

# Mineral composition and brittleness of Lower Cambrian Niutitang formation shale in northwest Hunan

*This paper determines the mineral composition and analyzes the brittleness of Lower Cambrian Niutitang formation shale in Northwest Hunan using XRD technology. The results show that Niutitang formation shale in Northwest Hunan has rather complicated composition, mainly consisting of quartz and clay minerals. The average content of quartz is 41.40%~60.81%, and the average content of clay minerals is 21.43%~26.40%.*

*Calcite, plagioclase, dolomite, pyrite, potassium feldspar and other minerals are also commonly found in the samples. The brittle minerals of Niutitang formation shale are featured by rich species, high content (greater than 40% in average) and large brittleness index (higher than 50% in average). Compared to the shales in different regions both at home and abroad, Niutitang formation shale in Northwest Hunan has better fracability.*

**Keywords:** *Shale; components of rock and mineral; brittleness; Niutitang formation; Lower Cambrian; northwest Hunan.*

## 1. Introduction

In recent years, driven by the large-scale commercial development of shale gas in North America, China has also accelerated the exploration and development of shale gas. Some important progress has been made in the evaluation of shale gas resource potentials and major breakthroughs have been achieved in some areas. Unlike conventional oil and gas systems, the shale gas system is mainly composed of compacted mud shale rich in clay minerals, and its distinctive feature is the large area containing gas, diversified rocks and relatively short transport

distances[1]. Minerals in shale directly controls shale porosity and the development of microstructure, which is of great significance to the gas-bearing and reservoir property of shale. Minerals in the shale are mainly clay minerals and quartz, and carbonate minerals and pyrite are also found as well. Clay minerals are lamellar silicate minerals, with relatively larger specific surface and pore volume to adsorb vapour molecular, which is helpful for shale gas preservation [2-4]. Although the organic matter can generate and preserve shale gas, it is necessary to connect the isolated microporous reservoirs in the shale by means of fracturing to obtain considerable yield [5]. And quartz and calcite and other brittle minerals are easy to produce cracks in the external stress, thus becoming an important seepage channel to natural gas [6]. Therefore, the mineral composition and rock brittleness evaluation are an important research content in shale gas exploration and development.

Domestic scholars have carried out researches on Niutitang formation shale in Northwest Hunan in terms of sedimentary environment, geochemistry, formation conditions, geological analysis and constituency evaluation, and have obtained some achievements [7-10]. However, systematic studies on the mineral composition of Niutitang formation shale are lacking.

In this paper, the mineral composition features and brittleness of Niutitang formation shale are studied through the X-ray diffraction analysis of the core samples collected from a number of wells in Niutitang formation in Northwest Hunan.

## 2. Geological background

The tectonic location of Northwest Hunan and surrounding areas belongs to Jiangnan fault zone of the Middle Yangtze plate. Northwest Hunan takes Baojing-Cili fault zone as the boundary. Its northwest side is the western Hubei and Hunan fold belt on the Yangtze Platform and southeast side is Jiangnan-Xuefeng Nappe belt zone. Strong thrust, folds, and uplift erosion were generated in this region after multistage torsional structural movements in Wuling Period and Yanshan

Messrs. Zhen Zhang, Peng Jiao and Xikai Wang, Key Laboratory of Metallogenic Prediction of Nonferrous Metals and Geological Environment Monitoring, Central South University, Ministry of Education, Central South University, Changsha 410 083, Zhen Zhang, Peng Jiao and Xikai Wang, School of Geosciences and Info-Physics, Central South University, Changsha 410 083, Wen Pang, Shandong Shengli Vocational Institute of Training Department, Dongying 257 000 and Linting Zhang, North China University of Science and Technology, Tangshan 063 210, China. E-mail: slzz0906@vip.qq.com

Period, which have formed current fold and fracture system with an NNE or NE strike and affected the enrichment and preservation of shale gas (Fig.1).

