesis is closely associated with organic diagenesis.

(3) Several diagenetic indicators show that the reservoir has already been in period B of the middle diagenetic stage to late diagenetic stage.

(4) Diagenesis has a great effect on shale reservoir. Firstly, it controls the conservation, development, and evolution of reservoir porosity. Secondly, it affects the mechanical property of the shale. Moreover, it also has a certain influence on adsorption capacity of the rock. Hydrocarbons generated during the organic diagenesis are the material source of shale gas. During the same process, a large number of nanopores in organic matter are generated, which increase the total porosity and the adsorption capacity of the rock.

Foundation item

Supported by National Key Basic Research Program of China (2012CB214706, 2013CB228006); China National Science & Technology Special Project (2012ZX05018-006).

Conflict of interest

The authors declare no conflict of interest.

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