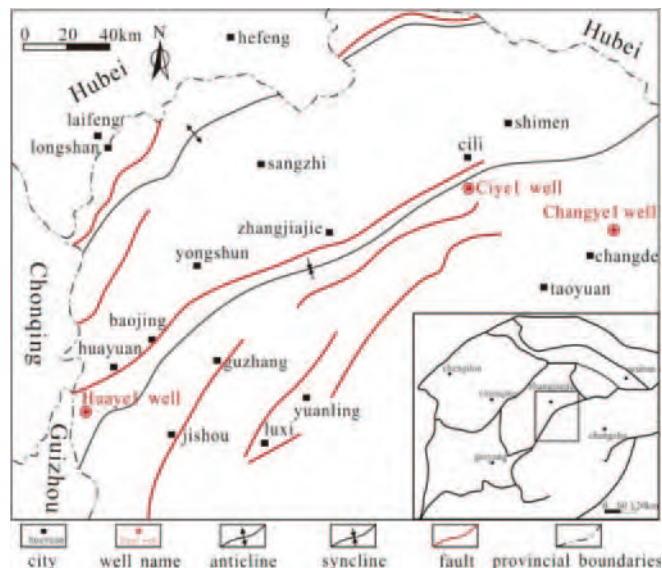


Fig.1 Location of the study area and distribution of sampling wells

In the early Cambrian, the South China Pangaea was disintegrated and the regional tectonic subsidence was intensified. Niutitang formation presented as an anoxic and reducing marine sediment[11] with deep water and low energy. Bounded by the line of Baojing~Zhangjiajie~North of Changde, the sedimentary environment in this area gradually transits from clastic rock deep-water continental shelf facies to bathyal sea-deep sea facies from northwest to southeast. The northwest is the region with continental shelf facies. The upper part and lower part of the area have different rock types. The lithology of the lower part is black, gray black, and dark gray shale, thin layer of carbonaceous shale sandwiched by siliceous shale, gray limestone, and stone coal, indicating deep shelf in the early stage. It is found from Hueye well 1 and Ciye well 1 that high-angle fractures are developed in the core of Niutitang formation shale, which are filled by clear calcite. Horizontal fractures are also developed. It can be seen

the pyrite presents a radial-pattern, star-point or banding distribution. The horizontal bedding is developed. Barite mineral can be seen at its surface. Energy spectrum analysis indicates that the content of Ba is 15.06%, and the content of S in pyrite is 50.58% (Fig.2), suggesting a fairly high productivity[11] of this segment of shale; the lithology of the upper part is inter-bedded black, gray black, grey, dark grey calcareous shale, shale, grey, dark grey, yellow grey, grayish yellow limestone containing marl, argillaceous limestone, and limestone, indicating shallow shelf in the late stage. The Southeast is the deep-water quiet deep-sea basin facies. Changye No.1 well found rocks with grey black and black colours. Partial layer section is greyish-green. The rock type mainly contains carbonaceous shale, siliceous rock, bottom stone coal and phosphate concretion. Horizontal subtle bedding is developed generally. There are not many bioturbation structures and the biological fossils are rare. The diagnostic mineral pyrite distributes in a lamellar way, signifying deep water reductive environment. The preliminary exploration practice shows that the reservoir forming conditions of Niutitang formation shale gas are superior (Table 1), and the exploration potential is huge.

(a) 2729.43m, Ciye No.1 well, stripped pyrite in carbonaceous shale; (b) 2,277.02m, Ciye No. 1 well, high-angle vertical joint filled with calcite veins; (c) 2,478.35m, black carbonaceous shale, with horizontal fractures developed; (d) 2,268.38m, Ciye No.1 well, star point and radial pattern pyrite; (e) 2,718.26m, Ciye No. 1 well, barite crystals; (f) 2,278.65m, Ciye No.1 well, horizontal bedding development in the shale; (g)(h)(i), 2,595m, Huaye No.1 well, Ba-rich energy spectrum analysis indicates the content of Ba is 15.06%; (j)(k)(l) 2,595m, Huaye No.1 well, pyrite energy spectrum analysis indicates that the content of S is 50.58%.

### 3. Sample and experiment

The experimental samples are mainly Niutitang formation cores and rock debris (Fig.1) from Hueye No.1 well and Ciye No.1 well. The samples were milled to about 200 meshes and dried in an oven at 70°C for 24 hours. XRD test was performed using a D8 ADVANCE type X-ray diffractometer

TABLE 1: THE ORGANIC GEOCHEMISTRY ANALYSIS OF NIUTITANG FORMATION IN NORTHWEST HUNAN

Well name	Depth /m	Thickness /m	TOC/%	Ro/%	Organic matter type	Sedimentary facies
Changye no.1	1103~1224	121	1.10~17.6/10.10	2.02~3.13/2.60	□	Deepwater shelf - slope
	1288~1344	66	2.62~9.85/8.20			
Huaye no.1	2485~2508	23	1.27~3.94/2.4	2.00~3.60/2.80	□, □	Deepwater shelf
	2526~2595	69	1.16~13.30/7.00			
Ciye no.1	2244~2272	28	0.46~2.69/1.16	1.68~2.63/2.08	□	Deepwater shelf
	2465~2500	35	0.28~2.45/1.46			
	2690~2732	42	1.79~6.44/4.83			

Notes: minimum value ~ maximum value/average value

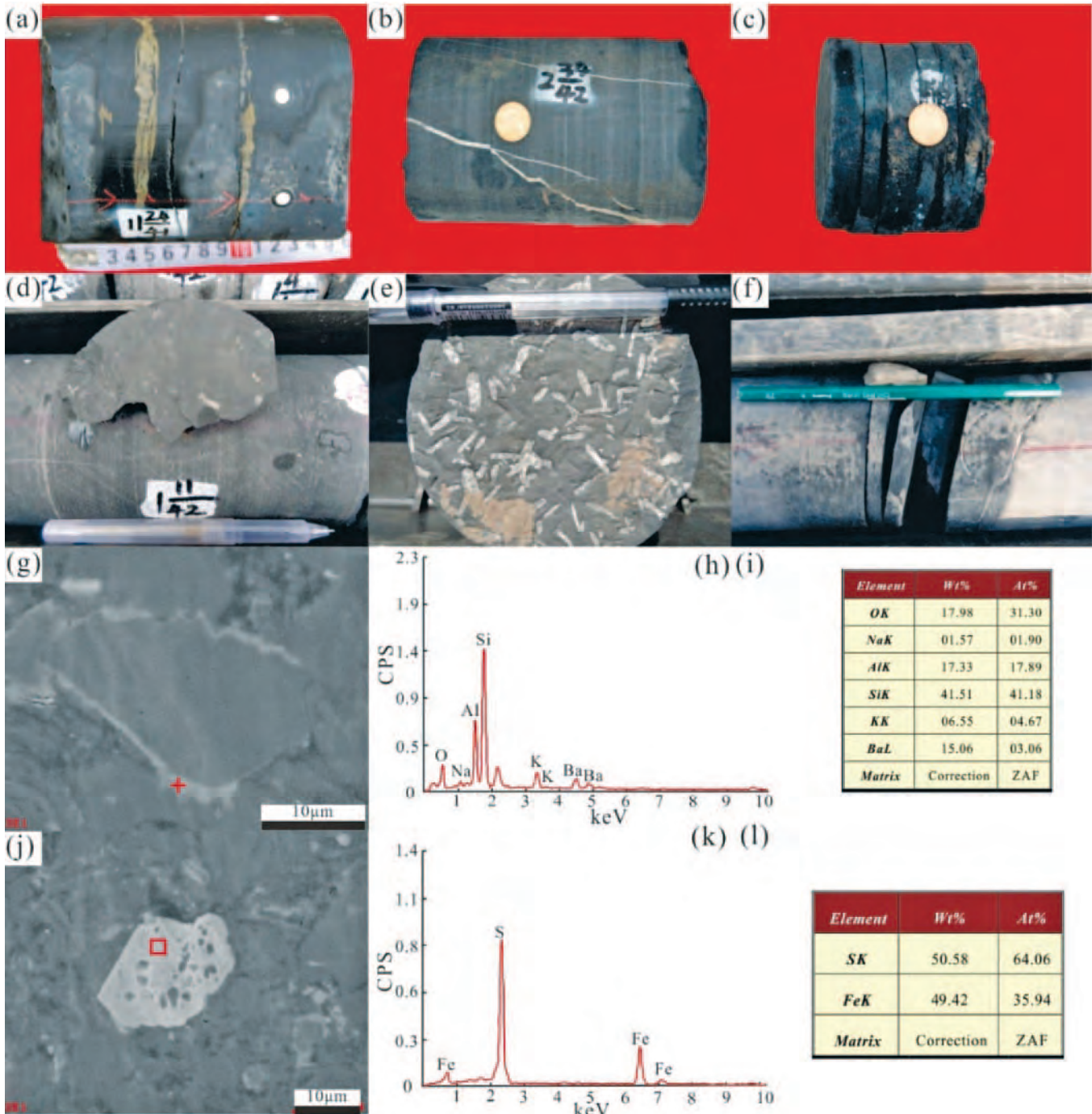


Fig.2 Core and microscopic photograph of Niutitang formation in northwest Hunan

of Bruker in Germany. The test conditions are as follows: Cu target (monochrome); working voltage, 40 kV; working current, 30 mA; 2 $\theta$ =3.850; slit: 1 mm. The qualitative analysis was carried out through continuous scanning at a speed of 40/min and with a sampling interval of 0.02°. The mineral compositions of the samples were confirmed through comparing the diffraction data with the standard power diffraction data provided by the International Centre for Diffraction Data of Joint Committee on Powder Diffraction

Standards (JCPDS-ICDD); the quantitative analysis was carried out using step scanning at a speed of 0.25°/min and with a sampling interval of 0.01°. The K value method based on national standard (GB5225-86) was adopted to make quantitative analysis.

#### 4. Results and analysis

##### 4.1 MINERAL COMPOSITION FEATURES

The X-ray diffraction quantitative analysis of the whole

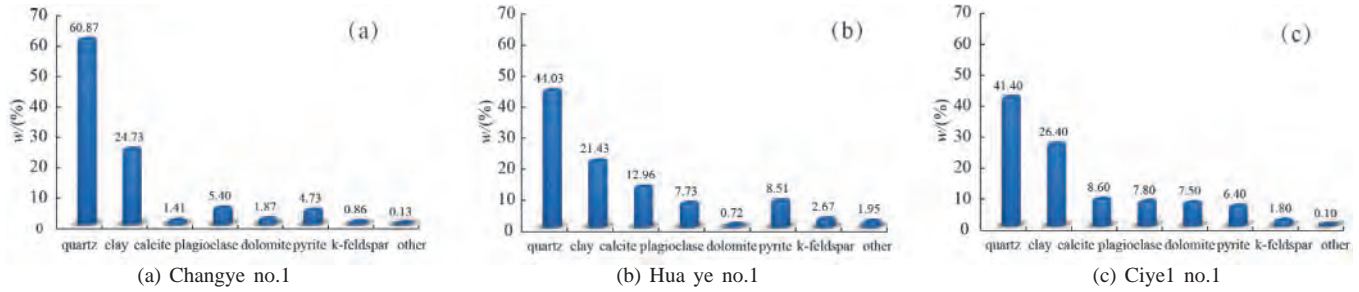


Fig.3 Minerals average composition of Niutitang formation in northwest Hunan

rock-mineral shows that the main mineral components of Niutitang formation in three wells is quartz, with an average percentage of more than 40%, and clay mineral with an average percentage of less than 28%. In addition, calcite, plagioclase, dolomite, pyrite and potassium feldspar are also generally found (Fig.3). The mineral composition is similar to that of Barnett shale in North America (Fig.4).

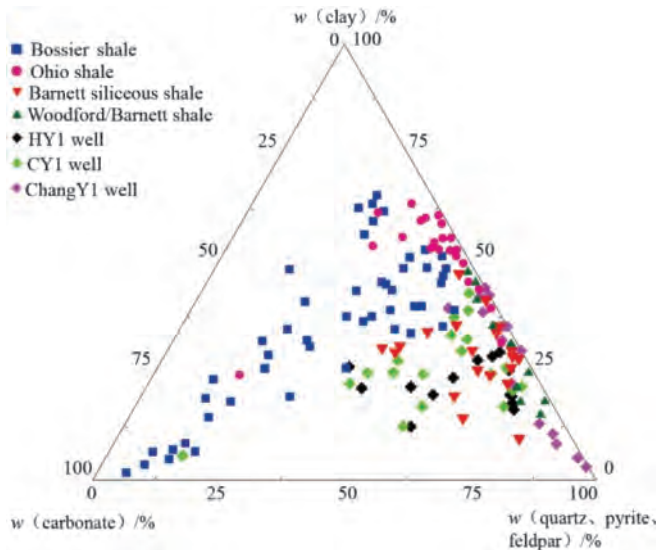


Fig.4 The mineral composition of Niutitang formation in northwest Hunan and typical American shales

There are both similarities and differences in the mineral composition of Niutitang formation shale in the three wells: the mineral composition of Niutitang formation shale in Ciye No.1 well is similar to that of Niutitang formation shale in Huaye No.1 well, while the mineral composition of Niutitang formation shale in Changye No.1 well are significantly different from those of Niutitang formation shale in both Ciye No.1 well and Huaye No.1 well. According to the whole-rock

X-ray diffraction analysis results, the rank of quartz content of Niutitang formation shale in three wells is as follows: Changye No. 1 well>Huaye No. 1 well>Ciye No.1 well; and the rank of clay minerals is as follows: Ciye No.1 well>Changye No.1 well>Huaye No.1 well.

#### 4.2 FEATURES OF CLAY MINERAL COMPOSITION

The quantitative analysis of X-ray diffraction of clay minerals in Niutitang formation shale from three wells signifies large difference in the clay mineral composition (Table 2). Although the three wells are all dominated by illite minerals, the content of illite mineral in Huaye No.1 well is closer to that of illite mineral in Ciye No.1 well and the content of illite mineral in these two wells are far greater than that in Changye No.1 well. The content of illite/smectite inter-stratified minerals and chlorite in Changye No.1 well is the highest (the mean of mass fraction is 28.4% and 11.2% respectively), followed by that in Ciye No.1 well. No illite/smectite inter-stratified minerals and kaolinite were found in Huaye No.1 well, and Changye No.1 well and Ciye No.1 well only contain a small amount of kaolinite.

#### 4.3 MINERAL DIFFERENTIAL ANALYSIS

The composition and distribution of shale minerals are mainly controlled by sedimentary environment, sediment provenance and diagenesis, which have great effect on the reservoir quality of the shale. The quartz in shale mainly includes detrital quartz and biogenic quartz according to their sources. The detrital quartz takes up a large proportion. It is derived from the weathering product of parent rock deposited during the sedimentary stage, and the biogenic quartz is derived from the precipitate of biological secretion. There is a positive correlation between the detrital quartz and porosity, while biogenic quartz and authigenic quartz are negatively correlated with porosity. In addition, some quartz can also be formed in the diagenesis process. A large amount of free

TABLE 2: CLAY MINERAL COMPOSITION OF NIUTITANG FORMATION IN NORTHWEST HUNAN

Well name	Sample	w(K)/%	w(C)/%	w(S)/%	w(It)/%	w(I/S)/%
Changye no.1	15	0~8.95(0.4)	1~23(11.2)	-	37~75(60)	12~49(28.4)
Huaye no.1	16	-	0~17(2.37)	-	84~100(97.63)	-
Ciye no.1	18	0~6(0.44)	0~24(8.11)	-	76~100(89.12)	0~14(2.33)

Notes: K - kaolinite, C - chlorite, S - smectite, It - illite, I/S - illite/smectite

silicon can be released during the process of transformation from smectite to illite and from illite to muscovite. The mass of silicon dioxide produced in these two kinds of reactions can reach 17%~28% and 17%~23% of that of the reacting minerals respectively, demonstrating a desiliconizing and alumina-enriching trend [12]. Due to the densification of the shale, the silica released from the reaction will bond in situ or in neighbouring shale with better permeability to form authigenic quartz [13], which results in the secondary enlargement in the quartz edge. In the maceral observation of Niutitang formation shale samples, some secondary enlargement phenomena of quartz particles were also found, proving that the authigenic quartz exists extensively, but the content was not high. The difference of quartz content in the three wells is mainly due to the difference of the content of detrital quartz, which is mainly related to the different depositional environment. The possibility of clay minerals to form authigenic clay minerals directly from colloidal SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> during the deposition process is negligible. However, in the process of diagenesis, the increase of buried depth and temperature will promote the conversion of clay minerals, mainly manifesting as the chloritization and illitization of smectite and muscovitization of illite. Only a small amount of chlorite was detected in the samples of Niutitang formation shale, and the content of chlorite in the samples of Niutitang formation shale in Changye No.1 well and Ciye No.1 well is much higher than that of Huaye No.1 well, indicating that the diagenetic stage of these three wells is different. In the diagenetic process, the smectite mainly transforms to illite, instead of chlorite.

#### 4.4 SHALE GAS RESERVOIR BRITTLINESS ANALYSIS

The matrix porosity and microfracture development degree, gas content and fracturing transformation methods of the shale are all affected by the content of brittle minerals in shale minerals [14-16]. The higher the content of brittle minerals such as quartz, feldspar, calcite and dolomite in shale minerals, the stronger will be the brittleness of the shale [5]. A large amount of practical experience of shale gas exploration and development at home and abroad shows that the higher the brittleness of the shale reservoir, the more easily it will be to generate induced fissures and natural fractures under the action of external forces, which is conducive to the seepage and desorption of shale gas to achieve the purpose of increasing production [17]. The content of brittle minerals in Banett shale and Woodford shale which have met the commercial development conditions abroad, is generally greater than 40% and the clay mineral content is lower than 30% [14].

The brittleness index is an important parameter to evaluate the brittleness of shale gas reservoirs. There are two calculation formulas:

$$\text{Brittleness index } \square = \frac{w(\text{quartz})}{(w(\text{quartz}+\text{calcite}+\text{clay mineral}) 100\%)} \dots (1)$$

Formula (1) is mainly applied to the shale in the area with relatively simple varieties of brittle minerals, such as multiple sets of shale in North America.

$$\text{Brittleness index II} = \frac{w(\text{quartz}+\text{feldspar}+\text{calcite}+\text{dolomite})}{(w(\text{quartz}+\text{feldspar}+\text{calcite}+\text{dolomite}+\text{clay mineral}) 100\%)} \dots (2)$$

Formula (2) is generally applied to calculate the brittleness index of the shale with complicated composition. Domestic shale gas evaluation mainly adopts formula (2) to calculate the brittleness index of the shale.

In view of the complicated composition of Niutitang formation shale in Northwest Hunan, it is easy to evaluate the brittleness of the shale by using the formula (2). The calculation results show that the brittleness index of Niutitang formation shale in the study area is within the range of 50%~100%. As shown in Fig. 4, the average content of quartz in Niutitang formation shale from Changye No.1 well is the highest. The brittleness index range is 55.01%~96.94% and the main frequency is 50%~70%. The average content of quartz in the shale from Huaye No.1 well and Ciye No.1 well is close. The former's index range is 67.71%~86.70% and main frequency is 70%~80%, while the latter's index range is 55.08%~86.48% and main frequency is 60%~80% (Fig.5).

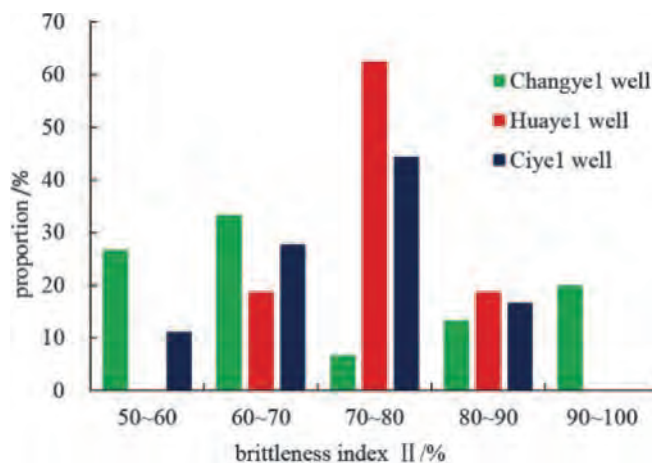


Fig.5 Brittleness index □ distribution of Niutitang formation in northwest Hunan

In order to make a comparison with the brittleness of the shale in North America, the brittleness of Niutitang formation shale in the study area was also calculated using the formula (1). Changye No.1 well (brittleness index I range 45.60%~96.37%), Huaye No.1 well (brittleness index I range 31.79%~71.24%) and Ciye No.1 well (brittleness index I range 36.32%~73.16%). It is found that the higher the quartz content in the shale, the closer the brittleness index □ and the brittleness index □ is, and the better the correlation is shown in Fig. 6. The value of the brittleness index II is larger than that of brittleness index I. Julia et al. [18] suggested that when

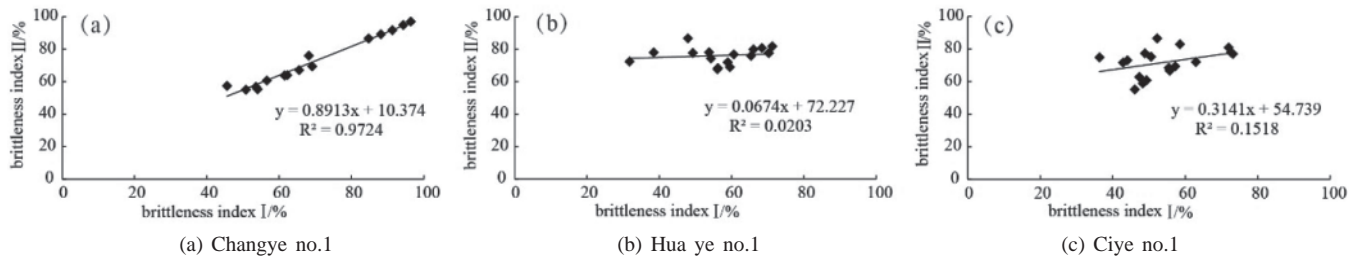


Fig.6 Correlation between two brittleness indexes of Niutitang formation in northwest Hunan

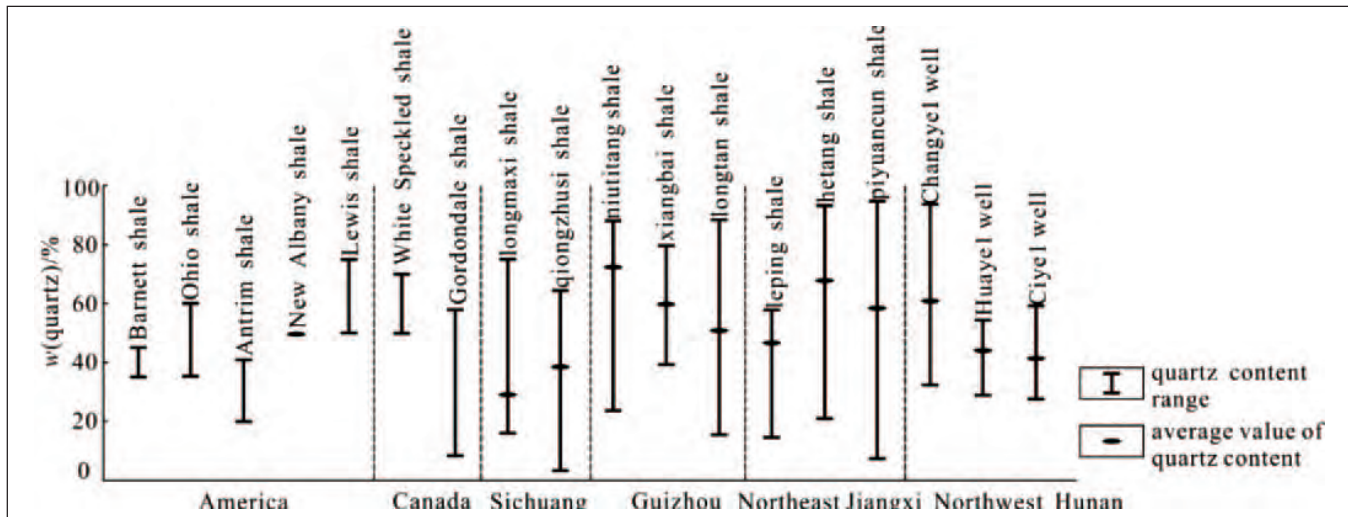


Fig.7 Quartz content comparison of Niutitang formation in northwest Hunan with North American shales and southern China

the brittleness index  $\square$  is higher than 40%, the fracturing effect of the shale is good. The average value of the brittleness index  $\square$  of multiple sets of shales in North America is within the range of 44.6%~70.8%, and the brittleness index  $\square$  of Niutitang formation shale is close to that, which is conducive to the fracturing stimulation of the shale. After comprehensive comparison with multiple sets of marine facies shales both at home and abroad, Niutitang formation shale in the study area is an ideal position for exploration and development of shale gas in South China.

### 5. Conclusions

Lower Cambrian Niutitang formation shale in Northwest Hunan has rather complicated composition, mainly consisting of quartz and clay minerals. The average content of quartz is 41.40%~60.81%, and the average content of clay minerals is 21.43%~26.40%. The clay mineral components are dominated by the illite, and the second one is chlorite mineral. Calcite, plagioclase, dolomite, pyrite, potassium feldspar and other minerals are also commonly found in the samples.

Compared to the shales in different regions both at home and abroad, the brittle minerals of Lower Cambrian Niutitang formation shale are featured by rich species, high content (greater than 40% in average) and large brittleness index (higher than 50% in average), indicating this shale gas reservoir has sound fracability.

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## STUDY ON THE FOUNDATIONS OF TRANSMISSION LINES IN MINING AREA WITH THE RISK OF GEOLOGICAL HAZARDS

(Continued from page 555)

### 4.3.3 Operational difficulty

The amount of concretes used in a combined foundation is 2.5 times that of an independent foundation while the amount of rebar used is 3 times that of an independent foundation. Therefore, the former workload is much heavier than the latter. When there is collapse or inclination, independent foundation is much easier to operate by adjusting anchor bolts or pushing a single foundation. However, combined foundation needs a large number of mechanical equipment and manpower during restoring. Despite the large input, it may fail to achieve the aim occasionally. Therefore, the independent foundation is better than the combined one in terms of operational difficulty.

### 5. Conclusion

According to the practice and experience gained in the mining areas in Guizhou, the depth to thickness ratio should be between 90 and 150. The location of the transmission tower should avoid fractured regions with steep terrains and structural deformation. In this way, the independent big slab foundation can effectively resist surface deformations caused by coal mining and thus ensure the safe operation of the transmission lines.

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